National Mission on use of Biomass in Thermal Power Plants (SAMARTH)
Ministry of Power, Government of India

Bureau of Indian Standards
The National Standards Body of India

SOLID BIOFUELS

COMPENDIUM OF INDIAN STANDARDS

Published by:

Bureau of Indian Standards,
Petroleum, Coal and Related Products Department
9, Bahadur Shah Zafar Marg, New Delhi - 110002

in collaboration with

National Mission on use of Biomass in Thermal Power Plants (SAMARTH)
E-3, Main Market Rd, Ecotech-II, Udyog Vihar,
Greater Noida, Uttar Pradesh- 201306

Contact Details:

Shri Chinmay Dwivedi,
Scientist E/ Director,
Head(Petroleum, Coal and Related Products Department)
01123235432/ hpcd@bis.gov.in

Ms Aditi Choudhary,
Scientist C/Deputy Director,
Member Secretary PCD 07
01123428478/pcd7@bis.gov.in

Shri Prafulchandra R. D
Member, National Mission on Use of Biomass in TPP, Ministry of Power
(Govt. of India) & Joint Director, CPRI
samarth.powermin.gov.in

FOREWORD

Pelletized biofuels have become a very important commodity as well as an integral part of our emerging new energy supply worldwide. As the trade of biomass pellets /briquettes has increased manifolds, the demand for reliable and agreed-upon standards for common ways of their evaluation is also growing. It is necessary to standardize quality requirements and testing of criteria for assessment of bio-mass for its commercial usage as a fuel along with its collection, storage and handling.

Bureau of Indian Standards through its Petroleum, Coal and Related Products Division Council (PCDC) is involved in preparation of Indian Standards in the field of Solid Biofuels. The technical Committee responsible for the preparation of these standardization of solid biofuels is Solid Mineral Fuels and Solid Biofuels Sectional Committee, PCD 07.

This handbook intends to provide technical guidance on published Indian Standards till date. This will help relevant stakeholders to refer to available Indian Standards.

It has also touched upon the existing policies on Solid Biofuels.

TABLE OF CONTENT

BUREAU OF INDIAN STANDARDS (BIS)

Bureau of Indian Standards (BIS), the National Standards Body (NSB) of India was established under the BIS Act, 1986 and came into existence on 1 April 1987 assuming the functions of the erstwhile Indian Standards Institution (ISI). The ISI came into being on 6 January 1947. The BIS Act, 2016 came into force on 12 October 2017 superseding BIS Act, 1986. BIS Act, 2016 provides for the establishment of a national standards body for the harmonious development of the activities of standardization, conformity assessment and quality assurance of goods, articles, processes, systems and services and for matters connected therewith or incidental thereto.

BIS through its core activities of standardization and conformity assessment, has been benefiting the national economy by providing safe, reliable and quality goods; minimizing health hazards to consumers; protecting the environment, promoting exports and imports substitutes; controlling over proliferation of varieties, etc. The standards and certification schemes of BIS apart from benefitting the consumers and industry also support various public policies especially in areas of product safety, consumer protection, food safety, environment protection, building and infrastructure development, etc.

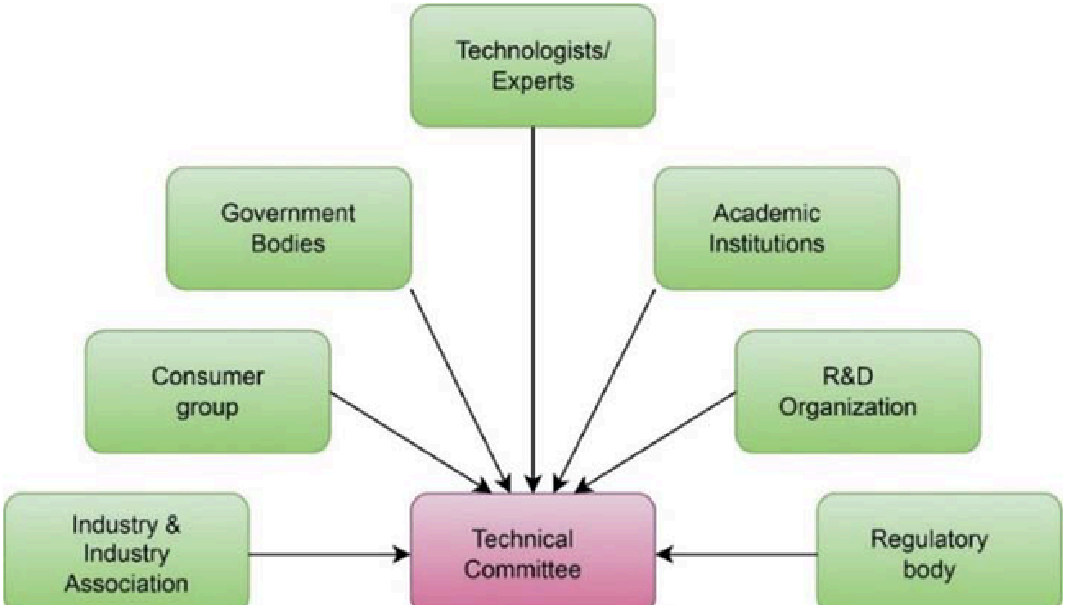
BIS also represents India in international standards bodies like the International Organization for Standardization (ISO) and via the Indian National Committee in the International Electrotechnical Commission (IEC) and participates actively in the international standardization work undertaken in these bodies. BIS presents the national viewpoints on new areas taken up for international standardization and on various draft international standards during the process of development of these standards so that the country's interest is protected and reflected in these standards. This also enables the BIS technical committees to consider adoption of the International Standards as Indian Standards, with or without modifications, in order to enable our products and services to integrate with global trade and commerce.

Aim of formulating a Standard:

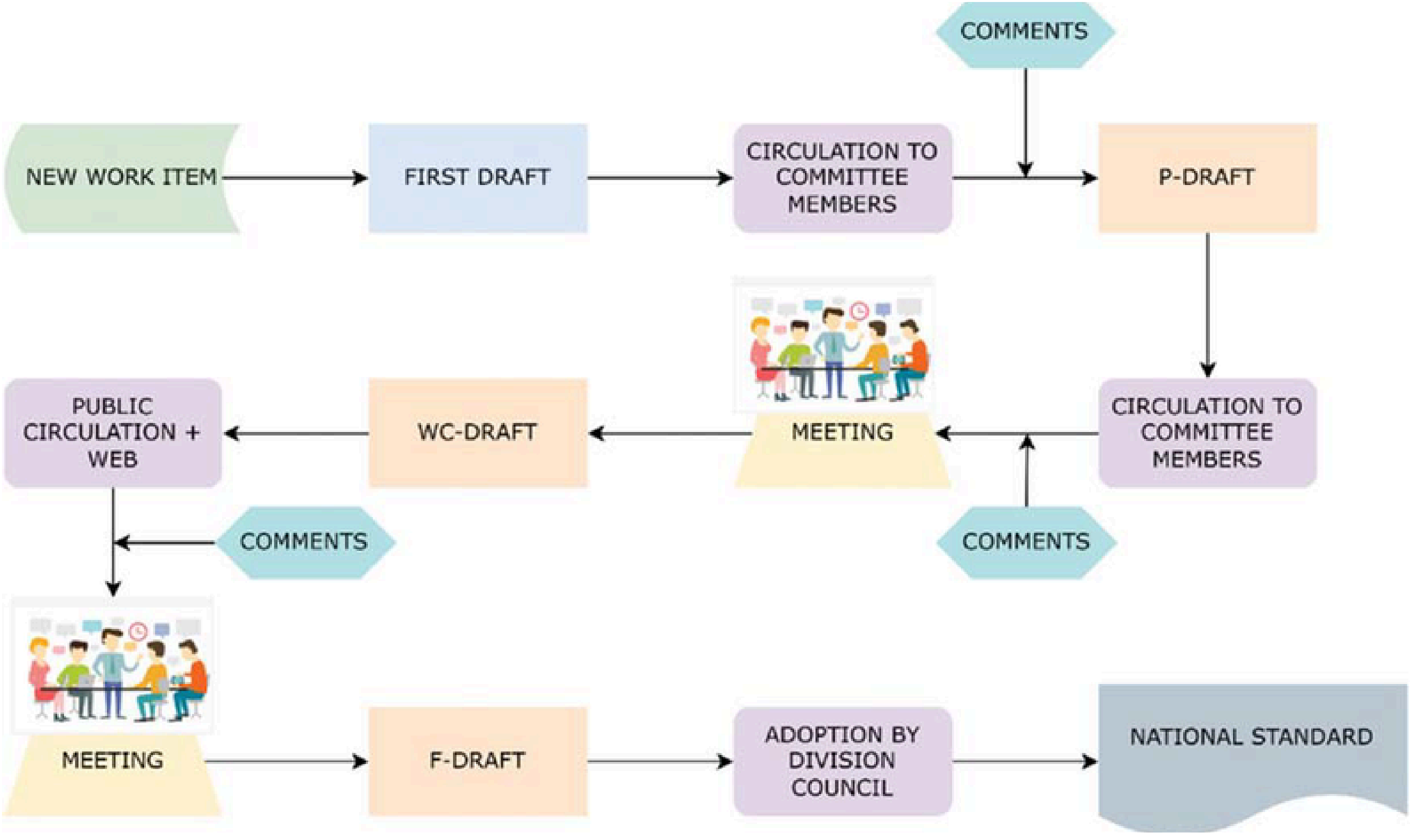
- Provision of means of communication amongst all interested parties;
- Promotion of economy in human effort, materials and energy in the production and exchange of goods
- Protection of consumer interests through adequate and consistent quality of goods and services;
- Promotion of the quality of life, safety, health and the protection of environment;
- Promotion of trade by removal of barriers caused by differences in national practices.

Process of Standards Formulation:

BIS formulates Indian Standards through sectional committees under a Division Council, which are set up to deal with specific group of subjects. The committee structure is designed to bring together all those with substantial interest in a particular field, so that standards are developed keeping in view the balance of interests among the relevant stakeholders.



COMMITTEE STRUCTURE



STANDARD DEVELOPMENT PROCESS

Standards formulation departments of BIS

There are 17 Technical Departments formulating standards in various subject areas. Corresponding to these departments 17 division councils exist. Each division council has several sectional committees working under it. The standards cover important segments of economy and help the industry in upgrading the quality of their goods and services.

PETROLEUM, COAL AND RELATED PRODUCTS DEPARTMENT

Petroleum, Coal and Related Products Department (PCD) is one of the 17 technical departments of BIS with the scope “Standardization in the field of petroleum, lubricants, bio-fuels, solid mineral fuels, gaseous fuels, bitumen, coal tar, organic chemicals, dye-intermediates, plastics (including adhesives and composites), safety of toys, rubber, cosmetics and fragrance & flavour ingredients”.

PCDC is the division council of PCD department.

The aspect wise breakup of the standards formulated by PCDC as on 03 February 2025 is given below:

Aspect	***No. of Standards***
Product Standards	694
Methods of Test	468
Codes of Practice	74
Terminology	35
Dimensions	6
System Standard	8
Safety Standard	26
Service/process Specification	19
Others	224
Total	***1554***

PCD, BIS is a ‘P’ member (Participating member) in 34 committees and ‘O’ Member (Observer member) in 5 committees of ISO.

Under PCD, there are 15 Sectional Committees working in various subject areas. List of committees with their scope is as follows:

- PCD 01 Methods for Sampling and test for Petroleum and related products of synthetic or biological origin including lubricants, greases, specialty products, additives and gaseous fuels (excluding bitumen and hydrogen)
- PCD 03 Petroleum and their Related Products of Synthetic or Biological Origin
- PCD 06 Bitumen, Tar and Related Products
- **PCD 07 Solid Mineral Fuels and Solid Biofuels**
- PCD 09 Organic Chemicals, Alcohols and Allied Products
- PCD 12 Plastics
- PCD 13 Rubber and Rubber Products
- PCD 18 Fragrance and Flavour
- PCD 19 Cosmetics
- PCD 21 Plastics Packaging
- PCD 25 Lubricants and their Related Products
- PCD 26 Dye Intermediates
- PCD 27 Methods of Sampling and Test for Plastics
- PCD 29 Methods of Test for Rubber and Rubber Products
- PCD 30 Toys and Related Test Methods Sectional Committee

Solid Mineral Fuels and Solid Biofuels Sectional Committee

(PCD 07)

Solid Mineral Fuels and Solid Biofuels Sectional Committee, PCD 07 is one of the technical committee under Petroleum, Coal and Related Products Department, with a scope to formulate Indian Standards for

a) Terminology, methods of sampling and test, codes of practice, classification by size and type, petrographic analysis and specification for solid mineral fuel

b) Terminology, methods of sampling and test, codes of practice, classification and specification for raw and processed materials originating from arboriculture, agriculture, aquaculture, horticulture and forestry to be used as a source for solid bio-fuels.

PCD 07 has developed approximately 95 standards covering Coal, Coke, and Solid Biofuels. Of these, 18 standards focus on Solid Biofuels, while 77 standards pertain to Solid Mineral Fuels, including coal, coke, and lignite.

The aspect-wise breakup of the standards formulated by PCD 07 as on 03 February 2025 is given below:

Aspect	**No. of Standards**	
	Solid Mineral Fuels	**Solid Biofuels**
Product Standards	7	2
Methods of Test	47	14
Codes of Practice	1	0
Terminology	6	1
Dimensions	2	0
Sampling/Sample Preparation	12	1
Others	2	0
Total	***77***	***18***

These standards are formulated by representatives of a balance of stakeholders including consumers, industry, R&D institutes, government departments/ regulators, technologies through various technical committees including sectional committee, subcommittee, panel and working group.

This handbook acknowledges the valuable contributions of the following experts in formulating the Indian Standards on Solid Biofuels:

Solid Mineral Fuels and Solid Biofuels Sectional Committee, PCD 07

Organization	Representative (s)
CSIR - Central Institute for Mining and Fuel Research, Dhanbad	Prof Arvind Kumar Mishra (***Chairperson***)
Bharat Coking Coal Limited, Dhanbad	Shri Samir Kumar Kundu Shri G. Venugopal (Alternate)
Bharat Heavy Electricals Limited, Tiruchirappalli	Dr Sivaji Seepana Shri Manda Teja Tarakesh (Alternate I) Shri Aman Seraphim Surin (Alternate II) Ms Aritra Chakraborty (Alternate III)
Cement Manufacturers Association, New Delhi	Shri Raju Goyal Shri Vipul Singh (Alternate I) Shri Ashutosh Shrivastava (Alternate II) Shri Shubho Chakravarty (Alternate III)
Central Electricity Authority, New Delhi	Shri Rajeev Kumar Mittal Ms Rita Nagdeve (Alternate)
Central Mine Planning and Design Institute Limited, Ranchi	Shri Vikash Rajak Shri Priyankar Upadhyay (Alternate)
Central Pollution Control Board, New Delhi	Shri Nazimuddin Shri Gaurav Gehlot (Alternate)

Organization	Representative (s)
Central Revenue Control Laboratory, New Delhi	Shri V. Suresh Dr. Mahesh Kumar (Alternate)
Coal Controller Organization, Kolkata	Shri Anjani Kumar
Coal India Limited, Kolkata	Shri Subhasis Sahu
CSIR - Central Institute for Mining and Fuel Research, Dhanbad	Dr Ashok Kumar Singh Dr Sujan Saha (Alternate)
CSIR - Institute of Minerals and Materials Technology, Bhubaneswar	Dr Suddhasatwa Basu Dr D. S. Rao (Alternate I) Dr Manish Kumar (Alternate II)
CSIR-National Metallurgical Laboratory, Jamshedpur	Dr Sanchita Chakravarty Ms Rupa Das Biswas (Alternate)
Geological Survey of India, Kolkata	Dr Sudip Bhattacharyya
Hindalco Industries Limited, Ranchi	Shri Mirajul Haque Dr Rahul Mitra (Alternate)
Indian Council of Agricultural Research, New Delhi	Dr Panna Lal Singh
Indian Institute of Technology (ISM), Dhanbad	Dr S. Bhattacharya
Indian Metals and Ferro Alloys Limited, Bhubaneswar	Shri Dinesh Kumar Mohanty
Mahanadi Coal Field Limited, Burla	Shri Ravikesh Kumar Raju Shri Neeraj Kalla (Alternate)
Ministry of Coal, New Delhi	Shri Anandi Prasad
Ministry of Environment Forest and Climate Change, New Delhi	Shri N. Subrahmanyam
Ministry of New and Renewable Energy, New Delhi	Shri Hiren Chandra Borah Shri Vikram Dhaka (Alternate)

Organization	Representative (s)
National Test House, Kolkata	Shri S. K. Laha Shri Ratul Bezbaruah (Alternate)
National Council for Cement and Building Materials, Faridabad	Shri Ankur Mittal Shri Gaurav Bhatnagar (Alternate)
NLC India Limited, Chennai	Shri R. Shanmugasundaram Shri G. Thirumurugan (Alternate)
Northern Coalfields Limited, Singrauli	Shri Satish Jha
Steel Authority of India Limited (SAIL), Research & Development Centre for Iron & Steel, Ranchi	Shri K. K. Manjhi Shri B. Chakraborty (Alternate)
Tata Power Limited, Mumbai	Shri Pravash Sinha
Tata Steel Limited, Kolkata	Shri B. K. Singh Shri H. N. Prasad (Alternate)
The Energy and Resources Institute, New Delhi	Shri Sunil Dhingra Shri N. K. Ram (Alternate)
The Singareni Collieries Company Limited, Kothagudem	Shri Syed Habeeb Hussain
BIS Directorate General	Shri Chinmay Dwivedi, Scientist 'E'/Director and Head (Petroleum, Coal and Related Products Department) [Representing Director General (Ex-officio)]
Member Secretary Ms Aditi Choudhary Scientist 'C'/Deputy Director (Petroleum, Coal And Related Products Department), BIS	

NATIONAL MISSION ON USE OF BIOMASS IN THERMAL POWER PLANTS

(SAMARTH: SUSTAINABLE AGRARIAN MISSION ON USE OF AGRI-RESIDUE IN THERMAL POWER PLANTS)

Background:

Biomass, derived from agricultural and organic residues, is central to India's renewable energy goals. With over 755 million tonnes of biomass produced annually, 228 million tonnes are surplus and much of which is burned in the fields, significantly contributing to air pollution and greenhouse gas emissions.

This leads to formulation of biomass co-firing policy and subsequently 'National Mission on Use of Biomass in Coal Based Thermal Power Plants'.

It was decided to explore the possibility of use of surplus biomass/stubble for co-firing with coal in thermal power plants.

MoP issued a policy on 17.11.2017 for biomass pellets co-firing and subsequent advisory was sent to all coal based power plants by CEA.

MoP set-up National Mission on Use of Biomass in TPPs on 12.07.2021.

To reduce the carbon footprint of thermal power plants and prevent stubble burning, the Ministry of Power has established the 'National Mission on Use of Biomass in Thermal Power Plants' also known as the SAMARTH Mission (Sustainable Agrarian Mission on the Use of Agro-Residue in Thermal Power Plants) on 12.07.2021, under which biomass pellets co-firing in TPPs is being promoted.

SAMARTH Mission Initiatives:

To accelerate the process of biomass co-firing in Thermal Power Plants, SAMARTH mission has taken following several steps in various domains:

Policy Formulation:

- Revised policy for Biomass co-firing issued on 08.10.2021 mandating 5 to 7 % biomass co-firing with coal in coal-based thermal power plants.
- Addendum to the Revised Policy issued on 03.05.2023 for inclusion of Bamboo and its by-products for manufacturing Biomass pellets.
- Modified Revised Biomass Policy issued on 16.06.2023 indicating price benchmarking of biomass pellets and procurement process of pellets.

Enabling Guidelines:

- Ministry of Power (MoP) has issued benchmarked price for procurement of non-torrefied biomass pellets for NCR, NR & WR Plants.
- Model SOP on co-firing of Biomass pellet in PF & FBC Boilers issued to all TPPs.
- Model Contract for Biomass procurement issued.

Financial Assistance:

- Financial grant to New Pellet plants through MNRE and CPCB schemes.
- Bankable Project report issued for Biomass pellet plants in association with SBI.
- Exclusive loan schemes for Biomass pellet manufacturing by SBI and other Government Banks.

Awareness campaigns & capacity building programs across nation

Research and Development

- *12 number of projects undertaken through Sub-Group 1 of Mission.*
- *8 number of research project under Department of Science and Technology.*

Testing & Standardization

- Issuance of 18 Indian Standards through BIS for testing of Solid biofuels (Biomass).
- Identification of 21 NABL accredited labs for testing of Biomass pellets.

Ease of Doing Business:

- Provisioning of necessary administrative approvals for Biomass Pellet Plant Installation through ‘National Single Window System’.
- Identification of ‘Biomass pellet manufacturing’ under ‘Priority Sector Lending’ (PSL) by RBI.
- Procurement Provision of Biomass pellets created on ‘GeM’ portal.
- Provision for trading of Agro-residue as commodity has been enabled by ‘e-NAM’ through its portal.
- Extended Storage Infrastructure facilities under AMI sub-scheme of ‘Integrated Scheme on Agricultural Marketing’ (ISAM) for Storage of Biomass.

INDIAN STANDARDS

SOLID BIOFUELS

IS 18721:2024

SUMMARY

Vocabulary promote clarity, quality assurance, and efficiency across production, trade, and utilization.

This standard prescribes the vocabulary used for solid biofuels, encompassing raw and processed material including woody, herbaceous, fruit and aquatic biomass, originating from agriculture, horticulture, forestry, arboriculture and aquaculture.

The terms and definitions in this standard are categorized based on the origin and source of solid biofuels in the supply chain; traded forms and biofuels produced during preparation; relevant properties, sampling, testing, classification, and specification; handling and processing based on biomass sources; and bioenergy resulting from solid biofuel conversion.

IS 18721 : 2024

SOLID BIOFUELS – VOCABULARY

Indian Standard

SOLID BIOFUELS — VOCABULARY

1 SCOPE

This standard prescribes the vocabulary used for solid biofuels. This standard includes only raw and processed material originating from agriculture, horticulture, forestry, arboriculture and aquaculture.

NOTES

1 Raw and processed material includes woody, herbaceous, fruit and aquatic biomass from the sectors mentioned above.

2 Chemically treated material does not include halogenated organic compounds or heavy metals at levels higher than those in typical virgin material values or higher than values of the country of origin.

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute the provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	*Title*
IS 3810	Solid mineral fuels — Vocabulary:
(Part 2) : 2002	Terms relating to sampling, testing and analysis (*first revision*)
(Part 3) : 2023	Glossary of terms relating to solid mineral fuels: Part 3 Coke (*first revision*)
IS 17654 : 2021/ ISO 18125 : 2017	Solid biofuels — Determination of calorific value

3 TERMS AND DEFINITIONS

The following are the most relevant terms used for solid biofuels (sources, processes, in-process material or finished or semi-finished products):

3.1 Additive — Material which has been intentionally introduced into the fuel feedstock to improve quality of fuel (for example, combustion properties), to reduce emissions or to make production more efficient.

3.2 Agrofuels — Biofuels obtained from energy crops and/or agricultural by-products (agricultural residues).

3.3 Air-dried — Condition in which the solid biofuel has dried in air to equilibrium moisture content.

3.4 Animal Biomass — Biomass obtained from livestock.

3.5 Animal by Products/Animal Residues — Agricultural and animal by-products (or residues) obtained during livestock operations.

NOTE — It includes among others solid excreta of animals.

3.6 Aquatic Biomass — Biomass from plants that have adapted to living in or on aquatic environments.

3.7 As Analyzed/As Determined Basis — Reference moisture content of the material at the moment of analysis/determination.

3.8 As Received/As Delivered — Calculation basis for a material in the delivery state.

3.9 Ash/Ash Content/Total Ash — Mass of inorganic residue remaining after combustion of a fuel under specified conditions, typically expressed as a percentage of the mass of dry matter in fuel.

NOTES

1 Depending on the combustion efficiency the ash may contain combustibles.

2 If a complete combustion is realized ash contains only inorganic, non-combustible components.

3.10 As Deformation Temperature/Deformation Temperature — Temperature at which first signs of melting occur.

NOTE — Ash deformation temperature can be seen as rounding of the edges, smoothing of surfaces, expansion of the cylinder or general changing of the cylinder shape. If the test piece starts to swell or bubble without the edges being rounded, the temperature is registered as DT (since swelling and bubbling only occur when a fraction of the ash is melted).

3.11 Ash Flow Temperature — Temperature at which the ash is spread out over the supporting tile in a layer, the height of which is half of the height of the test piece at the ash hemisphere temperature.

3.12 Ash Fusibility/Ash Melting Behavior — Characteristic set of temperatures at which the ash undergoes certain physical stages of melting during heating under specific conditions.

3.13 Ash Hemisphere Temperature/Hemisphere Temperature — Temperature at which the test piece forms approximately a hemisphere, that is, when the height is half of the base diameter.

3.14 Ash Shrinkage Starting Temperature — Temperature at which the area of a test piece falls below 95 percent of the original test piece area under specific conditions due to shrinking of a test piece.

3.15 Bagasse/Sugarcane Bagasse — Dry pulpy residue left after the extraction of juice from sugarcane.

3.16 Bag weight — Weight of solid biofuel plus bag.

3.17 Bale/Baled Biofuel — Solid biofuel which has been compressed and bound to keep its shape and bulk density.

Example: Straw bales, bales of energy grass, bales of logging residues.

3.18 Bark — Organic cellular tissue which is formed by taller plants (trees, bushes) on the outside of the growth zone (cambium) as a shell for the wooden body.

3.19 Biobased — Derived from biomass.

3.20 Biobased Content — Fraction of a fuel that is derived from biomass.

NOTE — Normally expressed as a percentage of the total mass of the product.

3.21 Biobased Product/Biobased Industrial Product/Bioproduct — Product wholly or partly derived from biomass.

NOTE — The biobased product is normally characterized by the biobased carbon content or the biobased content.

3.22 Biochar/Biocarbon/Biocoke/Bio-charcoal/ Biocoal/Charcoal — Solid carbonaceous biofuel derived by carbonization or distillation or pyrolysis of biomass.

3.23 Bioenergy — Energy derived from Biomass.

3.24 Biofuel — Solid, liquid or gaseous fuel produced directly or indirectly from biomass.

3.25 Biofuel Blend — Biofuel resulting from intentionally mixing of different biofuels, for example, straw or energy grass with wood, dried bio sludge with bark.

3.26 Biofuel Briquette — Densified biofuel made with or without additives in pre-determined geometric form with at least two dimensions (length, width, height) of more than 25 mm, produced by compressing biomass.

3.27 Biofuel Mixture — Biofuel resulting from natural or unintentional mixing of different biofuel and/or different types of biomasses.

3.28 Biofuel Pellet — Densified biofuels made with or without additives (3.2) usually with a cylindrical form, random length typically 5 mm to 40 mm and diameter up to 25 mm and broken ends, produced by compressing biomass.

3.29 Biomass — Organic material, that is, plant or animal based, including energy crops, agricultural crops and trees, food, feed and fibre crop residues, aquatic plants, algae, forestry and wood residues, agricultural wastes, processing by-products and other non-fossil organic matters.

3.30 Biomass By-product/Biomass Residue — Biomass from well-defined side-streams from forestry, agriculture or aquaculture or biomass product generated during the production of something else.

Example: Sawdust when sawing timber, olive press cake after pressing of oil, logging residues.

3.31 Biomass Resource Owner — Body or enterprise with the right to exploit the biomass resources for energy purposes.

NOTE — The biomass resource owner can be a land or forest owner, a company etc.

3.32 Biomethane — Methane produced from biomass (for example, solid biofuels).

3.33 Biosludge — Sludge formed in the aeration basin during biological wastewater treatment or biological treatment process and separated by sedimentation or flotation.

3.34 Black Liquor — Fluid containing lignin removed from the wood in the pulping process.

NOTES

1 Black liquor also contains pulping chemicals.

2 Black liquor is not a solid biofuel. The term is included for information only.

3.35 Bulk Density — Mass of a portion (that is, a large quantity of particulate material) of a solid fuel divided by the volume of the container which is filled by that portion under specific conditions.

3.36 Bulk Volume — Volume of a material including space between the particles.

3.37 Bundled Biofuel — Solid biofuels which has been bound together and where there is a lengthwise orientation of the material.

3.38 Calorific Value/Heating Value — Energy amount per unit mass or volume released on complete combustion (*see* IS 17654).

3.39 Cereal Crops — Annual crops grown with the main purpose of using the seed for food production.

Example: Barley, wheat, rye, oat.

NOTE — Some cereal crops can be used as a solid biofuel.

3.40 Certified Reference Material — Reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

3.41 Chemical Treatment — Any treatment with chemicals other than air, water or heat.

NOTE — The treatment with chemicals may take place in presence or absence of air, water or heat.

3.42 Chopped Straw — Straw which has been cut into small pieces.

3.43 Chunkwood — Wood cut with sharp cutting devices where most of the material have typical particle lengths of 50 mm to 150 mm, which are substantially longer and coarser than wood chips.

3.44 Coarse Pellet Fines — Particles with a size ranging from ≥ 3.15 mm to < 5.6 mm resulting from breakage of pellets during production or handling.

3.45 Coke — The solid residue of impure carbon obtained from carbon rich feedstock after removal of volatile material by destructive distillation. [*see* IS 3810 (Part 3)].

3.46 Combined Sample — Sample consisting of all the increments taken from a lot or a sub-lot.

NOTE — The increments may be reduced by division before being added to the combined sample.

3.47 Complete Tree — Tree, including limbs and root system.

3.48 Contamination — Occurrence of any undesirable matter such as chemical, physical and/or microbiological matter in the product.

3.49 Critical Control Point Quality Control Point — Point within or between processes at which relevant properties can be most readily assessed, quality control points also offer the greatest potential for quality improvement.

3.50 Cross Cut Ends — Short pieces of woody biomass which occur when the ends of logs or sawn timber are crosscut off, with or without bark.

3.51 Cutter Chips — Wood chips made as a by-product of the wood processing industry, with or without bark.

3.52 Delivery Lot — Solid biofuel batch on which the essential quality requirements for solid biofuel are focused.

NOTE — Delivery lot can be an individual delivery lot, which is an agreed quantity of solid biofuel (for example, a package, shipload or truck load) or continuous delivery, where several loads are delivered to the end-user during an agreed period of time (usually daily or weekly delivery).

3.53 Demolition Wood — Used wood arising from demolition of buildings or civil engineering installations.

3.54 Densified Biofuel Compressed Biofuel — Solid biofuel made by mechanically compressing (at normal or elevated temperature) biomass (or thermally treated biomass) to mould the solid biofuel into a specific size and shape such as cubes, pressed logs, biofuel pellets or biofuel briquettes.

3.55 Density — Ratio of mass to volume.

NOTES

1 It must always be stated whether the density refers to the density of individual particles or to the bulk density of the material and whether the mass of water in the material is included.

2 Also see bulk density and particle density.

3.56 Dry Ash Free basis — Calculation basis in which the solid biofuel is considered free from moisture and inorganic matter.

3.57 Dry Basis — Calculation basis in which the solid biofuel is considered free from moisture.

3.58 Dry Matter — Material remaining after removal of moisture under specific conditions.

3.59 Dry Matter Content — Portion of dry matter in the total material on mass basis expressed as a percentage of the total mass of the solid biofuel.

3.60 Dust — Fragmented material of small size passing a 500 µm sieve caused by a non-intentional process.

3.61 Edging Parts of Woody — Biomass which occur when trimming sawn timber and which show a remainder of the original rounded surface of the tree, with or without bark.

3.62 Energy Crops — Woody or herbaceous crops grown and harvested specifically for their fuel value.

3.63 Energy Density — Ratio of net energy content and bulk volume.

NOTE — The energy density is calculated using the net calorific value determined and the bulk density.

3.64 Energy Forest Trees — Woody biomass grown specifically for its fuel value in medium to long rotation forestry.

3.65 Energy Grain — Grain used for energy purpose.

3.66 Energy Grass — Herbaceous grass grown and harvested specifically for use as fuel.

Example: Sugarcane, miscanthus, reed canary grass.

3.67 Extraneous Ash — Ash from contaminants entering the material at harvest, logging, treatment, transport, storage etc.

3.68 Extraneous Substances — Foreign non biomass materials such as particles not belonging to the particular biomass such as stone, glass or clay etc entering the biomass or solid biofuel during any stage starting from harvesting to final storage for utilization.

3.69 Feedstock — Material that is further processed for conversion to bioenergy, biofuel and/or biobased products.

3.70 Fibre Saturation Point — Moisture content of woody material at which only the cell walls are completely saturated (all bound water) but no free water exists in the cell lumens typically about 27 percent total moisture (wet basis).

3.71 Fibre Sludge — Sludge formed in the sedimentation basin as a part of the wastewater treatment process in a pulp and paper mill and separated by sedimentation or flotation.

NOTE — The main component is pieces of wood fibres. The sludge can be dewatered and further processed into a solid biofuel.

3.72 Fines — Fraction of small sized particles as defined by a specification or user.

NOTE — In the solid biofuels, standards fines are always defined as particles passing a 3.15 mm round hole sieve.

3.73 Firewood — Cut and split oven-ready energy wood usually used in household wood burning appliances like stoves, fireplaces and central heating systems.

NOTE — Firewood usually has a uniform length, typically in the range of 15 cm to 100 cm.

3.74 Fixed Carbon — Remaining carbon calculated by subtracting total moisture, ash and volatile matter from 100 percent.

3.75 Flash Point — Temperature at which the vapor of a material will ignite when exposed to an ignition source.

3.76 Flammability — Propensity by gaseous, liquid or solid material to catch fire upon exposure to ignitable external source.

NOTE — For solids like dust from biomass the flammability is determined by testing method establishing the speed of burning in mm per time unit.

3.77 Foreign Material/Impurity — Material other than claimed, which has entered the fuel.

NOTE — Examples of impurities for biofuels are stones, soil, pieces of metal, plastics, rope.

3.78 Forest Chips — Forest wood in the form of wood chips.

3.79 Forest Fuels — Forest fuel is produced directly from forest wood or plantation wood by a mechanical process, the raw material has not previously had another use.

3.80 Forest Wood — Wood from forest, plantation and other virgin wood including segregated wood from gardens, parks, roadside maintenance, vineyards, fruit orchards and driftwood from freshwater.

3.81 Fruit Biomass — Part of a plant which holds seeds.

3.82 Fuel — Energy carrier intended for energy conversion.

NOTE — Fuels are solid, liquid or gaseous.

3.83 Fuel Powder/Fuel Flour — Pulverized fuel with a typical particle size less than 1 mm.

3.84 Fuel Specification — Description of origin and source, fuel traded form and fuel properties.

3.85 Fuel Wood/Energy Wood — Wood fuel where the original composition of the wood is maintained and which has only been threated mechanically.

3.86 General Analysis Sample — Sub-sample of a laboratory sample having a nominal top size of 1 mm or less and used for a number of chemical and physical analyses.

3.87 Green Biomass — Biomass with a moisture content close to fresh after harvesting but no further quality specified.

3.88 Green Chips/Fresh Chips — Wood chips produced from recently harvested woody biomass.

3.89 Grinding Dust — Dust-like wood residue formed in grinding/smoothening wood surfaces.

3.90 Gross Calorific Value (GCV)/Higher Heating Value (HHV) — Measured value of specific energy of combustion of a solid fuel burned in oxygen in a calorimetric bomb under such conditions that all the water of the reaction products is in the form of liquid water.

3.91 Gross Density — Ratio of the mass of a wooden body and its volume, including all cavities (pores and vessels), based on specific total moisture.

3.92 Heat Rate — Measure of the number of heating units required to generate output energy over a length of time expressed in kWh or MWh.

3.93 Heavy Extraneous Materials — Extraneous substances $\geq$ 3.15 mm with a specific density > 1 g/cm^3.

3.94 Herbaceous Biomass — Biomass from plants that has a non-woody stem and which dies back at the end of the growing season.

3.95 Herbaceous Fuels — All types of biofuels originating from herbaceous biomass.

3.96 Hog Fuel/Shred — Woodfuel that has pieces of varying size and shape, produced by crushing with blunt tools such as rollers, hammers or flails.

3.97 Hydrothermal Carbonized Biomass — Solid biofuel produced by hydrothermal carbonization of biomass, under specified conditions.

3.98 Increment — Portion of fuel extracted in a single operation of the sampling device.

3.99 Inorganic Matter — Non-combustible fraction of a fuel.

3.100 Laboratory Sample — Combined sample or a sub-sample of a combined sample for use in a laboratory.

3.101 Lignocellulose — Plant cell wall biomass composed primarily of cellulose, hemicelluloses and lignin.

3.102 Log Wood — Cut fuel wood in which most of the material has a length of 500 mm and above.

3.103 Logging Residues — Woody biomass residues created during wood harvesting.

NOTE — Logging residues include branches and treetops that can be salvaged when fresh or after seasoning.

3.104 Lot — Defined quantity of fuel for which the quality is to be determined.

3.105 Major Elements — The elements in the fuel that predominantly will constitute the ash, including aluminum (Al), calcium (Ca), iron (Fe), magnesium (Mg), phosphorus (P), potassium (K), silicon (Si), sodium (Na) and titanium (Ti).

3.106 Mass-reduction — Reduction of the mass of a sample or sub-sample.

3.107 Mechanical Durability — Ability of densified fuel units (for example, briquettes, pellets) to remain intact during handling and transportation.

3.108 Minor Elements — Elements in the fuel that are at small concentrations.

NOTE — Concerning solid biofuels, minor elements include, but not limited to elements (not all are metals) such as arsenic (As), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), mercury (Hg), manganese (Mn), molybdenum (Mo), nickel (Ni), lead (Pb), antimony (Sb), selenium (Se), tin (Sn), thallium (TI), vanadium (V) and zinc (Zn).

3.109 Moisture Sample — Sample taken specifically for the purpose of determining total moisture [*see* IS 3810 (Part 2)].

3.110 Municipal Solid Waste — Waste stream consisting of end-of-life-materials.

NOTE — Municipal solid waste can contain biomass fractions as well as non-biomass fractions.

3.111 Net Calorific Value (NCV)/Lower Heating Value (LHV) — Calculated value of specific energy of combustion of a solid fuel burned in oxygen under such conditions that all the water of the reaction products remain as water vapour.

3.112 Nominal Top Size — Smallest aperture size of the sieve through which at least 95 percent by mass of the material passes during the determination of particle size distribution of solid fuels.

3.113 Non-woody Biomass — Biomass originating from herbaceous, fruit or aquatic biomass as well as their blends or mixtures with woody biomass.

NOTE — Stem of fruit trees and energy crops, crop residues.

3.114 Non-woody Briquette — Biofuel briquette made from non-woody biomass

NOTES

1 The raw material for non-woody briquettes can be herbaceous, crop residues, fruit or aquatic biomass or biomass blends and mixtures.

2 Briquettes are usually manufactured in a piston press, with the total moisture content on wet basis usually being less than 15 percent of the mass.

3.115 Non-woody Pellet — Biofuel pellet made from non-woody biomass.

NOTE — The raw material for non-woody pellets is crop residues, herbaceous, fruits or aquatic biomass. Pellets are usually manufactured in a die, with total moisture content usually less than 15 percent of their mass on wet basis.

3.116 Off-gassing — Spontaneous emission of condensable (for example, terpenes) and non-condensable gases (for example, carbon monoxide, carbon dioxide, methane) from biomass.

3.117 Organic Matter — Combustible fraction of the fuel.

3.118 Oscillating Screen Classifier — Device containing one or multiple oscillating (flat) screens used to separate material into size classes for calculation of particle size distribution.

3.119 Oven Dry Matter — Biomass free of moisture, produced by drying to constant weight under specific conditions.

3.120 Oversize Particles — Particles exceeding a specific particle size limit value or values.

NOTE — Limit values may be given in three dimensions.

3.121 Particle — Any discrete unit of matter.

3.122 Particle Density — Density of a single particle.

NOTE — Pores within the particle are included.

3.123 Particle Size — Size of the particle as determined.

NOTES

1 Different methods of determination may give different results.
2 See also particle size distribution and fines.

3.124 Particle Size Distribution — Proportions of various particle sizes in a solid fuel.

3.125 Particle Size Reduction/Size Reduction — Reduction of the nominal top size of a sample or sub-sample.

3.126 Permeability in Storage/Bulk Permeability — Ability of gas such as air to pass through the void in biomass during storage.

NOTE — Permeability is measured in pressure (Pa) verses flow of gas ($m^3/s/m^2$) and depends on shape and orientation of particles and the bulk porosity of biomass.

3.127 Pressing Aid — Additive used for enhancing the production of densified fuels.

3.128 Primary Biomass — Biomass produced directly by photosynthesis and harvested or collected from the field or forest where it is grown.

Examples: Energy grain, perennial grasses, wood crops, crop residues and residues from logging.

3.129 Producer — Organization, unit or individual responsible for the production of the fuel.

NOTES

1 The producer can be responsible for any operation with the purpose of changing the biofuel properties.
2 The producer can also be the supplier of the fuel.

3.130 Product Declaration — Document dated and signed by the producer/supplier to the retailer or end-user, specifying origin and source, traded form and properties of defined lot, delivery period or delivery agreement.

3.131 Proximate Analysis — Quantitative analysis of a solid fuel reported in terms of prescribed methods for total moisture, volatile matter, ash content and fixed carbon measured at specified conditions.

3.132 Pulverized Fuel Grinded Fuel — Solid fuel in the form of dust and powder, produced by milling or grinding.

3.133 Recovered Construction Wood — Used wood arising from construction of buildings or from civil engineering works.

3.134 Reference Material — Material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method or for assigning values to materials.

3.135 Rotary Screen — Device with cylindrical screens used to separate material into size classes for calculation of particle size distribution.

3.136 Sample — Quantity of material (all increments), representative of a larger quantity for which the quality is to be determined.

3.137 Sample Division — Division of a sample or sub-sample to an appropriate size which normally leads to a mass reduction of a sample or sub-sample.

3.138 Sample Preparation — Actions taken to obtain representative laboratory samples or test portions from the original sample.

3.139 Sampling Form — Document that shall be used during sampling to record data about the way in which the sampling is actually being carried out

3.140 Sampling Plan — Predetermined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a population as a sample.

3.141 Sampling Certificate — Report which serves as a check list and provides the investigator with all necessary information about the sampling techniques applied at the site and any additional important information.

3.142 Sawdust — Fine particles created when sawing wood in which most of the material has a typical particle length of 1 mm to 5 mm.

3.143 Secondary Biomass — Residues and by-product streams from food, feed, fiber, wood and materials processing plants (such as sawdust, black liquor and cheese whey) and manures from animal feeding operations.

3.144 Short Rotation Coppice (SRC) — Production of woody biomass, generally on agricultural lands, by regenerating new stems (shoots) from the (stool) stump or roots and relying on rapid growth, (harvested) generally over a 1 year to 8 year cycle.

3.145 Short Rotation Forestry — Production of trees (generally) on forest land, that rely on rapid growth of individuals harvested in short cycles (of 5 year to 15 years).

3.146 Shredded Biofuel — Solid biofuel which has been mechanical treated into smaller particles with blunt tools.

Example: Chopped straw, shredded bark, wood and hog fuel.

3.147 Slab — Piece initially sawn from the length of a log when squaring up the log, with or without bark.

NOTE — Approximate length 200 cm to 800 cm.

3.148 Slag — Biofuel derived ash that is or has been in a molten (or liquid) state.

3.149 Smallwood — Fuel wood cut with sharp cutting devices and in which most of the material has a particle length typically 50 mm to 500 mm.

3.150 Solid Biofuel — Solid produced directly or indirectly from biomass.

3.151 Solid Recovered Fuel — Solid fuel prepared from non-hazardous waste to be utilized for energy recovery in incineration or co-incineration plants.

3.152 Solid Volume — Volume of a mass of particles excluding the volume of the void spaces between the particles.

NOTE — Typically determined by a fluid displaced by a specific amount of material.

3.153 Soot — Fine black particles, chiefly composed of carbon, produced by incomplete combustion of carbon rich feedstock.

3.154 Stacked Volume — Volume of stacked material including the space between the material pieces.

3.155 Stem Wood/Roundwood — Part of tree stem with the branches and top removed, with a length of more than 100 cm.

3.156 Stem Wood Chips — Wood chips made of stem wood, with or without bark.

3.157 Stump — Part of the tree stem below the felling cut, including the roots.

3.158 Supply Chain — The overall process of handling, transporting and processing raw materials from the point of collection to the point of delivery to the end-user.

3.159 Tertiary Biomass — Post consumer residues and wastes, such as fats, greases, oils, construction and demolition wood debris, other waste wood from urban environments, as well as packaging wastes, municipal solid wastes and landfill gases.

3.160 Test Sample — Laboratory sample after an appropriate preparation made by the laboratory.

3.161 Thermally Treated Biomass — Biomass whose chemical composition has been changed by heat (usually by temperatures of 200 °C to 300 °C and above).

3.162 Thinning Residues — Woody biomass residues originating from thinning operations.

3.163 Torrefied Biomass — Solid biofuel produced by torrefaction of biomass.

3.164 Total Carbon/Carbon Content — Content of carbon (C) within dry, moisture free fuel.

NOTES

1 Other element content (for example, hydrogen, oxygen, nitrogen, sulfur, chlorine) similarly defined.

2 For solid biofuels the amount of total oxygen is generally calculated as the remaining portion in the dry fuel from the sum of the total ash, the total carbon, the total hydrogen, the total nitrogen, the total sulphur and the total chlorine in the dry fuel.

3.165 Total Mass — Mass of all components of the solid fuel, including dry matter and moisture.

3.166 Total Moisture/Moisture Content — Total water in the fuel removable under specific conditions.

NOTE — Indicate reference (as received, as dried, as equilibrated) to avoid confusion.

3.167 Tree Section — Part of a tree (with branches) which has been cut into suitable length but not processed.

NOTE — Tree sections can be processed for example to pulpwood or forest fuel.

3.168 Ultimate Analysis/Elemental Analysis — Analysis of a fuel reported in terms of its total carbon, total hydrogen, total nitrogen, and total sulphur measured at specified conditions and total oxygen calculated by formula.

3.169 Used Wood — Wood substances or objects which have performed their intended purpose.

NOTE — *See* also recovered construction wood and demolition wood.

3.170 Volatile Matter — Material, which is released when a fuel is heated in the absence of oxygen under specific conditions and corrected for moisture of the material.

3.171 Whole Tree — Felled, undelimbed tree, excluding root system.

3.172 Whole Tree Chips — Wood chips made from whole trees.

3.173 Wood Briquette — Biofuel briquette made from woody biomass.

3.174 Wood Chips — Chipped woody biomass with a sub-rectangular shape and a typical length 5 mm to 50 mm typically in the form of pieces with a defined particle size produced by mechanical treatment with sharp tools such as knives.

3.175 Wood Fuels/Wood Based Fuels Wood Derives Biofuels — Solid wood-based fuel.

3.176 Wood Pellet — Biofuel pellet made from woody biomass.

3.177 Wood Processing Industry By-products Wood Processing Industry Residues — Woody biomass by-products (or residues) obtained from wood processing and from the pulp and paper industry.

3.178 Wood Shavings — Small slices or slivers from woody biomass created when shaping wood.

3.179 Woody Biomass — Biomass originating from trees, bushes and shrubs together with their fruit, leaves and needles inherent to the biomass **.**

NOTE — This definition includes forest, plantation and other virgin wood, wood processing industry by- products and residues, and used wood.

