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Solid biofuels — Determination of self-heating of pelletized biofuels —

Part 2:
Basket heating tests

Biocombustibles solides — Détermination de l'auto-échauffement des granulés de biocombustibles —

Partie 2: Essais utilisant la méthode du point de croisement

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 238, *Solid biofuels*.

A list of all parts in the ISO 20049 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

There is a continuous global growth in production, storage, handling, bulk transport and use of solid biofuels especially in the form of pelletized biofuels.

The specific physical and chemical characteristics of solid biofuels, their handling and storage can lead to a risk of fire and/or explosion, as well as health risks such as intoxication due to exposure to carbon-monoxide, asphyxiation due to oxygen depletion or allergic reactions.

Heat can be generated in solid biofuel by exothermic biological, chemical and physical processes. Biological processes include the metabolism of fungus and bacteria and occur at lower temperatures; the oxidation of wood constituents increases with temperature and dominates at higher temperatures; the heat production from biological and chemical processes leads to transport of moisture in the bulk material, with associated sorption and condensation of water, which both are exothermic processes. In, for example, a heap of stored forest fuel or a heap of moist wood chips, all of these processes can be present and contribute to heat production.

Solid biofuels such as wood pellets, however, are intrinsically sterile[6] due to the conditions during manufacturing (exposure to severe heat during drying, fragmentation during hammermilling and pressure during extrusion) but can attract microbes if becoming wet during handling and storage resulting in metabolism and generation of heat. Leakage of water into a storage of wood pellets can also lead to the physical processes mentioned above. Non-compressed wood like feedstock and chips typically have a fauna of microbes which under certain circumstances will result in heating. All the processes mentioned above contribute to what is called self-heating although oxidation is likely to be one of the main contributing factors in the temperature range under which most biofuels are stored. The heat build-up can be significant in large bulk stores as the heat conduction in the material is low. Under certain conditions the heat generation can lead to thermal runaway and spontaneous ignition.

The potential for self-heating seems to vary considerably for different types of solid biofuel pellets. The raw material used, and the properties of these raw materials have proven to influence the propensity for self-heating of the produced wood pellets. However, the production process (e.g. the drying process) also influences the potential for self-heating. It is therefore important to be able to identify solid biofuel pellets with high heat generation potential to avoid fires in stored materials.

Two intrinsically different types of tests methods can be used to estimate the potential of self-heating:

a) in the isothermal calorimetry method described in ISO 20049-1, the heat flow generated from the test portion is measured directly;

b) in the basket heating tests described in this document, the temperature of the test portion is being monitored and the critical ambient temperature (CAT), where the temperature of the test portion just does not increase significantly due to self-heating, is used for indirect assessment of self-heating.

These two methods are applied at different analysis temperature regimes. The operating temperature for an isothermal calorimeter is normally in the range 5 °C to 90 °C whereas basket heating tests are conducted at higher analysis (oven) temperatures. For basket heating tests with wood pellets, the CAT is found for a 1 l sample portion in the range 150 °C to 200 °C.

NOTE 1 The two types of test methods referred to above do not measure heat production from physical processes such as transport of moisture.

NOTE 2 It is likely that oxidation reactions taking place in the low respective high temperature regimes for solid biofuel pellets are of different character and thus have different reaction rates and heat production rates. In such a case, extrapolation of the data from a high temperature test series can lead to non-conservative results and might not be applicable without taking the low temperature reactions into account. In the general case of two reactions with different activation energies, the high activation energy is “frozen out” at low temperatures and the low activation energy reaction is “swamped” at higher temperatures[7].

NOTE 3 It has been shown for a limited number of different types of wood pellets that the reaction rates in the lower temperature regime measured by isothermal calorimetry were higher compared to the reaction rate data determined from basket heating tests in the higher temperature regime[8].

Basket heating tests have been used traditionally for characterization of the tendency for spontaneous ignition of predominantly coals, but also for other reactive organic materials such as, for example, cottonseed meal, bagasse and milk powder[9]. The principle used in this type of tests is to find the CAT for a self-heating sample material of specific size and geometry.

There are several different methods described in the literature with different degrees of sophistication. The variations span from simple pass and fail tests to more advanced tests from which data on reaction rates can be extracted[10].

Basket heating tests are useful for assessment of self-heating of solid biofuel pellets. The test method selected can be evaluated for its applicability based on the information given in this document.

A compilation of available basket heating test methods is given in this document. Guidance on the suitability for application of these methods for tests with pelletized biofuels is provided.

Basic theory of the use of basket heating test data for calculations of critical conditions in storages is provided in Annex B.

