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Solid recovered fuels — Determination of self-heating — Part 2: Basket heating tests

Combustibles solides de récupération — Détermination de l'autoéchauffement — Partie 2: Essais utilisant la méthode du point de croisement

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Foreword

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This document was prepared by Technical Committee ISO/TC 300, *Solid recovered materials, including solid recovered fuels*., in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 343, *Solid recovered fuels*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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Introduction

There is ~~a~~ continuous global growth in trading and use of solid recovered fuels (~~SRF~~SRFs). Therefore, intensive investigations about the risk of fires within SRF ~~-~~production, handling and storing have been conducted, see ÖNORM S 2098~~:2011~~. Recommendations ~~have been~~are given ~~by~~in ISO 21912.

Depending on the kind of input wastes, the treatment technology applied, the quality of the SRF produced and the realized storage versions, ~~SRF~~SRFs can generate heat spontaneously by exothermic biological, chemical and physical processes. The heat build-up can be significant in large storage volumes if the heat conduction in the material is low. During some conditions the heat generation can lead to pyrolysis and spontaneous ignition. The potential for self-heating varies considerably for different types and qualities of SRF and it is important to be able to identify SRF fractions with high heat generation potential to avoid fires in stored materials.

Avoiding fires throughout the production and supply chain will have positive consequences on the acceptance of ~~SRF~~SRFs and the costs for insurance coverage,

Application of SRF ~~-~~standards and the use of dedicated standards for the determination of self-heating will help to reduce the risk of fires and to develop tailor-made recommendations for SRF producers, logistics providers, SRF users, equipment suppliers~~/~~ or manufacturers, consultants, authorities and insurance providers.

As part of the determination and the assessment of risks for SRF, defined test methods and standards are established or need to be developed. However, ~~the~~ ageing and degradation due to handling and storage of SRF in actual environments ~~will affect~~affects their characteristics, so safety margins ~~have to~~should be established in relation to actual analysis results.

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

a) In the isothermal calorimetry method described in ISO 21911-1[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.

NOTE 1 ~~The~~These 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 recovered fuels~~SRFs are of different character and thus have different reaction rates and heat production rates. In such ~~a case~~cases, extrapolation of the data from a high-temperature test series can lead to non-conservative results and ~~might~~will possibly 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].

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

[1] In preparation. Stage at the time of publication: ISO/DIS 21911-1:2022.

There are several different methods described in the literature with different degreedegrees 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 recovered fuelsSRFs. 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 solid recovered fuelsSRFs is provided.

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

The test methods presented require representative samples for the conditions prevailing in the process (e.g. of SRF -storage). Sample preparation is necessary for this purpose. The methods presented are not suitable for assessing the fire hazard caused by impurities (disturbing materials),) as they occur mainly in the input area and the first steps of SRF -production.

Solid recovered fuels ~~—~~ — Determination of self-heating ~~—~~ — Part 2: Basket heating tests

1 Scope

This document gives guidance on basket heating tests for ~~characterisation~~characterization of self-heating properties of solid recovered fuels~~.~~ (SRFs).

This document includes:

a) a compilation of basket heating test methods~~,~~;

b) guidance on the applicability and use of basket heating tests for ~~solid recovered fuels,~~SRF;

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

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 intended for use in quality control and in hazard and risk assessments related to the procedures given in ISO 21912.

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 21646[2], *Solid recovered fuels — Sample preparation*

ISO 21637:2020, *Solid recovered fuels — Vocabulary*

ISO 21645, *Solid recovered fuels — Methods for sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 21637~~:2020~~ and the following apply.

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

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

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

[2] In preparation. Stage at the time of publication: ISO/FDIS 21646:2022.

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

Note 1 to entry: In EN 15188 the critical ambient temperature is defined as self-ignition temperature, T_{SI}.

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 beendomain omitted in the beginning of thefrom definition.]

3.5
spontaneous ignition
ignition caused by an internal exothermic reaction

[SOURCE: ISO 13943:2017, 3.24, modified — Notes to entry removed.]

3.6
test sample
laboratory sample [(3.7]) after an appropriate preparation made by the laboratory

Note 1 to entry: The test sample is here typically a representative sample from a batch of solid recovered fuel.

[SOURCE: ISO 21637:2020, 3.84, modified -— Note 1 to entry has been added.]

3.7
laboratory sample
combined sample or a sub-sample of a combined sample for use indelivered to a laboratory

[SOURCE: ISO 16559:2014, 4.124] 2022, 3.120, modified — Note 1 to entry removed.]

4 Symbols

Symbol	Quantity	Typical unit
A	pre-exponential factor in Arrhenius expression	s^{-1}
B	dimensionless adiabatic temperature rise	dimensionless
Bi	Biot number, ($Bi = \frac{h \cdot L}{\lambda}$)	dimensionless