IS 18640:2024

SUMMARY

Sample preparation is important to have reliable analysis of various properties of solid biofuel.

This standard prescribes two basic methods used during the sample preparation that are sample division and particle size-reduction of the sample.

It also establishes procedures for reducing samples (or increments) to laboratory samples and further into sub-samples and general analysis samples.

The samples prepared through these procedures are then used for various analyses, including calorific value, moisture content, ash content, bulk density, durability, particle size distribution, ash melting behavior, etc.

SOLID BIOFUELS – SAMPLE PREPARATION

Indian Standard

SOLID BIOFUELS — SAMPLE PREPARATION

1 SCOPE

1.1 This standard prescribes methods for reducing combined samples (or increments) to laboratory samples and laboratory samples to sub-samples and general analysis samples and is applicable to solid biofuels.

1.2 The methods prescribed in this standard are to be used for sample preparation, when the samples are to be tested for calorific value, moisture content, ash content, bulk density, durability, particle size distribution, ash melting behavior, chemical composition, and impurities.

2 REFERENCES

The standards given below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No./Other Standards	*Title*
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves
IS 17655	Solid biofuels — Determination of moisture content — Oven dry method:
(Part 1) : 2021/ ISO 18134-1 : 2015	Total moisture — Reference method
(Part 2) : 2021/ ISO 18134-2 : 2017	Total moisture — Simplified procedure
ISO 16559 : 2022	Solid biofuels — Vocabulary
ISO 18135 : 2017	Solid biofuels — Sampling

3 TERMINOLOGY

For the purpose of this standard, the definition as given in ISO 16559 and the following definition shall apply.

3.1 Nominal Top Size — Aperture size of the sieve through which at least 95 percent by mass of the material passes.

NOTE — For pellets (and other long materials), the diameter is used to determine the nominal top size.

4 PRINCIPLES OF CORRECT SAMPLE REDUCTION

4.1 The main purpose of sample preparation is that a sample is reduced to one or more test portions that are in general smaller than the original sample. The main principle for sample reduction is that the composition of the sample as taken on site shall not be changed during each stage of the sample preparation. Each sub-sample shall be representative of the original sample. To reach this goal, every particle in the sample before sample division shall have an equal probability of being included in the sub-sample following sample division. Two basic methods are used during the sample preparation are sample division and particle size-reduction of the sample.

NOTE — Avoid loss of moisture and fine particles during milling and other operations.

4.2 Because of the risk of changes in the moisture content (loss of moisture), a sub-sample (moisture analysis sample) shall be separated at the earliest possible stage of the sample preparation procedure. As an alternative, a separate moisture analysis sample may be taken. The sample reduction shall be carried out by a procedure that does not conflict with the requirements of IS 17655 (Part 1) or IS 17655 (Part 2).

4.3 For materials that have to be examined for moisture content, care should be taken to avoid any significant heat build-up and risk of drying.

5 APPARATUS

5.1 Apparatus for Sample Division

Sample division is the process of reducing the mass of the sample without reducing the size of the particles. The following apparatus are used for sample division.

NOTE — If the nominal top size of the sample is not known, the value has to be assumed. After the sample reduction, the assumed value should be compared to the actual value to assure that the requirements of the apparatus for sample division and sample size used are met.

5.1.1 *Riffle Boxes*

A riffle box should have an equal number of slots and at least six at each side (preferably more if possible), with adjacent slots directing material into different sub-samples, and the width of the slots shall be at least 2.5 times the nominal top size of the material to be riffled (*see* Fig. 1).

5.1.2 *Rotary Sample Dividers*

The inner dimensions of the equipment where the sample is fed shall be at least 2.5 times as wide as the nominal top size of the material to be processed and should be large enough to handle normal oversized material particles. The rotary sample divider shall have a feeder device adjusted, so that the number of compartments multiplied by the number of rotations shall not be less than 120 while the sample is being divided (for example *see* Fig. 2).

5.1.3 *Shovels and Scoops*

A shovel or scoop are tools used for manual sample division. They shall have a flat bottom, with edges raised high enough to prevent particles from rolling off, shall be at least 2.5 times as wide as the nominal top size of the material to be processed, and should be large enough to handle normal oversized material particles (for example *see* Fig. 3 and Fig. 4).

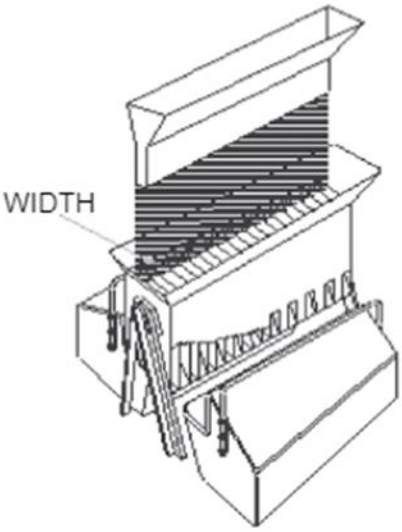


FIG. 1 EXAMPLE OF A RIFFLE BOX

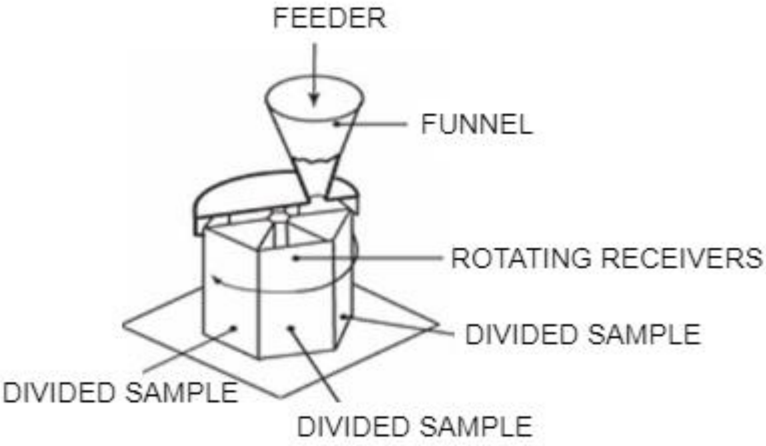


FIG. 2 EXAMPLE OF A ROTARY SAMPLE DIVIDER

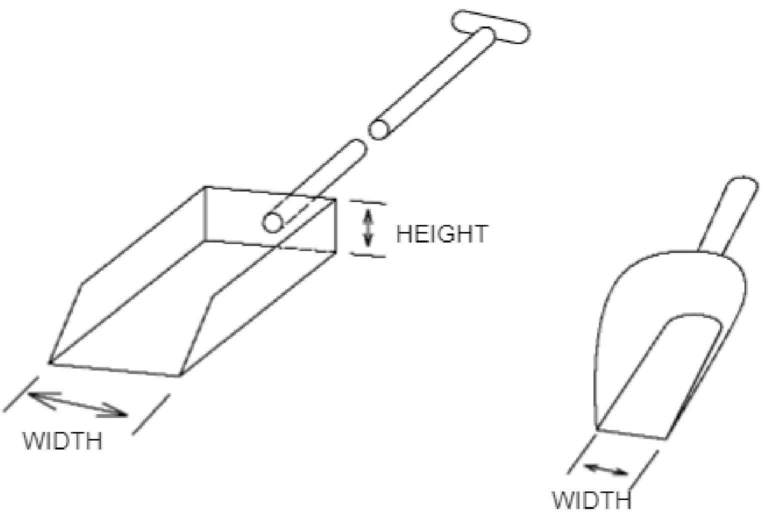


FIG. 3 EXAMPLE OF SCOOPS

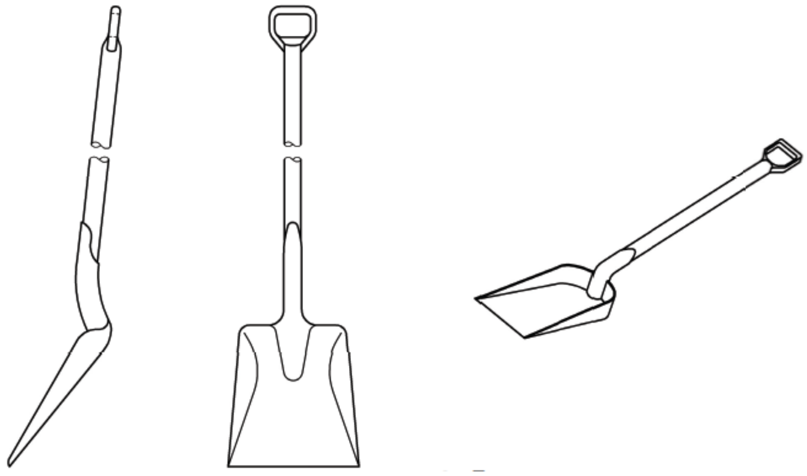


FIG. 4 EXAMPLE OF A SHOVEL

5.2 Apparatus for Particle Size-Reduction

NOTE — When analyzing metals (major or minor elements), take care that there is no contamination from the sample preparation or reduction equipment.

5.2.1 *Coarse Cutting Mill or Wood Crusher*

Coarse cutting mills can be used for cutting materials into lengths of about 10 mm to 30 mm (depending on the biofuel and the analyses to be performed). Drying of the material during coarse cutting shall be avoided by limiting heat production and air flow through the material. The equipment shall be designed so that it does not lose dust or contaminate the material with pieces of metal and shall be easy to clean.

NOTE — To prevent moisture losses during particle size reduction, a cutting mill or crushing mill with as low a cutting or crushing speed as possible is preferred.

5.2.2 *Cutting Mill*

Cutting mills can be used for reducing the nominal top size of materials used as biofuels from about 10 mm to 30 mm down to about 1 mm or less (depending on the biofuel and the analyses to be performed). The mill shall be provided with screens of various aperture sizes covering this range, including an appropriate sieve to control the nominal top size of the material produced. Other apparatus may be used provided that they are designed so that they do not get blocked with the material that is being processed. Avoid the use of cutting mills whose cutting faces contain significant quantities of an element that is to be determined in the analysis.

NOTE — Cross beater mills can be used without any excessive dusting, when fitted with dust filters (like a filter sock) between the mill and the receiving container. They are suitable for final grinding of hard, wood type materials after the pre-grinding with cutting type mills.

5.2.3 *Axe*

An axe is used for cutting wood logs or coarse material down to a maximum 30 mm thickness or suitable size to be processed in a cutting mill provided with a 30 mm sieve.

5.2.4 *Hand Saw*

5.2.4.1 A hand saw is used to saw off wood logs or coarse material down to a maximum 30 mm thickness or suitable size to be processed in a cutting mill provided with a 30 mm sieve.

5.2.4.2 A chain saw may contaminate the sample by chain oil and should therefore not be used. A saw machine should not be used for size reduction to avoid the risk of losing moisture in the sample as a result of heat caused by friction.

5.2.5 *Sieves*

A wire-mesh sieve with an aperture size of 1 mm [*see* IS 460 (Part 1)] is recommended to check the nominal top size of general analysis samples. A wire-mesh sieve with an aperture size of 0.25 mm will be recommended if sub-samples with this nominal top size are required.

5.2.6 *Balance*

A balance is required that is capable of determining the mass of samples to an accuracy of 0.1 percent of the sample mass, and the mass of sub-samples to an accuracy of 0.1 percent of the sub-sample mass.

6 SAMPLE REDUCTION — GENERAL PRINCIPLES

6.1 For every sample division stage to be carried out, it is important that a sufficient mass of material is retained, otherwise the sub-samples produced or the test portions to be taken may not be representative of the original sample. Due to various shape and size of different solid biofuels, different methods for determination of minimum sample masses have to be applied. Guidelines for minimum masses to be retained after each sample division stage, depending on the nominal top size of the material, are prescribed in Table 2.

6.2 Regarding pellets, the pellets diameter shall be considered the nominal top size and the opening of the equipment shall be large enough for the longest pellets to pass through.

6.3 In addition to the minimum masses as prescribed in Table 2, the mass after sample division shall be sufficiently large for the actual test or tests to be performed. Table 1 gives a guideline for the selection of the method to be applied to determinate the minimum sample mass to be retained after each sample division stage. Supplementary requirements concerning the masses of the test portions are given in international standards for test methods of solid biofuels.

Table 1 Guideline to Choose a Method to Determine the Minimum Masses to be Retained During Sample Division Stages

(*Clause* 6.3)

Sl No. (1)	Material/Method (2)	Sample Division Stage 1 (3)	Sample Division Stage 2 (4)	Sample Division Stage 3 (5)	Sample Division Stage 4 (6)
i)	Material	Bulk material of less than or equal to 100 mm nominal top size (*see* 3.1)	Large pieces greater than 100 mm material, for example, wood logs or briquettes.	Straw-like material with a low bulk density (less than 200 kg/m^3) and lengths greater than 31.5 mm	Irregular/mixed materials, for example, hogfuels, logging residues, bark
ii)	Method for determination of minimum sample mass	Calculate the minimum sample mass as given in Table 2.	Number of pieces should be stated, for example, 10 pieces randomly collected	Minimum sample mass should be defined, for example, 500 g	To be determined according to fuel type

Table 2 Guideline for Minimum Masses to be Retained During Sample Division Stages, Applicable for Material According to Col (2) in Table 1

(*Clauses* 6.1, 6.3, 7.1, 8.2, 8.5, 8.7 *and* Table 1)

Sl No. (1)	**Nominal top size,** mm (2)	**Minimum mass,** g (3)
i)	100	15 000
ii)	63	4 000
iii)	45	1 500
iv)	31.5	500
v)	16	350
vi)	8	250
vii)	3.15	100
viii)	1	30
ix)	0.25	10

6.4 According to particle size distribution, shape and density of particles, the calculation of minimum sample mass should be adapted to the methods given here at the best in practical terms. Fig. 5 summarizes the range of steps that can be taken during sample reduction.

7 METHODS FOR SAMPLE DIVISION

7.1 General

Combined samples may be divided into two or more laboratory samples and laboratory samples are in general further divided in sub-samples (test portions). This clause describes the methods and procedures for sample division. For every division step, the mass of retained material given in Table 2 shall be taken into account.

7.2 Riffling

This may be used for materials that can be passed through the riffle without bridging. It is not suitable for straw, bark, or other materials containing elongated particles, or for wet and/or sticky materials. Brittle materials should be handled with care to avoid the production of fine materials. Place the whole of the combined sample into one of the containers of the riffle so that it is evenly distributed throughout the container. Place the other two containers in position under the riffle. Pour the contents of the first container down the center line of the riffle. Pour the biofuel sufficiently slowly to avoid bridging occurring. Do not move the container from side to side (this would cause the end slots to receive less biofuel). Discard the biofuel that falls into one of the containers, chosen at random. Repeat the riffling process until a sub-sample of the required mass is obtained (see Fig. 1 for riffle box).

7.3 Strip Mixing

7.3.1 This may be used for all materials and will be a convenient method when a combined sample is to be divided into a small number of laboratory samples. Place the whole combined sample on a clean, hard surface and homogenize it by mixing with a shovel and form into a strip at least 20 times as long as it is wide. Distribute the sample along the length of the strip as evenly as possible, working randomly from end to end. Building up the strip with several layers will increase the quality of the division.

7.3.2 Obtain a laboratory sample by taking at least 20 increments from locations evenly spaced along the length of the strip. Take each increment by inserting two plates vertically into the strip and removing all the material from between the plates. The two plates should be inserted the same distance apart each time so that each increment contains the same quantity of material. The distance between the plates should be at least 2.5 times the nominal top size and at least the length of normal oversized material particles (see Fig. 6 for the principle of strip mixing).

7.4 Long Pile-Alternate Shovel Method

This sub-sampling method is recommended for the reduction of samples in excess of approximately 50 kg. Place the entire combined sample on a clean and even floor and mix it thoroughly by manually moving the material to form a long pile.

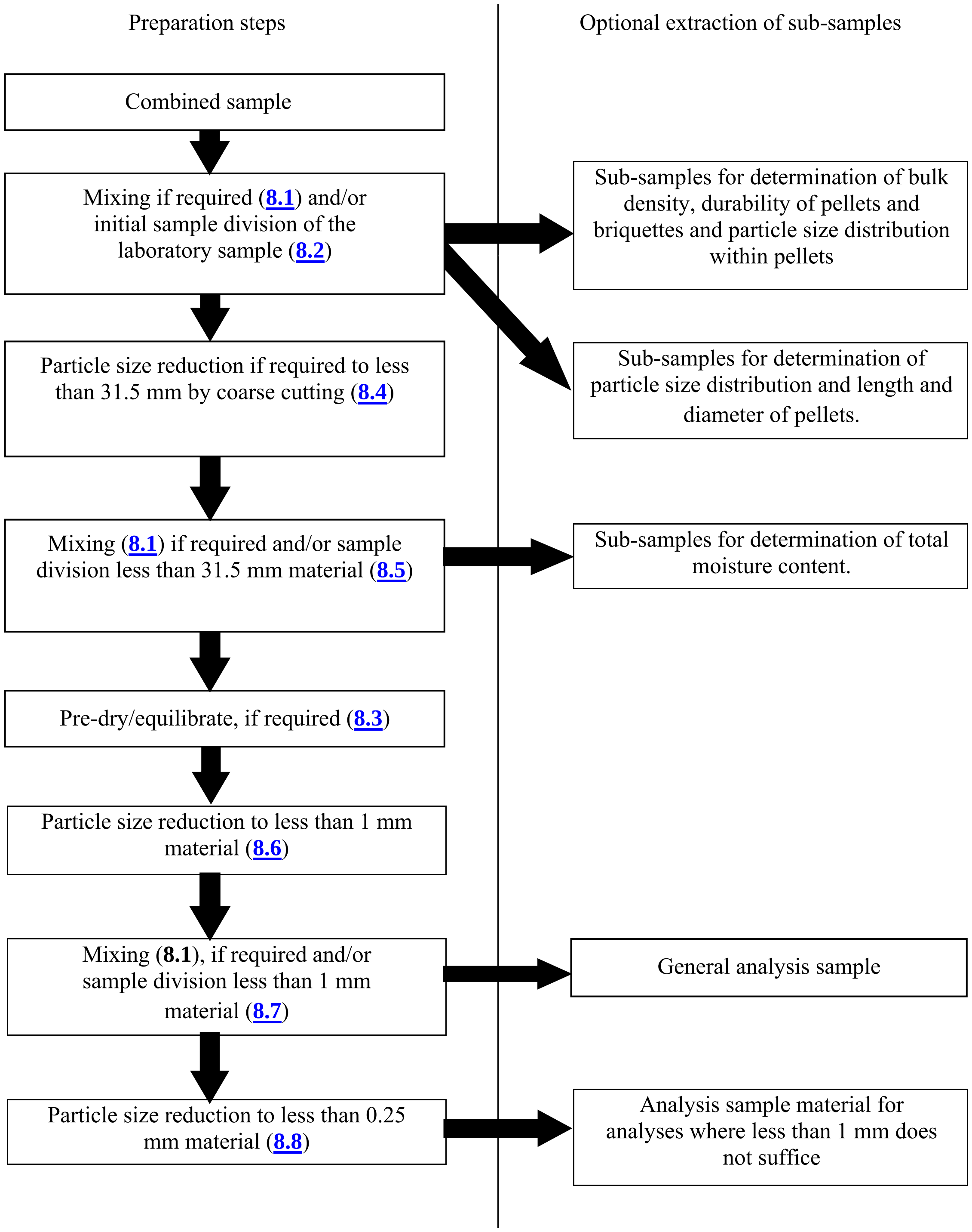


FIG. 5 FLOW SHEET FOR SAMPLE PREPARATION AND OPTIONAL EXTRACTION OF SUB-SAMPLES

NOTE — List of optional sub-sample extractions cannot be exhaustive.

Carry out this process at least three times to ensure thorough mixing. When forming new piles, deposit each shovelful on the end of the new pile. Divide the last pile into two equal piles by using a shovel. Place alternate shovel loads to either side and form into two piles with at least 10 shovel loads (increments) in each pile. One pile is randomly selected and the process is repeated, using appropriately smaller increments as the piles are getting smaller. For straw-like material, this method may require excessive manual work. For samples more than 500 kg, a mechanical shovel should be considered and used, if appropriate.

7.5 Rotary Divider

A rotary sample divider is a mechanical method that can be used to reduce the mass of a sample. This method is recommended for dry free flowing material. The rotary sample divider shall have a feeder device adjusted, so that the number of compartments multiplied by the number of rotations shall not be less than 120 while the sample is being divided (for example of a rotary divider *see* Fig. 2).

7.6 Coning and Quartering

7.6.1 This method may be used for materials such as sawdust and woodchips that can be manually worked with a shovel. It is suitable for producing sub-samples of these materials down to approximately 1 kg. Place the whole combined sample on a clean, hard surface. Shovel the sample into a conical pile, placing each shovelful on top of the preceding one in such a way that the biofuel runs down all sides of the cone and is evenly distributed and different particle sizes become well mixed. Repeat this process three times, forming a new conical pile each time. Flatten the third cone by inserting the shovel repeatedly and vertically into the peak of the cone to form a flat heap that has a uniform thickness and diameter and is no higher than the blade of the shovel. Quarter the flat heap along two diagonals at right angles by inserting the shovel vertically into the heap (*see* Fig. 7) (a sheet-metal cross may be used for this operation if available). Discard one pair of opposite quarters. Repeat the coning and quartering process until a sub-sample of the required size is obtained.

7.6.2 For materials and particles that tend to roll off the pile and cause segregation, this method should only be used when methods described in 7.2 to 7.5 cannot be applied.

7.7 Mass Reducing Straw-Like Material (Handful Sampling)

This method is suitable for small sample sizes of straw-like material. Place the whole sample in an air-tight bag and homogenize it by inverting and kneading the bag several times. Take several handfuls and put them alternately into two piles. Homogenize the material remaining in the bag again, then remove several more handfuls and add them alternately to the two piles. Continue like this until the material in the bag is all used. At least 20 handfuls shall be placed into each of the two sub-samples.

8 Method for Reducing Laboratory Samples to Sub-samples and General Analysis Samples

8.1 Mixing

8.1.1 Mixing is recommended before each step of sample division. It should be noted that the sample division does not ensure that samples are adequately mixed. Additional mixing can be achieved by pouring it through a riffle (*see* 7.2) or a container-type divider (*see* Fig. 2) three times, reuniting the parts after each pass.

8.1.2 Beware that mixing may result in loss or gain of moisture.

8.1.3 If the material is too wet to handle, pre-drying should be carried out first.

NOTES

1 In theory, thorough mixing of a sample prior to its division reduces errors occurring in sample preparation. In practice, this is not easy to achieve and some methods of hand mixing, for example, forming and reforming into a conical pile, can have the opposite effect, leading to increased segregation.

2 If mechanical sample dividers are used in the course of preparation, an additional mixing step is normally not necessary to meet the required precision.

3 Mechanical mixing can be useful at the final stage of preparation of test samples.

8.2 Initial Sample Division

If the initial mass of the laboratory sample exceeds the minimum mass as given in Table 2, the laboratory sample may be divided using one of the methods prescribed in 7. If the material is too wet to handle, pre-drying should be carried out first.

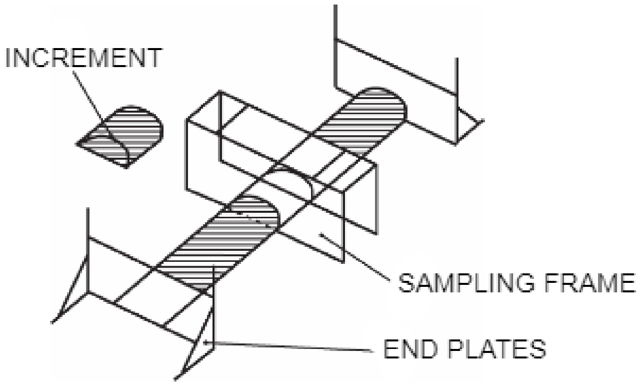


FIG. 6 STRIP MIXING

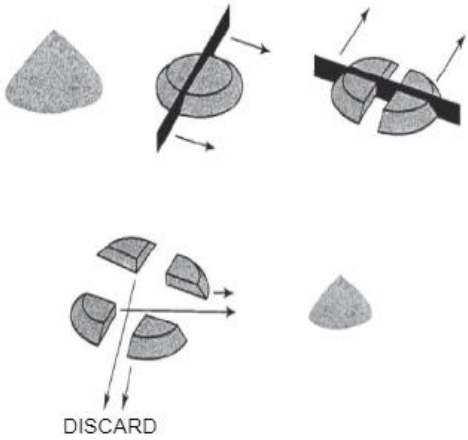


FIG. 7 CONING AND QUARTERING

8.3 Pre-drying

Pre-drying of wet samples is in general carried out to minimize moisture loss in the subsequent sample division processes, to facilitate the sample preparation processes and to minimize biological activity. If it is necessary to dry a sample by heating, it shall be dried in an oven at a temperature not exceeding 40 °C. All samples (including those that have been dried by heating) shall be spread out on a tray no more than a few particles deep and left for at least 24 h in the laboratory until they reach equilibrium with the temperature and humidity in the laboratory.

Before the laboratory sample or the divided laboratory sample according to 8.2 is subject to pre-drying, determine the mass of the sample by weighing on a balance accurate to at least 0.1 percent of the sample mass. Record this mass as $m_{\text{sample},1}$. After pre-drying and equilibrium with the surroundings, record the mass as $m_{\text{sample},2}$.

Calculate the moisture loss during pre-drying as a percentage of the initial mass of the sample as:

$$M_p = 100 \times \frac{m_{\text{sample},1} - m_{\text{sample},2}}{m_{\text{sample},1}}$$

where

Mp = moisture loss in percentage;
$m_{\text{sample},1}$ = initial mass, in g, of the sample; and
$m_{\text{sample},2}$ = mass, in g, of the sample after pre-drying.

Record the result to the nearest 0.1 percent.

NOTE — If the moisture content of the original sample is unimportant, for example, only properties of the dry matter or mechanical properties are to be determined or if a separate 'moisture analysis sample' is separated or sampled as well, the calculation of the moisture loss can be omitted. Also in this case, it is not necessary to obtain complete equilibrium with the temperature and humidity in the laboratory.

For coarse materials, for example, 24 h under laboratory conditions is not enough to reach equilibrium moisture content in all particles. Constancy in mass should be monitored by reweighing at intervals for example, 4 h. The sample material should be turned around from time to time during the equilibrium process.

8.4 Coarse Cutting (Particle Size Reduction to Less than 31.5 mm)

8.4.1 If the material contains particles that will be retained on a 31.5 mm sieve:

a) Use a 31.5 mm sieve to separate the sample into a coarse fraction (retained on the 31.5 mm sieve) and a fine fraction (passing the 31.5 mm sieve);

b) Process the coarse fraction using a coarse cutting mill so that it passes the 31.5 mm sieve; and
c) Re-combine the processed coarse fraction and the fine fraction and homogenize the sample.

8.4.2 If required, the above procedure may be used to achieve particle size reduction to a size below 31.5 mm by the use of a suitable coarse cutting mill and sieve.

NOTES

1 Depending on the material, other mills than the cutting mill can be used.

2 Grinding of the whole sample through the sieve fitted in the mill will produce the particle size needed.

8.5 Sample Division of Less than 31.5 mm Material

One of the methods described in 7 shall be used for sample division. Determine the masses of the containers into which the sub-samples are to be put before starting the division. Weigh the sub-sample masses using a balance, accurate to 0.1 percent. The masses of retained sub-samples shall comply with the values stated in Table 2.

Seal the container immediately if a sub-sample is required for moisture content determination or another test for which it is important not to lose moisture. When a sub-sample is to be used as a test portion, the sub-sample shall have a mass not less than the minimum test-portion mass specified in the appropriate test method.

8.6 Particle Size Reduction of Less than 31.5 mm Material to Less than 1 mm

Use a cutting mill for the preparation of the general analysis sample material less than 1 mm from the less than 31.5 mm material. Carry out the process in several steps, if necessary, using a finer sieve in the mill at each step, finishing with an appropriate sieve.

Example:

If the sample is to be reduced first to pass a5 mm screen and then to pass a 1 mm screen:

a) Use a 5 mm sieve to separate the sample into a coarse fraction (retained on the 5 mm sieve) and a fine fraction (passing the 5 mm sieve);
b) Process the coarse fraction using a cutting mill containing a 5 mm screen;
c) Re-combine the processed coarse fraction and the fine fraction and homogenize;
d) Use a 1 mm sieve to separate the sample into a coarse fraction (retained on the 1 mm sieve) and a fine fraction (passing the 1 mm sieve);
e) Process the coarse fraction using a cutting mill containing a 1 mm screen; and

f) Re-combine the processed coarse fraction and the fine fraction and homogenize the sample.

If the material contains seeds or grains, there is a risk that they will rotate in the mill or get stuck in the screen. Likewise, if the material contains straw, some straw may come to rest on the screen and not pass through. Examine the mill after the milling is completed. If any such particles are found, grind them manually until they pass the sieve and add this material to the sub-sample. Spread out the sub-sample in a tray no more than a few millimeters deep and leave it for at least 4 h in the laboratory until it reaches equilibrium with the temperature and humidity in the laboratory. Material that has been subjected to the particle size-reduction processes described in this sub-clause shall not be used for the determination of the total moisture content of the fuel.

NOTES

1 Depending on the material, other mills than the cutting mill can be used.

2 Grinding of the whole sample through the sieve fitted in the mill will produce the particle size needed and it also homogenizes the sample at the same time.

3 Sample division can be carried out between subsequent particle reduction steps as long as the minimum mass in Table 1 is respected.

8.7 Sample Division of Less than 1 mm Material

If required, for example, for back-up samples, divide the less than 1 mm material into two or more sub-samples using one of the methods as given in 7. It is strongly recommended to use division instead of simple extraction by scoop/spatula whenever possible.

If impurities (like sand, etc) are expected, then care should be taken especially for the segregation of particles, so the use of sample dividers is recommended. The sub-samples for further grinding shall keep the minimum masses stated in Table 2.

8.8 Particle Size Reduction of Less than 1 mm Material to Less than 0.25 mm

When a sub-sample is required with a nominal top size of 0.25 mm, use a cutting mill to reduce one of the less than 1 mm sub-samples to this size. Feed the cutting mill with small portions of material from the less than 1 mm sub-sample and let each portion pass through the 0.25 mm screen to prevent excess heat generation.

NOTE — Depending on the material, mills other than the cutting mill can be used.

9 STORAGE AND LABELLING

Samples shall be placed in air-tight plastic containers or bags. Each sample shall be labelled with a unique identification number of the sample, date and time of sampling and identification number or code of the lot or sub-lot number.

IS 18724:2024

SUMMARY

A biomass pellet is a small, cylindrical, and compressed form of organic material sourced from renewables, with specification standards set to ensure desired quality.

This standard specifies requirements of solid biofuels pellets produced from agro and other herbaceous residues, including blends and mixtures of woody and non-woody biomass. It does not cover thermally treated biomass pellets like torrefied pellets

The parameters as outlined include origin and source, diameter and length, moisture content, ash content, mechanical durability, fines, additives, gross calorific value, particle size, and bulk density. It also specifies requirements for reporting nitrogen, sulphur, chlorine, and various elements like arsenic, cadmium, chromium, copper, lead, mercury, nickel, and zinc

IS 18724 : 2024

SOLID BIOFUELS – FUEL SPECIFICATIONS AND CLASSES – PELLETS FROM AGRO AND HERBACEOUS RESIDUES

Indian Standard

SOLID BIOFUELS — FUEL SPECIFICATIONS AND CLASSES — PELLETS FROM AGRO AND HERBACEOUS RESIDUES

1 SCOPE

This standard prescribes the fuel quality classes and specifications of pellets produced from agro and other herbaceous residues.

NOTES

1 Blends and mixtures include blends and mixtures from the main origin-based solid biofuel groups like woody biomass, herbaceous biomass, fruit biomass and aquatic biomass. Blends are intentionally mixed biofuels, whereas mixtures are unintentionally mixed biofuels. The origin of the blend and mixture is to be described using ISO 17225-1 of Table 1. If solid biofuel blend or mixture contains chemically treated material, it shall be stated.

2 Thermally treated biomass pellets (for example, torrefied pellets) are not included in the scope of this standard.

2 REFERENCES

The standards listed in Annex A contain provisions, which through reference in the text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement, based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 18721 and in addition to, the following definitions shall apply.

3.1 Additive — Material which is intentionally introduced into the fuel feed stock to improve quality of fuel (for example, combustion properties), to reduce emissions or to make production more efficient.

NOTE — Trace amounts of grease or other lubricants that are introduced into the fuel processing stream as part of normal mill operations are not considered as additives.

3.2 Biofuel Pellet — Densified biofuel made with or without additives usually with a cylindrical form, random length typically 3 mm to 40 mm and diameter upto 25 mm and broken ends, produced by compressing biomass.

3.3 Chemical Treatment — Any treatment with chemicals other than air, water or heat.

NOTE — Examples of chemical treatment are listed in informative Annex C of ISO 17225-1.

3.4 Non-woody Biomass — Biomass originating from agriculture, herbaceous, fruit or aquatic biomass as well as blends or mixtures of woody and non-woody biomass.

3.5 Non-woody Pellet — Biofuel pellet (*see* **3.2**) made from non-woody biomass (*see* **3.4**).

4 REQUIREMENTS

4.1 The material shall comply with the requirements of non-woody pellets given in Table 1 and Fig 1.

4.2 In general, chemical treatment before harvesting of biomass does not need to be stated.

4.2.1 However, the process of chemical treatment before harvesting need to be mentioned in the following cases:

a) If any operator in the fuel supply chain has reason to suspect serious contamination of the biomass;
b) The soil (for example, coal slag heaps) or if planting has been done specifically for the chemical sequestration; and
c) Biomass has been fertilized by sewage sludge (originating from wastewater treatment or chemical process).

In such cases, fuel analysis shall be done to identify chemical impurities such as halogenated organic compounds or heavy metals.

4.2.2 In case of raw materials belonging to chemically treated herbaceous residue as per Table 1 of ISO 17225-1, the actual origin of the raw material shall be clearly reported.

4.3 Further analysis may not be required, if data for chemical or physical properties is available.

4.4 The amount of fines shall be reported before leaving the final point of loading for delivery to the end-user (*see* Table 1), to ensure that the end-user receives pellets with a low level of fines. While delivering pellets to the end-user, distributors should take appropriate measures to maintain this low level of fines.

4.5 To ensure that pellets maintain their quality, the handling and storage (including the equipment) shall be appropriate at different stages of supply or delivery chain ending to the end-user storage.

NOTE — The fines requirements are included to ensure small-scale users, while operating their combustion plant/appliances are protected from handling and combustion issues.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed as agreed between the purchaser and the supplier.

5.2 Marking

5.2.1 When the material is packed in packages, packages shall be marked with the following:

a) Name of the feedstock material or blend or mixtures;
b) Name of manufacturer and his recognized trade-mark, if any;
c) Month and year of manufacture;
d) Net mass of the material;
e) Dimensions of the pellets;
f) Lot number; and
g) Any other statutory requirements.

5.2.2 If pellets are available in general market for sale in addition to above information the following basic quality parameters shall be stated:

a) Gross calorific value;
b) Proximate analysis; and
c) Name and proportion (in percent) of the feedstock material or blend or mixtures used for preparation of the pellet.

5.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the rules and regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

The sampling and sample preparation shall be done as per ISO 18135 or ISO 21945 and IS 18640 respectively.

7 TEST METHODS

Tests shall be conducted according to the method of test referred in col (5) and col (6) of Table 1.

Table 1 Requirements of Pellets Derived from Agro and Herbaceous Residues.

(*Clauses* 4.1, 4.4 *and* 7, Annex B)

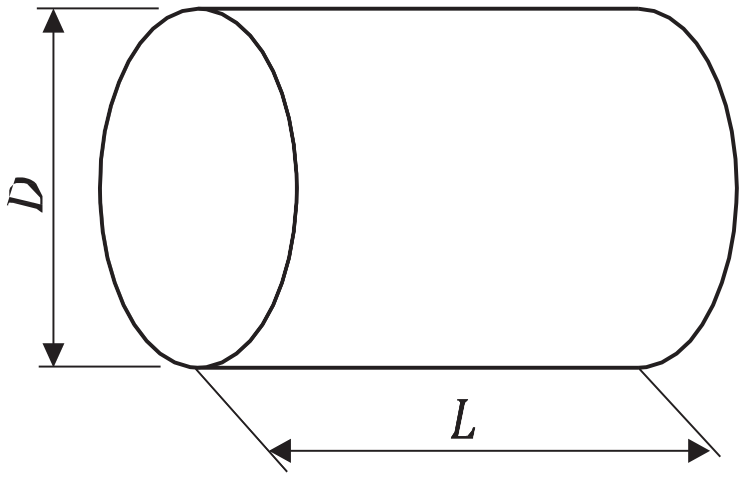
Sl. No	Characteristic	Requirements		Methods of Test, Ref to	
		A	B	IS/ Annex	ISO
(1)	(2)	(3)	(4)	(5)	(6)
i)	Origin and source1)	Agro residues and other herbaceous biomass		—	—
ii)	Diameter and length, mm	To be reported		IS 17643	—
iii)	Moisture, percent by mass, wet basis	≤ 14		≤ 14	IS 17655 (Part 1)/ IS 17655 (Part 2)

Table 1 (*Continued*)

Sl. No	Characteristic	Requirements		Methods of Test, Ref to	
		A	B	IS/ Annex	ISO
(1)	(2)	(3)	(4)	(5)	(6)
iv)	Ash, percent by mass, dry	≤ 15		≤ 15	IS 17653
v)	Mechanical durability2), percent mass	≥ 95		≥ 95	IS 18557 (Part 1)
vi)	Fines, percent mass	≤ 5		≤ 5	IS 17656
vii)	Additives3), percent mass	Type and amount to be reported.		—	—
viii)	Gross calorific value, kcal/kg	A1: $\geq$ 2 800 to < 3 000 A2: $\geq$ 3 000	B1: $\geq$ 2 800 to < 3 000 B2: $\geq$ 3 000	IS 17654	—
ix)	Particle size, mm	a) Pass through 2 mm mesh size, percent, *Min* = 75 b) Pass through 3 mm mesh size, percent = 100	—	B	—
x)	Bulk density, kg/m^3	To be reported		IS 17642	
xi)	Nitrogen, percent mass in dry	To be reported		IS 17832	—
xii)	Sulphur, percent mass in dry	To be reported		IS 17833	—
xiii)	Chlorine, percent mass in dry	To be reported		IS 17833	
xiv)	Arsenic (As), mg/kg dry	To be reported		—	ISO 16968
xv)	Cadmium (Cd), mg/kg dry	To be reported		—	ISO 16968
xvi)	Chromium (Cr), mg/kg dry	To be reported		—	ISO 16968
xvii)	Copper (Cu), mg/kg dry	To be reported		—	ISO 16968
xviii)	Lead (Pb), mg/kg dry	To be reported		—	ISO 16968
xix)	Mercury (Hg), mg/kg dry	To be reported		—	ISO 16968
xx)	Nickel (Ni), mg/kg dry	To be reported		—	ISO 16968

Table 1 (*Concluded*)

Sl. No	Characteristic	Requirements		Methods of Test, Ref to	
		A	B	IS/ Annex	ISO
(1)	(2)	(3)	(4)	(5)	(6)
xxi)	Zinc (Zn), mg/kg dry	To be reported		—	ISO 16968



All dimensions are in mm

FIG. 1 DIMENSIONS OF PELLETS

where

D = diameter; and

L = length.

---

1 Name and proportion (in percent) of the feedstock material or blend and mixtures used for preparation of the pellet shall be reported. Saw dust and other permitted woody biomass may be used in the blend to achieve the desired quality.

2 At final point of loading in bulk transport (at the time of loading) and in small (up to 20 kg) and big bags (at time of packing).

3 Type and amount of additive(s) to aid production, delivery or combustion (for example, pressing aids, slagging inhibitors or any other additives like starch, corn flour, potato flour, vegetable oil, and lignin) shall be reported.

ANNEX A

(*Clause 2*)

LIST OF REFERRED STANDARDS

IS No. / Other standards	*Title*
IS 17642 : 2021/ ISO 17828 : 2015	Solid biofuels — Determination of bulk density
IS 17643 : 2021/ ISO 17829 : 2015	Solid biofuels — Determination of length and diameter of pellets
IS 17653 : 2021/ ISO 18122 : 2015	Solid biofuels — Determination of ash content
IS 17654 : 2021/ ISO 18125 : 2017	Solid biofuels — Determination of calorific value
IS 17655 (Part 1) : 2021/ ISO 18134-1 : 2015	Solid biofuels — Determination of moisture content — Oven dry method: Part 1 Total moisture — Reference method
IS 17655 (Part 2) : 2021/ISO 18134-2 : 2017	Solid biofuels — Determination of moisture content — Oven dry method: Part 2 Total moisture — Simplified method
IS 17656 : 2021/ ISO 18846 : 2016	Solid biofuels — Determination of fines content in samples of pellets
IS 17832 : 2022/ ISO 16948 : 2015	Solid biofuels — Determination of total content of carbon, hydrogen and nitrogen
IS 17833 : 2022/ ISO 16994 : 2016	Solid biofuels — Determination of total content of sulfur and chlorine
IS 18557 (Part 1) : 2024/ISO 17831-1 : 2015	Solid biofuels — Determination of mechanical durability of pellets and briquettes: Part 1 Pellets
IS 18640 : 2024	Solid biofuels — Sample preparation
IS 18721 : 2024	Solid biofuels — Vocabulary
ISO 16968 : 2015	Solid biofuels — Determination of minor elements
ISO 17225-1 : 2021	Solid biofuels — Fuel specifications and classes : Part 1 General requirements
ISO 18135 : 2017	Solid biofuels — Sampling
ISO 21945 : 2020	Solid biofuels — Simplified sampling method for small scale applications

To access Indian Standards click on the link below:
https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/knowyourstandards/Indian_standards/isdetails/

ANNEX B

[*Table 1* and *Sl No.* (ix)]

DETERMINATION OF PARTICLE SIZE

B-1 SIEVE ANALYSIS

Sample of non-torrefied biomass pellets will be tested for particle size distribution after pulverizing 50 g crushed pellets sample in a lab pulverizer for 1.5 min or any fixed time period as found suitable by testing laboratory incharge and measuring the passing proportion of the pulverized sample by sieving it through 2 mm and 3 mm mesh size sieve.

IS 18725:2024

SUMMARY

A biomass briquette is a compact, block-shaped form of organic material made from renewable sources, with specification standards established to ensure consistent quality.

This standard specifies requirements of solid biofuels briquettes produced from agro and other herbaceous residues, including blends and mixtures of woody and non-woody biomass. It does not cover thermally treated biomass pellets like torrefied pellets

The parameters as outlined include origin and source, diameter and length, moisture content, ash content, additives, gross calorific value, and density. It also specifies requirements for reporting nitrogen, sulphur, chlorine, and various elements like arsenic, cadmium, chromium, copper, lead, mercury, nickel, and zinc.

SOLID BIOFUELS – FUEL SPECIFICATIONS AND CLASSES – BRIQUETTES FROM AGRO AND HERBACEOUS RESIDUES

IS 18725 : 2024

Indian Standard

SOLID BIOFUELS — FUEL SPECIFICATIONS AND CLASSES — BRIQUETTES FROM AGRO AND HERBACEOUS RESIDUES

1 SCOPE

This standard prescribes the fuel quality classes and specifications of briquettes produced from agro residues and other herbaceous residues.

NOTES

1 Blends and mixtures include blends and mixtures from the main origin-based solid biofuel groups like woody biomass, herbaceous biomass, fruit biomass and aquatic biomass. Blends are intentionally mixed biofuels, whereas mixtures are unintentionally mixed biofuels. The origin of the blend and mixture is to be described using ISO 17225-1, Table 1. If solid biofuel blend or mixture contains chemically treated material, it shall be reported.

2 Thermally treated biomass briquettes (for example, torrefied briquettes) are not included in the scope of this standard.

2 REFERENCES

The standards listed in Annex A contain provisions, which through reference in the text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard, are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 18721 and in addition to, the following definitions shall apply.

3.1. Additive — Material which is intentionally introduced into the fuel feed stock to improve quality of fuel (for example, combustion properties), to reduce emissions or to make production more efficient.

NOTE — Trace amounts of grease or other lubricants that are introduced into the fuel processing stream as part of normal mill operations are not considered as additives.

3.2 Biofuel Briquette — Densified biofuel made with or without additives in pre-determined geometric form with at least two dimensions of more than 25 mm, produced by compressing biomass.

3.3 Chemical Treatment — Any treatment with chemicals other than air, water or heat (for example, glue and paints).

NOTE — Examples of chemical treatment are listed in ISO 17225-1.

3.4 Non-woody Biomass — Biomass originating from agriculture, herbaceous, fruit or aquatic biomass as well as blends or mixtures of woody and non-woody biomass.

3.5 Non-woody Briquette — Biofuel briquette (*see* **3.2**) made from non-woody biomass (*see* **3.4**)

4 REQUIREMENTS

4.1 The material shall comply with the requirements of briquettes given in Table 1 and Fig 1.

4.2 In general, chemical treatment of biomass before harvesting need not to be reported.

4.2.1 However, the process of chemical treatment before harvesting need to be mentioned in the following cases:

a) If any operator in the fuel supply chain has reason to suspect serious contamination of the biomass;

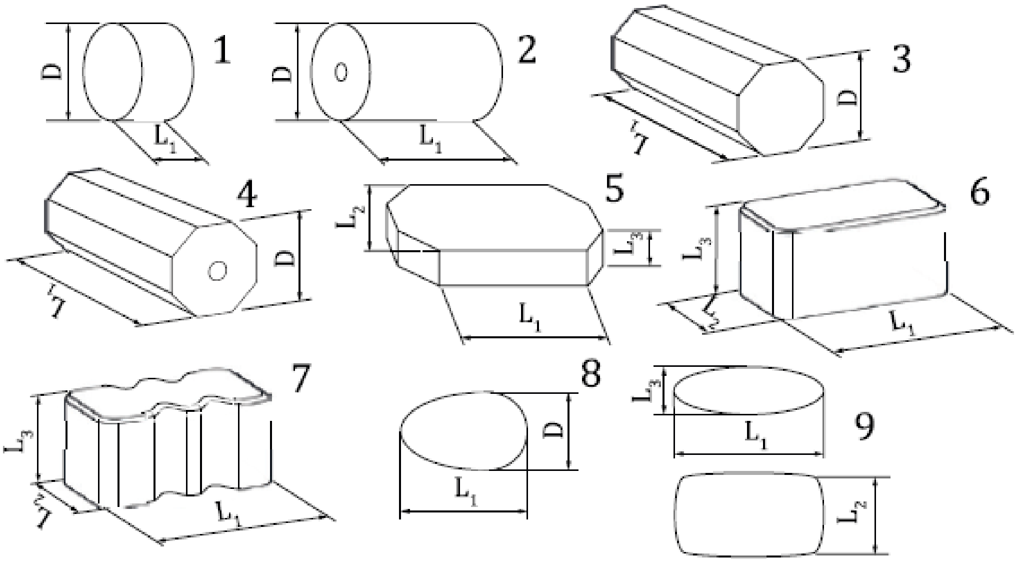
b) The soil (for example, coal slag heaps) or if planting has been done specifically for the chemical sequestration; and

c) Biomass has been fertilized by sewage sludge (originating from wastewater treatment or chemical process).