Solid biofuels — Determination of self-heating of pelletized biofuels —

Part 2:
Basket heating tests

1 Scope

This document specifies basket heating tests for the characterization of self-heating properties of solid biofuel pellets.

This document includes:

a) a compilation of basket heating test methods;

b) guidance on the applicability and use of basket heating tests for solid biofuel pellets;

c) information on the application of basket heating test data for calculations of critical conditions in storages.

Data on spontaneous heat generation determined using this document is only associated with the specific quality and age of the sample material.

The information derived using this document is for use in quality control and in hazard and risk assessments related to the procedures given in ISO 20024.

The described methods can be used for other substances than solid biofuel pellets (e.g. wood chips).

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, *Solid biofuels — Sample preparation*

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

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:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
analysis temperature
temperature of the analysis environment, i.e. the oven temperature

3.2

Biot number

quotient of the convective heat transfer coefficient (between the sample boundary and the surrounding air) and the conduction in the sample material normalized by the characteristic dimension of the test basket

3.3

critical ambient temperature

CAT

ambient temperature [the *analysis temperature* (3.1) or the temperature of a storage] where the internal temperature of the *test portion* (3.6) or the stored material increases significantly (due to *self-heating* (3.4))

3.4

self-heating

rise in temperature in a material resulting from an exothermic reaction within the material

[SOURCE: ISO 13943:2017, 3.341, modified — “<chemical>” has been deleted from the beginning of the definition.]

3.5

spontaneous ignition

ignition caused by an internal exothermic reaction

Note 1 to entry: See the definitions of ignition in ISO 13943.

[SOURCE: ISO 13943:2017, 3.24, modified — “spontaneous ignition” has replaced auto-ignition” has the preferred term and the other terms have been deleted. Notes 1 to 3 have been deleted and a new Note 1 to entry has been added.]

3.6

test portion

sub-sample either of a *laboratory sample* (3.8) or a *test sample* (3.7)

3.7

test sample

laboratory sample (3.8) after an appropriate preparation made by the laboratory

Note 1 to entry: In this document, the test sample is typically a representative sample from a batch of solid biofuel pellets.

3.8

laboratory sample

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

[SOURCE: ISO 16559:2014, 4.124]

4 Symbols

Symbol	Quantity	Typical unit
A	pre-exponential factor in Arrhenius expression	s^{-1}
B	dimensionless adiabatic temperature rise	dimensionless
Bi	Biot number, ($\mathrm{Bi}=\frac{hL}{\lambda}$)	dimensionless
c_0	ambient oxygen concentration by volume fraction	dimensionless
C	specific heat capacity of the reaction products	$\mathrm{J\ kg^{-1}\ K^{-1}}$
C_p	specific heat capacity of the bulk material	$\mathrm{J\ kg^{-1}\ K^{-1}}$
d	diameter of body	m
D	diffusion coefficient	$\mathrm{m^2\ s^{-1}}$
E_a	activation energy	$\mathrm{J\ mol^{-1}}$
H_0	gross calorific value	$\mathrm{J\ kg^{-1}}$
h	heat transfer coefficient	$\mathrm{W\ m^{-2}\ K^{-1}}$
h_r	radiative amount on heat transfer coefficient	$\mathrm{W\ m^{-2}\ K^{-1}}$
h_c	convective amount on heat transfer coefficient	$\mathrm{W\ m^{-2}\ K^{-1}}$
L	characteristic length	m
n	order of reaction	dimensionless
P	constant, see Formulae (2) and (3)	dimensionless
$\dot{q}'$	heat generation term, see Formula (B.1)	$\mathrm{W\ m^{-3}}$
Q	heat of reaction	$\mathrm{J\ kg^{-1}}$
Q_0	heat of reaction by volume of oxygen	$\mathrm{J\ m^{-3}}$
R	universal gas constant	$\mathrm{J\ mol^{-1}\ K^{-1}}$
Ra	Rayleigh number	dimensionless
t	time	s
T	temperature	K
T_0	ambient temperature	K
T_p	crossing point temperature	K
x	length coordinate	m
δ	Frank-Kamenetskii parameter, see Formula (B.4)	dimensionless
δ_c	critical value of δ	dimensionless
ε	activation energy parameter, ($\varepsilon=\frac{RT_0}{E}$)	dimensionless
Φ	oxygen diffusion parameter, see Formula (B.13)	dimensionless
λ	thermal conductivity of sample	$\mathrm{W\ m^{-1}\ K^{-1}}$
λ_air	thermal conductivity of air	$\mathrm{W\ m^{-1}\ K^{-1}}$
ρ	bulk density	$\mathrm{kg\ m^{-3}}$
σ	Stefan-Boltzmann coefficient	$\mathrm{W\ m^{-2}\ K^{-4}}$

5 Basket heating tests

The detailed test procedure varies between different isoperibolic and adiabatic methods. Isoperibolic methods include that the test portion is put in a wire-mesh basket, which is placed in an oven heated to a fixed elevated temperature. The oven is equipped with a fan to keep the temperature uniform and to give a relatively large convective heat transfer coefficient to the test specimen[9][10]. For adiabatic tests, the oven temperature is adjusted to the temperature at the centre of the sample[5].