In such cases, fuel analysis shall be done to identify chemical impurities such as halogenated organic compounds or heavy metals.

4.2.2 In case of raw materials belonging to chemically treated herbaceous residue as per Table 1 of ISO 17225-1, the actual origin of the raw material shall be clearly reported.

4.3 Further analysis may not be required, if data for chemical or physical properties are available.



All dimensions in millimetres.
FIG. 1 DIMENSIONS OF BRIQUETTES

where

D = diameter;
L_1 = length;
L_2 = width; and
L_3 = height.

Table 1 Requirements of Biomass Briquettes Derived from Agro and Herbaceous Residues

(*Clauses* 4.1 *and* 7)

Sl No.	**Characteristics**	**Requirements**			**Methods of test, Ref to**	
		A	B	C	IS	ISO
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Origin and source1)	Agro residues and other herbaceous biomass			—	—
ii)	Diameter (D); length (L1); width (L2) and height (L3), mm	Dimension and shape to be reported.			Dimension and shape to be specified as per Fig 1.	
iii)	Moisture (M), percent by mass, wet basis	≤ 15	≤ 15	≤ 15	IS 17655 (Part 1)/ IS 17655 (Part 2)	—

Table 1 (*Concluded*)

Sl No.	Characteristics	Requirements			Methods of test, Ref to	
		A	B	C	IS	ISO
(1)	(2)	(3)	(4)	(5)	(6)	(7)
iv)	Ash (A), percent in mass dry	≤ 20	≤ 20	≤ 20	IS 17653	
v)	Density of briquettes, g/cm^3	A1: ≥ 0.5 to < 0.7	B1: ≥ 0.5 to < 0.7	C1: ≥ 0.5 to < 0.7	—	ISO 18847
		A2: ≥ 0.7 to < 0.9	B2: ≥ 0.7 to < 0.9	C2: ≥ 0.7 to < 0.9		
		A3: ≥ 0.9	B3: ≥ 0.9	C3: ≥ 0.9		
vi)	Additives, percent by mass[2]	Type and amount to be reported	Type and amount to be reported	Type and amount to be reported	—	—
vii)	Gross calorific value, kcal/kg	≥ 2 500 to < 3 000	≥ 3 000 to < 3 500	≥ 3 500	IS 17654	—
viii)	Nitrogen (N), percent by mass in dry	To be reported			IS 17832	—
ix)	Sulfur (S), percent by mass in dry	To be reported			IS 17833	—
x)	Chlorine (Cl), percent by mass in dry	To be reported			IS 17833	—
xi)	Arsenic (As), mg/kg, dry	To be reported			—	ISO 16968
xii)	Cadmium (Cd), mg/kg, dry	To be reported			—	ISO 16968
xiii)	Chromium (Cr), mg/kg, dry	To be reported			—	ISO 16968
xiv)	Copper (Cu), mg/kg, dry	To be reported			—	ISO 16968
xv)	Lead (Pb), mg/kg, dry	To be reported			—	ISO 16968
xvi)	Mercury (Hg), mg/kg, dry	To be reported			—	ISO 16968
xvii)	Nickel (Ni), mg/kg, dry	To be reported			—	ISO 16968
xviii)	Zinc (Zn), mg/kg, dry	To be reported			—	ISO 16968

---

[1] Name and proportion (in percent) of the feedstock material or blend and mixtures used for preparation of the briquette shall be reported. Saw dust and other permitted woody biomass may be used in the blend to achieve the desired quality.

[2] Type and amount of additive(s) to aid production, delivery or combustion (for example, pressing aids, slagging inhibitors or any other additives like starch, corn flour, potato flour, vegetable oil, and lignin) shall be reported.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed as agreed between the purchaser and the supplier.

5.2 Marking

5.2.1 When the material is packed in packages, packages shall be marked with the following:

a) Name of the feedstock material or blend and mixtures;
b) Name of manufacturer and his recognized trade-mark, if any;
c) Month and year of manufacture;
d) Net mass of the material;
e) Dimensions of the briquette;
f) Lot number; and
g) Any other statutory requirements.

5.2.2 If briquettes are available in general market for sale, in addition to above information the following basic quality parameters shall be stated:

a) Gross calorific value;
b) Proximate analysis; and
c) Name and proportion (in percent) of the feedstock material or blend and mixtures used for preparation of the briquettes.

5.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the rules and regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

The sampling and sample preparation shall be done as per ISO 18135 or ISO 21945 and IS 18640 respectively.

7 TEST METHODS

Tests shall be conducted according to the methods of test referred in col (6) and col (7) of Table 1.

ANNEX A

(*Clause 2*)

LIST OF REFERRED STANDARDS

IS No./Other Standards	*Title*
IS 17653 : 2021/ ISO 18122 : 2015	Solid biofuels — Determination of ash content
IS 17654 : 2021/ ISO 18125 : 2017	Solid biofuels — Determination of calorific value
IS 17655 (Part 1) : 2021/ ISO 18134-1 : 2015	Solid biofuels — Determination of moisture content — Oven dry method : Part 1 Total moisture — Reference method
IS 17655 (Part 2) : 2021/ISO 18134-2 : 2017	Solid biofuels — Determination of moisture content — Oven dry method : Part 2 Total moisture — Simplified method
IS 17832 : 2022/ ISO 16948 : 2015	Solid biofuels — Determination of total content of carbon, hydrogen and nitrogen
IS 17833 : 2022/ ISO 16994 : 2016	Solid biofuels — Determination of total content of sulfur and chlorine
IS 18640 : 2024	Solid biofuels — Sample preparation
IS 18721 : 2024	Solid biofuels — Vocabulary
ISO 16968 : 2015	Solid biofuels — Determination of minor elements
ISO 17225-1 : 2021	Solid biofuels — Fuel specifications and classes — Part 1: General requirements
ISO 18135 : 2017	Solid biofuels — Sampling
ISO 18847 : 2016	Solid biofuels — Determination of particle density of pellets and briquettes
ISO 21945 : 2020	Solid biofuels — Simplified sampling method for small scale applications

To access Indian Standards click on the link below:

https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/knowyourstandards/Indian_standards/isdetails/

IS 17642:2021/
ISO 17828 : 2015

SUMMARY

Bulk density is an important parameter for fuel deliveries on volume basis, and together with the net calorific value, it determines the energy density.

This standard defines a method of determining the bulk density of solid biofuels by the use of a standard measuring container. The method is applicable to all pourable solid biofuels with a nominal top size of maximum 100 mm.

The standard emphasizes that bulk density is not an absolute value and is subject to variations from vibration, shock, pressure, biodegradation, drying, and wetting. Therefore, measured bulk density can therefore deviate from actual conditions during transportation, storage, or transhipment.

SOLID BIOFUELS – DETERMINATION OF BULK DENSITY

IS 17642 : 2021

Indian Standard

SOLID BIOFUELS — DETERMINATION OF BULK DENSITY

1 Scope

This International Standard defines a method of determining bulk density of solid biofuels by the use of a standard measuring container. This method is applicable to all pourable solid biofuels with a nominal top size of maximum 100 mm.

Bulk density is not an absolute value; therefore, conditions for its determination have to be standardized in order to gain comparative measuring results.

NOTE Bulk density of solid biofuels is subject to variation due to several factors such as vibration, shock, pressure, biodegradation, drying, and wetting. Measured bulk density can therefore deviate from actual conditions during transportation, storage, or transhipment.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14780, *Solid biofuels — Sample preparation*[1)]

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18134-1, *Solid biofuels — Determination of moisture content — Oven dry method, Part 1: Total moisture — Reference method*

ISO 18134-2, *Solid biofuels — Determination of moisture content — Oven dry method, Part 2: Total moisture — Simplified procedure*

ISO 18135, *Solid biofuels — Sampling*[1)]

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 apply.

4 Principle

A standard container is filled with the test portion of a given size and shape, densified by defined shock exposure and weighed afterwards. The bulk density is calculated from the net weight per standard volume and reported with the determined moisture content.

5 Apparatus

5.1 Measuring containers

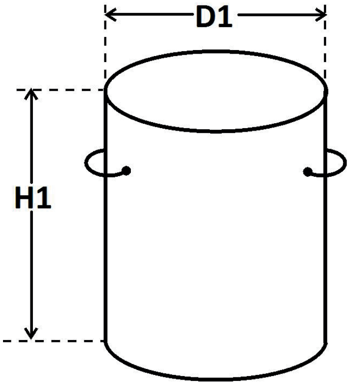
5.1.1 General

The container shall be cylindrically shaped and manufactured of a shock resistant, smooth-surfaced material. The container shall be resistant to deformation in order to prevent any variation in shape

---

1) In preparation.

and volume. The container has to be waterproof. For easier handling, grips can be fixed externally. The height-diameter-ratio shall be within 1,25 and 1,50.

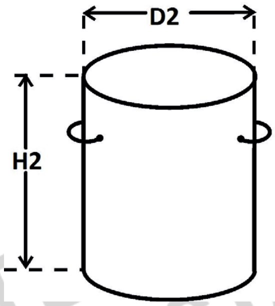


Key

D1 = 360 mm

H1 = 491 mm

Figure 1 — Large measuring container



Key

D2 = 167 mm

H2 = 228 mm

Figure 2 — Small measuring container

5.1.2 Large container

The large measuring container (see Figure 1) has a filling volume of 50 l (0,05 m^3) volume. The volume can deviate by 1 l (= 2 %). It shall have an effective (inner) diameter of 360 mm and an effective (inner) height of 491 mm (see Figure 1). Deviations from these dimensions are tolerable, if the height-diameter-ratio remains as given in 5.1.1.

5.1.3 Small container

The small measuring container (see Figure 2) has a filling volume of 5 l (0,005 m^3) volume. The volume can deviate by 0,1 l (= 2 %). It shall have an effective (inner) diameter of 167 mm and an effective (inner) height of 228 mm (see Figure 2). Deviations from these dimensions are tolerable, if the height-diameter-ratio remains as given in 5.1.1.

5.2 Balances

5.2.1 Balance 1

A balance shall be capable of reading to the nearest 10 g. This balance shall be used for measurements with the large container.

5.2.2 Balance 2

A balance shall be capable of reading to the nearest 1 g. This balance shall be used for measurements with the small container.

5.3 Scantlings

A rigid scantling with a length exceeding the diameter of the container in 5.1.1 shall be used for levelling the material in the measuring container by lateral movements of the scantling across the rim of the measuring container.

NOTE It is advisable to use a second scantling or other device for spacing the dropping height of 150 mm between the measuring container and the wooden board in 5.4.

5.4 Wooden board

A flat wooden board [e.g. oriented strand board (OSB)] with a thickness of approximately 15 mm and sufficient in size for dropping the container during shock exposure.

6 Sample preparation

Sampling shall be carried out in accordance with ISO 18135. If necessary, the sample can be divided in to test portions in accordance with ISO 14780. The test portion volume shall exceed the volume of the container measures by minimum of 30 %.

NOTE Precautions should be taken to ensure that the moisture is evenly distributed within the sample.

7 Procedure

7.1 Determination of the container volume

Before use, the mass and filling volume of the container shall be determined. Weigh the empty, clean, and dry container on the balance (5.2.1 or 5.2.2). Then fill the container with water and a few drops of wetting agent (e.g. liquid soap) until maximum capacity; then weigh it again. The water should be at a temperature between 10 °C and 20 °C. Calculate the volume (V) of the container from the net weight of water and the density of the water (1 kg/dm^3) and record the result rounded to the nearest 0,01 l (0,000 01 m^3) for the large container or 0,001 l (0,000 001 m^3) for the small container.

NOTE 1 The effect of temperature on the density of water is negligible.

NOTE 2 The container should be cleaned regularly and its volume should be checked regularly.

7.2 Container selection

The large container (5.2.1) can be used for larger materials within the scope of this International Standard. For materials with a nominal top size up to 12 mm and for pellets with a diameter equal or below 12 mm the small container (5.2.2) can be used (optional).

7.3 Measurement procedure

a) Fill the container by pouring the sample material from a height of 200 mm to 300 mm above the upper rim until a cone of maximum possible height is formed.

NOTE Make sure that the container is dry and clean before being (re)filled.

b) The filled container is then shock exposed to allow settling. This shall be done by dropping it freely from 150 mm height onto a wooden board (5.4). Before shock exposure, remove particles from the wooden board within the dropping area. Make sure that the container hits the board in a vertical position. Repeat the shock exposure two more times to apply a total of three shock exposures. Then refill the resulting empty space at the top of the container in accordance with 7.3 a).

NOTE The second scantling or device referred to in 5.3 can be used for spacing of the distance between the measuring container and the wooden board before dropping. Also, other mechanisms to create a comparable shock impact can be suitable.

c) Remove surplus material by using a scantling (5.3), which is shuffled over the container's edge in oscillating movements. When the test portion contains coarse material, all particles preventing the free passage of the scantling have to be removed by hand. If the removal of larger particles tears bigger holes into the leveled surface, the cavities shall be refilled and the removal procedure is repeated.

d) Weigh the container with the remaining sample material.

e) Unify the used sample material with the unused sample material and repeat the procedure from 7.3 a) to 7.3 d) at least once in order to get duplicate determinations.

f) Determine moisture content of the test sample material as received according to ISO 18134-1 or ISO 18134-2 immediately after bulk density determination.

8 Calculation

8.1 Calculation of bulk density as received

Calculate the bulk density of the sample as received (BD_{ar}) according to Formula (1):

$$BD_{ar} = \frac{(m_2 - m_1)}{V} \quad (1)$$

where

BD_{ar} is the bulk density as received in kg/m^3;

BD_d is the bulk density of the sample on dry basis in kg/m^3;

m_1 is the mass of the empty container in kg;

m_2 is the mass of the filled container in kg;

V is the net volume of the measuring container in m^3.

The result for each individual determination shall be calculated to 0,1 decimal place and for reporting purposes the mean value of the individual results shall be calculated and rounded to the nearest 10 kg/m^3.

8.2 Calculation of bulk density on dry basis

Calculate the bulk density of the test portion mass on dry basis (BD_d) in accordance with Formula (2):

$$BD_d = BD_{ar} \times \frac{(100 - M_{ar})}{100} \tag{2}$$

NOTE Formula (2) in 8.2 disregards shrinkage or expansion, which usually causes significant deviations when the test portion is measured at different drying stages. For wood fuels, these phenomena usually occur at a moisture content below the fibre saturation point, which is at around 25 % moisture content, depending on the wood species. A true comparison between material samples is therefore only possible when bulk density is measured at similar moisture contents. If material samples with different moisture content shall be compared and at least one sample is below the fibre saturation point, the usual effect of swelling or shrinkage is in the order of around 0,7 % volume change per percentage point of moisture difference below the fibre saturation point (see Reference [1]). For a comparison of measurements based on materials with similar moisture content, the application of this correction factor can be useful.

9 Performance characteristics

9.1 General

Table 1 — Repeatability and reproducibility limits

Bulk density	Maximum acceptable differences between results obtained	
	Repeatability limits	Reproducibility limits
Bulk density below 300 kg/m^3	3,0 %	6,0 %
Bulk density equal or above 300 kg/m^3	2,0 %	4,0 %

9.2 Repeatability

The results obtained for bulk density as received (BD_{ar}) of the duplicate determinations (see 7.3), performed within a short period of time by the same operator using the same apparatus in the same laboratory shall not differ by more than the values in Table 1 (see Reference [2]).

9.3 Reproducibility

The mean values of the results of duplicate determinations (see 7.3) for bulk density as received (BD_{ar}), performed in each of two different laboratories on representative test portions taken from the same sample material shall not differ by more than the values in Table 1 (see Reference [2]).

10 Test report

The test report shall include at least the following information:

a) identification of laboratory performing the test and the date of the test;

b) identification of the product (or sample) tested;

c) a reference to this International Standard, i.e. ISO 17828;

d) specification of the applied container size;

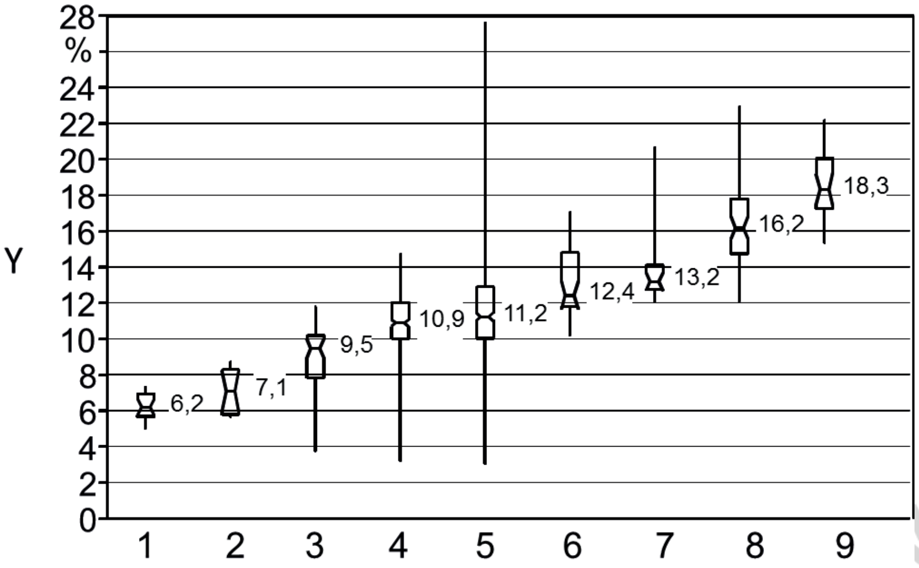
e) result of the test at moisture content as received according to 8.1 (required) or according to 8.2 (optional), expressed with relevant symbols;

f) any unusual features noted during the determination, which can affect the result;

g) any deviation from this International Standard or operations regarded as optional.

Annex A
(informative)

Measuring differences of sample treatment with and without shock impact



Legend

Y Relative deviation in % to non-shock application

	Number of replications
1 Wood pellets	21
2 Grain kernels	12
3 Herbaceous pellets	42
4 High density wood chips	312
5 Low density wood chips	492
6 Sawdust	39
7 Peat	18
8 Bark	63
9 Chopped miscanthus	24

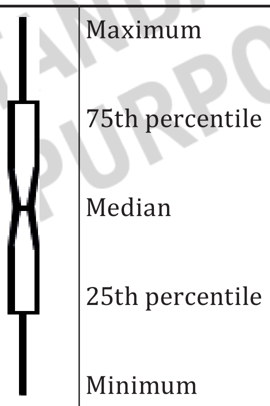


Figure A.1

Representation of the relative effect of shock impact compared to a non-shock application for bulk density determination, here given for the 50-l-container dropped three times before re-filling, surface levelling and weighing. The boundary value for high or low bulk density grouping of wood chips was 180 kg/m^3 (see Reference [1]).

IS 17643:2021/
ISO 17829 : 2015

SUMMARY

Determination of dimensions of pellets is important as dimensions outside of specifications can cause issues in various systems, such as transportation and burner feeding.

This standard focuses on determination of diameter and length of pellets including methods for determination of the proportion of oversized pellets and average length.

The method states use of caliper to measure the length and diameter of pellets. The length of a pellet shall be measured along the axis of the cylinder, while the diameter shall be measured perpendicular to the axis.

SOLID BIOFUELS – DETERMINATION OF LENGTH AND DIAMETER OF PELLETS

IS 17643 : 2021

Indian Standard

SOLID BIOFUELS — DETERMINATION OF LENGTH AND DIAMETER OF PELLETS

1 Scope

This International Standard specifies the methods for the determination of diameter and length of pellets. Concerning the pellet length methods for both determination of the proportion of oversized pellets and for determination of the average length are included.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 17225-1, *Solid biofuels — Fuel specifications and classes — Part 1: General requirements*

ISO 17225-2, *Solid biofuels — Fuel specifications and classes — Part 2: Graded wood pellets*

ISO 17225-6, *Solid biofuels — Fuel specifications and classes — Part 6: Graded non-woody pellets*

ISO 17225-8[1], *Solid biofuels — Fuel specifications and classes — Part 8: Thermally treated and densified biomass fuels*

EN 14778:2011, *Solid biofuels — Sampling*

EN 14780:2011, *Solid biofuels — Sample preparation*

ISO 18846[2], *Solid Biofuels — Determination of fines content in quantities of pellets*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

3 Terms and definitions

For the purpose of this document, the following terms and definitions given in ISO 16559 apply

3.1
test sample
original sample sent to the laboratory for analysis

3.2
test portion
sample extracted from the test sample and used during the analysis

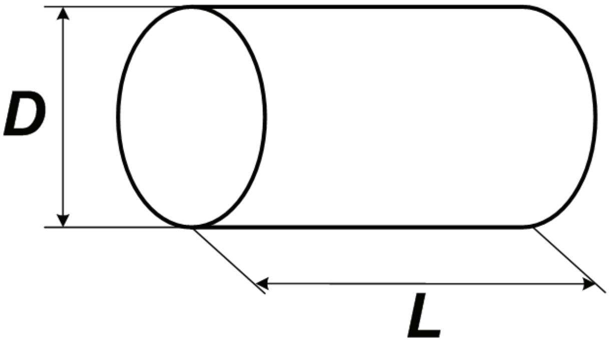
4 Principle

The length and diameter of fuel pellets of a representative test sample shall be measured by means of a caliper. The length of a pellet shall be measured along the axis of the cylinder. The diameter shall be measured perpendicular to the axis (see Figures 1 and 2).

---

[1] In preparation.

[2] To be published.



Key

D	diameter of a pellet
L	length of a pellet

Figure 1 — Length and diameter of a pellet

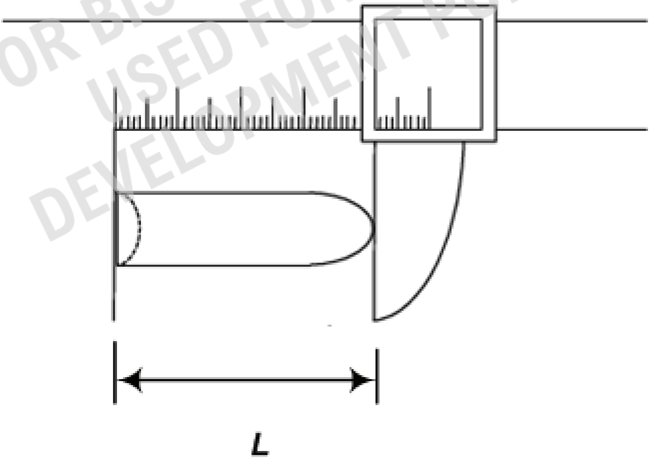


Figure 2 — Measuring the length between the mid-point of the convex and concave cross section of a pellet by means of a caliper.

5 Apparatus

5.1 **Measuring caliper**, shall have a resolution of at least 0,1 mm for measuring length and diameter.

5.2 **Balance**, shall be capable of reading to the nearest 0,01 g.

5.3 **Sieve**, shall have round holes of a diameter at 3,15 mm in accordance with ISO 3310-2.

6 Sample preparation

The test sample for determination of length and diameter shall be obtained in accordance with EN 14778 and a test portion shall be extracted in accordance with EN 14780. The test sample shall be divided into two test portions, one consisting of 10 pellets and the other consisting of a minimum mass in accordance with Table 1. The pellets shall be randomly selected.

Table 1 — Test portion size

Pellet size class	Estimated minimum test portion
≤6 mm	30 g - 40 g
>8 mm	40 g - 50 g
>10 mm	50 g - 70 g
>12 mm	70 g - 120 g
>25 mm	0 g - 500 g (minimum 50 pellets)

Both test portions shall be sieved in accordance with ISO 18846 using a sieve with punched round holes 3,15 mm in diameter in accordance with ISO 3310-2.

NOTE Rough handling during sample reduction and sieving could alter the surface of the pellets and thereby influence the result.

7 Procedure

7.1 Determination of pellet diameter class

In order to determine the diameter class for the test portion with 10 pellets, the diameter of each pellet shall be measured by means of a caliper (see 5.1). Record the result of each measurement. The average of the diameter measurements closest to the class defined in ISO 17225-2 determines the pellet diameter class.

7.2 Measuring of individual pellet length

Measure the length in mm of each pellet in the test portion with 40 - 50 pellets (see Clause 6) by means of a caliper (see 5.1). Record the result of each measurement.

7.3 Determination share of oversize length of pellets

7.3.1 Weighing

Determine the mass of the sieved test portion with 40 - 50 pellets by weighing to the nearest 0,01 g.

7.3.2 Sorting by length

Separate all pellets in the test portion with 40 - 50 pellets by means of a caliper (see 5.1) all pellets longer than the maximum length specified by ISO 17225-1, ISO 17225-2, ISO 17225-6, and ISO 17225-8.

NOTE Depending on the quality requirements, there can be more than one pellet length to be considered when sorting. Example: Concerning a D08 wood pellet, ISO 17225-2, Table 1 specifies 3,15 mm ≤ L ≤ 40 mm. Comment c in ISO 17225-2, Table 1 states:

“Amount of pellets longer than 40 mm can be 1 w-%. Maximum length shall be <45 mm. Pellets are longer than 3,15 mm, if they stay on a round-hole sieve of 3,15 mm. (For the) amount of pellets shorter than 10 mm, (the) w-% (is) recommended to be stated”.

7.3.3 Weighing of fractions by length

Weigh the mass of the pellets separated in 7.3.2 to the nearest 0,01 g and record the result.

8 Calculation

8.1 Pellet diameter class

Calculate the mean value and the standard deviation of the recorded pellets diameters class as determined in 7.1 and express the results to the nearest 0,1 mm.

In case the test portion contains pellets of different diameter classes (e.g. a mixture of D06 and D08), the average and the standard deviation of the pellet diameter for each of the classes shall be calculated separately and reported as a percent share of the number of measured pellets.

8.2 Share of oversize pellets

Calculate the w-% of oversize pellets by dividing the weight of the oversize pellets by length as established in 7.3.3 by the weight of the test portion as established in 7.3.1 and multiply by 100. Report the number of pellets above maximum dimension by length as specified in ISO 17225-1, ISO 17225-2, ISO 17225-6 or ISO 17225-8.

8.3 Average pellet length

Calculate the mean value and the standard deviation of the recorded pellet lengths as determined in 7.2 and express the result to the nearest 0,1 mm.

9 Performance characteristics

Because of the varying nature of the pellets covered by this document it is not possible to give a precision statement (repeatability or reproducibility) for this test method.

10 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of the product (sample) tested;

c) a reference to this International Standard, i.e. ISO 17829;

d) mean value and standard deviation of the pellet length as per 8.3;

e) mean value and standard deviation of the diameter for each of the class(es) (see 7.1) and each class reported in percentage share of the total number of measured pellets

f) w-% of pellets longer than 40 mm as specified in ISO 17225-1, ISO 17225-2, ISO 17225-6 and ISO 17225-8 (see 7.3.2);

g) number of pellets longer than the maximum length specified in ISO 17225-1, ISO 17225-2, ISO 17225-6 and ISO 17225-8 (see 7.3.2);

h) any unusual features noted during the determination, which may affect the result;

i) any operation not included in this International Standard, or regarded as optional.

IS 17653:2021/
ISO 18122 : 2015

SUMMARY

Ash content is a critical parameter for fuel deliveries because ash is a by-product of combustion that requires removal. The quantity of ash in a fuel can have economic implications related to disposal or utilization in other products. Furthermore, the chemical composition of the ash can contribute to slagging and corrosion in combustion equipment, making it important to know the amount of ash in the fuel.

The standard provides a method to determine the ash content for all solid biofuels. The method involves heating a sample in air under controlled conditions and then calculating the mass of the remaining residue.

IS 17653 : 2021

SOLID BIOFUELS – DETERMINATION OF ASH CONTENT

Indian Standard

SOLID BIOFUELS — DETERMINATION OF ASH CONTENT

1 Scope

This International Standard specifies a method for the determination of ash content of all solid biofuels.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18134-3, *Solid Biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

EN 14778[1], *Solid Biofuels — Sampling*

EN 14780[2], *Solid Biofuels — Sample preparation*

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 16559 and the following apply.

3.1
nominal top size
aperture of the sieve where at least 95 % by mass of the material passes

[SOURCE: ISO 16559]

3.2
laboratory sample
combined sample or a sub-sample of a combined sample for use in a laboratory

[SOURCE: ISO 16559]

3.3
test sample
laboratory sample after an appropriate preparation made by the laboratory

[SOURCE: ISO 16559]

3.4
test portion
sub-sample either of a laboratory sample or a test sample

[SOURCE: ISO 16559]

---

[1] To be replaced by ISO 18135.

[2] To be replaced by ISO 14780.

3.5

general analysis sample

sub-sample of a laboratory sample having a nominal top size of 1 mm or less and used for a number of chemical and physical analyses

[SOURCE: ISO 16559]

4 Principle

The ash content is determined by calculation the mass of the residue remaining after the sample is heated in air under rigidly controlled conditions of time, sample weight and equipment specifications to a controlled temperature of (550 ± 10) °C.

Automatic equipment (such as gravimetric analysers) may be used when the method is validated with biomass reference samples of an adequate biomass type. The automatic equipment shall fulfil all the requirements given in Clause 7 regarding sample size, heating procedure, atmosphere, temperature, and weighing accuracy.

NOTE Difference in the ash content if determined at a higher temperature, 815 °C, according to Reference [1] as compared to 550 °C, is explained by the decomposition of carbonates forming CO_2, by losses of volatile inorganic compounds and further oxidation of inorganic compounds (to higher oxidation states).

5 Apparatus

5.1 Dish

A dish of inert material, such as porcelain, silica, or platinum and of such size that the test sample loading does not exceed 0,1 g/cm^2 of bottom area.

NOTE If the test sample loading exceeds 0,1 g/cm^2 of bottom area there is a risk of incomplete incineration (in the lower sample layer) or absorption of CO_2 in the ash layer at the top (as $CaCO_3$) of calcium rich samples (as e.g. pure wood).

5.2 Furnace

The furnace shall be capable of providing a zone of uniform heat at the temperatures required and reaching these temperatures within the specified times. The ventilation rate through the furnace shall be such that no lack of oxygen for combustion arises during the heating procedure.

NOTE A ventilation rate of between five and 10 air changes per hour is sufficient.

5.3 Balance

The balance shall be capable of reading to the nearest 0,1 mg.

5.4 Desiccator and desiccant

A desiccator with appropriate desiccant is required to prevent absorption of moisture from the atmosphere by the test sample.

WARNING — Ash from solid biofuel is very hygroscopic and there is a risk that moisture bound in the desiccant can be absorbed in the sample. Therefore, the desiccant shall be controlled frequently and dried if necessary. In addition, lids shall be used to cover dishes while in the desiccator to prevent the absorption of moisture.

6 Sample preparation

A laboratory sample for the determination of ash content shall be obtained in accordance with EN 14778. From the laboratory sample a general analysis sample is prepared in accordance with EN 14780 and has a nominal particle top size of 1 mm or less.

6.1 Sample size

The general analysis sample shall include material sufficient for determination of ash content and moisture content.

6.2 Sample conditioning

The determination of ash content shall be done either

a) directly on a test portion of the general analysis sample, including a concurrent determination of the moisture content of a similar test portion in accordance with ISO 18134-3, or

b) from a test portion of the general analysis sample which has been dried using the same drying procedure as in the determination of the moisture content of the test portion and kept absolutely dry before the weighing for the ash content determinations (the test portion shall be kept in a closed container in a desiccator with desiccant).

NOTE For some solid biofuels it may be necessary to prepare a general analysis sample to a nominal top size of less than 1 mm (e.g. 0,25 mm) in order to keep the stated precision.

7 Procedure

7.1 Conditioning of dish

Heat the empty dish in the furnace to (550 ± 10) °C for at least 60 min. Remove the dish from the furnace. Allow the dish to cool on a heat resistant plate for 5 min to 10 min and then transfer to a desiccator with desiccant and allow to cool to ambient temperature. When the dish is cool weigh to the nearest 0,1 mg and record the mass.

NOTE 1 Several dishes can be handled at the same time.

NOTE 2 For determination of the ash content at 815 °C, see Reference [1].

7.2 Conditioning of the general analysis sample

The general analysis sample shall be mixed carefully before weighing the test portion. Place a minimum of 1 g of test portion at the bottom of the dish and spread in an even layer over the bottom surface. Weigh the dish plus the test portion to the nearest 0,1 mg and record the mass. If the test portion previously has been oven-dried, both the dish and the test portion shall be dried at 105 °C and then weighed as a precautionary measure for absorption of moisture.

NOTE If the ash content is expected to be very low, use a larger size test portion (and a larger dish) to improve the accuracy.

7.3 Ashing of test portion

Place the dish in a cold furnace and heat the test portion in accordance with the following temperature program.

— Raise the furnace temperature evenly to 250 °C over a period of 30 min to 50 min (i.e. heating rate of 4,5 °C/min to 7,5 °C/min). Maintain the temperature at this level for 60 min to allow the volatiles to leave the test portion before ignition.

— Continue to raise the furnace temperature evenly to (550 ± 10) °C over a period of 30 min (i.e. heating rate of 10 °C/min). Maintain temperature at this level for at least 120 min.

7.4 Weighing

Remove the dish with its content from the furnace. Allow the dish and its content to cool on a heat resistant plate for 5 min to 10 min and then transfer to a desiccator with desiccant and allow to cool to ambient temperature. Weigh the dish with ash to the nearest 0,1 mg as soon as ambient temperature is reached and record the mass. Calculate the ash content of the test portion as detailed in Clause 8.

7.5 Completion of ashing

If there is any doubt of complete incineration (for instance presence of soot at visual inspection) reload the dish with ash into the hot furnace (at 550 °C) for additional 30 min periods until the change in mass is lower than 0,5 mg.

For improved incineration, droplets of distilled water or ammonium nitrate solution shall be added to the sample before it is reloaded into the cold (at room temperature) furnace, and reheated to (550 ± 10) °C and kept at this temperature for further 30 min periods until the change in mass is lower than 0,5 mg.

A minimum of two determinations shall be carried out on the general analysis sample.

8 Calculation

The ash content on dry basis, A_d, of the sample expressed as a percentage by mass on a dry basis shall be calculated using Formula (1):

$$A_d = \frac{(m_3 - m_1)}{(m_2 - m_1)} \times 100 \times \frac{100}{100 - M_{ad}} \qquad (1)$$

where

m_1 is the mass in g of empty dish;

m_2 is the mass in g of the dish plus the test portion;

m_3 is the mass in g of the dish plus ash;

M_{ad} is the % moisture content of the test portion used for determination.

The result shall be calculated to two decimal places and the mean value shall be rounded to the nearest 0,1 % for reporting.

9 Performance characteristics

9.1 Repeatability

The result of duplicate determinations, carried out over a short period, but not simultaneously, in the same laboratory by the same operator with the same apparatus on two representative test portions taken from the same general analysis sample, shall not differ more than the values stated in Table 1.

9.2 Reproducibility

The mean value of results of duplicate determinations carried out in two different laboratories, on representative test portions taken from the same sample, shall not differ more than the values stated in Table 1, see Reference [2].

Table 1 — Repeatability and reproducibility of the method

Ash content %	Maximum acceptable differences between results	
	Repeatability	Reproducibility
<1 %	0,1 % absolute	0,2 % absolute
>1 %	10 % relative	20 % relative

10 Test Report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) a reference to this International Standard, i.e. ISO 18122;

d) results of the test on dry basis (alternatively for all standards: results of the test including the basis in which they are expressed, as indicated in Clause 8);

e) any unusual features noted during the determination; which may affect the result;

f) any deviation from this International Standard, or operations regarded as optional.

IS 17654:2021/
ISO 18125: 2017

SUMMARY

Gross calorific value (GCV) is important for solid biofuels as it measures the total energy content, helping determine fuel efficiency, combustion performance, and suitability for various applications.

This standard specifies a method for the determination of the gross calorific value of a solid biofuel at constant volume and at the reference temperature 25 °C in a bomb calorimeter calibrated by combustion of certified benzoic acid. The net calorific value is then calculated from the gross calorific value.

The determination involves two separate experiments: combustion of the calibrant (benzoic acid) and combustion of the biofuel, both performed under the same conditions.

IS 17654 : 2021

SOLID BIOFUELS – DETERMINATION OF CALORIFIC VALUE

Indian Standard

SOLID BIOFUELS — DETERMINATION OF CALORIFIC VALUE

1 Scope

This document specifies a method for the determination of the gross calorific value of a solid biofuel at constant volume and at the reference temperature 25 °C in a bomb calorimeter calibrated by combustion of certified benzoic acid.

The result obtained is the gross calorific value of the analysis sample at constant volume with all the water of the combustion products as liquid water. In practice, biofuels are burned at constant (atmospheric) pressure and the water is either not condensed (removed as vapour with the flue gases) or condensed. Under both conditions, the operative heat of combustion to be used is the net calorific value of the fuel at constant pressure. The net calorific value at constant volume may also be used; formulae are given for calculating both values.

General principles and procedures for the calibrations and the biofuel experiments are presented in the main text, whereas those pertaining to the use of a particular type of calorimetric instrument are described in Annexes A to C. Annex D contains checklists for performing calibration and fuel experiments using specified types of calorimeters. Annex E gives examples to illustrate some of the calculations.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 651, *Solid-stem calorimeter thermometers*

ISO 652, *Enclosed-scale calorimeter thermometers*

ISO 1770, *Solid-stem general purpose thermometers*

ISO 1771, *Enclosed-scale general purpose thermometers*

ISO 14780, *Solid biofuels — Sample preparation*

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18134-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

ISO 18135, *Solid biofuels — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1

gross calorific value at constant volume

absolute value of the specific energy of combustion, in joules, for unit mass of a solid biofuel burned in oxygen in a calorimetric bomb under the conditions specified

Note 1 to entry: The products of combustion are assumed to consist of gaseous oxygen, nitrogen, carbon dioxide and sulphur dioxide, of liquid water (in equilibrium with its vapour) saturated with carbon dioxide under the conditions of the bomb reaction, and of solid ash, all at the *reference temperature* (3.4).

3.2

net calorific value at constant volume

absolute value of the specific energy of combustion, in joules, for unit mass of the biofuel burned in oxygen under conditions of constant volume and such that all the water of the reaction products remains as water vapour (in a hypothetical state at 0,1 MPa), the other products being as for the gross calorific value, all at the *reference temperature* (3.4)

3.3

net calorific value at constant pressure

absolute value of the specific heat (enthalpy) of combustion, in joules, for unit mass of the biofuel burned in oxygen at constant pressure under such conditions that all the water of the reaction products remains as water vapour (at 0,1 MPa), the other products being as for the gross calorific value, all at the *reference temperature* (3.4)

3.4

reference temperature

international reference temperature for thermochemistry of 25 °C is adopted as the reference temperature for calorific values

Note 1 to entry: See 8.7.

Note 2 to entry: The temperature dependence of the calorific value of biofuels is small [less than 1 J/(g × K)].

3.5

effective heat capacity of the calorimeter

amount of energy required to cause unit change in temperature of the calorimeter

3.6

corrected temperature rise

change in calorimeter temperature caused solely by the processes taking place within the combustion bomb

Note 1 to entry: The corrected temperature rise is the total observed temperature rise corrected for heat exchange, stirring power, etc. (8.6).

Note 2 to entry: The change in temperature may be expressed in terms of other units: resistance of a platinum or thermistor thermometer, frequency of a quartz crystal resonator, etc., provided that a functional relationship is established between this quantity and a change in temperature. The *effective heat capacity of the calorimeter* (3.5) may be expressed in units of energy per such an arbitrary unit. Criteria for the required linearity and closeness in conditions between calibrations and fuel experiments are given in 9.3.

Note 3 to entry: A list of the symbols used and their definitions is given in Annex F.

4 Principle

4.1 Gross calorific value

A weighed portion of the analysis sample of the solid biofuel is burned in high-pressure oxygen in a bomb calorimeter under specified conditions. The effective heat capacity of the calorimeter is determined in calibration experiments by combustion of certified benzoic acid under similar conditions, accounted for in the certificate. The corrected temperature rise is established from observations of temperature before, during and after the combustion reaction takes place. The duration and frequency of the temperature observations depend on the type of calorimeter used. Water is added to the bomb initially to give a saturated vapour phase prior to combustion (see 8.2.1 and 9.2.2), thereby allowing all the water formed, from the hydrogen and moisture in the sample, to be regarded as liquid water.

The gross calorific value is calculated from the corrected temperature rise and the effective heat capacity of the calorimeter, with allowances made for contributions from ignition energy, combustion of the fuse(s) and for thermal effects from side reactions such as the formation of nitric acid. Furthermore, a correction is applied to account for the difference in energy between the aqueous sulphuric acid formed in the bomb reaction and gaseous sulphur dioxide, i.e. the required reaction product of sulphur in the biofuel. The corresponding energy effect between aqueous and gaseous hydrochloric acid can be neglected due to the usually low value for the correction regarding solid biofuels.

4.2 Net calorific value

The net calorific value at constant volume and the net calorific value at constant pressure of the biofuel are obtained by calculation from the gross calorific value at constant volume determined on the analysis sample. The calculation of the net calorific value at constant volume requires information about the moisture and hydrogen contents of the analysis sample. In principle, the calculation of the net calorific value at constant pressure also requires information about the oxygen and nitrogen contents of the analysis sample.

5 Reagents

5.1 Oxygen, at a pressure high enough to fill the bomb to 3 MPa, pure with an assay of at least a volume fraction of 99,5 %, and free from combustible matter.

Oxygen made by the electrolytic process may contain up to a volume fraction of 4 % of hydrogen.

5.2 Fuse.

5.2.1 Ignition wire, of nickel-chromium 0,16 mm to 0,20 mm in diameter, platinum 0,05 mm to 0,10 mm in diameter, or another suitable conducting wire with well-characterized thermal behaviour during combustion.

5.2.2 Cotton fuse, of white cellulose cotton, or equivalent, if required (see 8.2.1).

5.3 Combustion aids of known gross calorific value, composition and purity, like benzoic acid, n-dodecane, paraffin oil, combustion bags or capsules may be used.

5.4 Standard volumetric solutions and indicators, only for use when analysis of final bomb solutions is required.

5.4.1 Barium hydroxide solution, $c[\mathrm{Ba(OH)_2}] = 0{,}05$ mol/l.

5.4.2 Sodium carbonate solution, $c(\mathrm{Na_2CO_3}) = 0{,}05$ mol/l.

5.4.3 Sodium hydroxide solution, $c(\mathrm{NaOH}) = 0{,}1$ mol/l.

5.4.4 Hydrochloric acid solution, $c(\mathrm{HCl}) = 0{,}1$ mol/l.

5.4.5 Screened methyl orange indicator, 1 g/l solution.

Dissolve 0,25 g of methyl orange and 0,15 g of xylene cyanol FF in 50 ml of a volume fraction of 95 % ethanol and dilute to 250 ml with water.

5.4.6 Phenolphthalein, 10 g/l solution.

Dissolve 2,5 g of phenolphthalein in 250 ml of a volume fraction of 95 % ethanol.

5.5 Benzoic acid, of calorimetric-standard quality, certified by (or with certification unambiguously traceable to) a recognized standardizing authority.

Benzoic acid is the sole substance recommended for calibration of an oxygen-bomb calorimeter. For the purpose of checking the overall reliability of the calorimetric measurements, test substances, e.g. n-dodecane, are used. Test substances are mainly used to prove that certain characteristics of a sample, e.g. burning rate or chemical composition, do not introduce bias in the results. A test substance shall have a certified purity and a well-established energy of combustion.

The benzoic acid is burned in the form of pellets. It is normally used without drying or any treatment other than pelletizing; consult the sample certificate. It does not absorb moisture from the atmosphere at relative humidities below 90 %.

The benzoic acid shall be used as close to certification conditions as is feasible; significant departures from these conditions shall be accounted for in accordance with the directions in the certificate. The energy of combustion of the benzoic acid, as defined by the certificate for the conditions utilized, shall be adopted in calculating the effective heat capacity of the calorimeter (see 9.2).

6 Apparatus

6.1 General

The **calorimeter** (see Figure 1) consists of the assembled combustion bomb, the calorimeter can (with or without a lid), the calorimeter stirrer, water, temperature sensor, and leads with connectors inside the calorimeter can required for ignition of the sample or as part of temperature measurement or control circuits. During measurements, the calorimeter is enclosed in a thermostat. The manner in which the thermostat temperature is controlled defines the working principle of the instrument and hence the strategy for evaluation of the corrected temperature rise.

In aneroid systems (systems without a fluid), the calorimeter can, stirrer and water are replaced by a metal block. The combustion bomb itself constitutes the calorimeter in some aneroid systems.

In combustion calorimetric instruments with a high degree of automation, especially in the evaluation of the results, the calorimeter is in a few cases not as well-defined as the traditional, classical-type calorimeter. Using such an automated calorimeter is, however, within the scope of this document as long as the basic requirements are met with respect to calibration conditions, comparability between calibration and fuel experiments, ratio of sample mass to bomb volume, oxygen pressure, bomb liquid, reference temperature of the measurements and repeatability of the results. A print-out of some specified parameters from the individual measurements is essential. Details are given in Annex C.

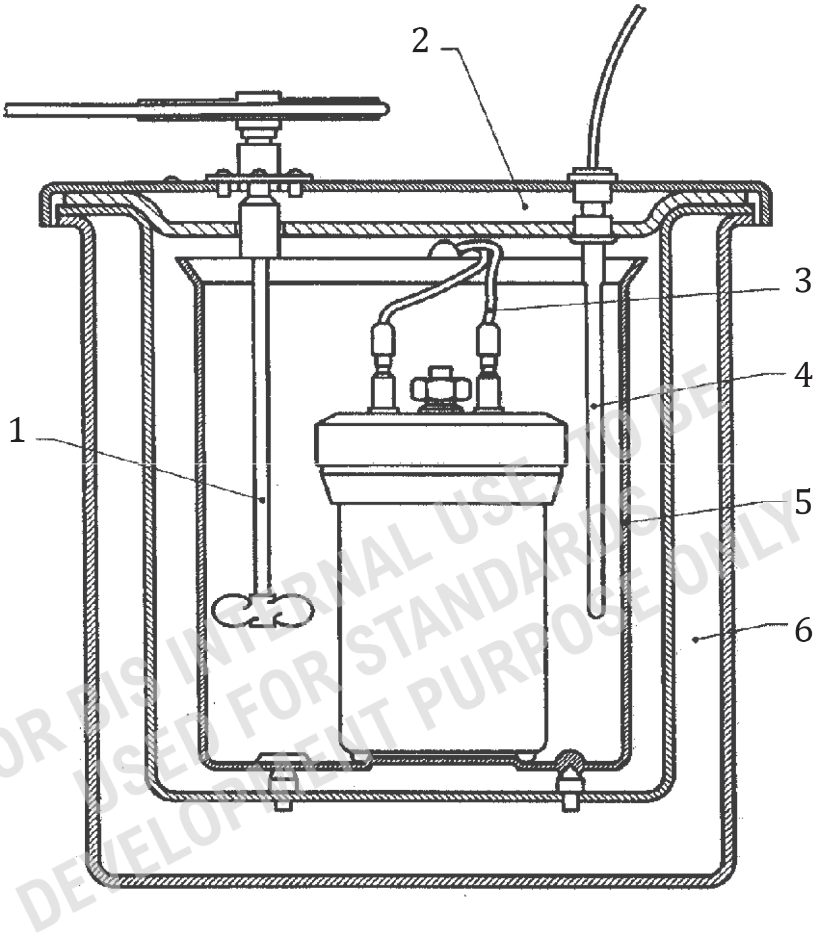
As the room conditions (temperature fluctuation, ventilation, etc.) may have an influence on the precision of the determination, the manufacturer's instructions for the placing of the instrument shall always be followed.

Equipment, adequate for determinations of calorific value in accordance with this document, is specified in 6.2 to 6.8.

6.2 Calorimeter with thermostat

6.2.1 Combustion bomb, capable of withstanding safely the pressures developed during combustion. The design shall permit complete recovery of all liquid products. The material of construction shall resist corrosion by the acids produced in the combustion of biofuels. A suitable internal volume of the bomb would be from 250 ml to 350 ml.

WARNING — Bomb parts shall be inspected regularly for wear and corrosion; particular attention shall be paid to the condition of the threads of the main closure. Manufacturers' instructions and any local regulations regarding the safe handling and use of the bomb shall be observed. When more than one bomb of the same design is used, it is imperative to use each bomb as a complete unit. Swapping of parts may lead to a serious accident.



Key

1	stirrer	4	thermometer
2	thermostat lid	5	calorimeter can
3	ignition leads	6	thermostat

Figure 1 — Classical-type bomb combustion calorimeter with thermostat

6.2.2 Calorimeter can, made of metal, highly polished on the outside and capable of holding an amount of water sufficient to completely cover the flat upper surface of the bomb while the water is being stirred. A lid generally helps reduce evaporation of calorimeter water, but unless it is in good thermal contact with the can, it lags behind in temperature during combustion, giving rise to undefined heat exchange with the thermostat and a prolonged main period.

6.2.3 Stirrer, working at constant speed. The stirrer shaft should have a low-heat conduction and/or a low-mass section below the cover of the surrounding thermostat to minimize transmission of heat to or from the system; this is of particular importance when the stirrer shaft is in direct contact with the stirrer motor. When a lid is used for the calorimeter can, this section of the shaft should be above the lid.

The rate of stirring for a stirred-water type calorimeter should be large enough to make sure that hot spots do not develop during the rapid part of the change in temperature of the calorimeter. A rate of stirring such that the length of the main period can be limited to 10 min or less is usually adequate (see Annexes A and B).

6.2.4 Thermostat (water jacket), completely surrounding the calorimeter, with an air gap of approximately 10 mm separating calorimeter and thermostat.

The mass of water of a thermostat intended for isothermal operation shall be sufficiently large to outbalance thermal disturbances from the outside. The temperature should be controlled to within ±0,1 K or better throughout the experiment. A passive constant temperature ("static") thermostat shall have a heat capacity large enough to restrict the change in temperature of its water. Criteria for satisfactory behaviour of this type of water jacket are given in Annex B.

NOTE 1 For an insulated metal static jacket, satisfactory properties are usually ensured by making a wide annular jacket with a capacity for water of at least 12,5 l.

NOTE 2 Calorimeters surrounded by insulating material, creating a thermal barrier, are regarded as static-jacket calorimeters.

When the thermostat (water jacket) is required to follow closely the temperature of the calorimeter, it should be of low mass and preferably have immersion heaters. Energy shall be supplied at a rate sufficient to maintain the temperature of the water in the thermostat to within 0,1 K of that of the calorimeter water after the charge has been fired. When in a steady state at 25 °C, the calculated mean drift in temperature of the calorimeter shall not exceed 0,000 5 K/min (see A.3.2).

6.2.5 Temperature measuring instrument, capable of indicating temperature with a resolution of at least 0,001 K so that temperature intervals of 2 K to 3 K can be determined with a resolution of 0,002 K or better. The absolute temperature shall be known to the nearest 0,1 K at the reference temperature of the calorimetric measurements. The temperature measuring device should be linear, or linearized, in its response to changes in temperature over the interval it is used.

As alternatives to the traditional mercury-in-glass thermometers, suitable temperature sensors are platinum resistance thermometers, thermistors, quartz crystal resonators, etc. which together with a suitable resistance bridge, null detector, frequency counter or other electronic equipment provide the required resolution. The short-term repeatability of this type of device shall be 0,001 K or better. Long-term drift shall not exceed the equivalent of 0,05 K for a period of 6 months. For sensors with linear response (in terms of temperature), drift is less likely to cause bias in the calorimetric measurements than are nonlinear sensors.

Mercury-in-glass thermometers shall conform to ISO 651, ISO 652, ISO 1770 or ISO 1771. A viewer with magnification about 5× is needed for reading the temperature with the resolution required.

A mechanical vibrator to tap the thermometer is suitable for preventing the mercury column from sticking (see 8.4). If this is not available, the thermometer shall be tapped manually before reading the temperature.

6.2.6 Ignition circuit

The electrical supply shall be 6 V to 12 V alternating current from a step-down transformer or direct current from batteries. It is desirable to include a pilot light in the circuit to indicate when current is flowing.

Where the firing is done manually, the firing switch shall be of the spring-loaded, normally open type, located in such a manner that any undue risk to the operator is avoided (see warning in 8.4).

6.3 Crucible, of silica, nickel-chromium, platinum or similar unreactive material.

The crucible should be 15 mm to 25 mm in diameter, flat based and about 20 mm deep. Silica crucibles should be about 1,5 mm thick and metal crucibles about 0,5 mm thick.

If smears of unburned carbon occur, a small low-mass platinum or nickel-chromium crucible, for example 0,25 mm thick, 15 mm in diameter and 7 mm deep, may be used.

6.4 Ancillary pressure equipment

6.4.1 Pressure regulator, to control the filling of the bomb with oxygen.

6.4.2 Pressure gauge (e.g. 0 MPa to 5 MPa), to indicate the pressure in the bomb with a resolution of 0,05 MPa.

6.4.3 Relief valve or bursting disk, operating at 3,5 MPa, and installed in the filling line, to prevent overfilling the bomb.

CAUTION — Equipment for high-pressure oxygen shall be kept free from oil and grease (high vacuum grease recommended by the manufacturer can be used according to the operating manual of the instrument). Do not test or calibrate the pressure gauge with hydrocarbon fluid.

6.5 Timer, indicating minutes and seconds.

6.6 Balances

6.6.1 Balance for weighing the sample, fuse, etc., with a resolution of at least 0,1 mg; 0,01 mg is preferable and is recommended when the sample mass is of the order of 0,5 g or less (see 8.2.1).

6.6.2 Balance for weighing the calorimeter water, with a resolution of 0,5 g (unless water can be dispensed into the calorimeter by volume with the required accuracy; see 8.3).

6.7 Thermostat (optional), for equilibrating the calorimeter water before each experiment to a predetermined initial temperature, within about ±0,3 K.

6.8 Pellet press, capable of applying a force of about 10 t, either hydraulically or mechanically, and having a die suitable to press a pellet having a diameter about 13 mm and a mass of (1,0 ± 0,2) g.

7 Preparation of test sample

Samples for the determination of calorific value shall be sampled in accordance with ISO 18135 and shall be received in the laboratory in sealed air-tight containers or packages. The biofuel sample used for the determination of calorific value shall be the general analysis sample (ground to pass a test sieve with an aperture of 1,0 mm) prepared according to the procedure given in ISO 14780. Sieve with an aperture less than 1,0 mm (0,5 mm or 0,25 mm) might be necessary for some solid biofuels to ensure the requisite repeatability and a complete combustion.

Due to the low density of solid biofuels, they shall be tested in a pellet form. As a test portion, a pellet of mass (1,0 ± 0,2) g is pressed with a suitable force to produce a compact, unbreakable test piece. Alternatively, the test may be carried out in powder form test portion closed in a combustion bag or capsule.

The general analysis sample shall be well mixed and in reasonable moisture equilibrium with the laboratory atmosphere. The moisture content shall either be determined simultaneously with the weighing of the samples for the determination of calorific value, or the sample shall be kept in a small, effectively closed container until moisture analyses are performed, to allow appropriate corrections for moisture in the analysis sample.

Determination of the moisture content (M_{ad}) of the general analysis sample shall be carried out by the method specified in ISO 18134-3.

A flow chart for a routine calorific value determination can be found in Annex H.

8 Calorimetric procedure

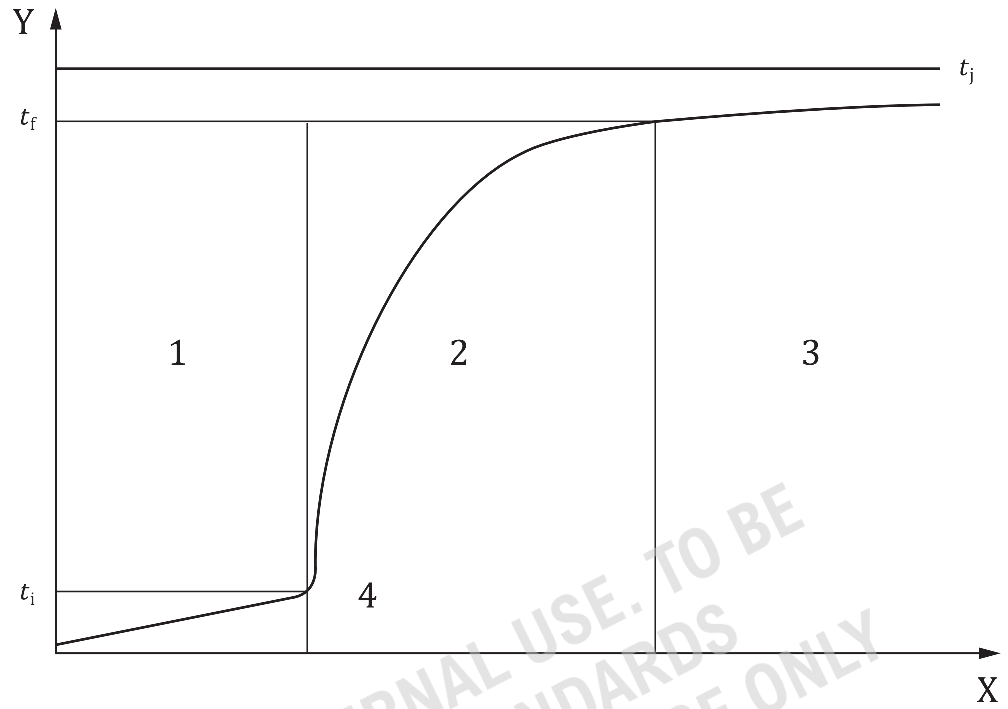
8.1 General

The calorimetric determination consists of two separate experiments, combustion of the calibrant (benzoic acid) and combustion of the biofuel, both under same specified conditions. The calorimetric procedure for the two types of experiment is essentially the same. In fact, the overall similarity is a requirement for proper cancellation of systematic errors caused, for example, by uncontrolled heat leaks not accounted for in the evaluation of the corrected temperature rise θ .

The experiment consists of carrying out quantitatively a combustion reaction (in high-pressure oxygen in the bomb) to defined products of combustion and of measuring the change in temperature caused by the total bomb process.

The temperature measurements required for the evaluation of the corrected temperature rise θ are made during a fore period, a main (= reaction) period, and an after period as outlined in Figure 2. For the adiabatic type calorimeter, the fore and after periods need, in principle, be only as long as required to establish the initial (firing) and final temperatures, respectively (see Annex A). For the isoperibol (isothermal jacket) and the static-jacket type calorimeters, the fore and after periods serve to establish the heat exchange properties of the calorimeter required to allow proper correction for heat exchange between calorimeter and thermostat during the main period when combustion takes place. The fore and after periods then have to be longer; see Annex B.

The power of stirring shall be maintained constant throughout an experiment which calls for a constant rate of stirring. An excessive rate of stirring results in an undesirable increase in the power of stirring with ensuing difficulties in keeping it constant. A wobbling stirrer is likely to cause significant short-term variations in stirring power.



Key

X	time	1	fore period
Y	temperature	2	main period
t_f	temperature at the end of main period	3	after period
t_i	ignition temperature	4	ignition
t_j	jacket temperature		

Figure 2 — Time-temperature curve (isoperibol calorimeter)

During combustion, the bomb head will become appreciably hotter than other parts of the bomb, and it is important to have enough well-stirred water above it to maintain reasonably small temperature gradients in the calorimeter water during the rapid part of the rise in temperature. For aneroid systems, the particular design determines to what extent hot spots may develop (see Annex C).

Certain biofuels may persistently burn incompletely, “exploding” and/or leaving residues that contain significant amounts of unburned sample or soot. By adding known amounts of an auxiliary material (e.g. benzoic acid, n-dodecane or paraffin oil), by using bags or capsules or cotton fuse, by omitting the distilled water from the bomb, or by using a lower oxygen filling pressure, a clean combustion can, in most instances, be achieved.

The auxiliary material shall be chemically stable, have known composition and purity, have a low vapour pressure and have a well-established energy of combustion; the energy should be known to within 0,10 % for particular material used. The amount used should be limited to the minimum amount required to achieve complete combustion of the sample. It should not exceed an amount that contributes half of the total energy in an experiment. The optimum proportion of the sample to auxiliary material depends on the properties of the fuel, and needs to be determined by experiment.

The mass of the auxiliary material shall be determined as accurately as possible so that its contribution can be correctly accounted for; this is particularly important when a hydrocarbon oil is used as its specific energy of combustion is considerably higher than that of the biofuel.

8.2 Preparing the bomb for measurement

8.2.1 General procedure

Weigh the test portion, pellet or the filled combustion bag or capsule, in the crucible, with a weighing resolution of 0,01 % or better. For 1 g test portions (see 9.2 and 10.2), this means weighing to the nearest 0,1 mg. Weigh the combustible fuse and/or ignition wire either with a precision comparable with that for weighing the test portion, or keep its mass constant, within specified limits, for all experiments (see 9.4 and 9.6.1).

Fasten the ignition wire tautly between the electrodes in the bomb (see also Note hereafter). Check the resistance of the ignition circuit of the bomb; for most bombs, it should not exceed 5 Ω to 10 Ω, measured between the outside connectors of the bomb head, or between the connector for the insulated electrode and the bomb head.

Tie, or attach firmly, the fuse (if needed, see Note hereafter) to the ignition wire, place the crucible in its support and bring the fuse into contact with the sample pellet or capsule. Make sure that the position of the crucible in the assembled bomb will be symmetrical with respect to the surrounding bomb wall.

When the ignition wire is combustible as well as electrically conducting, an alternative procedure may be adopted. A longer piece of wire, enough to make an open loop, is connected to the electrodes. After mounting of the crucible, the loop is brought in contact with the sample pellet or capsule. (In some cases, the ignition process is better controlled when the wire is kept at a small distance above the sample pellet.) Care should be taken to prevent any contact between ignition wire and crucible, in particular when a metal crucible is used since this would result in shorting the ignition circuit. A special fuse is superfluous under these conditions. The resistance of the ignition circuit of the bomb will be increased by a small amount only. For closer details of preparing the bomb, refer also to the manufacturer's instructions.

Add a defined amount of distilled water to the bomb. The amount shall always be exactly the same both in calibration and in determinations (see 9.2.1 and 9.2.2). As a main principle for biofuels, (1,0 ± 0,1) ml distilled water is added into the bomb. With some biofuels (and some calorimeters), the complete combustion can be achieved by omitting the distilled water out from the bomb or by using combustion aid. In some cases, the total absorption of the gaseous combustion products might provide the use of a larger amount of distilled water (e.g. 5 ml).

Assemble the bomb and charge it slowly with oxygen to a pressure of (3,0 ± 0,2) MPa without displacing the original air, or flush the bomb (with the outlet valve open, see manufacturer's instructions) with oxygen for about 30 s, close slowly the valve and charge the bomb to the pressure of (3,0 ± 0,2) MPa. The same procedure shall be used both in calibration and in determinations. If the bomb is inadvertently charged with oxygen above 3,3 MPa, discard the test and begin again.

WARNING — Do not reach over the bomb during charging.

The bomb is now ready for mounting in the calorimeter can.

8.2.2 Using combustion aid

Liquid combustion aid: After the mass of the pellet has been determined, the auxiliary liquid material shall be added drop by drop on the pellet placed in the crucible (allowing the liquid to be absorbed) and the added amount to be determined precisely by weighing.

Solid combustion aid: Use of solid combustion aids (benzoic acid recommended) without combustion bags or capsules is not recommended (a homogenous mixture of sample material and combustion aid before pressing the test pellet might be difficult to achieve).

Combustion bags or capsules: Combustion capsules or bags, or combustible crucibles with precisely known calorific value (gelatin, acetobutyrate or polyethylene) may be used as combustion aids (as such or with, e.g. benzoic acid) according to the manufacturer's instructions. They shall be weighed precisely before filling (see also 8.1). The sample material and the combustion aid like benzoic acid shall be mixed cautiously in the bag or capsule before testing.

8.3 Assembling the calorimeter

Bring the calorimeter water to within ±0,3 K of the selected initial temperature and fill the calorimeter can with the required amount. The quantity of water in the calorimeter can shall be the same to within 0,5 g or better in all experiments (see 9.6.1). Make sure that the outer surface of the can is dry and clean before the latter is placed in the thermostat. Mount the bomb in the calorimeter can after the can (containing the correct amount of water) has been placed in the thermostat.

Alternatively, the system may be operated on a constant total-calorimeter-mass basis (see 9.6.2). The bomb is then mounted in the calorimeter can before this is weighed with the water. The total mass of the calorimeter can, with the assembled bomb and the calorimeter water, shall then be within the 0,5 g or better limit in all experiments.

The assembled calorimeter shall contain enough water to well cover the flat upper surface of the bomb head and cap.

NOTE Weighing the water to within 0,5 g applies when the effective heat capacity is in the order of 10 kJ/K.

Check the bomb for gas leaks as soon as its top becomes covered with water. If the gas valves are not fully submerged, check for leaks with a drop of water across the exposed opening. Connect the leads for the ignition circuit and mount the thermometer.

WARNING — If gas escapes from the bomb, discard the test, eliminate the cause of leakage and begin again. Apart from being a hazard, leaks will inevitably lead to erroneous results.

Cooling water, temperature controls, stirrers, etc. are turned on and adjusted, as outlined in the instrument manual. Make sure that the calorimeter stirrer works properly. A period of about 5 min is normally required for the assembled calorimeter to reach a steady state in the thermostat or jacket, irrespective of the type of calorimeter. The criteria for when steady state has been attained depend on the working principle of the calorimeter (see Annexes A and B).

8.4 Combustion reaction and temperature measurements

Start taking temperature readings, to the nearest 0,001 K or better, as soon as the calorimeter has reached steady-state conditions. Readings at 1 min intervals normally suffice to establish the drift rate of the fore period or check the proper functioning of an adiabatic system. When a mercury-in-glass thermometer is used for the temperature measurements, tap the thermometer lightly for about 10 s before each reading and take care to avoid parallax errors.

At the end of the fore period, when the initial temperature t_i has been established, the combustion is initiated by firing the fuse. Hold the switch closed only for as long as it takes to ignite the fuse. Normally, the current is automatically interrupted as the conducting wire starts burning or partially melts. As long as the resistance of the ignition circuit of the combustion bomb is kept at its normal low value, the electrical energy required to initiate the reaction is so small that there is no need to measure and account for it separately.

WARNING — Do not extend any part of the body over the calorimeter during firing, nor for 20 s thereafter.

Continue taking temperature readings at 1 min intervals. The time corresponding to t_i marks the beginning of the main period. During the first few minutes after the charge has been fired, when the temperature is rising rapidly, readings to the nearest 0,02 K are adequate. Resume reading temperatures to the nearest 0,001 K or better as soon as is practicable, but no later than 5 min after the beginning of the main period. Criteria for the length of the fore, main and after periods, and hence the total number of temperature readings required, are given in Annexes A and B.

8.5 Analysis of products of combustion

At the end of the after period, when all the required temperature readings have been completed, remove the bomb from the calorimeter, release the pressure slowly (following manufacturers' manual) and dismantle the bomb. Examine the interior of the bomb, the crucible and any solid residue carefully for signs of incomplete combustion. Discard the test if unburned sample or any soot deposit is visible. Remove and measure any unreacted pieces of combustible ignition wire.

NOTE Another symptom of incomplete combustion is the presence of carbon monoxide in the bomb gas. Slow release of the gas through a suitable detector tube reveals any presence of carbon monoxide and indicates the concentration level. 0,1 ml/l of carbon monoxide in the combustion gas from a 300 ml bomb corresponds to an error of about 10 J.

Wash the contents of the bomb quantitatively into a beaker with distilled water. Make sure that the underside of the bomb head, the electrodes and the outside of the crucible are also washed.

In the case of calibration experiments, determine the formed nitric acid from the combined bomb washings either by ion-chromatography (as nitrate) as described in ISO 10304-1 or dilute the combined washings to about 50 ml and analyse for nitric acid, e.g. by titration with the sodium hydroxide solution (5.4.3) to a pH of about 5,5 or by using the screened methyl orange solution (5.4.5) as an indicator.

When the "sulphur" and/or nitric acid corrections are based on the actual amounts formed in the bomb process, the bomb washings from fuel combustions are analysed by the procedure described in the next three paragraphs [methods a) to c)] or by an equivalent method. If the sulphur content of the biofuel and the nitric acid correction are known, analysis of the final bomb liquid may be omitted (see 10.1).

a) Determine the formed nitric and sulphuric acid (as nitrate and as sulfate, respectively) by ion-chromatography as in ISO 10304-1.

b) Dilute the combined bomb washings to about 100 ml. Boil the washings to expel carbon dioxide and titrate the solution with barium hydroxide solution (5.4.1) while it is still hot using the phenolphthalein solution (5.4.6) as an indicator. Add 20,0 ml of the sodium carbonate solution (5.4.2), filter the warm solution and wash the precipitate with distilled water. When cold, titrate the filtrate with the hydrochloric acid solution (5.4.4), using the screened methyl orange solution (5.4.5) as an indicator, ignoring the phenolphthalein colour change.

c) If the sulfur content of the biofuel is known, the boiled bomb washings may be titrated, while still hot, with a simplified method using sodium hydroxide solution (5.4.3) and phenolphthalein as indicator (5.4.6).

8.6 Corrected temperature rise θ

8.6.1 Observed temperature rise

The temperature at the end of the main period t_{f} gives, together with the initial or firing temperature t_{i}, the observed temperature rise $t_{\mathrm{f}} - t_{\mathrm{i}}$.

8.6.2 Isoperibol and static-jacket calorimeters

In addition to the rise in temperature caused by the processes in the combustion bomb, the observed temperature rise contains contributions from heat exchange between calorimeter and thermostat and from stirring power. Allowance for heat exchange is made by the so-called heat-leak correction Δt_{ex} which includes the contribution from stirring power, i.e. Formula (1):

$$t_{\mathrm{f}} - t_{\mathrm{i}} = \theta + \Delta t_{\mathrm{ex}} \tag{1}$$

and hence Formula (2):

$$\theta = (t_{\mathrm{f}} - t_{\mathrm{i}}) - \Delta t_{\mathrm{ex}} \tag{2}$$

There are various ways of evaluating the term Δt_{ex}. The most common procedures used are the Regnault-Pfaundler and the Dickinson extrapolation methods.

NOTE The Regnault-Pfaundler method automatically accounts for variations in the time-temperature relationship for different types of samples and is hence the more reliable of the two methods.

Detailed instructions for the numerical evaluation of Δt_{ex} and the corrected temperature rise θ for isoperibol and static-jacket calorimeters are given in Annex B. The resulting formulae for Δt_{ex} are summarized in Formulae (3) and (4).

Regnault-Pfaundler method (see B.5.2)

$$\Delta t_{\mathrm{ex}} = (\tau_{\mathrm{f}} - \tau_{\mathrm{i}}) \times g_{\mathrm{f}} + \frac{g_{\mathrm{i}} - g_{\mathrm{f}}}{t_{\mathrm{mf}} - t_{\mathrm{mi}}} \times \left[n \times t_{\mathrm{mf}} - \frac{(t_{\mathrm{i}} + t_{\mathrm{f}})}{2} - \sum_{k=1}^{k=n\text{-}1} t_{\mathrm{k}} \right] \tag{3}$$

where

g_{i} is the drift rate in the fore (initial rating) period, in K/min;

g_{f} is the drift rate in the after (final rating) period, in K/min;

t_{mi} is the mean temperature in the fore period, in °C;

t_{mf} is the mean temperature in the after period, in °C;

t_{i} $(= t_0)$ is the temperature at the beginning of the main period (the time for ignition), in °C;

t_{f} $(= t_{\mathrm{n}})$ is the temperature at the end of the main period, in °C;

t_{k} is the successive temperature readings, in °C, taken at 1 min intervals during the main period (t_1 being the temperature 1 min after the beginning of the main period and $t_{\mathrm{n}} = t_{\mathrm{f}}$);

τ_{i} is the time at the beginning of the main period (time of ignition), in min;

τ_{f} is the time at the end of the main period, in min;

n is the number of 1 min intervals in the main period.

Dickinson extrapolation method (see B.5.3)

$$\Delta t_{\mathrm{ex}} = g_{\mathrm{i}}(\tau_{\mathrm{x}} - \tau_{\mathrm{i}}) + g_{\mathrm{f}}(\tau_{\mathrm{f}} - \tau_{\mathrm{x}}) \tag{4}$$

where

τ_{x}	is the time, in min, where the change in temperature $(t_{\mathrm{x}} - t_{\mathrm{i}})$ is 0,6 times the observed temperature rise $(t_{\mathrm{f}} - t_{\mathrm{i}})$;
g_{i} and g_{f}	represent, in principle, the drift rate, in K/min, at τ_{i} and τ_{f}, respectively. They are calculated as for the Regnault-Pfaundler method.

Alternatively, temperature may be expressed in some arbitrary unit throughout (see 9.6.1).

8.6.3 Adiabatic calorimeters

In adiabatic systems, heat exchange is by definition negligible. It is, however, common practice to compensate for the stirring power by an offset in temperature in the adiabatic control system (see Annex A). The corrected temperature rise θ then becomes Formula (5):

$$\theta = t_{\mathrm{f}} - t_{\mathrm{i}} \tag{5}$$

Stirring power is otherwise manifested as a constant drift in temperature throughout the experiment and is easily corrected for, but may prolong the total period of temperature observations.

Detailed instructions for the numerical evaluation of the corrected temperature rise θ for adiabatic calorimeters are given in Annex A.

8.6.4 Thermometer corrections

When a mercury-in-glass thermometer is used, the corrections specified in the certificate issued with the thermometer shall be applied to the observed initial temperature t_{i} and the final temperature t_{f}.

8.7 Reference temperature

The temperature at the end of the main period, the final temperature t_{f}, is the reference temperature of the individual experiment.

9 Calibration

9.1 Principle

Combustion of certified benzoic acid under specified conditions to gaseous carbon dioxide and liquid water serves to make a change in temperature of the calorimeter of one unit interpretable in defined units of energy. The classical type of combustion calorimeter can be maintained unchanged over extended periods of time in terms of mass (heat capacity), geometry and heat exchange surfaces. This allows for calibration of the instrument to be carried out as a separate series of measurements, establishing the effective heat capacity ε of the calorimeter.

This calibration constant ε should not change significantly over time, provided minor repairs or other changes in the system are correctly accounted for. Some of the fully automated calorimetric instruments are, however, physically less well-defined and therefore require more frequent calibrations, for some systems even daily.

Systematic errors may arise, for example, from evaporation of calorimeter water, from uncontrolled heat exchange along various paths and/or imperfections and lag in an adiabatic temperature control system during the reaction period. Cancellation of this type of error depends largely on the similarity between the calibration experiments and combustion of the test portions with respect to time-temperature profile and total change in temperature of the calorimeter. Systematic variation in the mass of benzoic acid used in the calibration experiments is an expedient way of establishing the requirements for "similarity" for a particular calorimetric system (see 9.3).

9.2 Calibrant

9.2.1 Certification conditions

The certificate value for the energy of combustion of benzoic acid refers to a process where the mass of sample and initial water, respectively, is 3 g/l of free bomb volume, the initial pressure of oxygen is 3,0 MPa and the reference temperature is 25 °C. The products of combustion are defined as gaseous carbon dioxide, liquid water and an equilibrium amount of carbon dioxide dissolved in the aqueous phase. Any nitric acid formed is corrected for by the energy for the process, where the acid is decomposed to form liquid water and gaseous nitrogen and oxygen. When calibrations are performed under different conditions, the certificate value shall be adjusted. A numerical expression to correct for such deviations is given in the certificate.

9.2.2 Calibration conditions

The calibration conditions determine the overall calorimetric conditions for the subsequent fuel determinations. For bombs with an internal volume of about 300 ml, 1 g of calibrant and 1 ml of water initially in the bomb are normally used (see 8.2.1). For bombs with a volume nearer to 200 ml, 0,6 g of benzoic acid is preferable; the amount of water should then be reduced accordingly (both in calibration and in determinations).

NOTE 1 The correction terms (per g of benzoic acid) for deviations from certificate conditions, quoted from a typical benzoic acid certificate, are for the initial pressure, mass of test portion, mass of water and reference temperature of the experiment, respectively 5 J/MPa, 1,1 J/g·l^{-1}, 0,8 J/g·l^{-1} and −1,2 J/K.

NOTE 2 As long as the initial pressure of oxygen and the reference temperature are kept within (3,0 ± 0,3) MPa and (25 ± 2) °C, respectively, the departure from certification conditions caused by pressure and/or temperature deviations is within ±3 J/g and need not be accounted for.

NOTE 3 The most significant deviation from the certification conditions is usually caused if larger amounts of water, e.g. 5 ml/g calibrant, are used. For a 300 ml bomb, this causes an increase in the certified value of 11 J/g. If 1,0 g of benzoic acid and 5,0 ml of water are used in a 200 ml bomb, the certified value increases by 20 J/g. The change is mostly caused by an increase in the fraction of carbon dioxide dissolved in the bomb liquid. If no water is used, the certified value decreases by 2 J/g.

NOTE 4 When the total heat capacity of the calorimeter is small, for example, in aneroid systems, the sample mass may have to be reduced in order to limit the total change in temperature (see Annex C).

9.3 Valid working range of the effective heat capacity ε

It ought to be possible to vary the amount of calibrant at least ±25 % without getting a significant trend in the values obtained for the effective heat capacity. If this is not the case, the working limits for a constant value of ε shall be defined in terms of total temperature rise measured. All subsequent measurements of calorific value shall be kept within these temperature rise limits by adjusting the sample weight of biofuel to be measured.

A plot of ε values, as a function of mass of calibrant used, reveals whether there is a significant trend in the effective heat capacity for a particular calorimeter. In this test, the calibrant mass should be varied from 0,7 g to 1,3 g, or an equivalent relative amount, and a minimum of eight experiments should be performed. There is no need to vary the initial amount of water in the bomb.

A convenient way of checking a system already calibrated by combustion of, for example, 1,0 g samples, is to use the benzoic acid as an unknown. The mean values from triplicate runs on 0,7 g and 1,3 g sample masses, respectively, are compared with the certificate values. This normally suffices to ascertain whether the effective heat capacity is constant for the range of heat produced. Deviations are generally expected to be in the direction of “low” calorific values for larger sample masses, equivalent to obtaining ε values on the high side when derived from large samples. Using benzoic acid as a test substance is particularly useful in checking the performance of highly automated systems.

The required range for a verified (validated) value of ε depends on the total variation in calorific value of the fuels normally analysed. A moderate trend in ε, e.g. ±0,3 % for a ±30 % variation in the observed temperature rise, may be compensated for by expressing the effective heat capacity ε as a function of $(t_\mathrm{f} - t_\mathrm{i})$ over some defined range. Similarly, if a non-linearized temperature sensor is used, ε may be expressed as a (linear) function of $(t_\mathrm{f} - t_\mathrm{i})$, provided stringent criteria are also established for how much t_f or t_i is allowed to vary.

Deviation of ε from a constant value, as discussed here, is caused by the physical design of the calorimeter and/or shortcomings in the temperature control of the instrument. For a particular set-up, examination of the applicable range of ε from a given set of calibration conditions should be carried out when the instrument is new or has been subjected to major repair or moved to a different location, and when changes in the temperature control system have occurred. Some adiabatic systems need to be checked on a more regular basis (see Annex A). Some automated calorimeters require calibration with a prescribed variation in sample mass (see Annex C).

9.4 Ancillary contributions

In addition to the energy from combustion of benzoic acid, there are contributions from combustion of the fuse(s) and the formation of nitric acid (from “air” nitrogen in the gaseous phase). The contribution from a fuse is derived from the amount involved and the appropriate energy of combustion. Any unreacted fuse wire has to be taken into account, i.e. subtracted from the initial amount.

The amount of nitric acid formed is determined on the final bomb solution, for example, by acid-base titration (see 8.5).

In most systems, the contribution from the fuse(s) can be kept nearly the same in all experiments (fuel and calibration) and can consequently be assigned a constant value. For a given bomb configuration, the amount of nitric acid formed in calibration experiments is approximately proportional to the amount of benzoic acid burned.

9.5 Calibration procedure

For the ordinary series of calibrations, five satisfactory combustions on benzoic acid shall be carried out. The sample shall be burned as pellets (see 5.5). The calorimetric procedure described in Clause 8 shall be followed. Recommendations concerning sample mass and initial amount of bomb water are given in 9.2.2. It is advantageous to use a crucible of low mass for the benzoic acid combustions. The initial temperature shall be chosen such that the reference temperature of the experiment (defined as t_f; see 8.7) is within the chosen range for the reference temperature.

The design of the calibration experiment, in terms of oxygen pressure, amount of bomb water, reference temperature, duration of the fore, main and after periods, etc., defines the detailed procedure for subsequent fuel combustions.

When the effective heat capacity ε of a calorimeter cannot be regarded as constant over the required working range but needs to be expressed as a function of $(t_\mathrm{f} - t_\mathrm{i})$ (see 9.3), the number of calibration experiments shall be increased to eight or more. The mass of sample for the individual experiments is chosen to yield values for the change in temperature over the entire intended working range, with a few replicate measurements around the end points, to define the slope of the ε versus $(t_\mathrm{f} - t_\mathrm{i})$ relationship.

9.6 Calculation of effective heat capacity for the individual experiment

9.6.1 Constant mass-of-calorimeter-water basis

For systems where the quantity of water in the calorimeter vessel is kept the same in all experiments, ε is calculated using Formula (6):

$$\varepsilon = \frac{m_{\text{ba}} \times q_{V,\text{ba}} + Q_{\text{fuse}} + Q_{\text{ign}} + Q_{\text{N}}}{\theta} \tag{6}$$

where

m_{ba} is the mass, in g, of benzoic acid (5.5);

$q_{V,\text{ba}}$ is the certified gross calorific value at constant volume, in J/g, for the benzoic acid (see 9.2.1);

Q_{fuse} is the contribution from combustion of the fuse, in J;

Q_{ign} is the contribution from oxidation of the ignition wire, in J;

Q_{N} is the contribution from formation of nitric acid (from liquid water and gaseous nitrogen and oxygen; see 8.5 and 9.2.1), in J;

θ is the corrected temperature rise, in K or in an arbitrary unit (see 3.6 and 8.6).

NOTE ε is normally expressed in J/K. When θ is expressed in arbitrary units, ε, will, of course, be expressed in J per this arbitrary unit, e.g. J/Ω.

The contribution from combustion of a cotton fuse is 17 500 J/g and from a nickel-chromium wire 6 000 J/g. Platinum wire melts and resolidifies and gives no net contribution.

When the sum of $Q_{\text{fuse}} + Q_{\text{ign}}$ is nearly the same, within a few joules, in all experiments, it can be assigned a constant value. It is not generally recommended to incorporate $Q_{\text{fuse}} + Q_{\text{ign}}$ in the value of ε, unless it is in itself small and the variation in θ is less than ±20 %.

For the formation of nitric acid from liquid water and gaseous nitrogen and oxygen, the contribution is 60 J/mmol.

Q_{N}, in J, is calculated from the found nitrate content $w(\text{NO}_3)$, in mg, determined by ion-chromatography using Formula (7):

$$Q_{\text{N}} = 0{,}97 \times w(\text{NO}_3) \tag{7}$$

or

from the titration result of [c(NaOH) = 0,1 mol/l], in ml, using formula $Q_{\text{N}} = 6{,}0 \times \text{ml}$ (see 8.5).

9.6.2 Constant total-calorimeter-mass basis

When the system is operated such that the calorimeter can with the assembled bomb and the water always has the same total mass, the amount of water in the can will vary slightly, depending chiefly upon the mass of the crucible used. It is then convenient to define ε_{o} as the effective heat capacity for the hypothetical calorimeter with no crucible in the bomb, as given by Formula (8):

$$\varepsilon_{\text{o}} = \varepsilon_{\bullet} + m_{\text{cr}} \times c_{p,\text{aq}} \tag{8}$$

where

$\varepsilon_{\bullet}$ is equal to ε as defined in 9.6.1;

m_{cr} is the mass, in g, of the crucible used in the calibration experiment (see Note hereafter);

$c_{p,\text{aq}}$ is the specific heat capacity of water, in J/(g·K), when the ε values are in J/K. At 25 °C, it is equal to 4,18 J/(g·K).

When arbitrary “units of temperature” are used, the value of $c_{p,\text{aq}}$ shall be adjusted accordingly. The relation between the kelvin and the unit utilized need only be known to within ±10 % for this purpose.

NOTE In the formula for ε_{o}, the second term is derived from:

$$m_{\text{cr}} \times \left(c_{p,\text{aq}} - c_{p,\text{cr}}\right) + m_{\text{sample}} \times \left(c_{p,\text{aq}} - c_{p,\text{sample}}\right)$$

The second part of this expression may be incorporated in ε_{o} without loss in accuracy, as its value will not vary significantly between calibration and fuel experiments. The expression is then reduced to Formula (9):

$$m_{\text{cr}} \times \left(c_{p,\text{aq}} - c_{p,\text{cr}}\right) \tag{9}$$

which, in most cases, may be simplified to $m_{\text{cr}} \times c_{p,\text{aq}}$ as given in the formula for ε_{o}. However, when a wide variety of crucibles is used, the heat capacity of the crucible may have to be accounted for. For instance, if a 10 g platinum crucible is used for the calibration experiments and a 10 g quartz crucible is used for the fuel combustions, an error of 6 J/K is introduced if $c_{p,\text{cr}}$ is not taken into account, corresponding to 18 J for a 3 K temperature rise. The correct formula is then shown as Formula (10):

$$\varepsilon_{\text{o}} = \varepsilon_{\bullet} + m_{\text{cr}} \times \left(c_{p,\text{aq}} - c_{p,\text{cr}}\right) \tag{10}$$

The specific heat capacities for platinum, quartz and steel are 0,133 J/(g·K), 0,74 J/(g·K) and 0,45 J/(g·K), respectively.

9.7 Precision of the mean value of the effective heat capacity ε

9.7.1 Constant value of ε

Calculate the arithmetic mean $\varepsilon_{(\text{n})}$ and the standard deviation from the results of the individual calibration experiments, ε (see 9.6.1) or ε_{o} (see 9.6.2), respectively. The standard deviation shall not exceed 0,20 %. All results from the current series of calibrations shall be included in the calculations; only experiments with evidence of incomplete combustion may, and shall, be discarded.

Provided the precision requirement is met, the arithmetic mean $\varepsilon_{(\text{n})}$ or $\varepsilon_{\text{o}(\text{n})}$, respectively, is regarded as the value for the effective heat capacity of the calorimeter.

If the precision requirement is not met, the cause for the unsatisfactory results shall be identified and corrected, and a new series of calibration experiments shall be performed.

9.7.2 ε as a function of the observed temperature rise

When ε cannot be regarded as constant, list the individual values of ε (see 9.6.1) or ε_0 (see 9.6.2), respectively, together with the corresponding values for the observed temperature rise $(t_f - t_i)$, for clarity denoted Δt. Fit the results to a straight line by linear regression with Δt as the independent variable. In addition to calculating the coefficients a and b as shown in Formula (11):

$$\hat{\varepsilon} = a + b \times \Delta t \tag{11}$$

the estimate of the variance s^2 about the line shall be calculated. For convenience, θ may be used instead of Δt.

The standard deviation s shall not exceed 0,20 %. Only results from experiments with evidence of incomplete combustion may, and shall, be discarded in the calculations.

Provided the precision requirement is met, $\hat{\varepsilon}$, as defined above, is regarded as the value for the effective heat capacity of the calorimeter to be used in the calculations of the calorific values for the fuels. The valid working range in terms of the observed temperature rise shall be clearly specified.

If the precision requirement is not met, the cause for the unsatisfactory results shall be identified and corrected, and a new series of calibration experiments shall be performed.

9.8 Redetermination of the effective heat capacity

When any significant part of the system is changed, the mean effective heat capacity shall be re-determined (see 9.3). It shall also be re-determined at intervals not longer than 6 months.

NOTE It is recommended, especially on a new system, to check the calibration regularly by performing a few monthly experiments using benzoic acid as a test substance (see 9.3).

Where a change to the system is not involved, the new mean value of ε shall be within 0,25 % of the previous value. If the difference is greater than 0,25 %, experimental procedures shall be examined and the cause of the problem identified and dealt with.

10 Gross calorific value

10.1 General

The calorimetric conditions for the biofuel combustions shall be consistent with those of the calibration experiments (see 8.2.1, 9.2.2 and 9.5). With the calorimetric procedure under satisfactory control, ascertaining complete combustion of the biofuel is the most important issue.

Some biofuels might be difficult to burn completely in the bomb and may have to be burned with a combustion aid or by using a combustion bag or capsule, with well-established energy of combustion, or by omitting the distilled water in the bomb (see Clause 7 and 8.2) or by reducing the oxygen pressure.

The bomb washings for sulphuric and nitric acid shall be analysed using procedures described in 8.5 or alternatively, a constant value for nitric acid (see below) together with a determined, typical or default value for sulfur shall be used for corrections (see 10.3.2 and E.4).

Nitric acid formation is largely dependent on the combustion temperature and enhanced by the nitrogen content of the sample. The nitric acid correction is normally different for fuel and benzoic acid combustions, respectively, and may also vary significantly for different types of biofuels.

The variation in the correction for nitric acid might be due to biofuels having low nitrogen content on the borderline of significance. When the sulfur content is determined separately on the sample, the nitric acid correction may be assigned a constant per-gram-of-sample value. A similar strategy shall then be adopted for the calibration experiments.

10.2 Combustion

Duplicate combustions shall be made. A representative sample shall be taken from the analysis sample (see Clause 7) and a pellet shall be pressed or capsule shall be used. The mass of the sample burned (or the sample together with combustion aid or capsule) shall be such that the observed temperature rise is within the range of the calibration experiments. The calorimetric procedure described in 8.2.1 to 8.6 shall be followed, with the same calorimetric conditions as in the calibration experiments (see 9.2.2).

Usually, a mass of around 1 g of biofuel sample is adequate. If the observed temperature rise falls outside the valid range for ε, the sample size of the biofuel shall be adjusted to meet the valid temperature rise range or the calibration shall be confirmed for the extended range of ε (see 9.3).

10.3 Calculation of gross calorific value

10.3.1 General

The energy change for the total bomb process is given by the effective heat capacity ε multiplied by the corrected temperature rise θ. To derive the energy of combustion of the biofuel sample, the energy contributions from all the ancillary reactions shall be subtracted from $\varepsilon \times \theta$ (see 9.4). When a combustion aid is used, its contribution is usually the largest ancillary quantity and shall be accurately accounted for.

Moreover, sulphur in the sample quantitatively yields sulphuric acid in the bomb, whereas the required state of sulphur for the calorific value of the fuel is gaseous sulphur dioxide (see 4.1). This is accounted for by a term representing the decomposition, at constant volume, of the aqueous sulphuric acid into gaseous sulphur dioxide and oxygen, and liquid water.

The derived calorific value for the biofuel is the gross calorific value at constant volume.

10.3.2 Constant mass-of-calorimeter-water basis

Calculate the gross calorific value at constant volume from the individual experiment by substituting into Formula (12):

$$q_{V,\mathrm{gr}} = \frac{\varepsilon_{(\mathrm{n})} \times \theta - Q_{\mathrm{fuse}} - Q_{\mathrm{ign}} - Q_{\mathrm{N}} - m_2 \times q_{V,2}}{m_1} - \frac{Q_{\mathrm{S}}}{m_1} \tag{12}$$

where

$q_{V,\mathrm{gr}}$	is the gross calorific value at constant volume of the fuel as analysed, in J/g;
$\varepsilon_{(\mathrm{n})}$	is the mean value of the effective heat capacity of the calorimeter as determined in the calibrations (see 9.6.1), in J/K, or, alternatively, in J per some arbitrary unit (see Note in 9.6.1);
Q_{S}	is the correction for taking the sulfur from the aqueous sulphuric acid to gaseous sulfur dioxide, in J;
m_1	is the mass, in g, of the sample of the fuel;

m_2	is the mass, in g, of the combustion aid (if relevant);
$q_{V,2}$	is the gross calorific value at constant volume of the combustion aid (if relevant), in J/g;
θ, Q_{fuse}, Q_{ign} and Q_{N}	are defined in 9.6.1.

NOTE When no buoyancy correction is applied to m_2, care should be taken to ensure that $q_{V,2}$ is valid for "per gram weighed in air".

The energy quantities required to calculate the contributions from fuse, ignition wire and formation of nitric acid are given in 9.6.1. Specific heat capacities for water and some common crucible materials are given in 9.6.2.

To account for the reaction where sulphuric acid decomposes into liquid water and gaseous sulphur dioxide and oxygen, the correction is 302 J/mmol, equivalent to 9,41 J/mg of sulphur, which in turn corresponds to a Q_{S} / m_1 value of 94,1 J/g of sample for 1 % of sulphur in the analysis sample, as given in Formula (13).

$$Q_{\mathrm{S}} = 94{,}1 \times w(\mathrm{S}) \times m_1 \tag{13}$$

where

$w(\mathrm{S})$ is the sulfur content of the sample (as analysed), as a percentage.

The sulfur content of the sample shall be determined or alternatively, a typical or default value may be used (see E.4 and Annex G). If the sulfur content is not determined, the value used shall be stated in the test report.

When the analytical procedures described in 8.5 are used, the contributions from sulphuric and nitric acids are given by Formulae (14) to (19):

a) determination by ion-chromatography

$$Q_{\mathrm{S}} = 3{,}14 \times w(\mathrm{SO}_4) \tag{14}$$

$$Q_{\mathrm{N}} = 0{,}97 \times w(\mathrm{NO}_3) \tag{15}$$

where

$w(\mathrm{SO}_4)$ is the sulfate content and $w(\mathrm{NO}_3)$ the nitrate content found, in mg, respectively;

b) titration with barium hydroxide and hydrochloric acid

$$Q_{\mathrm{S}} = 15{,}1(V_1 + V_2 - 20{,}0)\text{, and} \tag{16}$$

$$Q_{\mathrm{N}} = 6{,}0(20{,}0 - V_2)\text{, respectively,} \tag{17}$$

where

V_1 is the volume used, in ml, of the barium hydroxide solution (5.4.1);

V_2 is the volume used, in ml, of the hydrochloric acid solution (5.4.4);

c) titration with sodium hydroxide.

The corrections for nitric acid and sulphuric acid are calculated using a combined correction $Q_{\mathrm{N,S}}$ instead of Q_{N} together with an additional correction for sulphur ($Q_{\mathrm{S,\,add.}}$) in the formula of $q_{V,\mathrm{gr}}$ (one part of Q_{S}, namely 2 × 60 J/mmol is included in $Q_{\mathrm{N,S}}$; the additional correction needed is $302 - 2 \times 60 = 182$ J/mmol which is equal to 5,7 J/mg sulphur in the sample).

$$Q_{\mathrm{N,S}} = 6{,}0 \times V \tag{18}$$

$$Q_{\mathrm{S,\,add.}} = 57 \times w(\mathrm{S}) \times m_1 \tag{19}$$

where

V is the volume used, in ml, of sodium hydroxide solution (5.4.3);

$w(\mathrm{S})$ is the sulfur content of the sample (as analysed), as a percentage.