Basket heating tests are based on the Frank-Kamenetskii theory of criticality of a self-heating isotropic slab (see Annex B) and have been developed to determine the reaction kinetics of the global reaction responsible for heat production in a self-heating material.

NOTE 1 The large gap volume of pelletized material can lead to convective heat transport in the bulk if the furnace is equipped with a fan. In this case, it is recommended to keep the air flow in the vicinity of the sample at a low level and to correct the critical Frank-Kamenetskii parameter (see B.1.3) or to prevent convective transport within the sample by further measures (e.g. finer mesh wire of the basket).

NOTE 2 The CAT for the test portion in a basket heating tests is not equal to the CAT for spontaneous ignition in, for example, large-scale storage. The critical size for spontaneous ignition (if only heat transfer is considered) is directly related to the surface area-volume ratio of the self-heating specimen where heat is produced distributed in the volume and heat is dissipated from the surface area only. The test sample in laboratory size basket heating test has a very high surface area-volume ratio and has consequently a high CAT compared to a larger specimen.

6 Tests for product classification

6.1 UN classification

6.1.1 General

The United Nations (UN) Globally Harmonized System of Classification and Labelling of Chemicals (GHS)[11] is the international convention for hazard communication and labelling of gases, vapours, solid and liquid substances, and mixtures. The GHS defines limit values, classes and categories and related measures in relation to the level of hazards during transportation, handling and storage.

The UN Manual of Tests and Criteria (MTC)[12] prescribes specific test procedures in support of the GHS.

6.1.2 Test method for self-heating substances — UN MTC Test N.4

Test N.4 is described in the UN MTC Part III, 33.3.1.6[12], sometimes called the “basket test”.

This basket heating test determines the ability of a substance to undergo oxidative self-heating with exposure of it to air at temperatures of 100 °C, 120 °C or 140 °C in a 25 mm or 100 mm wire mesh cube.

The Test N.4 basket heating test is not intended for determination of self-heating kinetics but rather prescribed to classify a material (e.g. solid biofuels) as meeting the criteria for self-heating set out by the GHS[11] for hazard communication and labelling purposes.

The test set-up consists of a hot-air circulating oven, cubic sample containers of 25 mm and 100 mm sides made of stainless-steel net with a mesh opening of 0,05 mm, and thermocouples of 0,3 mm diameter for the measurement of the oven temperature and the temperature of the centre of the sample. The sample container is housed in a cubic container cover made from stainless-steel net with a mesh opening of 0,60 mm, and is slightly larger than the test container. To avoid the effect of air circulation, this cover is installed in a second steel cage, made from a net with a mesh size of 0,595 mm and 150 mm × 150 mm × 250 mm in size.

The normal procedure is to start with a test at 140 °C with a 100 mm cube sample. The container is housed in the cover and hung at the centre of the oven. The oven temperature is raised to 140 °C and kept there for 24 h. A positive result is obtained if spontaneous ignition occurs or if the temperature of the sample exceeds the oven temperature by 60 °C. If a negative result is obtained, no further test is necessary.

If a positive result is obtained at 140 °C with a 100 mm cube sample, the substance is classified as a self-heating substance and further testing shall be made to find the correct classification (see 6.1.3).

NOTE The bulk density tested can influence the test results. prEN 15188 suggests adjusting the bulk density of the sample to the respective practical conditions (if known) and recording the tested bulk density. The UN MTC contains no information on the bulk density to be tested.

6.1.3 Classification criteria — GHS

The classification criteria are given in chapter 2.11.2 of the GHS[11]. The criteria are summarized in Table 1.