The certification-condition value may be used for benzoic acid utilized as a combustion aid, provided 1 ml of water is used initially in the bomb. For larger amounts of water, it is recommended to adjust the per-gram value in accordance with the amount-of-water term in the certificate.

The mean value of duplicate determinations is regarded as the gross calorific value for the general analysis sample of the biofuel.

10.3.3 Constant total-calorimeter-mass basis

In this case, the mean value of the effective heat capacity $\varepsilon_{\mathrm{o(n)}}$ is the one derived from the individual ε_{o} results (see 9.6.2) and represents the calorimeter without a crucible. The value of ε valid for the actual fuel experiment is shown in Formula (20):

$$\varepsilon_{\bullet} = \varepsilon_{\mathrm{o(n)}} - m_{\mathrm{cr}} \times c_{p,\mathrm{aq}} \tag{20}$$

where

m_{cr} is the mass, in g, of the crucible used in the fuel combustion;

the other symbols are defined in 9.6.2.

If the heat capacity of the crucible has been taken into account (see Note to 9.6.2) in computing the ε_{o} values, $\left(c_{p,\mathrm{aq}} - c_{p,\mathrm{cr}}\right)$ should be substituted for $c_{p,\mathrm{aq}}$ in the calculations of $\varepsilon_{\bullet}$.

$\varepsilon_{\bullet}$ replaces $\varepsilon_{\mathrm{(n)}}$ in the formula given in 10.3.2 for the calculation of the gross calorific value at constant volume for the biofuel test portion from an individual experiment. The mean value of duplicate determinations is regarded as the resulting value for the general analysis sample of the biofuel.

10.3.4 ε as a function of the observed temperature rise

When it is required that the effective heat capacity of the calorimeter is expressed as a function of the observed temperature rise (see 9.3 and 9.7.2), $\varepsilon_{(\mathrm{n})}$ in the formula in 10.3.2 and $\varepsilon_{\mathrm{o(n)}}$ in the formula in 10.3.3, respectively, shall be replaced by Formula (21):

$$\hat{\varepsilon} = a + b \times \Delta t \tag{21}$$

where the coefficients a and b are derived from the calibrations (see 9.7.2).

Δt is the notation for the observed temperature rise $(t_{\mathrm{f}} - t_{\mathrm{i}})$, in K or the arbitrary unit used, for the actual fuel experiment. For convenience, θ may be used instead of Δt (see 9.7.2).

The mean value of duplicate determinations is regarded as the resulting value for the general analysis sample of the biofuel.

10.4 Expression of results

As the moisture content of the actual analysis sample is of interest merely in connection with the calculation to other bases, it is recommended to calculate a value for the gross calorific value at constant volume for the dry fuel (dry basis, in dry matter), using Formula (22):

$$q_{V,\mathrm{gr,d}} = q_{V,\mathrm{gr}} \times \frac{100}{100 - M_{\mathrm{ad}}} \tag{22}$$

where

$q_{V,\mathrm{gr,d}}$ is the gross calorific value at constant volume of the dry (moisture-free) fuel, in J/g;

M_{ad} is the moisture content in the analysis sample, in percentage by mass;

$q_{V,\mathrm{gr}}$ is defined in 10.3.2.

The calorific value at constant volume required for any particular moisture content basis $q_{V,\mathrm{gr,m}}$ is derived from Formula (23):

$$q_{V,\mathrm{gr,m}} = q_{V,\mathrm{gr,d}} \times (1 - 0{,}01M) \tag{23}$$

where M is the moisture content, in percentage by mass, for which the calorific value is required, normally for the fuel as sampled or as fired (total moisture content as received, M_{ar}), and Formula (24):

$$(1 - 0{,}01M_{\mathrm{ar}}) = \frac{100 - M_{\mathrm{ar}}}{100} \tag{24}$$

The result shall be reported to the nearest multiple of 10 J/g with unambiguous statements concerning the states constant volume, gross (liquid water), and moisture basis (dry, and also “as sampled” if it is reasonable).

Determination of the moisture content (M or M_{ar}) shall be carried out by the method specified in ISO 18134-1 or ISO 18134-2.

10.5 Calculation to other bases

For the calculation of results to other bases, refer to ISO 16993.

11 Performance characteristics

11.1 Repeatability limit

The results of duplicate determinations, carried out in the same laboratory by the same operator with the same apparatus within a short interval of time on the same analysis sample, shall not differ by more than 120 J/g for wood pellets and saw dust. For all other solid biofuels, the repeatability limit is 140 J/g.

11.2 Reproducibility limit

The means of the results of duplicate determinations carried out in each of two laboratories, on representative portions taken from the same sample at the last stage of sample preparation, shall not differ by more than 300 J/g for wood pellets and saw dust. For all other solid biofuels, the reproducibility limit is 400 J/g.

12 Calculation of net calorific value at constant pressure

12.1 General

The main difference between the **gross** and **net** calorific values is related to the physical state of water in the reaction products (compare definitions 3.1 to 3.3). The calorific value of the fuel most commonly used for practical purposes is the net calorific value at **constant pressure** for the fuel with some specified moisture content. This value may be derived from the gross calorific value at **constant volume** for the dry sample, provided that the total hydrogen content of the moisture-free sample can be determined by experiment or, for the particular biofuel, reliably estimated. In addition, the oxygen and nitrogen contents of the moisture-free sample "add" to the gaseous phase of the product system and should, in principle, be taken into account. For this purpose, the nitrogen may be included in the term for oxygen.

NOTE The net calorific value at constant volume (see 3.2) for the biofuel at some specified moisture level is as easily calculated, once a measure of the hydrogen content is available. In this case, the oxygen and nitrogen content is of no consequence.

12.2 Calculations

The net calorific value at constant pressure at required moisture levels may be calculated by the following formulae. For calculations to other bases, see ISO 16993.

Net calorific value at constant pressure for a dry sample (dry basis, in dry matter) is calculated using Formula (25):

$$\begin{aligned} q_{p,\text{net,d}} &= q_{V,\text{gr,d}} + 6{,}15 \times w(\text{H})_\text{d} - 0{,}8 \times \left[w(\text{O})_\text{d} + w(\text{N})_\text{d} \right] - 218{,}3 \times w(\text{H})_\text{d} \qquad (25) \\ &= q_{V,\text{gr,d}} - 212{,}2 \times w(\text{H})_\text{d} - 0{,}8 \times \left[w(\text{O})_\text{d} + w(\text{N})_\text{d} \right] \end{aligned}$$

Net calorific value at constant pressure at a required moisture content M (e.g. as received, M_ar, whereupon the symbol of calorific value is $q_{p,\text{net,ar}}$) is calculated using Formula (26):

$$\begin{aligned} q_{p,\text{net,m}} &= \left\{ q_{V,\text{gr,d}} - 212{,}2 w(\text{H})_\text{d} - 0{,}8 \left[w(\text{O})_\text{d} + w(\text{N})_\text{d} \right] \right\} \times (1 - 0{,}01 M) - 24{,}43 M \qquad (26) \\ &= q_{p,\text{net,d}} \times (1 - 0{,}01 M) - 24{,}43 \times M \end{aligned}$$

where

$q_{p,\text{net,m}}$ is the net calorific value at constant pressure, in J/g, of the biofuel with moisture content M (usually as received M_{ar});

$q_{V,\text{gr,d}}$ is the gross calorific value at constant volume, in J/g, of the moisture-free fuel (see 10.4);

$w(\text{H})_{\text{d}}$ is the hydrogen content, in percentage by mass, of the moisture-free (dry) biofuel (including the hydrogen from the water of hydration of the mineral matter as well as the hydrogen in the biofuel substance);

$w(\text{O})_{\text{d}}$ is the oxygen content, in percentage by mass, of the moisture-free biofuel;

$w(\text{N})_{\text{d}}$ is the nitrogen content, in percentage by mass, of the moisture-free biofuel;

M is the moisture content, in percentage by mass, for which the calculation is required. On the dry basis, $M = 0$; on the air-dried basis, $M = M_{\text{ad}}$ (see 10.4); on the as-sampled or as-fired (as received, ar) basis, $M = M_{\text{ar}}$ (total moisture content as received).

The enthalpy of vaporization (constant pressure) for water at 25 °C is 44,01 kJ/mol. This corresponds to 218,3 J/g for a mass fraction of 1 % of hydrogen in the biofuel sample or 24,43 J/g for a mass fraction of 1 % of moisture, respectively.

$\left[w(\text{O})_{\text{d}} + w(\text{N})_{\text{d}}\right]$ may be derived by subtracting from 100 the percentages of ash, carbon, hydrogen and sulfur.

NOTE The net calorific value at constant volume may be calculated from the following formula:

$$q_{V,\text{net,m}} = [q_{V,\text{gr,d}} - 206w(\text{H})_{\text{d}}] \times (1 - 0,01M) - 23,0M$$

where $q_{V,\text{net,m}}$ is the net calorific value at constant volume, in J/g, of the fuel with a moisture content of M. The other symbols are defined above.

13 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date when the test was undertaken;

b) identification of the biofuel and the sample tested;

c) a reference to this document, i.e. ISO 18125;

d) results with reference to the state(s) valid for the calorific value(s), i.e. with relevant symbols:

    1) gross calorific value (at constant volume) of dry biofuel (dry basis, in dry matter) ($q_{V,\text{gr,d}}$);

    2) net calorific value (at constant pressure) of dry biofuel (dry basis, in dry matter) ($q_{p,\text{net,d}}$);

    3) net calorific value of the moist biofuel (as sampled or as received, ar) ($q_{p,\text{net,ar}}$);

e) values of hydrogen, oxygen, nitrogen, sulphur used to calculate gross calorific value and net calorific value;

f) if typical or default values for hydrogen, oxygen, nitrogen, sulphur are used in the calculations, this shall be clearly stated;

g) any unusual features noted during the determination.

Annex A
(normative)

Adiabatic bomb calorimeters

A.1 Principle

In a truly adiabatic calorimeter, there is no heat exchange between the calorimeter and its surrounding thermostat (water jacket). Heat exchange takes place via common boundaries, the driving force being a net difference in temperature. Ideally, therefore, the whole of the outside surface of the calorimeter can, including the lid, should have a uniform temperature which at all times, during an experiment, is to be matched by the, also uniform, temperature of the inner wall of the thermostat well and lid that are facing the calorimeter. Without any difference in temperature, i.e. with zero thermal heat, there is no net flow of heat between calorimeter and thermostat. However, there will still be a slow rise in calorimeter temperature caused mainly by the stirring power, with additional positive or negative contributions, from thermometer probe self-heating and from conduction of heat along stirrer shaft, ignition leads, thermometers, etc. For convenience, "adiabatic" calorimeters are often operated with a small negative thermal head to balance, i.e. offset, this upward drift in temperature.

A.2 Sources of error for the real calorimeter

Truly adiabatic conditions are difficult to achieve in practice, in particular during the rapid part of the rise in calorimeter temperature upon ignition of the sample. The design of the thermostat and the way it is operated determine how effectively it responds to the change in calorimeter temperature and hence also the extent of uncontrolled heat exchange.

When the calorimeter itself has no lid, its upper heat exchange properties are largely determined by the surface of the calorimeter water together with the surface of, for example, bomb parts extending above the water. With such an "open" calorimeter, there is always some uncontrolled evaporation of calorimeter water during the main period, accompanied by a corresponding "heat loss". The magnitude of this error is mainly a function of how much the thermostat lid lags behind in temperature during the main period.

Unless special precautions have been taken in its design and mounting, a calorimeter lid is usually in poor thermal contact with the calorimeter itself. It then lags behind in temperature and may, for instance, be responsible for uncontrolled heat leakage from the thermostat. It may also prolong the time required for the calorimeter to reach thermal equilibrium or steady state. On the other hand, a lid prevents a net heat loss from evaporation of calorimeter water since this will condense on the inside of the lid, restoring the evaporation energy to the calorimeter. In fact, the condensing water assists the thermal equilibration of the lid with the rest of the calorimeter.

To minimize heat exchange caused by temporary temperature differences that cannot be prevented entirely, it is important to keep the outside surface of the calorimeter and the "inside" of the thermostat clean (polished) and dry. Generally, errors and insufficiencies that differ or vary between calibration and fuel experiments are the ones that in the end affect the accuracy of the final results.

A.3 Adiabatic conditions

A.3.1 Thermostat

When the thermostat is heated by passing an electrical current directly through the thermostat water, care shall be taken to keep the salt concentration (usually Na_2C0_3) at the specified level in order to maintain the heating power about the same in all experiments. A diminishing salt concentration may significantly hamper the heating rate, eventually leading to difficulties in achieving adiabatic conditions during the combustion of the sample.

Inadequate adiabatic control during the first half of the main period is easily overlooked. Irrespective of the mode of heating the thermostat, checks should be made at regular intervals (weekly) to ascertain, for example, that the time it takes for the thermostat to catch up with the rapidly rising calorimeter temperature during combustion does not gradually increase.

A.3.2 Adiabatic control

The controls for achieving adiabatic conditions shall be adjusted as prescribed in the instrument manual. In particular, select that setting of the bridge circuit that results in zero or minimum drift in calorimeter temperature at the final temperature of the experiments (see A.5).

NOTE Non-linear sensors are often used in the temperature control circuits. Unless the two sensors are perfectly matched, it is not possible to obtain zero drift in temperature over the whole of the selected working range. Neither is it then possible to achieve zero thermal head over the whole range with one bridge setting. Imperfectly matched sensors also put restrictions on the acceptable variation in the final temperature of the experiments.

In a well-behaved calorimeter, the adiabatic control settings usually need little or no short-term adjustment. This shall, however, be verified by regularly checking the drift rate at the final temperature, for example, by following the temperature over a 5 min to 10 min period in excess of the normal duration of the experiment. A drift rate of 0,001 K/min or more at the final temperature shall be eliminated by adjustment of the control settings, or corrected for (see 6.2.4 and A.5).

A.4 Initial steady state and length of the main period

The equilibration period serves to let the various components of the assembled calorimeter reach a uniform temperature. Simultaneously, the adiabatic controls work to bring the thermostat to its working temperature, close to that of the calorimeter. Let a few minutes pass after the controls have indicated that the temperature of the thermostat and of the calorimeter are about the same before taking readings of the calorimeter temperature at 1 min intervals.

When three consecutive readings yield the same value, within 0,001 K or better, or when they all change by the same (limited) amount (constant drift rate), the charge may be fired.

NOTE The expected duration of the combined equilibration and fore period for most adiabatic systems is in the order of 8 min to 10 min. However, subjecting any part of the calorimeter to substantially deviating temperatures in between experiments may significantly prolong the time for thermal equilibration of the calorimeter.

Depending on the type of sample, the combustion in the bomb takes from about 10 s to 25 s. The time required for the total amount of heat released to become uniformly distributed, i.e. for all parts of the calorimeter to attain a uniform temperature, is primarily a function of stirring pattern and stirrer efficiency. The main period shall cover this temperature equalization time but there is no merit in making it longer than necessary.

The length of the main period is determined in a series of calibration experiments where readings of temperature are taken at 1 min intervals from the time of firing the charge in each experiment. From these observations, note the time, in min, from the time of firing until the second of three consecutive readings that do not differ by more than 0,001 K is reached. The largest of these specific times from five calibration experiments defines the length of the main period. It shall not exceed 10 min, nor shall the time periods evaluated from the individual experiments differ by more than 2 min.

When normal operation involves a slight drift of the final temperature of the experiment, the requirement of "constant temperature" changes to one of constant drift rate to within 0,001 K/min for three consecutive 1 min intervals.

A.5 Correction for drift at the final temperature

When the adiabatic controls are set to give zero drift at the final temperature, the corrected temperature rise is $\theta = t_\mathrm{f} - t_\mathrm{i}$ (see 8.6.3) where t_i is the calorimeter temperature at the time of firing the charge and t_f is the temperature at the end of the main period.

A limited drift in temperature prior to ignition of the sample need not be accounted for in the calculations. Significant drift at the end of and beyond the main period shall, however, be taken into account. It may be regarded as a constant contribution throughout most of the main period. A reasonable approach is to make a correction commencing 1 min after ignition of the sample. The drift rate should, in principle, be determined for the individual run. But insofar as the final drift rate has been established as constant over extended periods of time for a defined range of final temperature, the correction may be based on such a fixed rate.

NOTE 1 A drift rate of 0,001 K/min unaccounted for would, with a main period of about 10 min, result in an error in 6 of approximately 0,01 K. For ε values of about 10 kJ/K, the resulting error in the calorific value of the fuel would be in the order of 100 J/g. If exactly the same error from the same source is made in the calibrations and in all fuel experiments, it will of course be of no consequence for the final result, at least as long as the variation in θ stays within about ±30 %.

The final drift rate g_f in K/min shall be determined over a time period that is at least half of what the correction is supposed to cover. For a main period of 9 min, this gives a rating period of 4 min.

NOTE 2 When the total temperature change of the calorimeter is expressed in units other than temperature (see 9.6.1), g_f is the corresponding per minute value of that unit.

The corrected temperature rise θ, corrected for drift at the final temperature, is calculated from Formula (A.1):

$$\theta = t_\mathrm{f} - t_\mathrm{i} - g_\mathrm{f} \times (\Delta\tau - 1) \tag{A.1}$$

where

$\Delta\tau$ is the length of the main period, in min;

g_f is calculated from Formula (A.2):

$$g_\mathrm{f} = \frac{t_\mathrm{f+a} - t_\mathrm{f}}{a} \tag{A.2}$$

where $t_\mathrm{f+a}$ is the temperature a min after the end of the main period.

Another way of evaluating g_f is as the slope of a linear regression fitting of time-temperature readings at 1 min intervals from the end of the main period onwards.

A.6 Strategy for checking on bias

For adiabatic combustion calorimeters, the main source for systematic error in the measurement is related to difficulties in maintaining adiabatic conditions during the rapid part of the temperature change in the calorimeter. This is manifested as an upward trend in the values obtained for the effective heat capacity with increasing sample mass. Fast-burning samples, such as paraffin oil, usually aggravate this problem and this type of heat-leak error shall not cancel between calibration and fuel experiments.

In most calorimeters, a check on temperature lag in the thermostat as a function of sample mass and type is readily made. The change in thermostat temperature upon ignition of the sample is measured for about 3 min and plotted as a function of time together with time-temperature values for the calorimeter. For adiabatic calorimeters, readings of calorimeter temperature are, in fact, not required during the first part of the main period for other than diagnostic purposes. For the check on thermostat lag, they are required at a frequency sufficient to outline the features of the time-temperature curve.

No particular calibration of the thermostat thermometer is required, but it shall have a response time comparable to that of the calorimeter thermometer. On the graph plot, the two temperature “scales” are simply made to coincide at the time for ignition of the sample. The two temperatures should, of course, be close at the upper end where the system is approaching thermal equilibrium. The area between the two curves is a measure of potential heat leak, and a significant increase of this area as a function of sample mass, i.e. of θ, or sample type for comparable values of θ, indicates that there is a risk of systematic error in the determinations of calorific value. Special care is then required in restricting the variation in heat evolved per experiment to a safe level and range.

Annex B
(normative)

Isoperibol and static-jacket bomb calorimeters

B.1 Principle

The characteristic feature of isoperibol calorimeters is the isothermal jacket. The temperature of the surrounding thermostat is kept constant throughout the experiment by active control. The thermostat of a static-jacket calorimeter has a thermal capacity such that, even without active control, its temperature remains nearly constant during measurements. In both cases, there is a flow of heat between the calorimeter itself and the thermostat. Calorimeters surrounded by thermally insulating material behave largely as static-jacket calorimeters.

Heat exchange between calorimeter and thermostat takes place via common boundaries, the driving force being the thermal head. Ideally, the whole of the outside surface of the calorimeter can, including the lid, should have a uniform temperature equal to that measured by the temperature sensor in the calorimeter. The temperature of the inner wall of the thermostat well and lid facing the calorimeter should remain constant and uniform throughout the experiment.

To make it possible to evaluate and correct for the actual heat exchange, the calorimeter, as a whole, shall behave in conformity with Newton's law of cooling, i.e. the heat flow between calorimeter and thermostat shall be directly proportional to the actual temperature difference for a sufficiently large range of thermal head. For the calorimeter, this is expressed as:

$$\frac{\mathrm{d}q}{\mathrm{d}\tau} = k(t_\mathrm{j} - t) \qquad \text{(B.1)}$$

where

$\frac{\mathrm{d}q}{\mathrm{d}\tau}$	is the heat flow into the calorimeter;
t_j	is the jacket temperature;
t	is the calorimeter temperature;
$(t_\mathrm{j} - t)$	is the thermal head;
k	is the Newton's law cooling constant.

In Formula (B.1), $\mathrm{d}q$ (heat change) may be replaced by $c_p\mathrm{d}t$ (heat capacity × temperature change). As the heat capacity of the calorimeter ε can be regarded as constant over the temperature range of an experiment, the formula then becomes Formula (B.2):

$$\frac{\mathrm{d}q}{\mathrm{d}\tau} = G(t_\mathrm{j} - t) + P_\mathrm{st} \qquad \text{(B.2)}$$

where

$\frac{dq}{d\tau}$ is the rate of temperature change (drift) in the calorimeter caused by the flow of heat;

G is a constant generally referred to as the specific rate constant;

P_{st} is the power of stirring.

The requirement that the power of stirring shall be constant throughout an experiment (see 8.1) allows $dq / d\tau$ to be expressed as Formula (B.3):

$$dq / d\tau = G(t_\infty - t) \tag{B.3}$$

where

t_∞ is the temperature that the calorimeter would eventually attain if left running for an extended period of time;

G and t_∞ are evaluated from the time-temperature measurements of the rating periods, the fore and the after period, respectively (see Figure 2 in 8.1).

The contribution from heat exchange to the total observed temperature rise in the main period is obtained by integration [see Formula (B.4)]:

$$\Delta t_{ex} = \int_{\tau_i}^{\tau_f} (dt / d\tau) d\tau = \int_{\tau_i}^{\tau_f} G(t_\infty - t) d\tau \tag{B.4}$$

using the time-temperature readings (t, τ) of the main period.

B.2 Sources of error for the real calorimeter

Making the isothermal jacket of an isoperibol calorimeter behave as required in terms of constant and uniform temperature presents no real problem, provided that the thermostat fluid is circulated through the thermostat lid at a reasonable rate.

In a static-jacket calorimeter, the thermostat temperature will change slightly during an experiment, with a somewhat different profile when the calorimeter temperature rises upon firing the charge. The thermal capacity of the thermostat shall be such that for a specific rate (cooling) constant G of 0,002 0 min^{-1}, the rise in temperature of the jacket water is less than 0,16 K from the time of firing the charge to the end of the after period; for a specific rate constant of 0,003 0 min^{-1}, it shall be less than 0,11 K. The drift in temperature of the thermostat is proportional to the thermal head.

A calorimeter lid in poor thermal contact with the main part of the calorimeter lags behind when the temperature changes rapidly in the main period, and may give rise to an unpredictable heat exchange with the thermostat. It may also prolong the time required for the calorimeter to reach thermal equilibrium or steady state. On the other hand, a lid prevents a net heat loss from evaporation of calorimeter water since this will condense on the inside of the lid, restoring the evaporation energy to the calorimeter. In fact, the condensing water assists the thermal equilibration of the lid with the rest of the calorimeter. The choice of thermostat temperature affects the evaporation losses when the calorimeter has no lid.

Variations in heat exchange properties are minimized by keeping the outside surface of the calorimeter and the “inside” of the thermostat clean (polished) and dry. The specific rate constant G then should not vary by more than ±3 % from one experiment to the other. Larger deviations may, for example, be indicative of stirrer malfunction. It should be emphasized that the errors which really affect the accuracy of the final results are those that differ or vary between calibration and fuel experiments.

B.3 Choice of jacket temperature

It is good calorimetric practice to run the thermostat of an isoperibol calorimeter at a temperature that is 0,2 K to 0,4 K higher than the final temperature of the calorimeter. In this way, the calorimeter will be the colder part throughout the experiment, hence minimizing evaporation losses. This is particularly important when the calorimeter has no lid.

The same argument applies to static-jacket calorimeters.

B.4 Rating periods

B.4.1 Initial steady state and fore period

A few minutes should be allowed to let the various components of the assembled calorimeter reach a uniform temperature after turning the stirrer on, before readings of temperature are taken at 1 min intervals. The initial rating period, the fore period, in principle, begins as soon as the calorimeter reaches a steady state in terms of temperature drift rate. For successive 1 min intervals, the temperature increments should then not differ by more than 0,002 K/min or the average difference should not exceed 0,001 K/min. A fore period of 5 min (six readings; five increments) should suffice to establish the value of $(\mathrm{d}t / \mathrm{d}\tau)_\mathrm{i} = g_\mathrm{i}$, the initial drift rate. For an increase in temperature, g_i has a positive value (> 0).

NOTE The calorimeter temperature, as a function of time as a whole, is an exponential going asymptotically to t_∞. However, during rating periods of 10 min or less, the curvature is negligible except in cases of a large thermal head, in excess of 5 K, in combination with a large value for the specific rate constant, greater than 0,005 min^{-1}.

The charge is fired directly upon taking the last reading of temperature in the fore period (see 8.4).

B.4.2 After period and length of the main period

The final rating period (the after period) begins when all parts of the calorimeter have attained a uniform temperature after combustion of the sample, i.e. when the calorimeter has reached a new steady state in terms of temperature drift rate. The time required for the total amount of heat released to become uniformly distributed is primarily a function of stirring pattern and stirrer efficiency. The duration of the main period shall be chosen so that temperature equalization is ensured, but there is no merit in making the main period longer than necessary.

The main period begins at the last reading of temperature in the fore period and ends with the beginning of the after period. The latter is determined in a series of calibration experiments and is taken as the time when, for a subsequent 5 min period, the average deviation of the individual 1 min temperature increments is not more than 0,001 K/min. The mean of the length of time for the main period determined from five calibration experiments, rounded to the nearest minute, defines the length of the main period. It shall not exceed 10 min. Nor shall the time intervals evaluated from the individual experiments differ by more than 2 min.

The length of the main period shall be the same in the calibration and in the fuel experiments. When θ is meant to vary over a wide range, it is advisable to determine the length of the main period at the larger values of θ.

The duration of the after period should be 5 min to 7 min in order to establish the final drift rate g_f well enough for the calculation of the correction for heat exchange Δt_ex. For an increase in temperature with time, g_f has a positive value (> 0).

B.5 Calculation of the corrected temperature rise θ

B.5.1 General

The observed temperature rise, $t_f - t_i$, is the sum of θ, the change in temperature caused by the processes in the combustion bomb, and Δt_{ex}, the contribution from heat exchange with the surrounding thermostat (including the contribution from stirring power). The time-temperature readings taken during the fore, main and after periods contain the information required for the evaluation of Δt_{ex}, and hence θ from Formula (B.5):

$$\theta = t_f - t_i - \Delta t_{ex} \tag{B.5}$$

The rating periods yield, as shown in Formulae (B.6) and (B7):

$$g_i = \left(\frac{dt}{d\tau}\right)_i = G(t_\infty - t_{mi}) \tag{B.6}$$

and

$$g_f = \left(\frac{dt}{d\tau}\right)_f = G(t_\infty - t_{mf}) \tag{B.7}$$

utilized in the calculation of the specific rate constant, as shown in Formula (B.8):

$$G = \frac{g_i - g_f}{t_{mf} - t_{mi}} \tag{B.8}$$

where

g_i is the drift rate in the fore (initial rating) period, in K/min;

g_f is the drift rate in the after (final rating) period, in K/min;

t_{mf} is the mean temperature in the after period, in °C;

t_{mi} is the mean temperature in the fore period, in °C.

Temperature may be expressed in some arbitrary unit throughout (see 9.6.1).

g_i and g_f are preferably evaluated as the slope of a linear least-squares fitting of the time-temperature values of the fore and after periods, respectively. Alternatively, they are taken as the mean values of the 1 min temperature increments in the rating periods.

B.5.2 Regnault-Pfaundler method

For time-temperature readings in the main period all taken at equal time intervals (e.g. 1 min), Δt_{ex} may be expressed as Formula (B.9):

$$\Delta t_{\mathrm{ex}} = G\int_{\tau_{\mathrm{i}}}^{\tau_{\mathrm{f}}} \left(t_{\infty} - t\right)\mathrm{d}\tau = \left[g_{\mathrm{f}} + G(t_{\mathrm{mf}} - t_{\mathrm{m}})\right]\times(\tau_{\mathrm{f}} - \tau_{\mathrm{i}}) \tag{B.9}$$

where t_{m} (the integrated mean temperature) is calculated from Formula (B.10):

$$t_{\mathrm{m}} = \frac{1}{n}\left[\frac{t_0 + t_{\mathrm{n}}}{2} + \sum_{k=1}^{k=n-1} t_{\mathrm{k}}\right] \tag{B.10}$$

where

$t_0 (= t_{\mathrm{i}})$ is the temperature at the beginning of the main period;

$t_1, t_2, \dots, t_{\mathrm{k}}, \dots t_{\mathrm{n}}$ are the successive temperature readings taken during the main period, $t_{\mathrm{n}} (= t_{\mathrm{f}})$ being that at the end;

τ_{i} and τ_{f} are the times at the beginning and end of the main period, respectively.

B.5.3 Dickinson extrapolation method

In the Dickinson extrapolation method, the objective is to find a time τ_{x} such that Formula (B.11) is solved:

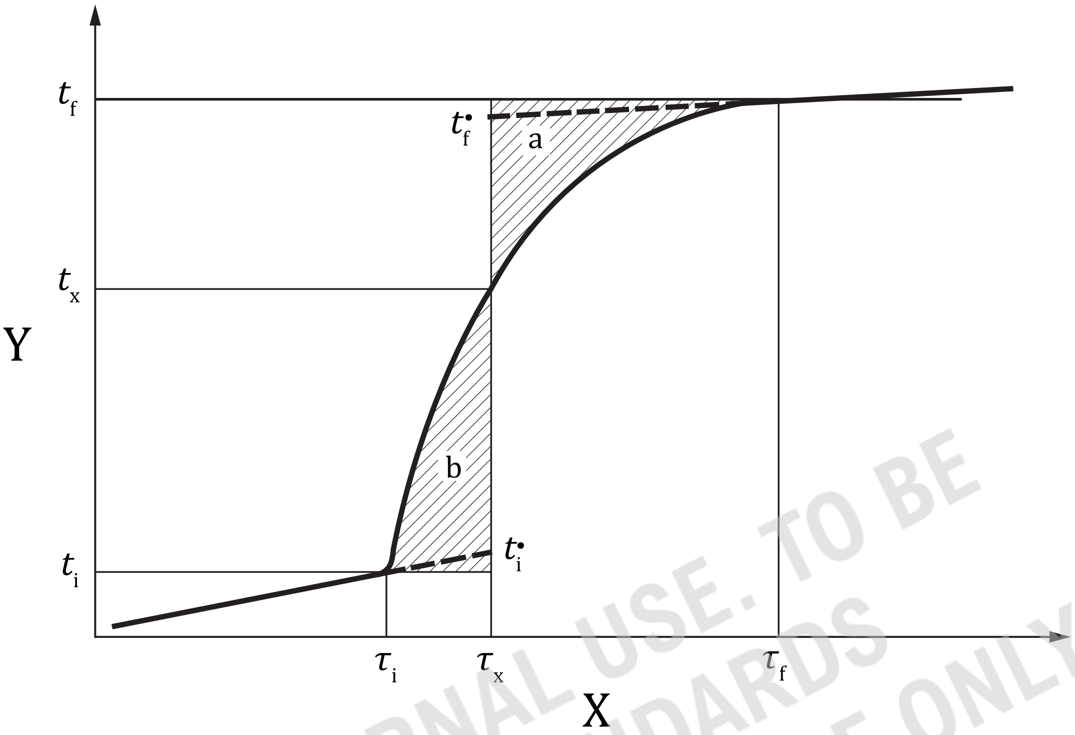
$$g_{\mathrm{i}}(\tau_{\mathrm{x}} - \tau_{\mathrm{i}}) + g_{\mathrm{f}}(\tau_{\mathrm{f}} - \tau_{\mathrm{x}}) = G\int_{\tau_{\mathrm{i}}}^{\tau} \left(t_{\infty} - t\right)\mathrm{d}\tau = \Delta t_{\mathrm{ex}} \tag{B.11}$$

This is accomplished when the hatched areas *a* and *b* in Figure B.1 are of equal size. The corrected temperature rise θ becomes Formula (B.12)

$$\theta = t_{\mathrm{f}} - t_{\mathrm{i}} - g_{\mathrm{i}}(\tau_{\mathrm{x}} - \tau_{\mathrm{i}}) - g_{\mathrm{f}}(\tau_{\mathrm{f}} - \tau_{\mathrm{x}}) = t_{\mathrm{f}}^{\bullet} - t_{\mathrm{i}}^{\bullet} \tag{B.12}$$

where g_{i} and g_{f} represent, in principle, the drift rates at τ_{i} and τ_{f}, respectively.

For a combustion reaction, the time-temperature curve is close to being an exponential which means that τ_{x} is the time associated with the temperature where the change in temperature $\left(t_{\mathrm{x}} - t_{\mathrm{i}}\right)$ is 0,6 times the total (observed) temperature rise $\left(t_{\mathrm{f}} - t_{\mathrm{i}}\right)$. The quantity $\left(\tau_{\mathrm{x}} - \tau_{\mathrm{i}}\right)$ varies with the kinetic behaviour of the combustion reaction of the type of sample studied.



Key

X time, τ

Y temperature, t

Figure B.1 — Dickinson extrapolation

Annex C
(normative)

Automated bomb calorimeters

C.1 Instrument

Among the various types of fully automated bomb combustion calorimeters, there are instruments that fulfil all the basic requirements regarding a physically well-defined calorimeter, as well as instruments whose thermal behaviour has to be described empirically. The former type of instrument usually demands less in terms of comparability, for example, in the amount of heat released in calibrations and in fuel experiments, respectively, in order to yield reliable results. Also, the effective heat capacity ε of a well-defined calorimeter, as a rule, remains constant over long periods of time.

There is no particular reason to assume that instruments with a less well-defined calorimeter could not produce calorific values with the required accuracy, provided that the repeatability is within some set limits and the user is aware of and adheres to restrictions in the choice of operating conditions. Normally, a calorimeter of this kind requires more frequent calibrations, in some cases every day that it is used.

Aneroid calorimeters (see 6.1) are convenient for automated operation, as they require no apportioning of calorimeter water, thereby also eliminating evaporation errors. They are usually operated as adiabatic or quasi-adiabatic systems but may equally well be of the isoperibol type. Characteristically, they have a small heat capacity, leading to large changes in calorimeter temperature, thus facilitating the measurement of θ with a relatively high resolution. Conversely, large values of θ tend to increase the risk for introducing systematic error in aneroid systems aggravated by difficulties in achieving uniform calorimeter surface temperature during combustion of the sample. A countermeasure is to limit the sample mass, bearing in mind that for smaller samples, particular attention shall be given to their being representative.

In certain cases, well-defined stable calorimetric systems allow operation in dynamic mode, i.e. already a few minutes into the main period, it is possible to predict the final outcome of the experiment in terms of θ, without any loss in accuracy of the results.

C.2 Calibration

The effective heat capacity ε shall, in principle, be determined as specified in Clause 9, with particular reference to 9.2, 9.4 and 9.5.

The instrument manufacturer may specify bomb conditions (ratio of sample mass to bomb volume, initial bomb water, oxygen pressure) that deviate significantly from those defined in 9.2.1. When these bomb conditions cause changes in the energy of combustion of the calibrant (benzoic acid) larger than ±5 J/g (see 9.2.2), it shall be possible to adjust any preset value for benzoic acid, i.e. to input the correct value for the calculations of ε.

Recommendations to exclude the initial amount of water in the bomb should primarily be disregarded. The amount, however, may be kept quite small (or even zero) but should be the same both in calibration and in all experiments.

The reference temperature of the experiments (equal to the final temperature t_f of the main period) should be kept the same, within ±1 K, in all experiments. If necessary, it may be chosen arbitrarily within ±10 K from 25 °C without seriously affecting the numerical values of the determinations of calorific value (see 3.4). A deviation in excess of ±5 K from 25 °C should be quoted with the test result.

NOTE Ancillary quantities given in 9.6.1, 9.6.2 and 10.3.2 refer, in principle, to states and reactions at 25 °C.

Some instruments call for calibration using samples differing by about a factor of 2 in mass. Correctly implemented, this offers considerable flexibility for subsequent fuel measurements. Establishing a valid working range for the effective heat capacity ε is always required (see 9.3). When the range is narrow in terms of the amount of heat released, special attention shall be given to performing all experiments within these limits.

For instruments that require frequent calibration, the manufacturer may provide benzoic acid pellets of appropriate mass with an assigned value for the energy of combustion. As a rule, these pellets do not qualify as the calibrant (see 5.5 and 9.2) but are convenient for everyday use. An alternative is to check the calibration by making a series of measurements on a pelletized sample of certified benzoic acid at regular intervals and whenever a new batch of the manufacturer's sample is used. The mean value from a series of five combustions, with the sample mass about the same throughout, shall not differ by more than ±50 J/g from the certified value, recalculated when applicable, to the actual bomb conditions.

Some instruments require preconditioning by combustion of a few samples before yielding stable results. Almost any benzoic acid (pelletized) or combustion aid (see 8.1) may be used for this purpose. The results from these conditioning runs should be disregarded.

Combustion of certified benzoic acid as an "unknown" is generally the most convenient way of checking the performance of a calorimeter (see 9.3).

C.3 Precision requirements for calibrations

The values of ε for the individual calibration experiments should be printed or displayed so that they can be manually recorded (in J/K or in arbitrary units together with θ in these units). Generally, the precision requirements for ε, as given in 9.7, apply.

Some systems compensate for significant drift by using the mean of the previous mean value and the value for ε from the latest calibration experiment as the measure for the effective heat capacity. In such a case, the individual values of ε for a series of calibration experiments cannot be used to evaluate the precision characteristics of the measurements. Instead, a series of individual measurements using certified benzoic acid as the sample shall be performed over a period of 1 day or, at the most, 2 days. For a series of five benzoic acid combustions, the standard deviation shall not exceed 0,20 %. The mean value shall not differ by more than ±50 J/g from the certificate value (see C.2).

C.4 Comparability of calibration and fuel experiments

The conditions specified in 10.1 to 10.3 apply, including arguments about whether thermal contributions from combustion of the fuse and/or side reactions, such as the formation of nitric acid, need to be taken into account in the evaluations (see 9.6.1).

In the calculational procedures of automated instruments, there are normally no provisions to allow specifically for the use of crucibles of widely different material and mass.

In aneroid systems or systems working on a constant mass-of-water basis, the error from disregarding a difference in heat capacity of individual crucibles is as shown in Formula (C.1):

$$\frac{\theta \times \Delta C}{m_1} \tag{C.1}$$

where

ΔC is the difference in heat capacity $\left(m_{\mathrm{cr}} \times c_{p,\mathrm{cr}}\right)$ of the crucible used in the calibrations and that used in combustion of the fuel;

m_1 is the mass of fuel burned.

For calorimeters working on a constant total-calorimeter-mass basis, the error is estimated by using Formula (C.2) (see 9.6.2):

$$\frac{\theta \times \Delta m_{\mathrm{cr}} \times c_{p,\mathrm{aq}}}{m_1} \tag{C.2}$$

Getting clean combustions is the first priority. Optimizing the overall conditions to achieve it is usually worthwhile.

C.5 Documentation and print-out

The evaluation of the gross calorific value at constant volume $q_{V,\mathrm{gr}}$ for the analysis sample shall, in principle, be in accordance with 10.4. The value shall be given in J/g or another convenient unit.

The printed or otherwise recorded information on the individual experiment shall allow the user to verify the calculations starting from values of θ, ε, mass of sample, fuse and any combustion aid. The formulae used should be given in the manual itself or in an annex. Ancillary quantities used in the calculations shall be unambiguously identifiable, and it shall be possible to make the necessary alterations in the program required by changes in procedure, including a change in the numerical value used for the energy of combustion of the calibrant in calibration experiments. Corrections applied for ignition energy, side reactions, etc. shall be clearly stated.

The reference temperature of the experiment shall be identified to the nearest 0,2 K.

C.6 Precision requirements for fuel experiments

The precision requirements in terms of repeatability limit of the results of duplicate measurements are stated in Clause 11.

Annex D
(informative)

Checklists for the design and procedures of combustion experiments

D.1 General

This annex contains checklists intended as aids in setting up and carrying out a complete determination of a calorific value, including calibration of the instrument, using a specified type of calorimeter. Formulae, identical to those given in the main text, are repeated here for clarity.

The general experimental conditions are defined in D.2 which are common to the use of all types of bomb calorimeters. D.3 contains information pertinent to the use of adiabatic calorimeters, D.4 applies to isoperibol calorimeters and D.5 deals with the highly automated bomb-calorimetric systems. Static-jacket calorimeters may be treated as isoperibol systems.

Numbers in parentheses refer to clauses or subclauses in the main text or Annexes A to C. The basic calorimetric procedure is described in Clause 8. The calibration procedures are described in 9.5 and 9.6. The experimental and calculational procedures for the fuel combustions are specified in 10.2 to 10.4. Additional information required for the particular type of calorimeter is given in:

- Annex A and D.3 for adiabatic calorimeters;
- Annex B and D.4 for isoperibol or static-jacket calorimeters;
- Annex C and D.5 for other types of calorimeters.

D.2 Choice of general parameters

Calibration conditions, the basis for the conditions of subsequent fuel experiments (see 9.2.2 and 9.3 for general calibration requirements):

- bomb volume, V_{bomb}, expressed in litres;
- mass of benzoic acid, m_{ba}, expressed in grams;
- mass of bomb water, m_{aq}, expressed in grams; V_{aq}, expressed in millilitres, may be substituted for m_{aq};
- initial pressure of oxygen, p_0, expressed in megapascals;
- reference temperature, t_{ref}, expressed in degrees Celsius.

Calculation of the bomb condition value of benzoic acid:

This value is used in the calculations of the effective heat capacity of the calorimeter, ε. Consult the particular benzoic acid certificate; see also 9.6.1 and 9.6.2.

Certificate value of benzoic acid, expressed in joules per gram (see 9.2.1)

- $m_{\text{ba}}/V_{\text{bomb}}$, expressed in grams per litre, equal to 3,0 g/l;
- $V_{\text{aq}}/V_{\text{bomb}}$, expressed in millilitres per litre, equal to 3,0 m/l;
- p_0, expressed in megapascals, equal to 3,0 MPa;

- t_{ref}, expressed in degrees Celsius, equal to 25 °C; see 8.7;
- adjustment to certificate value, expressed in joules per gram, in accordance with the formula in the certificate.

This yields, $q_{V,\text{ba}}$, expressed in joules per gram.

Amount of calorimeter water (8.1 and 8.3; not relevant for aneroid systems):

The amount of calorimeter water is determined either

a) on a constant mass-of-calorimeter-water basis, i.e. the mass of calorimeter water, expressed in grams; see 8.3, 9.6.1 and 10.3.2; or, alternatively

b) on a constant total-calorimeter-mass basis, i.e. the mass of the (calorimeter + water + assembled bomb), expressed in grams; see 8.3, 9.6.2 and 10.3.3.

Additional parameters to consider:

- ignition wire (fuse), l_{wire}, expressed in centimetres, or a constant Q_{ign}, expressed in joules; see 9.4 and 9.6.1;
- fuse, m_{fuse}, expressed in grams, or a constant Q_{fuse}, expressed in joules; see 9.4 and 9.6.1.

Decide whether the correction for nitric acid Q_N needs to be determined by analysis for the individual experiment or assigned a constant **per-gram** value (not necessarily the same for calibrant and fuel experiments, respectively) or **per-experiment** value (see 9.4 and 10.1).

D.3 Adiabatic calorimeters

D.3.1 Determination of the corrected temperature rise θ

Make the necessary adjustments to achieve adiabatic conditions (see A.3.1 and A.3.2).

Estimate the heat capacity of the system and, from the choice of mass of sample, make a prediction of the expected temperature rise Δt in order to determine the starting temperature $(t_{\text{ref}} - \Delta t)$.

Determine what the conditions are for an initial steady state (see A.4).

Make a series of experiments to determine the length of the main period (see A.4, 8.2 to 8.5 and 9.5).

From the time-temperature measurements (τ_k, t_k) for a set of benzoic acid combustions, calculate the corrected temperature rise θ for the individual experiments (see A.5) using Formula (D.1):

$$\theta = t_f - t_i \tag{D.1}$$

For a significant (but limited) drift at the end of the main period, θ is derived using Formula (D.2) (see A.5):

$$\theta = t_f - t_i - g_f \times (\Delta\tau - 1) \tag{D.2}$$

D.3.2 Evaluation of the effective heat capacity ε

Calculate the effective heat capacity ε for the individual experiments.

For alternative D.2 a), the constant mass-of-calorimeter-water basis (9.6.1), ε is calculated using Formula (D.3):

$$\varepsilon = \frac{m_{\mathrm{ba}} \times q_{V,\mathrm{ba}} + Q_{\mathrm{fuse}} + Q_{\mathrm{ign}} + Q_{\mathrm{N}}}{\theta} \tag{D.3}$$

For alternative D.2 b), the constant total-calorimeter-mass basis (9.6.2), ε_{o} is calculated using Formula (D.4):

$$\varepsilon_{\mathrm{o}} = \varepsilon_{\bullet} + m_{\mathrm{cr}} \times c_{p,\mathrm{aq}} \tag{D.4}$$

where

$\varepsilon_{\bullet}$ is equal to ε as defined above;

m_{cr} is the mass of the crucible used in the individual calibration experiment.

Compare Note in 9.6.2.

Calculate the mean value $\varepsilon_{(\mathrm{n})}$ or $\varepsilon_{\mathrm{o(n)}}$, respectively, and make sure that the precision requirements are met (9.7).

The system is now calibrated and the main calorimetric parameters set for subsequent combustion measurements on fuel samples.

Ancillary quantities required in the calculations are given in 9.6.1.

D.3.3 The gross calorific value at constant volume $q_{V,\mathrm{gr}}$

Perform the fuel combustions in accordance with the instructions in 10.2 and 10.3. θ is calculated in the same way as for the calibrations.

For alternative D.2 a), a calorimeter operated on the constant mass-of-calorimeter-water basis, calculate the calorific value (see 10.3.2) using Formula (D.5):

$$q_{V,\mathrm{gr}} = \frac{\varepsilon_{(\mathrm{n})} \times \theta - Q_{\mathrm{fuse}} - Q_{\mathrm{ign}} - Q_{\mathrm{N}} - m_2 \times q_{V,2}}{m_1} - \frac{Q_{\mathrm{S}}}{m_1} \tag{D.5}$$

For alternative D.2 b), a calorimeter operated on the constant total-calorimeter-mass basis, calculate the calorific value (see 10.3.3) using Formula (D.6):

$$q_{V,\mathrm{gr}} = \frac{\varepsilon_{\bullet} \times \theta - Q_{\mathrm{fuse}} - Q_{\mathrm{ign}} - Q_{\mathrm{N}} - m_2 \times q_{V,2}}{m_1} - \frac{Q_{\mathrm{S}}}{m_1} \tag{D.6}$$

where

$\varepsilon_{\bullet}$ is derived from $\varepsilon_{\bullet} = \varepsilon_{\mathrm{o(n)}} - m_{\mathrm{cr}} \times c_{p,\mathrm{aq}}$;

m_{cr} is the mass, in g, of the crucible in the individual experiment.

Always use the crucible best suited for the particular sample under investigation.

Ancillary quantities required in the calculations are given in 9.6.1 and 10.3.2.

D.4 Isoperibol calorimeters

D.4.1 Determination of the corrected temperature rise θ

Set the jacket temperature to the value chosen for the experiments (see B.3).

Estimate the heat capacity of the system and, from the choice of mass of sample, make a prediction of the expected temperature rise Δt in order to determine the starting temperature ($t_{\mathrm{ref}} - \Delta t$).

Investigate what the conditions are for an initial steady state and decide upon the length of the fore or initial rating period (B.4.1).

Make a series of experiments to determine the length of the main period (see B.4.2, 8.2 to 8.5 and 9.5).

From the time-temperature measurements ($\tau_{\mathrm{k}}, t_{\mathrm{k}}$) for a set of benzoic acid combustions, calculate the corrected temperature rise θ for the individual experiments, utilizing either the Regnault-Pfaundler or the Dickinson method.

Regnault-Pfaundler method (see B.5.1 and B.5.2)

Determine the drift rates g_{i} and g_{f} and the mean temperatures t_{mi} and t_{mf} of the rating periods and calculate the specific rate constant G according to Formula (D.7):

$$G = \frac{g_{\mathrm{i}} - g_{\mathrm{f}}}{t_{\mathrm{mf}} - t_{\mathrm{mi}}} \tag{D.7}$$

Then calculate t_{m} (the integrated mean temperature) and Δt_{ex} (the contribution from heat exchange) according to Formulae (D.8) and (D.9):

$$t_{\mathrm{m}} = \frac{1}{n}\left[\frac{t_0 + t_{\mathrm{n}}}{2} + \sum_{k=1}^{k=n-1} t_{\mathrm{k}}\right] \tag{D.8}$$

and

$$\Delta t_{\mathrm{ex}} = G\int_{\tau_{\mathrm{i}}}^{\tau_{\mathrm{f}}} (t_{\infty} - t)\mathrm{d}\tau = \left[g_{\mathrm{f}} + G(t_{\mathrm{mf}} - t_{\mathrm{m}})\right] \times (\tau_{\mathrm{f}} - \tau_{\mathrm{i}}) \tag{D.9}$$

Finally, calculate θ using Formula (D.10):

$$\theta = t_{\mathrm{f}} - t_{\mathrm{i}} - \Delta t_{\mathrm{ex}} \tag{D.10}$$

Dickinson extrapolation method (see B.5.1 and B.5.3)

Make a graph of the time-temperature (τ_k, t_k) values of the main period and determine the time for $t_i + 0{,}6 \times (t_f - t_i)$. This time is taken as τ_x. Determine the drift rates, i.e. the slopes of the rating periods, using Formulae (D.11) and (D.12):

$$g_i = (\mathrm{d}t / \mathrm{d}\tau)_i \tag{D.11}$$

and

$$g_f = (\mathrm{d}t / \mathrm{d}\tau)_f \tag{D.12}$$

Then calculate θ using Formula (D.13):

$$\theta = t_f - t_i - g_i(\tau_x - \tau_i) - g_f(\tau_f - \tau_x) \tag{D.13}$$

NOTE The extrapolation time τ_x for the fuel experiments is likely to differ from that for the calibrations.

D.4.2 Evaluation of the effective heat capacity ε

Calculate the effective heat capacity for the individual experiments using the appropriate formula [alternative D.2 a) or D.2 b)] as given in D.3.2.

Calculate the mean value $\varepsilon_{(n)}$ or $\varepsilon_{o(n)}$, respectively, and make sure that the precision requirements are met (see 9.7).

The system is now calibrated and the main calorimetric parameters set for subsequent combustion measurements on fuel samples.

D.4.3 The gross calorific value at constant volume $q_{V,\mathrm{gr}}$

Perform the fuel combustions in accordance with the instructions in 10.2 and 10.3. θ is calculated in the same way as for the calibrations.

Calculate the calorific value using the appropriate formula [alternative D.2 a) or D.2 b)] as given in D.3.3.

D.5 Automated bomb calorimeters

Operate the calorimeter according to the instructions. The corrected temperature rise θ is usually derived automatically by the system.

Make sure that the correct value is used for the energy of combustion of the calibrant under the bomb conditions utilized (see D.2) in the evaluation of the calibration constant.

Make sure that the precision requirements are met. If necessary, check the system by burning benzoic acid as an unknown. Any restrictions set by the manufacturer, on the amount of sample burned, shall be adhered to.

Define the valid working range for subsequent measurements.

Make a check on the calculations with respect to fuse wire and nitric acid corrections. Unless the correction for sulphuric acid to sulphur dioxide (Q_S / m_1) is already taken care of by the system, use the value given in 10.3.2.

Annex E
(informative)

Examples to illustrate the main calculations used in this document when an automated bomb calorimeter is used for determinations

The given example is based on an adiabatic calorimeter. The differences between automatic calorimeter types (adiabatic, isoperibol or isothermal) and manufacturers appear as they do give out both of the critical temperatures (t_{i} and t_{f}) or only the corrected temperature rise θ. Attention should be paid to the handling of corrections, especially Q_{fuse} and Q_{ign} (there might be small differences between manufacturers).

E.1 Gross calorific value at constant volume

The calibration data are given in Table E.1.

Table E.1 — Calibration data

No.	m_{ba} g	t_{i} K	t_{f} K	θ K	Q_{fuse} J	Q_{ign} J	NaOH ml	Q_{N} J	ε J/K
1	1,028 2	1,059	4,102	3,043	0	21,5	6,5	39,0	8 962
2	1,052 5	0,454	3,568	3,114	0	21,5	5,9	35,4	8 963
3	1,001 9	0,892	3,859	2,967	0	21,5	6,4	38,4	8 957
4	1,022 9	0,942	3,970	3,028	0	21,5	5,7	34,2	8 959
5	1,014 6	0,373	3,375	3,002	0	21,5	6,4	38,4	8 964
								Average $\varepsilon_{(\mathrm{n})}$	**8 961**

$q_{V,\mathrm{ba}}$ = 26 465 J/g

l_{wire} = 10 cm of ignition wire; of which 8 cm is burned (Q_{ign} = 2,69 J/cm)

No fuse, cotton, etc. was used.

NaOH, [c = 0,1 mol/l]

Corrected temperature rise θ

Difference of initial and final temperatures ($t_{\mathrm{f}} - t_{\mathrm{i}}$) or directly read temperature rise θ (see above).

Calculation of the effective heat capacity ε

The effective heat capacity ε is derived from the total energy change of the overall bomb process, e.g.

(1,028 2 × 26 465 + 21,5 + 39,0) divided by θ (3,043), i.e. ε = 8 962 J/K.

Calculation of the gross calorific value $q_{V,\mathrm{gr}}$

For the combustion of about 1 g of a biofuel sample, the parameters of the experiment are

m_1 = 1,192 4 g Q_{fuse} = 0

l_{wire} = 8 cm Q_{ign} = 21,5 J

θ = 2,630 K $Q_{\text{N,S}}$ = 29,4 J

The nitric acid correction (and a part of the sulphuric acid correction) was determined by titration with NaOH (method c) as $Q_{\text{N,S}}$.

The “combustion energy” from the biofuel sample is derived from $(\varepsilon_{(\text{n})} \times \theta)$ minus the contributions from the ignition wire and the formation of nitric acid, i.e. (21,5 + 29,4) J, divided by the mass of the biofuel sample m_1.

Hence

$$\frac{8\ 961 \times 2,630 - 21,5 - 29,4}{1,192\ 4} = 19\ 722\ \text{J/g}$$

where ε was used as $\varepsilon_{(\text{n})}$.

The sulphur content of the biofuel analysis sample was a mass fraction of 0,02 %. The correction to account for the reaction from aqueous sulphuric acid to gaseous sulphur dioxide is 0,02 × 57 = 1 J/g ($Q_{\text{S}} / m_1 = (57 \times \text{S} \times m_1) / m_1$), which is to be subtracted from 19 722 J/g, yielding 19 721 J/g as the gross calorific value at constant volume for the analysis sample, $q_{V,\text{gr}}$.

The total moisture content as received (M_{ar}) for the biofuel in question was a mass fraction of 40,0 %; the moisture content of the analysis sample (M_{ad}) was a mass fraction of 3,0 %. For the dry sample (dry basis, in dry matter), the gross calorific value at constant volume is

$$q_{V,\text{gr,d}} = 19\ 721 \times \frac{100}{100 - 3,0}\ \text{J/g} = 20\ 330\ \text{J/g or } 20,33\ \text{MJ/kg}$$

For the original (moist, as received) biofuel sample, the gross calorific value at constant volume is

$$q_{V,\text{gr,ar}} = 20\ 330 \times (1 - 0,01 \times 40,0) = 12\ 198\ \text{J/g or } 12,20\ \text{MJ/kg.}$$

E.2 Gross calorific value at constant pressure

If the biofuel were burned in oxygen at constant pressure, instead of at constant volume in the bomb, the volume of the system would change. The hydrogen in the fuel, reacting with gaseous oxygen to give liquid water, causes a decrease in the volume of the system. When the biofuel carbon reacts with gaseous oxygen, an equal volume of gaseous carbon dioxide is formed and hence no change in volume occurs in combustion of the carbon. The oxygen and nitrogen in the biofuel both give rise to an increase in volume. The change in the volume of the gaseous phase for the combustion reaction may be expressed as

$$\Delta n_{\text{g}} = 0,01 \times \left[-\frac{0,5 \times w(\text{H})}{2,016} + \frac{w(\text{O})}{31,999} + \frac{w(\text{N})}{28,013} \right] \text{mol per g of sample}$$

where $w(\text{H})$, $w(\text{O})$ and $w(\text{N})$ are the total percentages of hydrogen, oxygen and nitrogen, respectively, in the biofuel substance in the state specified for the conversion from a constant volume calorific value to a constant pressure value.

The Δn_{g} value is to be multiplied by $\text{R}T$ to interpret the volume change in terms of the associated change in energy. T in this context is the reference temperature for calorific values, i.e. 298,15 K (25 °C).

For convenience, the dry state values are used to show the relation between the gross calorific value at constant pressure and that at constant volume:

$$q_{p,\mathrm{gr,d}} = q_{V,\mathrm{gr,d}} + \left\{6,15 \times w(\mathrm{H})_\mathrm{d} - 0,8 \times \left[w(\mathrm{O})_\mathrm{d} + w(\mathrm{N})_\mathrm{d}\right]\right\} \ \mathrm{J/g}$$

where $w(\mathrm{H})_\mathrm{d}$, $w(\mathrm{O})_\mathrm{d}$ and $w(\mathrm{N})_\mathrm{d}$ have the same significance as in 12.2. $w(\mathrm{H})$, $w(\mathrm{O})$ and $w(\mathrm{N})$ do not contain any contributions from the mineral matter of the fuel sample. The uncertainty introduced by substituting $w(\mathrm{H})_\mathrm{d}$, $w(\mathrm{O})_\mathrm{d}$ and $w(\mathrm{N})_\mathrm{d}$ is, however, negligible. The coefficient for $w(\mathrm{N})_\mathrm{d}$, strictly, is 0,9 but may be taken to be the same as that for oxygen since the term as such is small.

E.3 Net calorific value

E.3.1 Net calorific value at constant volume

The energy of vaporization (constant volume) for water at 25 °C is 41,53 kJ/mol. This corresponds to 206,0 J/g for a mass fraction of 1 % of hydrogen in the fuel sample or 23,05 J/g for a mass fraction of 1 % of moisture, respectively.

The net calorific value at constant volume, $q_{V,\mathrm{net}}$, is derived from the corresponding gross calorific value, for example:

$$q_{V,\mathrm{net,d}} = q_{V,\mathrm{gr,d}} - 206,0 \times w(\mathrm{H})_\mathrm{d}$$

where $w(\mathrm{H})_\mathrm{d}$ is the hydrogen content, in percentage by mass, of the moisture-free fuel (see 12.2).

For the required moisture content M (e.g. total moisture content as received, M_ar), the net calorific value at constant volume is calculated from

$$q_{V,\mathrm{net,m}} = \left[q_{V,\mathrm{gr,d}} - 206,0 \times w(\mathrm{H})_\mathrm{d}\right] \times (1 - 0,01 \times M) - 23,05 \times M$$

E.3.2 Net calorific value at constant pressure

The enthalpy of vaporization (constant pressure) for water at 25 °C is 44,01 kJ/mol. This corresponds to 218,3 J/g for a mass fraction of 1 % of hydrogen in the fuel sample or 24,43 J/g for a mass fraction of 1 % of moisture, respectively.

The net calorific value at constant pressure for the dry sample may be derived from that at constant volume according to

$$q_{p,\mathrm{net,d}} = q_{V,\mathrm{gr,d}} + 6,15 \times w(\mathrm{H})_\mathrm{d} - 0,8 \times \left[w(\mathrm{O})_\mathrm{d} + w(\mathrm{N})_\mathrm{d}\right] - 218,3 \times w(\mathrm{H})_\mathrm{d}$$

$$= q_{V,\mathrm{gr,d}} - 212,2 \times w(\mathrm{H})_\mathrm{d} - 0,8 \times \left[w(\mathrm{O})_\mathrm{d} + w(\mathrm{N})_\mathrm{d}\right]$$

For the required moisture content M (e.g. total moisture content as received, M_ar), the net calorific value at constant pressure is calculated from

$$q_{p,\mathrm{net,m}} = \left\{q_{V,\mathrm{gr,d}} - 212,2 \times w(\mathrm{H})_\mathrm{d} - 0,8 \times \left[w(\mathrm{O})_\mathrm{d} + w(\mathrm{N})_\mathrm{d}\right]\right\} \times (1 - 0,01M) - 24,43 \times M$$

E.4 Use of typical or default values to calculate calorific values

a) Well-defined and characterized virgin solid biofuels

For well-defined and characterized virgin biofuels, typical values with well-documented references, or if such are not available, default values for hydrogen (H), nitrogen (N), oxygen (O) and sulfur (S) can be used instead of determined values on the calculations of gross calorific values and net calorific values.

The default values in Annex G are given on dry, ash free basis and shall be converted to the stage required in this document using formulae given in ISO 16993.

b) Other solid biofuels and solid biofuel blends

For other biofuels and biofuel blends and mixtures, the determination of hydrogen (H), nitrogen (N) oxygen (O), sulphur (S) and ash shall be done simultaneously with the determination of calorific value.

The oxygen content (O) on dry basis is calculated as given in ISO 16993.

The content of chlorine $w(\mathrm{Cl})_\mathrm{d}$ and sulfur $w(\mathrm{S})_\mathrm{d}$ may be ignored in the calculation of the oxygen content because of their usually low content in solid biofuels and the negligible effect to the calculation.

Annex F
(informative)

List of symbols used in this document

$q_{V,\mathrm{gr}}$	Gross calorific value at constant volume of the biofuel as analysed
$q_{V,\mathrm{gr,d}}$	Gross calorific value at constant volume of the dry (moisture-free) fuel (dry basis, in dry matter)
$q_{V,\mathrm{gr,m}}$	Gross calorific value at constant volume of the biofuel with moisture content M
$q_{p,\mathrm{gr,d}}$	Gross calorific value at constant pressure of the dry (moisture-free) fuel (dry basis, in dry matter)
$q_{V,\mathrm{net,m}}$	Net calorific value at constant volume of the biofuel with moisture content M
$q_{p,\mathrm{net}}$	Net calorific value at constant pressure
$q_{p,\mathrm{net,d}}$	Net calorific value at constant pressure of the dry (moisture-free) fuel (dry basis, in dry matter)
$q_{p,\mathrm{net,m}}$	Net calorific value at constant pressure of the biofuel with moisture content M (e.g. as received, $q_{p,\mathrm{net,ar}}$)
$q_{V,\mathrm{ba}}$	Certified gross calorific value at constant volume for benzoic acid
$q_{V,2}$	Gross calorific value at constant volume of a combustion aid
Q_{fuse}	Contribution from combustion of the fuse
Q_{ign}	Contribution from oxidation of the ignition wire
Q_{N}	Contribution from formation of nitric acid (from liquid water and gaseous nitrogen and oxygen)
Q_{S}	Correction for taking the sulfur from the aqueous sulphuric acid in the bomb to gaseous sulfur dioxide
$Q_{\mathrm{N,S}}$	Combined correction for nitric and sulphuric acids
$Q_{\mathrm{S,\ add.}}$	Additional correction for sulfur to be used with $Q_{\mathrm{N,S}}$
M	Moisture content of the biofuel for which the calculation is required
M_{ad}	Moisture in the general analysis sample

M_{ar}	Total moisture as received (as sampled)
m_{ba}	Mass of benzoic acid
m_1	Mass of biofuel sample
m_2	Mass of combustion aid
m_{cr}	Mass of crucible
ε	Effective heat capacity of the calorimeter
ε_{o}	Effective heat capacity of hypothetical calorimeter with no crucible in the bomb
$\varepsilon_{\bullet}$	Effective heat capacity of calorimeter on a “total-calorimeter-mass” basis
$\varepsilon_{(\mathrm{n})}$	Mean effective heat capacity of the calorimeter based on *n* determinations of ε
$\varepsilon_{\mathrm{o(n)}}$	Mean effective heat capacity of the calorimeter based on *n* determinations of ε_{o}
$\hat{\varepsilon}$	Best estimate (corresponds to “mean” value) of ε from linear regression of ε as a function of the observed temperature rise $(t_{\mathrm{f}} - t_{\mathrm{i}})$
$c_{p,\mathrm{cr}}$	Specific heat capacity of the crucible
$c_{p,\mathrm{aq}}$	Specific heat capacity of water
θ	Corrected temperature rise
T	Reference temperature for the calculation of calorific value (T = 298,15 K)
t	Calorimeter temperature
t_{i}	Initial temperature of the main period
t_{f}	Final temperature of the main period (= reference temperature)
$t_{\mathrm{f}} - t_{\mathrm{i}}$	Observed temperature rise
Δt_{ex}	Heat-leak correction
t_{j}	Thermostat (jacket) temperature
$t_{\mathrm{j}} - t$	Thermal head
t_{∞}	Asymptotic temperature of an isoperibol calorimeter (at “infinite” time)
t_{mi}	Mean temperature in the fore period
t_{mf}	Mean temperature in the after period
$t_{\mathrm{f+a}}$	Temperature *a* minutes after the end of the main period

t_x	Temperature at the time τ_x
τ	Time
τ_i	Time at the beginning of the main period
τ_f	Time at the end of the main period
τ_x	Dickinson extrapolation time
R	Gas constant (R = 8,314 47 J/mol × K)
G	Specific rate constant
g	Drift rate ($\mathrm{d}t / \mathrm{d}\tau$) in the rating periods
g_i	Drift rate in the fore period
g_f	Drift rate in the after period
P_{st}	Power of stirring
daf	Dry and ash free
$w(\mathrm{A})_d$	Ash content of the moisture free biofuel
$w(\mathrm{C})_d$	Carbon content of the moisture-free biofuel
$w(\mathrm{Cl})_d$	Chlorine content of the moisture-free biofuel
$w(\mathrm{H})_d$	Hydrogen content of the moisture-free biofuel (includes the hydrogen from the water of hydration of the mineral matter as well as hydrogen in the biofuel substance)
$w(\mathrm{O})_d$	Oxygen content of the moisture-free biofuel
$w(\mathrm{N})_d$	Nitrogen content of the moisture-free biofuel
$w(\mathrm{S})_d$	Sulfur content of the moisture-free biofuel

Annex G
(informative)

Default values of most used solid biofuels for the calculations of calorific values

Solid biofuel, **origin**		**Hydrogen (H)** mass fraction daf	**Oxygen (O)** mass fraction daf	**Nitrogen (N)** mass fraction daf	**Sulfur (S)** mass fraction daf
Forest, plantation and other virgin wood (1.1) Chemically untreated by-products and residues from wood processing industry (1.2.1)	Stemwood without bark, needles and leaves (1.1.3.3, 1.1.3.4, 1.2.1.3, 1.2.1.4)	6,2	43	0,1	0,02
	Whole trees (1.1.1, 1.1.2)	6,2	42	0,2	0,02
	Logging residues (1.1.4)	6,1	41	0,5	0,04
	Bark (1.1.6, 1.2.1.5)	6,1	40	0,4	0,1
	Short rotation coppice (1.1.1.3, 1.1.2.3)	6,3	44	0,5	0,05
Cereal crops, straw parts (2.1.1.2) Grasses, straw parts (2.1.2.2)	Wheat, rye, barley (2.1.1.2)	6,3	43	0,5	0,1
	Rice (2.1.1.2)	6,2	44	1,0	0,1
	Rape (2.1.1.2)	6,3	43	0,8	0,3
	Grasses (2.1.2.2)	6,3	43	1,0	0,2
Cereal crops, grains and seeds (2.1.1.3)	Wheat, rye, barley (2.1.1.3)	6,6	45	2,0	0,1
Olive stones (3.1.2.3, 3.2.1.2)	Virgin stones and chemically untreated stones (3.1.2.3, 3.2.1.2)	6,2	42	0,2	0,02

NOTE 1 The above listed values are documented and approved by ISO/TC 238/WG4.

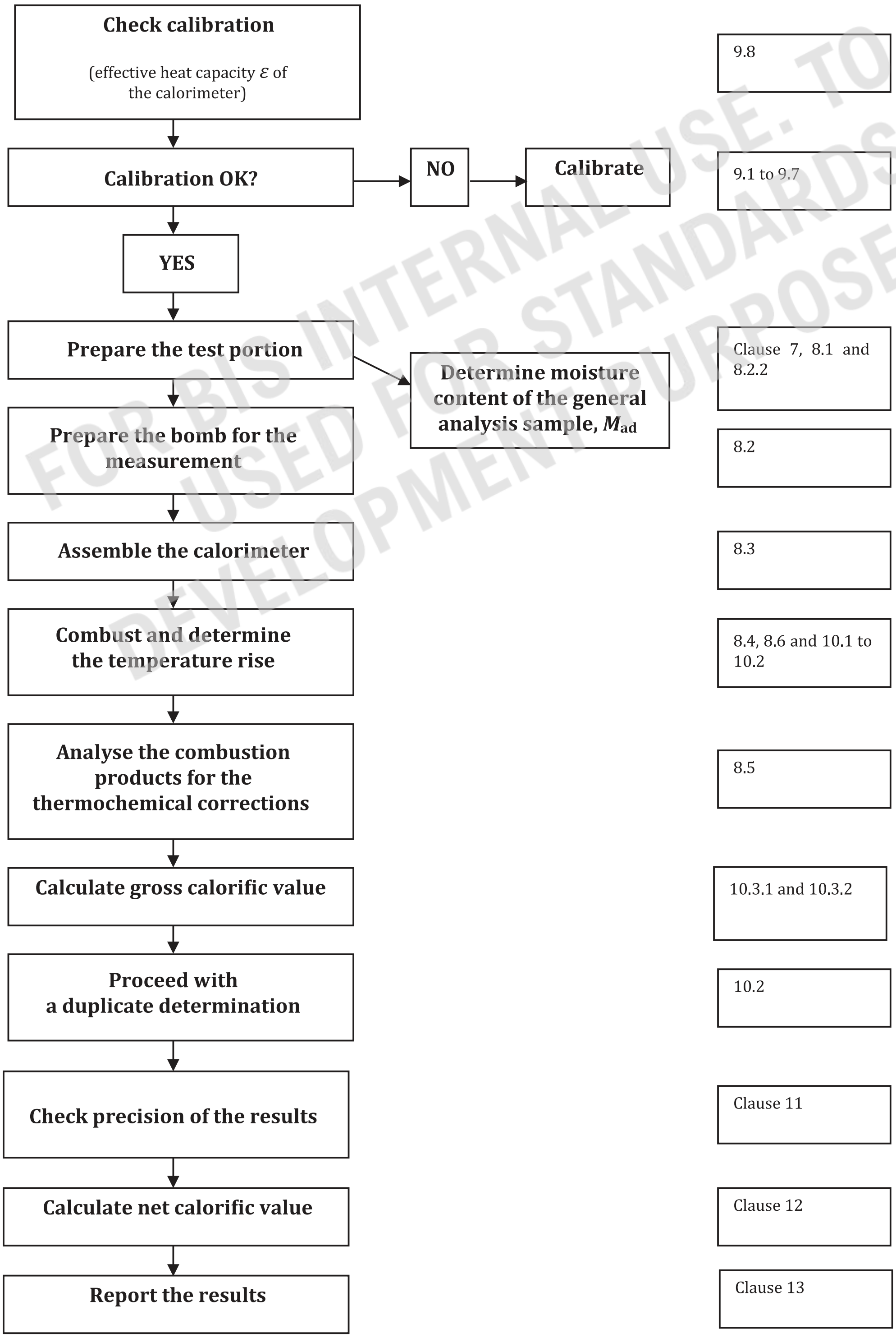
NOTE 2 The above listed values are given on a dry, ash free basis. ISO 16993 provides formulae for conversion to “as analysed” and “dry” basis.

NOTE 3 The specification of the origins of solid biofuels is in accordance with ISO 17225-1:2014, Table 1.

NOTE 4 Default values for olive stones are valid only if ash content is <1,5 w-% dry basis.

Annex H
(informative)

Flow chart for a routine calorific value determination



IS 17655 (PART 1):2021/
ISO 18134-1 : 2015

SUMMARY

Moisture value is crucial for solid biofuels as it affects combustion efficiency, energy output, storage stability, and transportation costs.

This part of IS 17655 describes the method for determining the total moisture content of solid biofuels using an oven dry method and may be used when high precision of the determination of moisture content is necessary.

The procedure involves drying a test portion of the solid biofuel at 105 °C in an air atmosphere until a constant mass is achieved. The moisture content is then calculated based on the mass loss of the test portion, with a correction for buoyancy effects.

SOLID BIOFUELS – DETERMINATION OF MOISTURE CONTENT – OVEN DRY METHOD: PART 1 TOTAL MOISTURE – REFERENCE METHOD

Indian Standard

SOLID BIOFUELS — DETERMINATION OF MOISTURE CONTENT — OVEN DRY METHOD

PART 1 TOTAL MOISTURE — REFERENCE METHOD

1 Scope

This part of ISO 18134 describes the method of determining the total moisture content of a test sample of solid biofuels by drying in an oven and may be used when high precision of the determination of moisture content is necessary. The method described in this International Standard is applicable to all solid biofuels. The moisture content of solid biofuels (as received) is always reported based on the total mass of the test sample (wet basis).

NOTE The term moisture content, when used with biomass materials, can be misleading since untreated biomass frequently contains varying amounts of volatile compounds (extractives) which might evaporate when determining moisture content by oven drying (see References [2] and [3]).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

EN 14780[1], *Solid biofuels — Sample preparation*

EN 14778[2], *Solid biofuels — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

3.1
nominal top size
aperture size of the sieve where at least 95 % by mass of the material passes

[SOURCE: ISO 16559:2014, definition 4.137, modified]

3.2
test sample
original sample sent to the laboratory for analysis

3.3
test portion
sample extracted from the test sample and used during the analysis

---

[1] To be replaced by ISO 14780.

[2] To be replaced by ISO 18135.

4 Principle

The test portion of solid biofuel shall be dried at a temperature of 105 °C in air atmosphere until constant mass is achieved and the percentage moisture shall be calculated from the loss in mass of the test portion and includes a procedure for correction of the buoyancy effects.

5 Apparatus

5.1 Drying oven, capable of being controlled at a temperature within the range of (105 ± 2) °C and in which the air atmosphere changes between three to five times per hour. The air velocity shall be such that the test portion particles are not dislodged from their tray.

5.2 Dishes and trays, of non-corrodible and heat-resistant material and of such dimension that they will hold the total test portion in a layer not exceeding 1 g of material per cm^2. The surface of the trays shall be such that the possibility to adsorption/absorption is minimised (very clean and even surface).

5.3 Balance, capable of reading to the nearest 0,1 g.

6 Sample preparation

6.1 Sample reduction

Test samples for the determination of total moisture content shall be obtained in accordance with ISO 18135 and shall be received in the laboratory in sealed air-tight containers or bags. A test portion shall be prepared in accordance with ISO 14780 and the nominal top size reduced to below 31,5 mm.

6.2 Pre-dried test sample

During the course of its preparation, the test sample might have been pre-dried (see ISO 14780) in which case, Formula (2) detailed in 8.3 shall be used to calculate the total moisture content.

WARNING — Dried solid biofuels are hygroscopic. Precautions shall be taken to ensure that moisture is not lost during preparation of the test portion. Significant losses of moisture from test portion will occur after a few minutes in room atmosphere.

6.3 Size of test portion

The mass of the test portion shall have a minimum mass of 300 g.

NOTE For fine particulate solid biofuels (e.g. sawdust and fuel powder), the test portion can be reduced to 200 g or 100 g, respectively, if using a balance capable of reading to the nearest 0,01 g.

7 Procedure

7.1 Handling of test portion

Weigh an empty and clean drying tray to the nearest 0,1 g.

Transfer the test portion from the package (container or bag) in which it is delivered to the empty and clean drying tray and spread the material evenly, not to exceed 1 cm^2 of surface area per 1 g of material. In case moisture remains on the inside surfaces of the package, shake the package to allow the material to re-absorb the moisture prior to emptying the package.

7.2 Weighing of test portion and correction for buoyancy of trays

Weigh the tray with the test portion to the nearest 0,1 g before heating.

Weigh an identical empty and clean tray (reference tray) to the nearest 0,1 g before heating.

NOTE 1 A reference tray is included in the procedure for the purpose of correction of buoyancy. The weight of a tray when still hot is less than the weight of the cold tray due to buoyancy. The magnitude of the buoyancy effect depends on the size and the weight of the tray.

Thereafter, place the tray with the test portion together with the reference tray in the temperature controlled oven at (105 ± 2) °C. Heat the trays until constant mass has been achieved. Constant mass is defined as a change not exceeding 0,2 % absolute of the initial mass of the test portion during a heating period of 60 min. The drying time required will depend on particle size of the material, rate of atmospheric change in the oven, and thickness of the layer of material.

Remove the two trays from the oven and weigh each when they are still hot to the nearest 0,1 g within 10 s to 15 s in order to avoid absorption of moisture. Use heat-insulating material on the balance pan to avoid direct contact with the hot tray.

NOTE 2 The required drying time can be determined in pre-tests on similar fuel types with comparable particle size.

Do not overload the drying oven. The space above the trays and between the trays shall allow free flow of air and moisture.

To prevent unnecessary losses of volatile compounds, generally, the drying time should not exceed 24 h.

The moisture content determination shall be conducted in duplicate.

8 Calculation

8.1 General

The total moisture content shall be calculated on a wet basis in accordance with Formula (1) detailed in 8.2. The determination of moisture content for pre-died test sample is detailed in 8.3. The result shall be reported on a wet basis and reported in accordance with Clause 10.

8.2 Moisture content on a wet basis

The moisture content, M_{ar}, of the test portion, as received, expressed as a percentage by mass, shall be calculated in accordance with Formula (1):

$$M_{ar} = \frac{(m_2 - m_3) - (m_4 - m_5)}{(m_2 - m_1)} \times 100 \qquad (1)$$

where

m_1 is the mass of the empty tray used for the test portion, in g;

m_2 is the mass of the tray and test portion before drying (weight in room temp), in g;

m_3 is the mass of the tray and test portion after drying (weight when still hot), in g;

m_4 is the mass of the reference tray before drying (weight at room temp), in g;

m_5 is the mass of the reference tray after drying (weight when still hot), in g.

The result shall be calculated to two decimal places and the mean value of both determinations shall be rounded to the nearest 0,1 % for reporting.

8.3 Moisture content for pre-dried material

If the test sample has been pre-dried before this moisture determination (according to 6.2), the total moisture, M_T, expressed as a percentage by mass is given by Formula (2):

$$M_T = M_p + M_r x \left(1 - M_p / 100\right) \qquad (2)$$

where

M_p is the moisture loss of pre-drying, expressed as a percentage by mass of the original test sample;

M_r is the residual moisture determined in the pre-dried test sample by this procedure, expressed as a percentage by mass.

9 Performance characteristics

Because of the varying nature of the solid biofuels covered by this part of ISO 18134, it is not possible to give a precision statement (repeatability or reproducibility) for this test method.

10 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) reference to this part of ISO 18134, i.e. ISO 18134-1;

d) results of the test on wet basis;

e) any unusual features noted during the determination which might affect the result;

f) any deviation from this part of ISO 18134 or operations regarded as optional.

IS 17655 (PART 2):2021/
ISO 18134-2 : 2017

SUMMARY

Moisture value is crucial for solid biofuels as it affects combustion efficiency, energy output, storage stability, and transportation costs.

This part of IS 17655 describes the method of determining the total moisture content of a test sample of solid biofuels by drying in an oven and is used when the highest precision is not needed, e.g. for routine production control on site.

The portion is dried at 105 °C until a constant mass is achieved, and the moisture percentage is calculated from the mass loss. This simplified method neglects the effect of buoyancy and requires only a single determination, unlike the reference method IS 17655 (Part 1). The weight of a tray when still hot is less than the weight of the cold tray due to buoyancy. The magnitude of the buoyancy effect depends of the size and the weight of the tray.

SOLID BIOFUELS – DETERMINATION OF MOISTURE CONTENT – OVEN DRY METHOD: PART 2 TOTAL MOISTURE – SIMPLIFIED METHOD

IS 17655 (PART 2): 2021

Indian Standard

SOLID BIOFUELS — DETERMINATION OF MOISTURE CONTENT — OVEN DRY METHOD

PART 2 TOTAL MOISTURE — SIMPLIFIED METHOD

1 Scope

This document describes the method of determining the total moisture content of a test sample of solid biofuels by drying in an oven and is used when the highest precision is not needed, e.g. for routine production control on site. The method described in ISO 18134 (all parts) is applicable to all solid biofuels. The moisture content of solid biofuels (as received) is always reported based on the total mass of the test sample (wet basis).

NOTE The term moisture content, when used with biomass materials, can be misleading since untreated biomass frequently contains varying amounts of volatile compounds (extractives) which might evaporate when determining moisture content by oven drying (see References [1] and [3]).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14780[1], *Solid biofuels — Sample preparation*

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18135[2], *Solid biofuels — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
nominal top size
aperture of the sieve where at least 95 % by mass of the material passes

[SOURCE: ISO 16559:2014, 4.137, modified.]

3.2
test sample
original sample sent to the laboratory for analysis

---

[1] To be published.

[2] To be published.

3.3
test portion
sample extracted from the *test sample* (3.2) and used during the analysis

4 Principle

The test portion of solid biofuel shall be dried at a temperature of 105 °C in air atmosphere until constant mass is achieved and the percentage moisture shall be calculated from the loss in mass of the test portion.

The difference with this procedure compared to the reference method ISO 18134-1 is that the effect due to buoyancy is neglected and only a single determination is required. The weight of a tray when still hot is less than the weight of the cold tray due to buoyancy. The magnitude of the buoyancy effect depends of the size and the weight of the tray.

5 Apparatus

5.1 Drying oven, capable of being controlled at a temperature within the range of (105 ± 2) °C and in which the air atmosphere changes between three to five times per hour. The air velocity shall be such that the test portion particles are not dislodged from their tray.

5.2 Dishes and trays, of non-corrodible and heat-resistant material and of such dimension that they will hold the total test portion in the proportion of not exceeding 1 g of material per cm^2. The surface of the trays shall be such that the possibility to adsorption/absorption is minimized (very clean and even surface).

5.3 Balance, capable of reading to the nearest 0,1 g.

6 Sample preparation

6.1 Sample reduction

Test samples for the determination of total moisture content shall be obtained in accordance with ISO 18135 and shall be received in the laboratory in sealed air-tight containers or bags. A test portion shall be prepared in accordance with ISO 14780 and the nominal top size reduced to below 31,5 mm.

6.2 Drying of test portion

The necessary drying time of the test portion depends, among other things, on the particle size of the test sample material. In order to reduce the necessary drying time, the particle size of the material may be reduced to below 31,5 mm by cutting up the material, provided that the cutting procedure does not change the moisture content of the material. To minimize loss of moisture, the cutting shall be carried out as fast as possible while avoiding air ventilation as much as possible. Visibly wet samples cannot be cut up without loss of moisture and shall therefore be pre-dried.

If the above procedure is not practical, larger test sample sizes and longer drying times shall be used. Deviations from the established procedures shall be documented on the test report.

WARNING — Dried solid biofuels are hygroscopic. Precautions shall be taken to ensure that moisture is not lost during preparation of the test portion. Significant losses of moisture from test portion will occur after a few minutes in room atmosphere.

6.3 Size of test portion

The mass of the test portion shall have a minimum mass of 300 g.

NOTE For fine particulate solid biofuels (e.g. sawdust and fuel powder), the test portion can be reduced to 200 g or 100 g, respectively, if using a balance capable of reading to the nearest 0,01 g.

7 Procedure

7.1 Handling of test portion

Weigh an empty and clean drying tray to the nearest 0,1 g.

Transfer the test portion from the package (container or bag) in which it is delivered to the empty and clean drying tray and spread the material evenly, not to exceed 1 cm^2 of surface area per 1 g of material. In case moisture remains on the inside surfaces of the package, shake the package to allow the material to re-absorb the moisture prior to emptying the package.

7.2 Weight of test portion

Weigh the tray with the test portion to the nearest 0,1 g before heating.

NOTE 1 Do not use larger dimension of the drying tray than necessary in relation to the size of the test portion due to buoyancy when hot weighing is undertaken (see ISO 18134-1).

Thereafter, place the tray in the temperature-controlled oven at (105 ± 2) °C. Heat the tray until constant mass has been achieved. Constant mass is defined as a change not exceeding 0,2 % absolute of the initial mass of the test portion during a heating period of 60 min. The drying time required will depend on particle size of the material, rate of atmospheric change in the oven, and thickness of the layer of material.

Remove the tray from the oven and weigh when still hot to the nearest 0,1 g within 10 s to 15 s in order to avoid absorption of moisture. Use heat-insulating material on the balance pan to avoid direct contact with the hot tray.

NOTE 2 The required drying time can be determined in pre-tests on similar fuel types with comparable particle size.

Do not overload the drying oven. The space above the trays and between the trays shall allow free flow of air and moisture.

To prevent unnecessary losses of volatile compounds, generally, the drying time should not exceed 24 h.

8 Calculation

The moisture content, M_{ar}, of the test portion, as received, shall be reported on a wet basis and expressed as a percentage by mass and shall be calculated in accordance with Formula (1):

$$M_{ar} = \frac{(m_2 - m_3)}{(m_2 - m_1)} \times 100 \qquad (1)$$

where

m_1 is the mass of the empty drying container, in g;

m_2 is the mass of the drying container and test portion before drying, in g;

m_3 is the mass of the drying container and test portion after drying (weigh when still hot), in g.

The result shall be calculated to two decimal places and rounded to the nearest 0,1 % for reporting.

9 Performance characteristics

Because of the varying nature of the solid biofuels covered by this document, it is not possible to give a precision statement (repeatability or reproducibility) for this test method.

10 Test report

The test report shall include at least the following information:

a) an identification of the laboratory performing the test and the date of the test;

b) an identification of the product (or sample) tested;

c) a reference to this document, i.e. ISO 18134-2;

d) the results of the test on wet basis;

e) any unusual features noted during the determination which might affect the result;

f) any deviation from this document or operations regarded as optional.

IS 17655 (PART 3):2021/
ISO 18134-3 : 2015

SUMMARY

Moisture value is crucial for solid biofuels as it affects combustion efficiency, energy output, storage stability, and transportation costs.

This part of IS 17655 describes the method of determining the moisture in the analysis test sample by drying in an oven. It is intended to be used for general analysis samples.

Since biofuels in small particle size are very hygroscopic, their moisture content will change with humidity in the atmosphere and therefore, the moisture of the test portion is determined simultaneously with determination of for example calorific value, carbon content, and nitrogen content.

The test portion of solid biofuel is dried at a temperature of 105 °C in air atmosphere until constant mass is achieved and the percentage moisture is calculated from the loss in mass of the test portion.

SOLID BIOFUELS DETERMINATION OF MOISTURE CONTENT OVEN DRY METHOD: PART 3 MOISTURE IN GENERAL ANALYSIS SAMPLE

IS 17655 (PART 3): 2021

Indian Standard

SOLID BIOFUELS — DETERMINATION OF MOISTURE CONTENT — OVEN DRY METHOD

PART 3 MOISTURE IN GENERAL ANALYSIS SAMPLE

1 Scope

This part of ISO 18134 describes the method of determining the moisture in the analysis test sample by drying in an oven. It is intended to be used for general analysis samples in accordance with EN 14780. The method described in this part of ISO 18134 is applicable to all solid biofuels. The moisture content of solid biofuels (as received) is always reported based on the total mass of the test sample (wet basis).

Since biofuels in small particle size are very hygroscopic, their moisture content will change with humidity in the atmosphere and therefore, the moisture of the test portion is determined simultaneously with determination of for example calorific value, carbon content, and nitrogen content.

NOTE The term moisture content when used with biomass materials can be misleading since untreated biomass frequently contains varying amounts of volatile compounds (extractives) which can evaporate when determining the moisture content by oven drying (see References [1] and [2]).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

EN 14778,[1]*Solid Biofuels — Sampling*

EN 14780,[2]*Solid Biofuels — Sample preparation*

ISO 11722, *Solid mineral fuels — Hard coal — Determination of moisture in the general analysis test sample by drying in nitrogen*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

3.1
nominal top size
aperture size of the sieve where at least 95 % by mass of the material passes

[SOURCE: ISO 16559]

---

[1] To be replaced by ISO 18135.

[2] To be replaced by ISO 14780.

3.2
test sample
original sample sent to the laboratory for analysis

[SOURCE: ISO 16559]

3.3
test portion
sample extracted from the *test sample* (3.2) and used during the analysis

[SOURCE: ISO 16559]

4 Principle

The test portion of solid biofuel is dried at a temperature of 105 °C in air atmosphere until constant mass is achieved and the percentage moisture is calculated from the loss in mass of the test portion.

Automatic equipment such as gravimetric analyzers can be used when the method is validated with biomass reference test samples of selected biomass type. Analysis with such equipment shall fulfil all the requirements given in Clause 7 regarding sample size, temperature, atmosphere, and weighing accuracy.

The analysis sample can be dried in air atmosphere or in nitrogen atmosphere. If the sample material is susceptible to oxidation (at 105 °C), drying in nitrogen atmosphere is preferred in accordance with ISO 11722. The used drying atmosphere should be reported in accordance with Clause 10.

5 Apparatus

5.1 Drying oven, capable of being controlled at a temperature within the range of (105 ± 2) °C and in which the air atmosphere changes between three and five times per hour.

The air velocity shall be such that the test portion particles are not dislodged from their tray.

The use of nitrogen atmosphere is detailed in ISO 11722.

5.2 Dishes and trays, of non-corrodible and heat-resistant material and of such dimension that they will hold the total test portion in a layer not exceeding 0,2 g of material per cm^2 and covered by a well fitted lid. The surface of the trays shall be such that the possibility to adsorption/absorption is minimized (very clean and even surface).

5.3 Balance, capable of reading to the nearest 0,1 mg.

5.4 Desiccator with desiccant to minimize absorption by the test portion of moisture from the atmosphere.

6 Sample preparation

6.1 General

Test samples for the determination of total moisture content shall be obtained in accordance with EN 14778 and shall be received in the laboratory in sealed air-tight containers or bags. A test portion shall be prepared in accordance with EN 14780 and the nominal top size reduced to below 1 mm.

WARNING — Dried solid biofuels are hygroscopic. Precautions shall be taken to ensure that moisture is not lost during preparation of the test portion. Significant losses of moisture from test portion will occur after a few minutes in room atmosphere.

Before commencing the determination, mix the analysis sample in its container, preferably by mechanical means, to ensure a well-mixed sample.

6.2 Size of test portion

The mass of the test portion shall have a minimum mass of 1 g.

7 Procedure

Dry an empty weighing dish with its lid at (105 ± 2) °C until constant in mass and cool it to room temperature in a desiccator (5.4).

NOTE Several dishes can be handled at the same time.

Weigh the weighing dish with its lid to the nearest 0,1 mg.

Add minimum 1 g of the test portion into the weighing dish in an even layer and weigh the weighing dish with its lid plus the test portion to the nearest 0,1 mg.

Heat the uncovered dish and its lid with the test portion at (105 ± 2) °C in the temperature controlled oven until constant mass has been achieved. Constant mass is defined as a change not exceeding 1 mg in mass during a heating period of 60 min. The drying time required will depend on the particle size of the material, the rate of atmospheric change in the oven, and the thickness of the layer of material. Allow for up to 3 h.

Replace the lid immediately upon removal from the oven. Transfer the dish and its contents to a desiccator. Let it cool in the desiccator to room temperature.

Remove the dish and its lid with the test portion from the desiccator and weigh to the nearest 0,1 mg. Since small particle size biofuels are very hygroscopic, it is important to weigh rapidly once the test portion taken out of the desiccator.

The moisture content determination shall be conducted in duplicate.

8 Calculation

The moisture content, M_{ad}, of the test portion, *as analysed*, expressed as a percentage by mass, shall be calculated in accordance with Formula (1):

$$M_{\mathrm{ad}} = \frac{(m_2 - m_3)}{(m_2 - m_1)} \times 100 \tag{1}$$

where

m_1 is the mass in grams of the empty dish plus lid;

m_2 is the mass in grams of the dish plus lid plus test portion before drying;

m_3 is the mass in grams of the dish plus lid plus test portion after drying.

The result shall be calculated to two decimal places and the mean value of both determinations shall be rounded to the nearest 0,1 % for reporting.

9 Performance characteristics

9.1 Repeatability

The result of duplicate determinations, carried out in the same laboratory, by the same operator, with the same apparatus on representative portions weighed out at the same time from the analysis sample, shall not differ more than 0,2 % absolute.

9.2 Reproducibility

Since the humidity of the atmosphere and other factors in different laboratories may vary, it is not practical to quote a value regarding reproducibility.

10 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) a reference to this part of ISO 18134, i.e. ISO 18134-3;

d) results of the test *on wet basis* (alternatively for all standards: results of the test including the basis in which they are expressed, as indicated in Clause 8);

e) any unusual features noted during the determination, which can affect the result;

f) any deviation from this part of ISO 18134, or operations regarded as optional.

IS 17844 : 2022/
ISO 18123 : 2015

SUMMARY

The volatile matter content is determined as the loss in mass, less that due to moisture, when solid biofuel is subject to partial pyrolysis under standardized conditions.

This standard prescribes method used to determine the volatile matter content of solid biofuels.

It describes the method of heating a general analysis sample at 900 °C ± 10 °C for 7 minutes, mostly without air contact, and calculating the volatile matter percentage from the mass loss after deducting moisture. Automatic equipment is permitted if validated

SOLID BIOFUELS – DETERMINATION OF THE CONTENT OF VOLATILE MATTER

Indian Standard

SOLID BIOFUELS — DETERMINATION OF THE CONTENT OF VOLATILE MATTER

1 Scope

This International Standard aims to define the requirements and method used to determine the volatile matter content of solid biofuels. It is intended for persons and organisations that manufacture, plan, sell, erect or use machinery, equipment, tools, and entire plants related to solid biofuels, and to all persons and organisations involved in producing, purchasing, selling, and utilizing solid biofuels.