Table 1 — Criteria in the GHS for self-heating substances and mixtures

Category	Criteria
1	A positive result is obtained in a test using 25 mm sample cube at 140 °C.
2	a) A positive result is obtained in a test using a 100 mm sample cube at 140 °C and a negative result is obtained in a test using a 25 mm cube sample at 140 °C and the substance or mixture is to be packed in packages with a volume of more than 3 m^3; or b) A positive result is obtained in a test using a 100 mm sample cube at 140 °C and a negative result is obtained in a test using a 25 mm cube sample at 140 °C, a positive result is obtained in a test using a 100 mm cube sample at 120 °C and the substance or mixture is to be packed in packages with a volume of more than 450 litres; or c) A positive result is obtained in a test using a 100 mm sample cube at 140 °C and a negative result is obtained in a test using a 25 mm cube sample at 140 °C and a positive result is obtained in a test using a 100 mm cube sample at 100 °C.

NOTE Hazard packing groups classification is prescribed depending on the flammability characteristics of the material, see Table 32.1 of the GHS[11].

6.2 Classification criteria — IMO

Handling guidelines and hazard classifications for all cargoes, including solid biofuels, transported onboard ocean vessels are specified by the International Maritime Organization (IMO) in the International Maritime Solid Bulk Cargoes Code[13]. The code stipulates UN MTC Test N.4 to be used for testing but adds additional criteria for solid possessing hazards compared to the GHS criteria in Table 1, as follows:

a) Does the material undergo dangerous self-heating when tested in accordance with Test N.4 in a 100 mm sample cube at 140 °C?

   If yes, Class 4.2 applies. Materials in this class are materials, other than pyrophoric materials, which, in contact with air without energy supply, are liable to self-heating.

b) Does the material show a temperature increase of 10 °C or more when tested in accordance with Test N.4 in a 100 mm sample cube at 140 °C?

   If yes, test in a 100 mm sample cube at 100 °C and check the temperature increase is 10 °C or more:

   1) if yes, material hazardous in bulk (MHB) applies;

   2) if no, neither Class 4.2 nor MHB applies.

NOTE Wood pellets containing no binder and additives are given the designation MHB (OH) as a result of a high emission of carbon monoxide and not MHB (SH), since wood pellets are not classified as self-heating in accordance with the criteria specified under the GHS and the MTC.

6.3 Applicability of UN MTC Test N.4 for pelletized biofuels

Experience from the testing of wood pellets indicates that the CAT for this type of material always is higher than 140 °C in 1,0 l basket heating tests; see, for example, Reference [8]. The 140 °C criterion seems thus not to be generally applicable for solid biofuel pellets.

The reasons that this test is unsuitable as a general test method for solid biofuel pellets are the following:

a) the criteria in Test N.4 is based on fix reaction kinetics of coal, which is not directly transferable to solid biofuel pellets;

b) experience shows that the UN criteria based on self-ignition of the analytical sample in a 100 mm sample cube test at 140 °C is not valid for solid biofuel pellets since the CAT of 1000 cm^3 wood pellets is normally higher;

c) there is no published information on the selectivity and the correlation to large scale storage of this tests for solid biofuel pellets;

d) the self-heating process of wood pellets can undergo multi-step reactions at different temperature ranges. Low temperature reactions are not covered by tests in accordance with the Test N.4 method.

7 Tests for determination of reaction kinetics

7.1 General

There are different basket heating tests available for the determination of reaction kinetics for self-heating of reactive materials. The most important of these methods are summarized in 7.2 to 7.4.

7.2 Isoperibolic test methods

7.2.1 General

The original basket heating test method was developed at the Fire Research Station in UK and is sometimes referred to as the “FRS method”. This is a rather time-consuming method to use because of the large number of experiments that is needed for each material studied. This method does not exist in the form of a test standard but has been described in detail by Bowes[14] and Beever[9].

Several investigations and interlaboratory comparisons in the past have shown significant differences between the results of hot storage tests determined by different laboratories[15][16]. Laboratory-specific differences have been identified as possible reasons for the deviations, for example:

a) oven ventilation (enforced, natural convection);

b) oven size;

c) sample baskets (shape, size, construction);

d) radiation effects;

e) measuring precision (temperature difference between tests with ignition and no ignition);

f) minimum sample size.

For that reason, the original FRS method was modified and further developed in the European standard EN 15188. The main difference is the use of an additional mesh wire screen and special volumes of the sample baskets (cubes) to normalize/harmonize the test conditions in the surrounding of the samples independent from used oven type and size. This is, however, an important deviation from the Frank-Kamenetskii theory (see Annex B), which relies on a high Biot number of the test specimen to keep the boundary of the tests specimen at the analysis (oven) temperature. On the other hand, the air flow velocity in the vicinity of the sample is reduced to prevent convective mass and heat transport in the sample. For these reasons, the critical Frank-Kamenetskii parameter shall be corrected in accordance with B.1.3 if this method is used.