The volatile matter content is determined as the loss in mass, less that due to moisture, when solid biofuel is subject to partial pyrolysis under standardized conditions.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18134-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

EN 14778[1],*Solid Biofuels — Sampling*

EN 14780[2], *Solid biofuels — Sample preparation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

3.1
nominal top size
aperture of the sieve where at least 95 % by mass of the material passes

[SOURCE: ISO 16559]

3.2
laboratory sample
combined sample or a sub-sample of a combined sample for use in a laboratory

[SOURCE: ISO 16559]

3.3
test portion
sub-sample either of a laboratory sample or a test sample

[SOURCE: ISO 16559]

---

[1] To be replaced by ISO 18135.

[2] To be replaced by ISO 14780.

3.4
general analysis sample
sub-sample of a laboratory sample having a nominal top size of 1 mm or less and used for a number of chemical and physical analyses

[SOURCE: ISO 16559]

4 Principle

A portion of the general analysis sample is heated mostly out of contact with air at 900 °C ± 10 °C for 7 min. The furnace is normally not equipped with a vacuum however there is a partial vacuum created during heating to a degree depending on air influx during charging of the test portion into the chamber and the air trapped in the crucible before the lid is put on. The percentage of volatile matter is calculated from the loss in mass of the test portion after deducting the loss in mass due to moisture.

Automatic equipment may be used when the method is validated with biomass reference samples of an adequate biomass type. The automatic equipment shall fulfil all the requirements given in Clauses 5 to 8 regarding sample size, atmosphere, temperatures, and weighing accuracy.

5 Apparatus

5.1 Furnace

The furnace shall be heated by electricity, in which a zone of uniform temperature of 900 °C ± 10 °C can be maintained (see example in Figure 1).

NOTE It is important for furnaces to have flues so that the furnace door seals well. The flue should not reach far out of the oven and should be fitted with a butterfly valve to restrict airflow through the furnace.

Its heating capacity shall be such that, with an initial temperature of 900 °C ± 10 °C, the temperature is regained within 4 min after insertion of a cold stand and its crucibles. The temperature is measured with a thermocouple, as described in 5.2.

Normally the furnace will be designed specifically either for multiple determinations using a number of crucibles in one stand or for receiving one crucible and its stand.

The crucible stand shall be placed in the middle of the furnace. The temperature of 900 °C shall be attained as closely as possible with a specified tolerance of ±10 °C in order to compensate for inherent errors in the temperature measurement and lack of uniformity in the temperature distribution.

Dimensions in millimetres

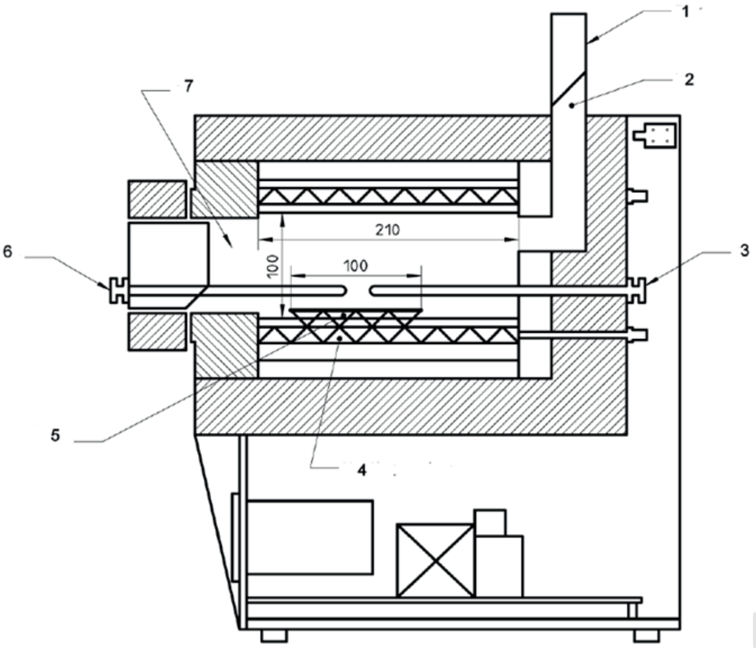


Figure 1 — Example of suitable furnace

Key

1 flue
2 valve
3 thermocouple
4 heating system
5 zone of uniform temperature
6 check thermocouple
7 chamber (width 700 mm)

5.2 Thermocouples and temperature calibration

A sheathed thermocouple shall be permanently installed in the furnace (see right hand side of Figure 1) with its thermo junction as close as possible to the centre of the heating chamber.

An unsheathed thermocouple (see left hand side of Figure 1) long enough to reach the centre of the heating chamber is used for calibration.

The furnace temperature readings shall be checked at regular time intervals with an unsheathed calibrated thermocouple. The unsheathed thermocouple (see Clause 5) shall be positioned as close as possible to the area of the permanently installed thermocouple.

NOTE The temperature/electromotive force (EMF) relationship of a thermo junction maintained at elevated temperatures gradually changes with time, which means that the time for the determination should not be longer than necessary.

5.3 Crucible

The crucible shall be cylindrical, with a well-fitting lid, both made of fused silica. The crucible with lid shall have a mass between 10 g and 14 g and dimensions approximate those shown in Figure 2. The fit of the lid on the crucible is critical to the determination and a lid shall be selected to match the crucible so that the horizontal clearance between them is no greater than 0,5 mm. After selection, the crucible and the lid shall be ground together to give smooth surfaces and then be given a common distinguishing mark.

Dimensions in millimetres

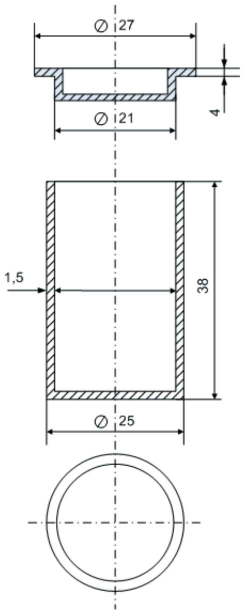


Figure 2 — Silica crucible and lid

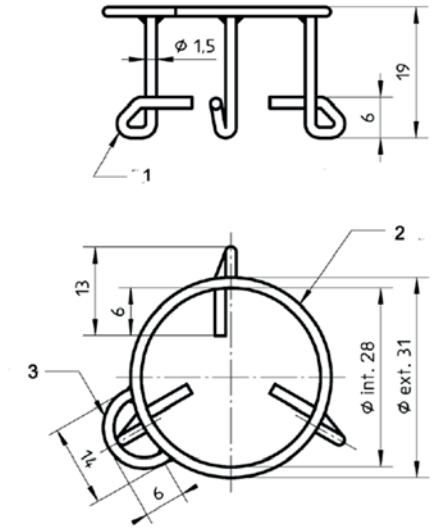
5.4 Crucible stand

The crucible stand to hold the crucible(s) in place in the furnace shall be such that the appropriate rate of heating can be achieved. For example, it may consist of

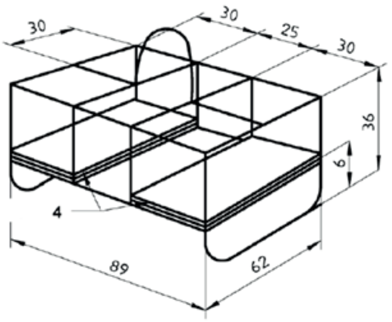
a) for single determinations, a ring of heat-resistant steel wire as shown in Figure 3 a), with ceramic discs, 25 mm diameter and 2 mm thick, resting on the inner projection of its legs, or

b) for multiple determinations, a tray of heat-resistant steel wire as shown in Figure 3 b), of appropriate size, with ceramic plates 2 mm thick supporting the crucibles.

Dimensions in millimetres



a) Suitable for single determination



b) Suitable for multiple determinations

Key

1 three legs paced 120° apart
2 ring
3 handle
4 ceramic plates

Figure 3 — Examples of crucible stands

5.5 Balance

The balance shall be capable of reading to the nearest 0,1 mg.

5.6 Desiccator and desiccant

A desiccator with appropriate desiccant to prevent absorption of moisture from the atmosphere by the test portion.

WARNING — Ash from solid biofuel is very hygroscopic and there is a risk that moisture bound in the desiccant can be absorbed in the sample. Therefore, the desiccant shall be controlled frequently and dried if necessary. In addition, lids shall be used to cover dishes while in the desiccator to prevent the absorption of moisture.

6 Sample preparation

The test sample shall be obtained in accordance with EN 14778. A general analysis sample shall be prepared in accordance with EN 14780, for moisture determination and for determination of volatile content. The material shall have a nominal top size of 1 mm or less.

6.1 Sample size

The general analysis sample shall include material sufficient for the determination of the volatile content and the moisture content.

6.2 Sample conditioning

The determination of volatile content shall be done either

a) directly on a test portion of a general analysis sample, including a concurrent determination of the moisture content in accordance with ISO 18134-3, or

b) from a test portion of the general analysis sample which has been dried using the same drying procedure as in the determination of the moisture content of the test portion and kept absolutely dry before the weighing for the volatile matter content determinations (the test portion shall be kept in a closed container in a desiccator with desiccant).

NOTE For some solid biofuels, it could be necessary to prepare a general analysis sample to a nominal top size of less than 1 mm (e.g. 0,25 mm) in order to keep the stated precision.

7 Procedure

7.1 Conditioning of crucibles

Fill either a stand with one empty crucible and lid [see Figure 3 a)] or a stand with the requisite number of empty crucibles and lids [see Figure 3 b)] and insert in the furnace. Maintain at 900 °C ± 10 °C for 7 min ± 5 s. Remove the stand with crucible(s) from the furnace and allow to cool to room temperature on a plate of thermo-resistant material and store the crucibles in a desiccator.

7.2 Charging of crucibles

Weigh empty crucible(s) with lid(s) in cool condition.

Fill crucible(s) with 1 g ± 0,1 g to the nearest 0,1 mg of material from the general analysis sample. Replace the lid(s) and tap the crucible(s) on a clean hard surface until the material forms a layer of even thickness at the bottom of the crucible.

7.3 Volatilization of test portion

Place the charged crucible(s) again in a cool stand and place in the furnace already maintained at 900 °C ± 10 °C. Close the door and leave for 7 min ± 5 s.

Remove the crucible(s) from the furnace and place on a thermo-resistant surface and allow to cool for approximately 5 min to 10 min and then place the crucible(s) in a desiccator and let cool to room temperature. When cool, weigh the crucible(s) to the nearest 0,1 mg.

NOTE 1 The initial heating and cooling of crucible(s) followed by storage in desiccator and final heating and cooling followed by storage in a desiccator minimizes the risk of distortion of results due to water adsorption on surfaces. Also, the rapid cooling and storage in desiccator minimizes the absorption of moisture by the solid biofuel material.

NOTE 2 If multiple determinations are being made, any vacant places in the stand are to be filled with empty crucibles.

NOTE 3 For some types of solid biofuels, it could be necessary to carry out the determination on a dry sample in order to avoid loss of material due to violent reactions during the heating process. In such case, the charged crucible(s) are dried at 105 °C and cooled in accordance with ISO 18134-3, before being placed in the furnace.

8 Calculation

The volatile matter V_d in the test portion, expressed as a percentage by mass on the dry basis, is given by Formula (1):

$$V_d = \left[\frac{100\left(m_2 - m_3\right)}{m_2 - m_1} - M_{\mathrm{ad}}\right] \times \left(\frac{100}{100 - M_{\mathrm{ad}}}\right) \qquad (1)$$

where

m_1 is the mass in g of the empty crucible and lid;

m_2 is the mass in g of the crucible and lid and test portion before heating;

m_3 is the mass in g of the crucible and lid and contents after heating;

M_{ad} is the moisture, as a percentage by mass, in the test portion as determined in accordance with ISO 18134-3.

The result shall be calculated to two decimal places and the mean value shall be rounded to the nearest 0,1 % for reporting.

9 Performance characteristics

9.1 Repeatability

The results of duplicate determinations (performed within a short period of time, but not simultaneously) in the same laboratory, by the same operator, using the same apparatus on two representative portions taken from the same general analysis sample shall not differ by more than the value given in Table 1, see References [2], [3], [4], [5], and [6].

9.2 Reproducibility

The mean value of the results of duplicate determinations carried out in two different laboratories on representative portions taken from the same general analysis sample, shall not differ by more than the value given in Table 1, see Reference[2]. The reproducibility is however somewhat depending on the flux of air in the crucible and the furnace. Diligence in the handling during the procedures may improve reproducibility, see References [2], [3], [4], [5], and [6].

Table 1 — Repeatability and reproducibility of the method

Volatile matter	**Maximum acceptable differences between results obtained** (calculated on dry basis)	
	Repeatability limit	**Reproducibility limit**
Solid biofuel	1,0 % of the mean result	3,0 % of the mean result

10 Test report

The test report shall include the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) a reference to this International Standard, i.e. ISO 18123;

d) results of the test on dry basis or alternatively as indicated in Clause 9;

e) any unusual features noted during the determination; which may affect the result;

f) any deviation from this International Standard, or operations regarded as optional.

IS 17656 : 2021/
ISO 18846 : 2016

SUMMARY

The amount of fines is an important quality parameter. Excessive amounts of fines in pellet consignments increase the risk of dust explosions and can pose a health problem if inhaled.

This standard focuses on the determination of fines content, defined as the percentage in mass of material below 3.15 mm in size, when passed through a sieve with 3.15 mm diameter round hole.

SOLID BIOFUELS – DETERMINATION OF FINES CONTENT IN SAMPLES OF PELLETS

IS 17656 : 2021

Indian Standard

SOLID BIOFUELS — DETERMINATION OF FINES CONTENT IN SAMPLES OF PELLETS

1 Scope

This International Standard specifies a method for determining the amount of material passing through a sieve with 3,15 mm diameter round hole.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 14780[1], *Solid biofuels — Sample preparation*

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18135[2], *Solid biofuels — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 apply.

4 Principle

A test sample is subjected to sieving by means of manually shaking a sieve with 3,15 mm diameter round holes by horizontal movements and the mass of the material passing through is determined.

5 Apparatus

5.1 Sieve. The sieve shall have round holes with a diameter of 3,15 mm and aperture geometry in accordance with ISO 3310-2.

A sieve with a diameter of 400 mm is recommended. The frame of the sieve shall have a height that enable the sieves to contain a sample and allows a free movement of the sample during the sieving process. Other sizes of sieves may be used for practical reasons but it is important to make sure the sieve is not overloaded which may result in insufficient agitation of the test sample which may impact the flow of fines through the apertures of the sieve.

5.2 Collecting pan, for collection of material passing through the sieve, a collecting pan of adequate size is required.

5.3 Weighing container, for weighing of the fractions, the collecting pan or a separate container of adequate size is required.

---

[1] To be published.

[2] To be published. (Until ISO 18135 is published, EN 14778 should be used.)

5.4 Balance. The balance shall be capable of reading to the nearest 0,1 g.

The balance shall have a weighing capacity of 2 kg or a weighing capacity equal to the selected size of the sieve fractions and the mass of the weighing container.

6 Sample preparation

6.1 Sample reduction

The laboratory sample used for the determination of the fines content shall be obtained in accordance with ISO 18135. The sampling strategy shall be such that special provisions are taken regarding the tendency for stratification of the fines in a consignment of pellets. For example, the entire quantity of pellets in a bag shall be sampled in order to obtain a representative test sample. Due to the special provisions necessary regarding sampling for fines determination, the combined (raw) sample may end up being very large. The combined sample may be divided into one or more smaller test portions using a procedure for sample division in accordance with ISO 14780[3].

NOTE In bulk handling of large volumes of pellets, there is often a risk for stratification of materials in storage bins. There is also the risk of incremental variations of fines in volumes of pellets due to batch-wise operation during transportation with rail cars, trucks and ocean vessels.

6.2 Size of the test portion

The size of the test portion shall be selected in relation to pellets diameter as given in Table 1.

NOTE A larger size of the test portion will yield a higher accuracy (e.g. 1 % according to ISO 17225-1, ISO 17225-2 and ISO 17225-6).

Table 1 — Minimum mass of test portion for pellet diameter classes in accordance with ISO 17225-1, ISO 17225-2 and ISO 17225-6

Pellet diameter mm	Sample size kg	
	Minimum	**Recommended**
<6	3	5 to 10
6 to 12	5	10 to 15
>12 (up to 25)	10	15 to 20

The total mass of the test portion shall be weighed to the nearest 0,1 g and recorded for the quality control calculation in 8.3.

7 Procedure

7.1 Sieving

The sieving shall be done in such a way that the fine particles are separated from the pellets but at the same time avoiding new fines being created. This is best achieved by 5 to 10 circular horizontal rotations of 0,5 kg of material in the sieve. If a sieve with a diameter other than 400 mm is used, the size of the sieved sub-portions (of the test portion) shall be adjusted to achieve the same degree of filling of the sieve.

After each sub-portion is sieved, the fines in the collecting pan shall be transferred to a weighing container or if practical the fines may be left in the collecting pan as fines from subsequent sub-portions

---

[3] To be published. (Until ISO 14780 is published, EN 14780 should be used.)

collected. The coarse material retained on the sieve from each sub-portion shall be transferred to another container.

7.2 Weighing

After completing the sieving of all sub-portions of the test portion, weigh the total amount of material which passed through the sieve (the fines fraction) and weigh the total amount of material retained on the sieve (the coarse fraction).

The weighing of the fines fraction can be performed either by weighing the collecting pan with the fines fraction and subtracting the mass of the empty collecting pan, or the fines fraction can be transferred to a tared weighing container for determining the mass of the fraction.

8 Calculations

8.1 Total mass of all fractions

Calculate the total mass of all fractions as the sum of the mass of the fines fraction and the mass of the coarse fraction.

8.2 Proportion of fines

Calculate the *w*-% of fines by dividing the mass of the fraction by the mass of all fractions (as per 8.1) and multiply by 100.

8.3 Quality control

Calculate the difference between the mass of the test portion (as per 8.2) and the total mass of all fractions (as per 8.1) and express the difference in percent of the mass of the test portion. If the difference is larger than 2 *w*-%, the causes for the deviation shall be investigated and the determination shall be repeated. If this is impractical or the difference after repeated determination still exceeds a mass fraction of 2 % of the mass of the test portion, the size of the difference in % mass fraction of the test portion shall be reported together with the fines content as per 8.2.

9 Performance characteristics

For the time being, not enough data are available for a precision statement regarding this test method.

10 Test report

The test report shall include at least the following information:

a) the identification of the laboratory performing the test and the date of the test;

b) the identification of product (or sample) tested;

c) a reference to this International Standard, i.e. ISO 18846;

d) the mass of the test portion (as per 6.2);

e) the result of the test on an as-received basis;

f) the difference between the mass of the test portion and the total mass of all fractions, in percent of the mass of the test portion if the difference exceeds a mass fraction of 2 %;

g) any unusual features noted during the determination, which may affect the result;

h) any deviation from this International Standard, or operations regarded as optional.

IS 18557 (PART 1) : 2024/
ISO 17831-1 : 2015

SUMMARY

Mechanical durability value is important for solid biofuels as it indicates the fuel's resistance to breaking or crumbling during handling, storage, and transport, ensuring quality and performance.

This part of IS 18557 defines a determination method for testing the mechanical durability of pellets.

The standard determines durability by subjecting a test portion to controlled shocks in a rotating test chamber. After tumbling, the durability is calculated based on the mass of the test portion after separation by sieving.

The document outlines the required apparatus, including a pellet tester and sieves, and details the sample preparation and testing procedures.

SOLID BIOFUELS – DETERMINATION OF MECHANICAL DURABILITY OF PELLETS AND BRIQUETTES: PART 1 PELLETS

IS 18557 (PART 1) : 2024

Indian Standard

SOLID BIOFUELS — DETERMINATION OF MECHANICAL DURABILITY OF PELLETS AND BRIQUETTES

PART 1 PELLETS

1 Scope

This part of ISO 17831 defines a determination method for testing the mechanical durability of pellets. The mechanical durability is a measure of the resistance of compressed fuels towards shocks and/or abrasion as a consequence of handling and transportation.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14780, *Solid biofuels — Sample preparation*[1)]

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18134-1, *Solid biofuels — Determination of moisture content — Oven dry method — Part 1: Total moisture — Reference method*

ISO 18134-2, *Solid biofuels — Determination of moisture content — Oven dry method — Part 2: Total moisture — Simplified method*

ISO 18135, *Solid Biofuels — Sampling*[1)]

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 apply.

4 Principle

A test portion is subjected to controlled shocks by collision of pellets against each other and against the walls of a specified rotating test chamber. The durability is calculated from the mass of test portion, after separation by sieving of particles less than 3,15 mm, and the mass of the test portion after tumbling.

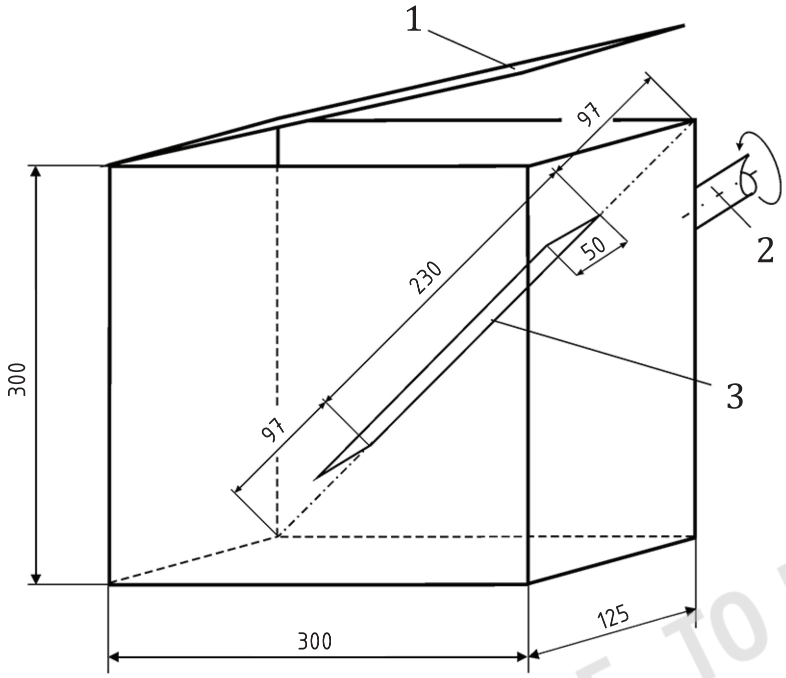
5 Apparatus

5.1 Pellets tester

The structure and dimensions of the pellet tester are shown in Figure 1 (see also Annex A).

[1)] In preparation.

Dimensions in millimetres



Key

1	filling door
2	drive shaft
3	baffle

Figure 1 — Structure of the main parts of the pellet tester

The pellets tester shall consist of a dust tight box with an internal baffle for mixing of the pellets and a driving mechanism.

The box shall have an internal smooth surface and projections such as rivets and screws shall be kept to a minimum and well rounded (alternatively flathead screws can be used). A door can be placed at any side.

Specification of box and inner dimensions:

Material: stainless steel (1,5 ± 0,1) mm thick

Width (300 ± 3) mm

Height (300 ± 3) mm

Breadth (125 ± 1,3) mm

Specification of baffle and dimensions:

Material: stainless steel (1,5 ± 0,1) mm thick

Length: (230 ± 2,3) mm

Width: (50 ± 1,0) mm

The baffle is affixed on a diagonal of one of the 300 mm × 300 mm side of the box. The baffle extends (50 ± 1,0) mm into the box (see Figure 1) and is securely fastened to the back of the box. The edges of the baffle shall not be sharp but rounded to avoid any cutting effect.

The box shall be capable of rotating at a constant speed of (50 ± 2) r/min by means of an electric motor with suitable pulleys or gear in order to avoid vibrations. A rotation counter shall be connected to the drum.

The rotation counter can also be connected to the motor for automatic shut-off after a defined number of rotations.

5.2 Sieve

The sieve shall have a screen with 3,15 mm diameter round holes and suitable for manual screening (see ISO 3310-2). The recommended diameter of sieve is 400 mm or above.

5.3 Balance

The balance shall be capable of reading to the nearest 0,1 g and a weighing capacity of 2 kg.

6 Sample preparation

A laboratory sample of minimum 2 kg material for the determination of mechanical durability shall be obtained in accordance with ISO 18135 and prepared in accordance with ISO 14780. The test sample shall be divided into four equal test samples.

One test sample shall be used for determination of the total moisture content in accordance with ISO 18134-1 or ISO 18134-2.

Simultaneously for the determination of the durability each of the remaining three test samples shall be sieved separately in order to remove the fines (particles less than 3,15 mm) using a sieve as described in 5.2.

The sieving shall be performed by manually shaking each of the test portions in about 5-10 circular movements. The recommended amount of material in the sieve shall be less than 0,8 g/cm^2 of sieve area. This requirement will be achieved for example by using a sample of 1,0 kg on a sieve 400 mm diameter. If a sieve with a different diameter is used, the amount of the material shall be adjusted to achieve the same degree of filling.

The three test samples may be unified and divided into two portions in preparation for the tumbling (7.1).

7 Procedure

7.1 Tumbling procedure

Select one of the test portion of (500 ± 10) g from the unified sample (see Clause 6). For pellets above 12 mm diameter a test portion of (500 ± 50) g is allowed. Weigh the test portion to the nearest 0,1 g and place it in the tumbling box of the pellet tester (see 5.1). Tumble the test portion at (50 ± 2) r/min for 500 rotations. After this, the test portion is removed and manually sieved (7.2) for separation of the fines.

The same procedure shall be performed for the second test portion from the unified sample (see Clause 6).

7.2 Sieving procedure

For sieving using a sieve described in 5.2. Sieving of the test portions after the tumbling procedure shall be done in such a way as to avoid generation of new fines. The sieving shall be performed by shaking each of the previously tumbled test portions one after the other with about 5-10 circular movements. The recommended sieve diameter shall be chosen in order to achieve a load of less than 0,8 g/cm^2 of sieve area (see also Clause 6).

The sieving has to be done completely. Weigh the material remaining on the sieve for each of the tumbled test portions. The pellets durability shall be calculated in accordance with Clause 8.

8 Calculation of the mechanical durability

The mechanical durability of pellets shall be calculated using Formula (1):

$$DU = \frac{m_A}{m_E} \times 100 \qquad (1)$$

where

DU is the mechanical durability, in %;

m_E is the mass of the sieved pellets before the tumbling treatment in g;

m_A is the mass of the sieved pellets after the tumbling treatment in g.

The result for each of the two test portions shall be calculated to two decimal places and the mean value result from the two test portions shall be rounded to the nearest 0,1 percent for reporting.

9 Performance characteristics

9.1 General

Table 1

Durability	Maximum acceptable differences between results	
	Repeatability limit	Reproducibility limits
Durability above or equal to 97,5 %	0,3 % absolute	0,6 % absolute
Durability under 97,5 %	2 % absolute	3 % absolute

9.2 Repeatability

The results of the duplicate determinations (performed within a short period of time, but not simultaneously) in the same laboratory by the same operator using the same apparatus on two representative test portions taken from the same laboratory sample, shall not differ by more than the values given in Table 1 (see References [3], [4], [5], [6], and [7]).

9.3 Reproducibility

The mean value of the results of duplicate determinations, performed in each of two different laboratories on representative test portions taken from the same laborytory sample shall not differ by more than the values given in Table 1 (see References [3], [4], [5], [6], and [7]).

10 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) a reference to this part of ISO 17831, i.e. ISO 17831-1;

d) result of the mechanical durability (as received) as mean value and the moisture content (as received);

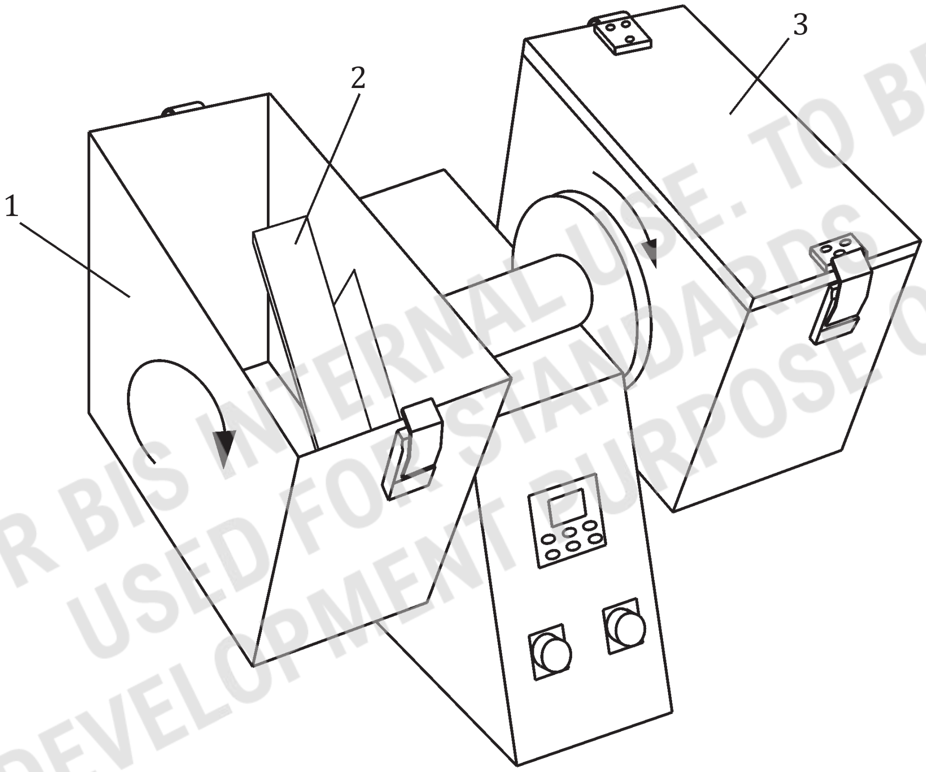
e) result of the mechanical durability (as received) for all individual replications (optional);

f) any unusual features noted during the determination which can affect the result;

g) any operation not included in this part of ISO 17831, or regarded as optional.

Annex A
(informative)

Example of pellets tester with two boxes



Key

1	tumbling box (with lid removed)
2	baffle
3	lid closing a tumbling box

Figure A.1

IS 18557 (PART 2) : 2024/
ISO 17831-2 : 2015

SUMMARY

Mechanical durability value is important for solid biofuels as it indicates the fuel's resistance to breaking or crumbling during handling, storage, and transport, ensuring quality and performance.

This part of IS 18557 defines a determination method for testing the mechanical durability of briquettes.

The standard determines durability by subjecting a test portion to controlled shocks in a rotating test chamber, which is then calculated from the mass of the sample remaining after separation of abraded and fine broken particles.

The document outlines the required apparatus, including a briquette tester and sieves, and details the sample preparation and testing procedures.

SOLID BIOFUELS – DETERMINATION OF MECHANICAL DURABILITY OF PELLETS AND BRIQUETTES: PART 2 BRIQUETTES

Indian Standard

SOLID BIOFUELS — DETERMINATION OF MECHANICAL DURABILITY OF PELLETS AND BRIQUETTES

PART 2 BRIQUETTS

1 Scope

This part of ISO 17831 defines a method for determining the mechanical durability of briquettes. The mechanical durability is a measure of the resistance of compressed fuels towards shocks and/or abrasion as a consequence of handling and transportation.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 14780, *Solid biofuels — Sample preparation*[1)]

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18134-1, *Solid biofuels — Determination of moisture content — Oven dry method — Part 1: Total moisture — Reference method*

ISO 18134-2, *Solid biofuels — Determination of moisture content — Oven dry method — Part 2: Total moisture — Simplified method*

ISO 18135, *Solid Biofuels — Sampling*[1)]

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 16559 apply.

4 Principle

The test portion is subjected to controlled shocks by collision of briquettes against each other and against the walls of a specified rotating test chamber. The durability is calculated from the mass of the sample remaining after separation of abraded and fine broken particles.

[1)] In preparation.

5 Apparatus

5.1 Briquette tester

The briquette tester (durability drum) shall be a cylindrical steel drum with a nominal volume of 160 l, having the following dimensions (see Figure 1):

— internal length or depth: (598 ± 8) mm;

— internal diameter: (598 ± 8) mm.

The drum shall be made of minimum 1 mm steel plate. The internal surface area of the drum shall be smooth and any disturbances of the surface such as ridges or furrows shall be avoided.

The durability drum shall be equipped with a rectangular steel baffle having the following dimensions:

— length: (598 ± 8) mm;

— height: (200 ± 2) mm;

— thickness: 2 mm.

The baffle shall be welded on its length to the full height of the internal curved surface of the drum, parallel to the axis of the drum and perpendicular to the tangent of the curve. The baffle tip shall be shaped in a smooth curve of 90° to obtain a rim perpendicular to the baffle. The curve shall have a radius of r = 10 mm and the curved tip shall have a total vertical height of 30 mm. The direction of rotation and further details are illustrated in Figure 1.

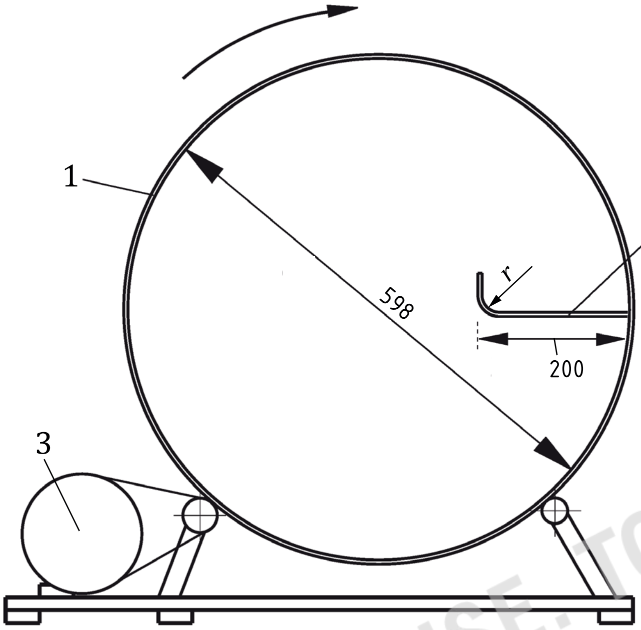
The drum is open on one of the ends. This opening shall be fitted with a dustproof lid of the same diameter as the internal diameter of the drum (598 ± 8) mm. When the lid is in position, it shall be approximately flush with the side of the cylinder. This lid shall be made of minimum 1 mm steel plate.

It shall be possible to fasten the lid securely by four rotating bolts (or any other suitable mechanism) fixed on the external part of the drum.

The drum shall be capable of being constantly driven at (21 ± 0,1) r/min by an electric motor, by suitable pulleys or gearings, in order to avoid vibrations. A rotation counter should be connected to the drum.

The rotation counter can also be connected to the motor so that the latter is automatically switched off after a defined number of rotations.

Dimensions en millimètres



Key

1 drum

2 baffle

3 motor

Figure 1 — Principle of the durability drum

5.2 Sieve

Depending on the briquette diameter, a sieve with a metal wire cloth conforming to ISO 3310-1 shall be chosen so that the aperture size is approximately equivalent to 2/3 of the briquettes diameter or diagonal but not exceeding 45 mm. The sieve is selected from the series between 16 mm and 45 mm in accordance with ISO 3310-1. The sieve shall have a diameter of 400 mm or above.

5.3 Balance

A balance with a weighing capability of at least 2 kg and capable of measuring the mass to the nearest 0,1 g.

6 Test sample preparation

The laboratory sample used for the determination of mechanical durability shall be obtained, and if necessary, divided in mass in accordance with ISO 18135 and prepared in accordance with ISO 14780. The size of the laboratory sample shall conform to the requirements of this part of ISO 17831 (depending on the nominal top size) but shall be at least 25 kg. For briquettes of type A, as defined in Table 1, the minimum required laboratory sample mass shall be 10 kg.

Divide the laboratory sample to obtain two test samples: one to be used for moisture content determination (5 kg), as received, the other for the mechanical durability test. The moisture content as received shall be determined on a non-sieved test sample simultaneously with the durability test; moisture shall be determined in accordance to ISO 18134-1 or ISO 18134-2.

The test sample for durability testing shall be stored in airtight containers to avoid moisture changes. While the test is being conducted, the test sample shall be at room temperature.

A test portion for a durability test shall be selected based on the average mass of a single briquette. The required size is determined after assigning the briquette to one of three briquette types A, B or C as described in Table 1. For this assignment, select 15 undestroyed briquettes from the test sample randomly. Then, weigh each briquette individually to the nearest 0,1 g using the balance as defined in 5.3. Calculate the average briquette mass of the 15 briquettes at moisture content as received. Then, select the applicable size of a test portion in accordance with Table 1. Do not cut or break any briquette of the test portion.

NOTE A briquette is undestroyed if all physical dimensions which define its shape are clearly recognizable. Abrasion and broken edges are not regarded as destruction. Cylindrical briquettes where the actual length is a result of natural breaking instead of cutting are also regarded as undestroyed.

Table 1 — Size of a test portion for briquette types A, B or C

Briquette type	Description	Size of a test portion
A	briquettes with a mass lower than 0,5 kg (as received)	≥2,0 kg (add max. 1 additional briquette to meet the requirement)
B	briquettes with mass of (0,5 to 1,0) kg (as received)	2 briquettes
C	briquettes with a mass higher than 1,0 kg (as received)	1 briquette

For briquettes of type A, prepare two test portions for the determination of durability. For briquettes of type B and type C, prepare five test portions for the determination.

The test portions shall not contain any small, broken particles. Small particles shall be separated from the test portion by the use of the sieve mentioned in 5.2 or by sorting out briquettes manually.

7 Procedure

7.1 Tumbling procedure

Select a test portion as described in Table 1. Weigh the test portion to the nearest 0,1 g and place it in the durability drum of the briquette tester (5.1). Tumble the test portion in the drum at (21 ± 0,1) r/min for 5 min (i.e. for 105 rotations ± 0,5 rotations). After this, the test portion is removed and manually sieved (7.2) for separation of the fines.

For briquettes of type A, as defined in Table 1, perform the same procedure for a second test portion.

For briquettes of type B or type C, as defined in Table 1, perform the same procedure with the other four test portions in order to achieve five test portion measurements in total.

NOTE Due to inconsistent sample sizes a comparison of results for briquettes of type A, B and C is not recommended.

7.2 Sieving procedure

For sieving use a sieve described in 5.2. The sieving of the test portions, after the tumbling procedure shall be done in such a way as to avoid generation of new particles. The sieving shall be performed by shaking the previously tumbled test portions one after the other in about 5 to 10 circular movements.

The sieving has to be done completely. The material remaining on the sieve shall be weighed and calculated as percent of the material loaded into the briquette tester. The durability of briquettes is defined according to Clause 8.

8 Calculation of the mechanical durability

The mechanical durability of briquettes shall be calculated using Formula (1):

$$DU = \frac{m_A}{m_E} \times 100 \quad (1)$$

where

DU is the mechanical durability, in percent;

m_E is the mass of sieved briquettes before the drum treatment, in g;

m_A is the mass of sieved briquettes after the drum treatment, in g.

The result for each test portion shall be calculated to two decimal places and the mean result for all test portions for a particular briquette type as per Table 1 shall be rounded to the nearest 0,1 % for reporting.

9 Performance characteristics

For the time being, not enough data is available for a precision statement regarding this test method.

10 Test report

The test report shall include at least the following information:

a) identification of laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) reference to this part of ISO 17831, i.e. ISO 17831-2;

d) result of the mechanical durability (at moisture as received) as mean value and the moisture content (as received);

e) results of the mechanical durability (at moisture as received) for all individual test portions;

f) shape and dimensions of the briquettes;

g) assignment of briquette to type A, B or C in accordance with Table 1;

h) any unusual features noted during the determination, which can affect the result;

i) any deviation not included in this International Standard, or regarded as optional.

IS 17832 : 2022/
ISO 16948 : 2015

SUMMARY

The reliable determination of nitrogen, carbon and hydrogen content is crucial for quality control, input parameters for combustion calculations, assessing nitrogen's environmental impact related to NOx emissions, calculating net calorific value using hydrogen content, and determining CO2 emissions based on carbon content.

This standard describes a method for the determination of total carbon, hydrogen and nitrogen contents in solid biofuels.

The method involves combusting a sample in oxygen or an oxygen/carrier gas mixture, converting it into ash and gaseous products like carbon dioxide, water vapor, and nitrogen. These products are then treated to remove interfering components, and the mass fractions of carbon dioxide, water vapor, and nitrogen are determined quantitatively using instrumental gas analysis.

SOLID BIOFUELS – DETERMINATION OF TOTAL CONTENT OF CARBON, HYDROGEN AND NITROGEN

IS 17832 : 2022

Indian Standard

SOLID BIOFUELS — DETERMINATION OF TOTAL CONTENT OF CARBON, HYDROGEN AND NITROGEN

1 Scope

This International Standard describes a method for the determination of total carbon, hydrogen and nitrogen contents in solid biofuels.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 14780[1], *Solid Biofuels — Sample preparation*

ISO 16993, *Solid biofuels — Conversion of analytical results from one basis to another*

3 Terms and definitions

For the purposes of this document the terms and definitions given in ISO 16559 and the following apply.

3.1
reference material
RM
material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials

3.2
certified reference material
CRM
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

3.3
NIST standard reference material
SRM
CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterisations and provides information regarding the appropriate use(s) of the material

Note 1 to entry: The National Institute of Standards and Technology (NIST), known between 1901 and 1988 as the National Bureau of Standards (NBS), is a measurement standards laboratory, also known as a National Metrological Institute (NMI), which is a non-regulatory agency of the United States Department of Commerce

---

[1] To be prepared.

4 Principle

A known mass of sample is burnt in oxygen, or in an oxygen/carrier gas mixture, under conditions such that it is converted into ash and gaseous products of combustion. These consist mainly of carbon dioxide, water vapour, elemental nitrogen and/or oxides of nitrogen, oxides and oxyacids of sulfur and hydrogen halides. The products of combustion are treated to ensure that any hydrogen associated with sulfur or halides products of combustion are liberated as water vapour. Oxides of nitrogen are reduced to nitrogen, and those products of combustion which would interfere with the subsequent gas-analysis procedures are removed. The carbon dioxide, water vapour and nitrogen mass fractions of the gas stream are then determined quantitatively by appropriate instrumental gas analysis procedures.

5 Reagents and calibration substances

5.1 General

WARNING — Care should be exercised when handling reagents, many of which are toxic and corrosive.

Unless otherwise stated, use only reagents and calibration standards of recognized analytical grade for the analysis.

5.2 Carrier gas

The carrier gas used is helium or another suitable gas as specified by the instrument manufacturer.

5.3 Oxygen

Oxygen is used as specified by the instrument manufacturer.

5.4 Additional reagents

Additional reagents are of types and qualities as specified by the instrument manufacturer.

5.5 Calibration substances

Examples of pure organic substances suitable for calibration are given in Table 1.

Table 1 — Examples of suitable calibration substances and their theoretical C, H and N contents

Name	Formula	% C	% H	% N
Acetanilide	C_8H_9NO	71,1	6,7	10,4
Atropin	$C_{17}H_{23}NO_3$	70,6	8,0	4,8
Benzoic acid	$C_7H_6O_2$	68,8	5,0	0,0
Cystine	$C_6H_{12}N_2O_4S_2$	30,0	5,0	11,7
Diphenyl amine	$C_{12}H_{11}N$	85,2	6,6	8,3
EDTA	$C_{10}H_{16}N_2O_8$	41,1	5,5	9,6
Phenylalanine	$C_9H_{11}NO_2$	65,4	6,7	8,5
Sulfanil amide	$C_6H_8N_2O_2S$	41,8	4,7	16,3
Sulfanilic acid	$C_6H_7NO_3S$	41,6	4,1	8,1
TRIS	$C_4H_{11}NO_3$	39,7	9,2	11,6

The materials shall be dry and of high purity, i.e. more than 99,9 %. For calibration purposes, the contents of C, H and N according to the certificate of the materials shall be used, not the theoretical contents. Other pure materials can be used provided that they meet the requirement of this standard.

5.6 Use of Certified Reference Materials (CRM or SRM)

Use certified reference materials, issued by an internationally recognized authority, to check if the accuracy of the calibration meets the required performance characteristics. Examples of certified reference materials are: NBS 1573 tomato leaves and NBS 1575 pine needles.

When, due to matrix effects or concentration range limitations, no good recoveries for the certified reference materials can be obtained, calibration with at least two CRM or SRM materials, may solve these problems. In that case CRM or SRM materials other than used for the calibration shall be used for verification purposes.

NOTE A CRM or SRM is prepared and used for three main purposes:

a) to help develop accurate methods of analysis;

b) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit;

c) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

6 Apparatus

No specific design of systems is presented here because there are a range of components and configurations available, which can be used to carry out the test method satisfactorily.

The apparatus shall, however, meet the following functional requirements:

a) The conditions of combustion of the sample shall be such that all of the carbon (including that in mineral carbonates), the hydrogen (including that in the water of constitution of the minerals), and the nitrogen present, shall be converted into carbon dioxide, water vapour (except for hydrogen associated with oxyacids of sulfur and volatile halides), and gaseous nitrogen and/or oxides of nitrogen respectively.

b) The combustion gases, or a representative aliquot, shall be treated to remove and/or separate out any components which would subsequently interfere with the detection and measurement of the carbon dioxide, water vapour or nitrogen in the gas stream.

c) Hydrogen present as hydrogen halides or sulfur oxyacids shall be liberated, as water vapour, into the gas stream prior to determination of water vapour content.

d) Any nitrogen oxides produced by the combustion process shall be reduced to nitrogen prior to presentation to the detection system.

e) The detection systems shall provide responses that correlate directly with the concentrations of the combustion gases, over the full range applicable and preferably in a linear manner.

f) If a nonlinear response is provided by a detection system, it shall include provisions for evaluating that response in a manner which correlates accurately with the concentration of the combustion gas.

g) It shall include a means of displaying the detector responses or of calculating and presenting the concentrations of carbon, hydrogen and nitrogen in the sample following the input of other appropriate data as necessary.

7 Preparation of the test sample

The test sample is the general analysis sample with a nominal top size of 1 mm or less, prepared in accordance with ISO 14780.

The moisture content of the test sample shall be determined concurrently by the method described in ISO 18134-3, using another portion of the test sample.

NOTE 1 For some instruments it may be necessary to prepare a test sample with a lower nominal top size than 1 mm, e.g. 0,25 mm, in order to keep the desired precision. For "new products" an adequate particle size should be determined by validation experiments.

NOTE 2 For some types of instruments it is necessary to carry out the determination of hydrogen on dried analysis samples to prevent erroneous low results. For a determination on dry sample, the analysis sample is dried as described in ISO 18134–3 immediately before the determination. When using dried samples there is a risk of obtaining erroneous low carbon results.

8 Procedure

8.1 Preparation of the test portion

Weigh, to the nearest 0,1 % (relative), a quantity of the test sample recommended by the instrument manufacturer as appropriate to the type of instrumentation and the expected concentrations of carbon, hydrogen and nitrogen. The test portion shall be weighed directly into the sample capsule in the case of a micro- or semi-micro analyser. Otherwise it may be weighed directly or transferred from a suitable weighing container.

8.2 Calibration of the apparatus

When the analytical system is evaluated for the first time, establish a calibration function for the measurement in accordance with the manufacturers' instructions. Adjust the established calibration function during the analysis if necessary. Check the performance of the instrument using the accepted standard procedures like replicate analysis, use of CRM and or Standard Reference Material (SRM), control samples and create control charts. The calibration and quality control scheme shall be organized and maintained in such a way that the required uncertainty of measurement can be obtained. The results of the validation study of BioNorm2 (Annex A) demonstrate what is achievable with commercial instruments that are used by experienced laboratories.

8.3 Analysis of test samples

Analyse test portions (8.1) of the test samples in accordance with the manufacturer's instructions.

At intervals between batches of samples, analyse a calibration substance (5.5), CRM (5.6) or an internal laboratory control sample.

The control sample shall have a carbon, hydrogen and nitrogen content comparable to the samples.

Example of an analysis run on an automated CHN analyser:

a) 2 dummy samples to condition the instrument

b) 3 samples of calibration substance to check or carry out the calibration

c) 1 laboratory control sample, to ensure the instrument performance for the actual type of samples

d) 9 samples (in duplicate)

e) 2 samples of calibration substance to check the calibration

f) Repetition of d) to e) until all samples are processed

g) 1 laboratory control sample

Calibration checks during an analysis run may be used for adjustment of the established calibration function cf. 8.2. Adjustments should only compensate for small drifts in the instrument performance; relative differences of more than 10 % usually will be an indication of possible malfunction of the instrument.

9 Expression of results

The total carbon, hydrogen and nitrogen contents of the biofuel sample, as analysed, shall be recorded as a percentage by mass.

Report the results on dry basis, as the mean of duplicate determinations. The following equations shall be used for the calculations to dry basis:

For the carbon content: $$C_{\mathrm{d}} = C_{\mathrm{ad}} \times \frac{100}{100 - M_{\mathrm{ad}}} \qquad (1)$$

For the nitrogen content: $$N_{\mathrm{d}} = N_{\mathrm{ad}} \times \frac{100}{100 - M_{\mathrm{ad}}} \qquad (2)$$

For the hydrogen content: $$H_{\mathrm{d}} = \left(H_{\mathrm{ad}} - \frac{M_{\mathrm{ad}}}{8{,}937} \right) \times \frac{100}{100 - M_{\mathrm{ad}}} \qquad (3)$$

where

d is dry basis;

ad is as determined;

M_{ad} is the moisture content of the general analysis sample when analysed.

The constant factor 8,937 is for calculating the hydrogen concentration in the water that is present in the sample. The factor is obtained from the molar formula of water (H_2O) and the atomic weight of hydrogen (1,008) and oxygen (15,999 4).

The results may be calculated to an as received basis according to ISO 16993.

10 Performance characteristics

The achievable performance of the method is given in Annex A showing the results obtained by a European intercomparison study carried out for a sample of wood chips and a sample of an exhausted olive residue. The content of carbon and hydrogen of both samples represents the typical range for solid biofuels. Concerning the content of nitrogen in solid biofuels the samples of wood chips and exhausted olive residues represent the lowest and highest values expected, respectively.

11 Test report

The test report shall contain at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (sample) tested;

c) reference to this International Standard (ISO 16948:2015);

d) method for the determination;

e) the results of the test including the basis in which they are expressed, as indicated in Clause 9;

f) any unusual features noted during the test procedure;

g) any operation not included in this International Standard, or regarded as optional.

Annex A
(informative)

Performance data

The round robin was carried out by laboratories in Austria, Belgium, Denmark, Finland, Germany, Ireland, Italy, The Netherlands, Spain, Sweden and the United Kingdom. The variety of instruments and other analytical conditions were used in accordance with the quality parameters specified in the method.

The tests were carried out using two samples, wood chips and exhausted olive residues produced in the EU-project BioNorm according to ISO 14780. The sample "wood chips" was made of German coniferous wood chips; the chips were dried and milled to 1 mm by means of cutting mill. The sample "exhausted olive residues" was obtained from olive oil industry in Spain from a typical outdoor storage facility. In the original sample stones and other natural impurities were present. These impurities and stones were removed manually and the sample was prepared from the residues in two steps using a coarse cutting mill equipped with a 10 mm sieve and a laboratory cutting mill equipped with WC cutting tools and a 1 mm sieve.

All data are reported on dry basis.

The performance data according to ISO 5725-2 are presented in Tables A.1, A.2 and A.3.

NOTE 1 See Table A.1 for definition of the symbols used in the tables of A.1 to A.3.

NOTE 2 A guideline can be found in to ISO 16993:2015, Annex C on how to use these validation parameters.

Table A.1 — Performance data for Carbon (C)

Sample	n	l	o %	x % m/m	s_R % m/m	CV_R %	s_r % m/m	CV_r %
wood chips	27	128	1,5	50,3	0,55	1,1	0,14	0,29
exhausted olive residue	26	123	1,6	48,0	0,55	1,2	0,27	0,56
Definition symbols								
n	is the number of laboratories after outlier elimination							
l	is the number of outlier free individual analytical values							
o	is the percentage of outlying values from replicate determination							
x	is the overall mean							
s_R	is the reproducibility standard deviation							
CV_R	is the coefficient of the variation of the reproducibility							
s_r	is the repeatability standard deviation							
CV_r	is the coefficient of the variation of the repeatability							

Table A.2 — Performance data for Hydrogen (H)

Sample	n	l	o	x	s_R	CV_R	s_r	CV_r
			%	% m/m	% m/m	%	% m/m	%
wood chips	28	135	0	6,1	0,36	5,9	0,07	1,2
exhausted olive residues	24	115	0	5,7	0,32	5,77	0,06	1,1

Table A.3 — Performance data for Nitrogen (N)

Sample	n	l	o %	x % m/m	s_R % m/m	CV_R %	s_r % m/m	CV_r %
wood chips	18	84	6,7	0,10	0,04	30	0,01	7,4
exhausted olive residues	25	115	7,3	1,40	0,11	8,1	0,04	3,2

NOTE 1 The high s_R value of 0,04 % m/m concerning the determination of nitrogen in the wood chip sample is obtained due to a contribution from elemental analysers with a small sample intake (typical 2 mg to 5 mg) in combination with a thermal conductivity detector and with an insufficient removal of the air in the sample capsule.

The performance at low levels can be improved using the Kjeldahl method or element analyzers working with higher sample intake and/or a high sensitive nitrogen detection.

NOTE 2 The high s_R value of about 0,36 % m/m concerning the determination of hydrogen could be due to the following conditions concerning the procedures of the participating laboratories:

— determination on the moist (as received) analysis sample using a micro-analyser with the problem stated in the NOTE 2 of Clause 7;

— determination on the moist analysis sample without correction for the moisture-hydrogen included;

— determination on dried analysis sample material which between the drying and the analysis have absorbed moisture.

An s_R value of 0,2 % m/m should be obtainable when using a correct procedure.

IS 17833 : 2022/
ISO 16994 : 2016

SUMMARY

Sulfur and chlorine are present in solid biofuels in varying concentrations. During the combustion process, they are usually converted to sulfur-oxides and chlorides. The presence of these elements and their reaction products can contribute significantly to corrosion and to environmentally harmful emissions.

This standard describes methods for the determination of the total sulfur and total chlorine content in solid biofuels. It specifies two methods for decomposition of the fuel and different analytical techniques for the quantification of the elements in the decomposition solutions. The use of automatic equipment is also included in the standard, provided that a validation is carried out as specified and that the performance characteristics are similar to those of the method described in the standard.

SOLID BIOFUELS – DETERMINATION OF TOTAL CONTENT OF SULFUR AND CHLORINE

Indian Standard

SOLID BIOFUELS — DETERMINATION OF TOTAL CONTENT OF SULFUR AND CHLORINE

1 Scope

This International Standard describes methods for the determination of the total sulfur and total chlorine content in solid biofuels. This International Standard specifies two methods for decomposition of the fuel and different analytical techniques for the quantification of the elements in the decomposition solutions. The use of automatic equipment is also included in this International Standard, provided that a validation is carried out as specified and that the performance characteristics are similar to those of the method described in this International Standard.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 14780[2], *Solid biofuels — Sample preparation*

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 16967:2015, *Solid biofuels — Determination of major elements — Al, Ca, Fe, Mg, P, K, Si, Na and Ti*

ISO 18125[2], *Solid biofuels — Determination of calorific value*

ISO 18134-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

CEN Guide 13:2008, *Validation of environmental test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

3.1
reference material
RM
material or substance one or more of whose property values are sufficiently homogeneous and well-established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials

---

[2] To be published.

3.2
certified reference material
CRM
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

3.3
NIST standard reference material
SRM
CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterisations and provides information regarding the appropriate use(s) of the material

4 Principle

4.1 General

The determination of total sulfur and total chlorine content is performed in two steps (4.2 and 4.3) or by using automatic equipment (see 4.4).

4.2 Decomposition of the biofuel

- Combustion in an oxygen atmosphere in a combustion vessel and absorption of the acidic gas components in an absorption solution (method A).
- Digestion in closed vessels as described in ISO 16967:2015, Part A (method B).

4.3 Determination of sulfate and chloride in the decomposition solution

- Ion chromatography, in accordance with the principles of ISO 10304-1.
- ICP, in accordance with the principles of ISO 11885 (determination as sulfur and chlorine).

4.4 Automatic equipment

Automatic equipment may be used when the method is validated with biomass reference samples of an adequate biomass type. If automatic equipment is used, sulfur and chlorine compounds may be detected as gaseous components (e.g. by infrared methods). Examples for automatic analysers include elemental analysers, AOX-analysers.

If automatic equipment or X-ray fluorescence are used, the method shall be validated for the respective main origin based biomass group (see ISO 17225-1:2014, Table 1: woody biomass, herbaceous biomass, or fruit biomass) according to CEN Guide 13:2008, Clause 3 validation of alternative methods with one of the following two approaches:

- full validation as applies to reference methods;
- relative validation in which a comparison is made to the reference method, e.g. by participation in inter-laboratory comparison tests.

NOTE Equipment validated only with, for example, straw reference materials, is not automatically suitable for the determination of sulfur and chlorine in, for example, wood samples, because of the usually significant lower concentrations of the elements in wood and/or the unknown influences of the different matrix.

5 Reagents

The reagents listed below relate to the digestion method specified in 8.1.1 (method A). Reagents for the digestion method B and the different detection methods according to 8.2 are specified in the corresponding standards.

5.1 General

All reagents shall be at least of analytical grade and suitable for their specific purpose. Particularly, they shall contain negligible amounts of chlorine and sulfur, i.e. amounts that do not contribute significantly to the determination.

5.2 Water, de-ionized water will normally fulfil the requirements of 5.1

5.3 Oxygen, pure oxygen with an assay of at least 99,5 % (V/V).

5.4 Combustion aid/enhancer, various substances may be used, e.g. benzoic acid, paraffin oil, acetobutyrate capsules, polyethylene bags.

5.5 Use of certified reference materials (CRM or SRM).

Use certified reference materials to check if the accuracy of the calibration meets the required performance characteristics. Examples of certified reference materials are SRM 1570 spinach leaves, SRM 1571 orchard leaves, SRM 1573 tomato leaves, and SRM 1575 pine needles.

When, due to matrix effects or concentration range limitations, no good recoveries for the certified reference materials can be obtained, calibration with at least two CRM or SRM materials, could solve these problems (for example, CRM 101 spruce needles and CRM 100 beech leaves). In that case, CRM or SRM materials other than used for the calibration shall be used for verification purposes.

NOTE A CRM or SRM is prepared and used for the following three main purposes:

a) to help develop accurate methods of analysis;

b) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit;

c) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

6 Apparatus

6.1 General

6.1.1 Analytical balance, with a resolution of at least 0,1 mg.

6.1.2 General laboratory equipment, such as volumetric flasks and measuring cylinders.

6.2 Method A

6.2.1 Pellet press, capable of applying a force of 0,1 Nm, equipped with a die to press a pellet with a diameter of about 13 mm.

6.2.2 Combustion vessel, suitable for the determination of sulfur and chlorine.

The combustion vessel may be the same as used for the determination of the calorific value (see ISO 18125).

The combustion vessel shall not leak during the test and shall permit a quantitative recovery of the liquid. Its inner surface may be made of stainless steel or any other material that will not be affected by the combustion process or products.

Note that not all combustion vessels of calorimeters can be used because the principle of construction, the materials used for construction, or the surfaces in the combustion vessels may adsorb or react with the acidic gases formed during combustion or it may not be possible to clean the combustion vessel completely.

6.3 Method B

Sample digestion vessels and an associated heating device (see ISO 16967).

7 Preparation of the test sample

The test sample is the general analysis test sample with a nominal top size of 1 mm prepared in accordance with ISO 14780.

NOTE It might be necessary to prepare a test sample with a lower nominal top size than 1 mm (e.g. 0,25 mm) in order to keep the stated precision and repeatability limits. If a nominal size below 1 mm is used, the correctness of results is to be controlled by a CRM prepared to the nominal size used for the sample.

As the results are to be calculated on dry basis, the moisture content of the test sample shall be determined concurrently by the method described in ISO 18134-3, using another portion of the test sample.

8 Procedure

8.1 Decomposition

8.1.1 Method A: Combustion in a closed combustion vessel

Solid biofuel samples are usually combusted in a pellet form due to the low density and their combustion behaviour.

— Take a sample of approximately 1 g (unless the combustion vessel is designed for other sample amounts).

— Press the sample with a suitable force to produce a compact unbreakable pellet that is weighed to 0,1 mg. If the calorific value is determined simultaneously, the sample amount may eventually be adjusted according to the specification in ISO 18125.

— Transfer the sample into a quartz glass or metal crucible.

The combustion may be carried out using a combustion aid (5.5).

a) Liquid combustion aid: After the mass of the sample pellet is determined, the auxiliary liquid material shall be added drop-wise on the pellet placed in the crucible (allowing the liquid to be adsorbed); the added amount has to be determined exactly by weighing.

b) Combustion bag or capsule: The sample may be filled in powdered form into a combustion bag or capsule with precisely known weight. The sample mass is calculated by weighing the sample in the combustion bag or capsule and subtracting the mass of the bag or capsule.

c) Solid combustion aid: After the mass of the sample is determined, add an appropriate amount of the aid (e.g. benzoic acid) and determine the added amount of the aid exactly by weighing. Mix the sample and the aid carefully and prepare a pellet of the whole mix as described above. Ensure that the weight of the pellet equals the weight of the sample and the weight of the aid.

- Add 1 ml water into the combustion vessel (also see ISO 18125 if the calorific value is determined simultaneously). Alternatively, the water can be omitted or more water (up to 5 ml) can be used. If the content of chlorine or sulfur exceeds a mass fraction of 2 %, alkaline solutions may be used to neutralize the acidic compounds produced. When ion chromatography is used for determination, the absorption solution may be the mobile phase, e.g. a carbonate/bicarbonate solution. In all cases, the calibration of the method and the blank tests has to be done with the same amount and the same kind of receiving solution.
- Place the crucible in position and arrange the firing wire. Assemble the combustion vessel and tighten the cover securely. Before ignition, the combustion vessel is filled with 30 bar oxygen.

NOTE 1 If the chlorine content of the sample is very low, the cotton thread usually used for ignition can contribute significantly to the measured chlorine content. This can be avoided by using highly pure combustible sample holder without cotton threats.

NOTE 2 The combustion vessel can be flushed with oxygen to reduce the nitrous oxide formation during the combustion process especially when the concentration of sulfur and chlorine is determined by ion chromatography (some peaks in the chromatogram might not be separated satisfactorily).

- After combustion, release the combustion vessel pressure slowly before opening it.
- Transfer the content of the combustion vessel to a volumetric flask (50 ml or 100 ml).
- Thoroughly rinse the combustion vessel, the cover, and the crucible, including the combustion residues in the crucible, with water and collect all the rinse water carefully. Transfer it into the volumetric flask and fill the flask to volume. While rinsing, take care that the ash that was formed after the combustion is collected in the flask as well.

If the sample contains high concentrations of sulfur and/or chlorine (>2 %), the combustion gas should be let through a gas washing bottle with a disk to ensure that all acidic gas components are dissolved. The solution from this gas washing bottle can be combined with the combustion vessel washings or can be analysed separately from the solution of the combustion vessel.

NOTE 3 If the sample contains high amounts of ash (>5 %), then chlorine and sulfur can be trapped in the combustion residues. In that case, the combustion residue can be analysed for the chlorine and sulfur content, e.g. with XRF or an ash fusion procedure. Alternatively, a low sample intake in combination with a combustion aid can be used.

NOTE 4 Special care is necessary when samples with low contents of chlorine and/or sulfur (e.g. virgin wood) are analysed after samples with high contents of these elements (e.g. herbaceous samples or waste samples). The most efficient way of cleaning the combustion vessel is multiple combustions of pure benzoic acid (see 8.1.3 blank test).

For some subsequent analytical methods, a chemical treatment of the solution is necessary before filling up to volume. Some analytical methods require a filtered solution.

The calorific value may be determined simultaneously. In this case, the requirements of ISO 18125 shall be observed. The content of other halogens (fluoride, bromide, and iodide) can be determined by a similar method (see EN 14582).

8.1.2 Method B: Digestion in a closed vessel

The method for the digestion in closed vessels is described in ISO 16967. For the determination of chlorine, an amount of 0,8 ml H_2O_2 per 100 mg of sample shall be used for the digestion. This larger amount compared to ISO 16967 is used to avoid losses of chlorine.

8.1.3 Blank test

Carry out a blank test, using the same procedures and methods as described in 8.1.1 (method A) or 8.1.2 (method B), respectively, using benzoic acid for method A. This assesses both the contents of the elements in the reagents and any contamination from equipment and the laboratory atmosphere. This contribution shall not be quantitatively significant.

The measured blank value has to be subtracted from the sample value. At high element level, the blank should be less than 10 % of the sample content. For low element level (a content below 500 mg/kg in the sample), it is adequate that the contents of the elements in the blank solution are 30 % or less of the contents of the elements in the sample solution.

8.2 Detection methods

8.2.1 Ion chromatography

Ion chromatography is the recommended method for the detection of sulfate and chloride. The determination should be according to the principles of ISO 10304-1.

NOTE The solution obtained from the digestion can be filtered using a syringe equipped with a 0,45 µm pore size filter tip to avoid damage of the ion chromatograph.

8.2.2 Other detection methods

The following methods are standardized at international levels and can be used provided that they have been validated and that the performance characteristics are similar to those of the method described in this International Standard.

Table 1 — Other methods for the detection of sulfate and chloride

Method	Cl	S	References (examples)
ICP	X	X	ISO 11885
Photometric (colourimetric)	X		DIN 51727
Turbidimetric		X	ASTM D516-07
Coulometric	X		DIN 38405-1 (method D1–3)
Potentiometric titration	X		DIN 38405-1 (method D1–2)

8.3 Calibration of the apparatus

When the analytical system is evaluated for the first time, establish a calibration function for the measurement in accordance with the manufacturers' instructions. Adjust the established calibration function during the analysis, if necessary. Check the performance of the instrument using the accepted standard procedures like replicate analysis, use of standard reference material (SRM) and/or CRM, control samples and create control charts. The calibration and quality control scheme shall be organized and maintained in such a way that the required uncertainty of measurement can be obtained. The results of the validation study of BioNorm2 (see Annex A) demonstrates what is achievable with commercial instruments that are used by experienced laboratories.

8.4 Analyses of the decomposition solutions

Analyse test portions of the digests in accordance with the manufacturer's instructions.

9 Expression of results

9.1 General

The results shall be reported as the mean of duplicate determinations. The results shall be calculated on dry basis according to 9.2 and 9.3. The results may be calculated to other bases, e.g. to as-received basis according to ISO 16993.

9.2 Total chlorine

The total content of chlorine in the sample on dry basis, $w_{\mathrm{Cl,d}}$, expressed in mass fraction (%) , is given by Formula (1):

$$w_{\mathrm{Cl,d}} = \frac{(c - c_0) \times V}{m} \times 100 \times \frac{100}{(100 - M_{\mathrm{ad}})} \tag{1}$$

where

c	is the concentration of chloride in the solution, in mg/l;
c_0	is the concentration of chloride in the solution of the blank experiment, in mg/l;
V	is the volume of the solution, in l;
m	is the mass of the test portion used, in mg;
M_{ad}	is the moisture content in the analysis test sample, in mass fraction (%).

9.3 Total sulfur

The total content of sulfur in the sample on dry basis, $w_{\mathrm{S,d}}$, expressed in mass fraction (%), is given by Formula (2):

$$w_{\mathrm{S,d}} = \frac{(c - c_0) \times V}{m} \times 0{,}3338 \times 100 \times \frac{100}{(100 - M_{\mathrm{ad}})} \tag{2}$$

where

c	is the concentration of sulfate in the solution, in mg/l;
c_0	is the concentration of sulfate in the solution of the blank experiment, in mg/l;
V	is the volume of the solution, in l;
m	is the mass of the test portion used, in mg;
0,3338	is the stoichiometric ratio of the relative molar masses of sulfur and sulfate;
M_{ad}	is the moisture content in the analysis test sample, in mass fraction (%).

10 Performance characteristics

The achievable performance of the method is given in Annex A showing the results obtained by a European comparison study carried out for a sample of wood chips and a sample of an exhausted olive residue. These two samples represent the extremity of the method. The wood chip sample represents samples with low contents of sulfur and chlorine and the olive residue samples, with high amounts of sulfur and chlorine.

11 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of sample tested;

c) a reference to this International Standard, i.e. ISO 16994:2016;

d) methods used for digestion and for determination;

e) results of the test including the basis in which they are expressed, as indicated in Clause 9;

f) any unusual features noted during the determination;

g) any operation not included in this International Standard, or regarded as optional.

Annex A
(informative)

Performance data

The round robin was carried out by laboratories in Austria, Belgium, Denmark, Finland, Germany, Ireland, Italy, The Netherlands, Spain, Sweden, and the United Kingdom. The variety of instruments and other analytical conditions were used in accordance with the quality parameters specified in the method.

The tests were carried out using two samples: wood chips and exhausted olive residues produced in the EU-project BioNorm according to ISO 14780. The sample "wood chips" was made of German coniferous wood chips; the chips were dried and milled to 1 mm by means of cutting mill. The sample "exhausted olive residues" was obtained from olive oil industry in Spain from a typical outdoor storage facility. In the original sample, stones and other natural impurities were present. These impurities and stones were removed manually and the sample was prepared from the residues in two steps using a coarse cutting mill equipped with a 10 mm sieve and a laboratory cutting mill equipped with WC cutting tools and a 1 mm sieve.

All data are reported on dry basis.

The performance data according to ISO 5725-2 are presented in Table A.1 and Table A.2.

NOTE 1 See Table A.1 for definition of the symbols used in Table A.1 and Table A.2.

NOTE 2 A guideline can be found in ISO 16993:2016, Annex C on how to use these validation parameters.

Table A.1 — Performance data for sulfur (S)

Sample	n	l	o %	x mass fraction %	s_R mass fraction %	CV_R %	s_r mass fraction %	CV_r %
wood chips	20	90	5,2	0,009	0,003	34	0,001	12
exhausted olive residues	23	111	1,8	0,12	0,021	17	0,007	5,4

Definition of symbols:

n	number of laboratories after outlier elimination;
l	number of outlier free individual analytical values;
o	percentage of outlying values from replicate determination;
x	overall mean;
s_R	reproducibility standard deviation;
CV_R	coefficient of the variation of the reproducibility;
s_r	repeatability standard deviation;
CV_r	coefficient of the variation of the repeatability.

Table A.2 — Performance data for chlorine (Cl)

Sample	*n*	*l*	*o* %	*x* mass fraction %	s_R mass fraction %	CV_R %	s_r mass fraction %	CV_r %
wood chips	17	75	7,4	0,006	0,003	52	0,001	13
exhausted olive residues	16	75	0	0,20	0,02	8,0	0,01	2,8

Table A.3 — List of techniques for the round robin

Chlorine (24 participating laboratories)		**Sulfur** (28 participating laboratories)	
Used method	**No. of laboratories**	**Used method**	**No. of laboratories**
Ion chromatography	16	Ion chromatography	13
Titration	2	S-Analyzer	12
ICP	2	ICP	3
Eschka	1	—	—
Photometry	1	—	—
Coulometry	1	—	—
Cl-Analyzer	1	—	—

IS 17834 : 2022/
ISO 16995 : 2015

SUMMARY

The presence of chlorine, sodium, and potassium in solid biofuels can lead to utilization problems like corrosion, fouling, and slagging in furnaces, and also affect gaseous emissions from thermal processes. Determining the water-soluble content of these elements helps estimate their aggressive potential in causing slagging and fouling.

The standard specifies a method for determining the content of water-soluble chloride, sodium, and potassium in solid biofuels. This is achieved through water extraction in a closed container, followed by quantification using various analytical techniques.

SOLID BIOFUELS – DETERMINATION OF THE WATER SOLUBLE CHLORIDE, SODIUM AND POTASSIUM CONTENT

IS 17834 : 2022

Indian Standard

SOLID BIOFUELS — DETERMINATION OF THE WATER SOLUBLE CHLORIDE, SODIUM AND POTASSIUM CONTENT

1 Scope

This International Standard describes a method for the determination of the water soluble chloride, sodium and potassium content in solid biofuels by extraction with water in a closed container and their subsequent quantification by different analytical techniques.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9964-1, *Water quality — Determination of sodium and potassium — Part 1: Determination of sodium by atomic absorption spectrometry*

ISO 9964-2, *Water quality — Determination of sodium and potassium — Part 2: Determination of potassium by atomic absorption spectrometry*

ISO 9964-3, *Water quality — Determination of sodium and potassium — Part 3: Determination of sodium and potassium by flame emission spectrometry*

ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 16993, *Solid biofuels — Conversion of analytical results from one basis to another*

ISO 18134-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis simple*

EN 14780, *Solid biofuels —Sample preparation*

Std. Meth. 4500-Cl- D Standard Methods For the Examination Of Water and Wastewater, 18th Edition 1992. 4500-Cl- D. Potentiometric Method

3 Terms and definitions

For the purposes of this document the terms and definitions given in ISO 16559 and the following apply.

3.1
water soluble chloride, sodium and potassium content
amount of the element which can be extracted with water using the extraction procedure specified in this International Standard

4 Principle

The fuel sample is heated with water in a closed container at 120 °C for 1 h. The concentrations of chloride, sodium and potassium in the obtained water extract are determined by one of the following techniques:

— chloride: ion chromatography (IC) or potentiometric titration with silver nitrate;

NOTE When potentiometric titration with silver nitrate is used, any contents of water soluble bromide and iodide will be included in the determination.

— sodium and potassium: flame emission spectroscopy (FES) or flame atomic absorption spectroscopy (FAAS) or inductively coupled plasma optical emission spectroscopy (ICP-OES).

5 Reagents

5.1 Water, containing negligible amounts of chloride, sodium and potassium i.e. amounts that do not contribute significantly to the determinations. Deionised water normally fulfil this requirement.

6 Apparatus

6.1 Heating oven or **autoclave**, capable of being maintained at a temperature of (120 ± 5) °C.

6.2 Vessel, made of fluoropolymer with a volume of about 100 ml and provided with a tight screw cap. The vessel and the cap shall be capable of withstanding at least 125 °C (232 kPa). If only the water soluble content of chloride is to be determined, an equivalent low-thermal-expansion borosilicate glass vessel can be used.

6.3 Balance, with a resolution of at least 1 mg.

6.4 General laboratory equipment, such as volumetric flasks and measuring cylinders. If sodium and potassium are to be determined, the use of equipment made of glass shall be avoided.

6.5 Membrane filtering apparatus, with membrane filters of mean pore size 0,45 µm.

7 Preparation of the test sample

The test sample is the general analysis sample with a nominal top size of 1 mm or less, prepared in accordance with EN 14780.

If the results are to be calculated other than on an “as determined” basis, the moisture content of the test sample shall be determined concurrently by the method specified in ISO 18134-3, using another portion of the test sample.

8 Procedure

8.1 Extraction

a) Weigh, in an empty clean vessel (see 6.2), 1,0 g of the analysis sample to the nearest 1 mg.

b) Add 50,0 ml water, swirl the content and close the vessel tight.

c) Leave the closed vessel in a heating oven or an autoclave at 120 °C for 60 min.

d) Take the closed vessel out of the oven or the autoclave and let it cool down to room temperature.

WARNING — Do not attempt to open the vessel before it is cold.

e) Transfer the content of the vessel to a 100 ml volumetric flask. Wash the inside of the vessel with small portions of water; add the washings to the volumetric flask and make it up to a volume of 100 ml with water.

f) Filter a portion of the solution [see 8.1 e)] through a membrane filter of pore size 0,45 µm, discarding the first portion of the filtrate. Alternatively the filtering can be carried out using a syringe equipped with a 0,45 µm pore size filter tip.

NOTE If only the water soluble content of chloride is to be determined, filtering may be omitted or a coarse folded filter paper may be used instead of the membrane filter.

8.2 Detection methods

8.2.1 General

Complete the determination by measuring the concentration of the elements in the prepared solution; for chloride by using one of the methods stated in 8.2.2 and for sodium and potassium by using one of the methods stated in 8.2.3.

8.2.2 Methods for the determination of chloride concentration

For the determination of the chloride concentration one of the following methods shall be used:

- ion-chromatographic determination according to the principles of ISO 10304-1;
- potentiometric titration with silver nitrate according to methods described in Standard Method 4500-Cl- D.

NOTE Equivalent national standards e.g. References [1] [2] or [3] can be used.

Other methods may be used provided that it can be proved that the results obtained are comparable to results obtained by determinations using one of the above stated methods, within the performance characteristics of these methods.

8.2.3 Methods for the determination of sodium and potassium concentration

For the determination of the concentration of sodium and potassium one of the following methods shall be used:

- ICP-OES according to the principles of ISO 11885;
- FAAS according to the principles of ISO 9964-1 and ISO 9964-2;
- FES according to the principles of ISO 9964-3.

For the instrumental technique used, an initial control for eventual interferences shall be performed using a standard addition method and/or a dilution method.

Other methods may be used provided that it can be proved that the obtained results are comparable to results obtained by determinations using one of the above stated methods, within the performance characteristics of these methods.

8.3 Blank test

Carry out a blank test, using the same procedure and methods as described in 8.1 and 8.2 but omitting the test portion. This assesses both the contents of the elements in the reagents and any contamination from equipment and in the laboratory atmosphere. This shall not be quantitatively significant.

The measured blank value has to be subtracted from the sample value. At high element level the blank should be less than 10 % of the sample content. For low element level (a content below 500 mg/kg in the sample), it is adequate that the contents of the elements in the blank solution are 30 % or less of the contents of the elements in the sample solution.

9 Calculation

The water soluble content of the element in the sample as analysed, w_X, expressed in mg/kg is given by Formula (1):

$$w_x = \frac{(c - c_0) \times V}{m} \tag{1}$$

where

c is the concentration of the element (chloride, sodium or potassium), in mg/l, in the extract solution [see 8.1 e)];

c_0 is the concentration of the element (chloride, sodium or potassium), in mg/l, in the extract solution of the blank experiment (see 8.3);

V is the volume, in ml, of the extract solution;

m is the mass, in g, of the test portion used.

Report the results as the mean of duplicate determinations.

The results shall be calculated to a dry basis or to an as received basis according to ISO 16993.

10 Performance characteristics

The achievable performance of the method is given in Annex A showing the results obtained by a European comparison study carried out for a sample of wood chips and a sample of an exhausted olive residue. These two samples represent the extremity of the method. The wood chip sample represents samples with low contents of water soluble chloride, sodium and potassium and the olive residue samples with high amounts of water soluble chloride and potassium.

11 Test report

The test report shall include at least the following information:

a) a reference to this International Standard (ISO 16995);

b) the identification of the laboratory performing the test and the date of the test;

c) the identification of product (or sample) tested;

d) the detection method(s) used for the determination;

e) the results of the test including the basis in which they are expressed, as indicated in Clause 9;

f) any unusual features noted during the determination;

g) any operation not included in this International Standard, or regarded as optional.

Annex A
(informative)

Performance data

A comparison study was carried out by laboratories in Austria, Belgium, Denmark, Finland, Germany, Ireland, Italy, The Netherlands, Spain, Sweden and the United Kingdom. The variety of instruments and other analytical conditions were used in accordance with the quality parameters specified in the method.

The tests were carried out using two samples, wood chips and exhausted olive residues produced in the EU-project BioNorm according to prEN 14780:2006. The "wood chips" sample was made of German coniferous wood chips; the chips were dried and milled to 1 mm by means of cutting mill. The "exhausted olive residues" sample was obtained from olive oil industry in Spain from a typical outdoor storage facility. In the original sample, stones and other natural impurities were present. These impurities and stones were removed manually and the sample was prepared from the residues in two steps using a coarse cutting mill equipped with a 10 mm sieve and a laboratory cutting mill equipped with tungsten carbide (WC) cutting tools and a 1 mm sieve.

All data are reported on dry basis.

The performance data according to ISO 5725-2 are presented in Tables A.1, A.2 and A.3.

NOTE A guideline can be found in ISO 16993:2015, Annex C on how to use these validation parameters.

Table A.1 — Performance data for water soluble chloride

Sample	n	l	O %	x mg/kg	s_R mg/kg	$C_{V,R}$ %	s_r mg/kg	$C_{V,r}$ %
Wood chips	7	35	15	26	13	45	5	19
Exhausted olive residues	7	35	0	2 100	150	7,4	49	2,4

Definitions of the symbols

n	is the number of laboratories after outlier elimination
l	is the number of outlier free individual analytical values
O	is the percentage of outlying values from replicate determination
x	is the overall mean
s_R	is the reproducibility standard deviation
$C_{V,R}$	is the coefficient of the variation of the reproducibility
s_r	is the repeatability standard deviation
$C_{V,r}$	is the coefficient of the variation of the repeatability

Table A.2 — Performance data for water soluble sodium

Sample	n	l	O %	x mg/kg	s_R mg/kg	$C_{V,R}$ %	s_r mg/kg	$C_{V,r}$ %
Wood chips	There is not enough data available							
Exhausted olive residues	8	40	0	99	16	16	8	8,4

NOTE See Table A.1 for definitions of the symbols.

Table A.3 — Performance data for water soluble potassium

Sample	n	l	O %	x mg/kg	s_R mg/kg	$C_{V,R}$ %	s_r mg/kg	$C_{V,r}$ %
Wood chips	6	29	4,4	634	20	3,1	9	1,4
Exhausted olive residues	7	35	0	22 400	1 060	4,8	524	2,3
NOTE See Table A.1 for definitions of the symbols.								

STANDARDIZATION – INTERNATIONAL LEVEL – SOLID BIOFUELS

Standardization in the field of Solid Biofuels at International level is done by

ISO/TC 238 'Solid Biofuels and Pyrogenic Biocarbon'

Scope of ISO/ TC 238 — Standardization of terminology, specifications and classes, quality assurance, sampling and sample preparation and test methods in the field of raw and processed materials originating from arboriculture, agriculture, aquaculture, horticulture and forestry to be used as a source for solid bio-fuels.

Excluded: Areas covered by ISO/TC 28/SC 7 Liquid biofuels, ISO/TC 193 Natural gas.

Secretariat: SIS - Swedish Institute for Standards [Sweden]
Committee Manager: Mrs Katarina Timåker
Chairperson (until end 2025): Mr Per Forsberg

Structure of ISO/TC 238

- ISO/TC 238/CAG Chairperson´s Advisory Group
- ISO/TC 238/WG 01 Terminology
- ISO/TC 238/WG 02 Fuel specifications and classes
- ISO/TC 238/WG 04 Physical and mechanical test methods
- ISO/TC 238/WG 05 Chemical test methods
- ISO/TC 238/WG 06 Sampling and sample preparation
- ISO/TC 238/WG 07 Safety of solid biofuels

The following countries are members of ISO/TC 238 in the respective capacities:

- ***Participating Members [right to vote] – 27 countries :***

Austria, Canada, Chile, China, Denmark, Finland, France, Germany, Hungary, Hungary, India, Ireland, Italy, Japan, Kazakhstan, Republic of Korea, Lithuania, Malaysia, Netherlands, Norway, Poland, South Africa, Spain, Sweden, Switzerland, Uganda, United Kingdom, United States

- ***Observing Members [no right to vote] – 22 countries:***

Argentina, Armenia, Barbados, Belgium, Bulgaria, Colombia, Costa Rica, Croatia, Czech Republic, Egypt, Estonia, Islamic Republic of Iran, Israel, Republic of Moldova, Mongolia, New Zealand, Philippines, Romania, Serbia, Slovakia, Sri Lanka, Thailand

Solid Mineral Fuels and Solid Bifouels Sectioanl Committee, PCD 7 has been co-coordinating with ISO/TC 238 “Solid Biofuels” which is one of amongst various technical committees of ISO dealing with the development and drafting of International Standards in the field of Solid Biofuels. BIS is representing the country as ‘P’ Member of ISO/TC 238.

ISO/TC 238 has published 46 standards. List of ISO Standards published till 4 February 2025 are as given below:

Sl No.	ISO Number	Title
1.	ISO 5370:2023	Solid biofuels — Determination of fines content in pellets
2.	ISO 14780:2017	Solid biofuels — Sample preparation
3.	**ISO 16559:2022**	Solid biofuels — Vocabulary
4.	**ISO 16948:2015**	Solid biofuels — Determination of total content of carbon, hydrogen and nitrogen
5.	**ISO 16967:2015**	Solid biofuels — Determination of major elements — Al, Ca, Fe, Mg, P, K, Si, Na and Ti
6.	**ISO 16968:2015**	Solid biofuels — Determination of minor elements
7.	**ISO 16993:2016**	Solid biofuels — Conversion of analytical results from one basis to another
8.	**ISO 16994:2016**	Solid biofuels — Determination of total content of sulfur and chlorine
9.	**ISO 16995:2015**	Solid biofuels — Determination of the water soluble chloride, sodium and potassium content
10.	**ISO/TS 16996:2015**	Solid biofuels — Determination of elemental composition by X-ray fluorescence

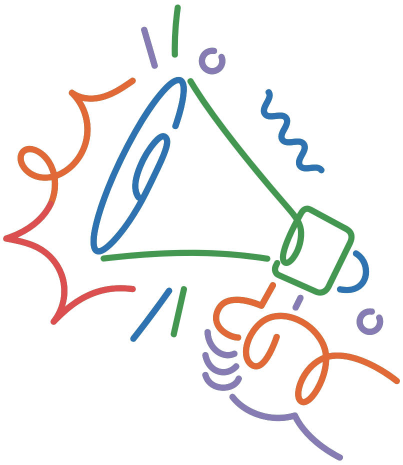
Sl No.	ISO Number	Title
11.	**ISO 17225-1:2021**	Solid biofuels — Fuel specifications and classes — Part 1: General requirements
12.	**ISO 17225-2:2021**	Solid biofuels — Fuel specifications and classes — Part 2: Graded wood pellets
13.	**ISO 17225-3:2021**	Solid biofuels — Fuel specifications and classes — Part 3: Graded wood briquettes
14.	**ISO 17225-4:2021**	Solid biofuels — Fuel specifications and classes — Part 4: Graded wood chips
15.	**ISO 17225-5:2021**	Solid biofuels — Fuel specifications and classes — Part 5: Graded firewood
16.	**ISO 17225-6:2021**	Solid biofuels — Fuel specifications and classes — Part 6: Graded non-woody pellets
17.	**ISO 17225-7:2021**	Solid biofuels — Fuel specifications and classes — Part 7: Graded non-woody briquettes
18.	**ISO 17225-8:2023**	Solid biofuels — Fuel specifications and classes — Part 8: Graded thermally treated and densified biomass fuels for commercial and industrial use
19.	**ISO 17225-9:2021**	Solid biofuels — Fuel specifications and classes — Part 9: Graded hog fuel and wood chips for industrial use
20.	**ISO 17827-1:2024**	Solid biofuels — Determination of particle size distribution for uncompressed fuels — Part 1: Oscillating screen method using sieves with apertures of 3,15 mm and above

Sl No.	ISO Number	Title
21.	**ISO 17827-2:2024**	Solid biofuels — Determination of particle size distribution for uncompressed fuels — Part 2: Vibrating screen method using sieves with apertures of 3,15 mm and below
22.	**ISO 17828:2015**	Solid biofuels — Determination of bulk density
23.	**ISO 17829:2015**	Solid Biofuels — Determination of length and diameter of pellets
24.	**ISO 17830:2024**	Solid biofuels — Particle size distribution of disintegrated pellets
25.	**ISO 17831-1:2015**	Solid biofuels — Determination of mechanical durability of pellets and briquettes — Part 1: Pellets
26.	**ISO 17831-2:2015**	Solid biofuels — Determination of mechanical durability of pellets and briquettes — Part 2: Briquettes
27.	**ISO 18122:2022**	Solid biofuels — Determination of ash content
28.	**ISO 18123:2023**	Solid biofuels — Determination of volatile matter
29.	**ISO 18125:2017**	Solid biofuels — Determination of calorific value
30.	**ISO 18134-1:2022**	Solid biofuels — Determination of moisture content — Part 1: Reference method
31.	**ISO 18134-2:2024**	Solid biofuels — Determination of moisture content — Part 2: Simplified method

Sl No.	ISO Number	Title
32.	**ISO 18134-3:2023**	Solid biofuels — Determination of moisture content — Part 3: Moisture in general analysis sample
33.	**ISO 18135:2017**	Solid Biofuels — Sampling
34.	**ISO 18847:2024**	Solid biofuels — Determination of particle density of pellets and briquettes
35.	**ISO 19743:2017**	Solid biofuels — Determination of content of heavy extraneous materials larger than 3,15 mm
36.	**ISO 20023:2018**	Solid biofuels — Safety of solid biofuel pellets — Safe handling and storage of wood pellets in residential and other small-scale applications
37.	**ISO 20024:2020**	Solid biofuels — Safe handling and storage of solid biofuel pellets in commercial and industrial applications
38.	**ISO/TS 20048-1:2020**	Solid biofuels — Determination of off-gassing and oxygen depletion characteristics — Part 1: Laboratory method for the determination of off-gassing and oxygen depletion using closed containers
39.	**ISO 20048-2:2023**	Solid biofuels — Determination of off-gassing and oxygen depletion characteristics — Part 2: Operational method for screening of carbon monoxide off-gassing
40.	**ISO 20049-1:2020**	Solid biofuels — Determination of self-heating of pelletized biofuels — Part 1: Isothermal calorimetry

Sl No.	ISO Number	Title
41.	**ISO/TS 20049-2:2020**	Solid biofuels — Determination of self-heating of pelletized biofuels — Part 2: Basket heating tests
42.	**ISO 21404:2020**	Solid biofuels — Determination of ash melting of behaviour
43.	**ISO/TS 21596:2021**	Solid biofuels — Determination of grindability — Hardgrove type method for thermally treated biomass fuels
44.	**ISO 21945:2020**	Solid biofuels — Simplified sampling method for small scale applications
45.	**ISO 23343-1:2021**	Solid biofuels — Determination of water sorption and its effect on durability of thermally treated biomass fuels — Part 1: Pellets
46.	**ISO/TR 23437:2020**	Solid biofuels — Bridging behaviour of bulk biofuels

Join the Future of Standardization!



Make your mark on national and international standards.

We invite experts and professionals to contribute to the advancement of standards in the Solid Biofuels sector.

Get Involved:

For national level:

https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/dgdashboard/committee_sso/

For international level:
https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/balot_data_public?param=jexpt

or

📩 Email us at pcd7@bis.gov.in

📝 Mention your area of specialization and interest.

Together, let's shape the standards of tomorrow!

DIGITAL PLATFORMS OF BIS

BIS website: www.bis.gov.in

BIS care App:
https://play.google.com/store/apps/details?id=com.bis.bisapp

Facebook:
https://www.facebook.com/IndianStandards/

Instagram:
https://www.instagram.com/indianstandards/

YouTube:
http://bit.ly/BISYouTubeOfficial

LinkedIn:
http://bit.ly/BISLinkedInOfficial

Twitter:
https://x.com/jagograhakjago

eBIS - Search, download Indian Standard :
https://standardsbis.bsbedge.com/

Comment on wide circulation document:
https://www.services.bis.gov.in/php/BIS_2.0/dgdashboard/draft/wcdraftDepartment

New Work Item Proposal:
https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/all-proposals-dg

Programme of Work:
https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/pow_new/

Indigenous Indian Standards can be downloaded for free of cost by visiting the URL: Bureau of Indian Standards - e-Sale (bsbedge.com)

VERIFY HUID

KNOW YOUR STANDARD

PRODUCTS UNDER SIMPLIFIED PROCEDURE

LABORATORIES

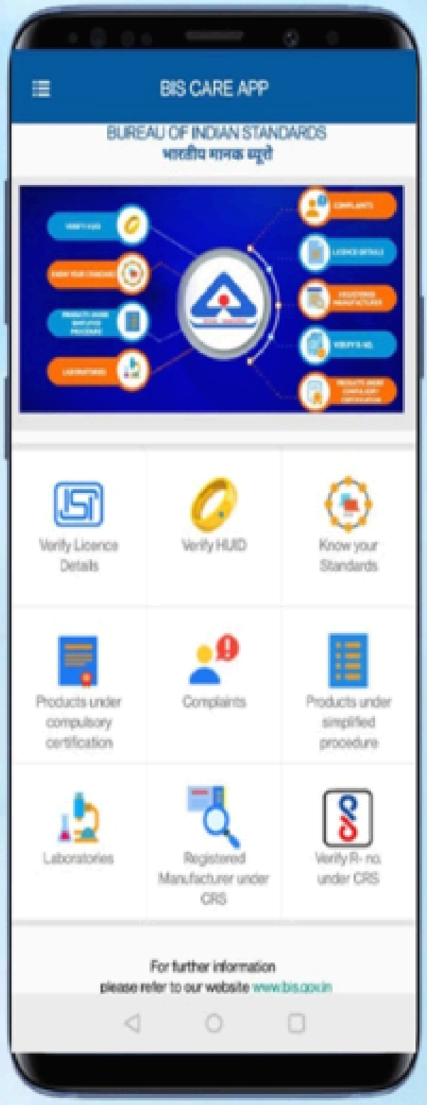
COMPLAINTS

LICENCE DETAILS

PUBLIC-ALERT PRODUCT RECALLS

VERIFY R-NO.

PRODUCTS UNDER COMPULSORY CERTIFICATION



BIS CARE APP

BUREAU OF INDIAN STANDARDS
भारतीय मानक ब्यूरो

Verify Licence Details	Verify HUID	Know your Standards
Products under compulsory certification	Complaints	Products under simplified procedure
Laboratories	Registered Manufacturer under CRS	Verify R- no. under CRS

For further information
please refer to our website www.bis.gov.in

Download **BIS CARE APP**
from Play Store

BIS CARE APP

A tool for Consumer Empowerment

Main features of the App

- Check the authenticity of the product with ISI mark by using 'Verify Licence Details.'
- Check the authenticity of Hallmarked Jewellery items with HUID number by using 'verify HUID'.
- Select 'Know your Standards' for information on any Indian Standard, licenses against it and laboratories for this product.
- Check the authenticity of electronic products with R-number by using 'Verify R-number under CRS'.
- Register complaints regarding quality of product or misuse of ISI mark by using 'Complaints'.

Bureau of Indian Standards

www.bis.gov.in

75 years of serving the Nation