~~c_0~~	~~ambient oxygen concentration by volume fraction~~	~~dimensionless~~
~~C~~	~~specific heat capacity of the reaction products~~	~~J kg^{-1} K^{-1}~~
~~C_p~~	~~specific heat capacity of the bulk material~~	~~J kg^{-1} K^{-1}~~
~~d~~	~~diameter of body~~	~~m~~
~~D~~	~~diffusion coefficient~~	~~m^2 s^{-1}~~
~~E_a~~	~~activation energy~~	~~J mol^{-1}~~
~~H_0~~	~~Gross calorific value~~	~~J kg^{-1}~~
~~h~~	~~heat transfer coefficient~~	~~W m^{-2} K^{-1}~~
~~h_r~~	~~radiative amount on heat transfer coefficient;~~	~~W m^{-2} K^{-1}~~
~~h_c~~	~~convective amount on heat transfer coefficient;~~	~~W m^{-2} K^{-1}~~
~~L~~	~~characteristic length~~	~~m~~
~~n~~	~~order of reaction~~	~~dimensionless~~
~~P~~	~~constant~~	~~dimensionless~~
~~$\dot{q}'$~~	~~heat generation term~~	~~W m^{-3}~~
~~Q~~	~~heat of reaction~~	~~J kg^{-1}~~
~~Q_0~~	~~heat of reaction by volume of oxygen~~	~~J m^{-3}~~
~~R~~	~~universal gas constant~~	~~J mol^{-1} K^{-1}~~
~~Ra~~	~~Rayleight number~~	~~dimensionless~~
~~S~~	~~surface~~	~~m^2~~
~~t~~	~~time~~	~~s~~
~~T~~	~~temperature~~	~~K~~
~~T_0~~	~~ambient temperature~~	~~K~~
~~T_p~~	~~crossing point temperature~~	~~K~~
~~V~~	~~volume~~	~~m^3~~
~~x~~	~~length coordinate~~	~~m~~
~~δ~~	~~Frank-Kamenetskii parameter~~	~~dimensionless~~
~~δ_c~~	~~critical value of δ~~	~~dimensionless~~
~~ε~~	~~activation energy parameter, ($\varepsilon = \frac{R\,T_0}{E_a}$)~~	~~dimensionless~~
~~Φ~~	~~oxygen diffusion parameter~~	~~dimensionless~~
~~λ~~	~~thermal conductivity of sample~~	~~W m^{-1} K^{-1}~~
~~λ_{air}~~	~~thermal conductivity of air~~	~~W m^{-1} K^{-1}~~
~~ρ~~	~~bulk density~~	~~kg m^{-3}~~
~~σ~~	~~Stefan-Boltzmann coefficient~~	~~W m^{-2} K^{-4}~~

Symbol	**Quantity**	**Typical unit**
A	pre-exponential factor in Arrhenius expression	s^{-1}
B	dimensionless adiabatic temperature rise	dimensionless

Bi	Biot number, ($\mathrm{Bi}=\frac{h\cdot L}{\lambda}$)	dimensionless
c_0	ambient oxygen concentration by volume fraction	dimensionless
C	specific heat capacity of the reaction products	J kg^{-1} K^{-1}
C_p	specific heat capacity of the bulk material	J kg^{-1} K^{-1}
d	diameter of body	m
D	diffusion coefficient	m^2 s^{-1}
E_a	activation energy	J mol^{-1}
H_0	gross calorific value	J kg^{-1}
h	heat transfer coefficient	W m^{-2} K^{-1}
h_r	radiative amount on heat transfer coefficient	W m^{-2} K^{-1}
h_c	convective amount on heat transfer coefficient	W m^{-2} K^{-1}
L	characteristic length	m
n	order of reaction	dimensionless
P	constant	dimensionless
$\dot{q}'$	heat generation term	W m^{-3}
Q	heat of reaction	J kg^{-1}
Q_0	heat of reaction by volume of oxygen	J m^{-3}
R	universal gas constant	J mol^{-1} K^{-1}
Ra	Rayleight number	dimensionless
S	surface	m^2
t	time	s
T	temperature	K
T_0	ambient temperature	K
T_p	crossing point temperature	K
T_{si}	self-ignition temperature	K
V	volume	m^3
x	length coordinate	m
δ	Frank-Kamenetskii parameter	dimensionless
δ_c	critical value of δ	dimensionless
ε	activation energy parameter, $\left(\varepsilon=\frac{RT_0}{E_a}\right)$	dimensionless
Φ	oxygen diffusion parameter	dimensionless
λ	thermal conductivity of sample	W m^{-1} K^{-1}
λ_{air}	thermal conductivity of air	W m^{-1} K^{-1}
ρ	bulk density	kg m^{-3}
σ	Stefan-Boltzmann coefficient	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, see EN 15188.

Basket heating tests are based on the Frank-Kamenetskii theory of criticality of a self-heating isotropic slab (see Annex C) and have been developed to determine the reaction kinetics of the global reaction responsible for heat production in a self-heating material. 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 air flow in the vicinity of the sample should be kept at a low level and the critical Frank-Kamenetskii parameter should be corrected (see C.1.3) or the convective transport within the sample should be prevented by further measures (e.g. finer mesh wire of the basket).

NOTE The ~~critical ambient temperature (~~CAT~~)~~ for the test portion in a basket heating ~~tests~~test is not equal to the CAT for spontaneous ignition in ~~e.g.~~, 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 a 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 Globally Harmonized System of Classification and Labelling of Chemicals (GHS~~)~~)[11] is the international convention for hazard communication and labelling of ~~gaseous~~gases and vapours, solid and liquid substances ~~as well as~~and mixtures. GHS defines limit values, classes and categories and related measures in relation to the level of hazards during transportation, handling and storage.

The United Nations Manual of Test and Criteria (MTC~~) is prescribing~~)[12] prescribes specific test procedures in support of GHS.

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

Test N.4 is described in the ~~United Nations Manual of Tests and Criteria (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 N.4 test basket heating test is not intended for determination of self-heating kinetics but rather prescribed to classify a material (e.g. ~~solid recovered fuels~~SRFs) 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 with sides 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 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 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 cube. 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 cube, the substance is classified as a self-heating substance and further testing shall be made to find the correct classification (see 6.1.3).

The bulk density tested can influence the test results. ~~According EN 15188 the~~The bulk density of the sample shall be adjusted according to EN 15188 to the respective practical conditions (if known) and the tested bulk density shall be recorded. ~~The United Nations Manual of Tests and Criteria~~The 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 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~~1	~~a)~~ A positive result is obtained in a test using ~~100~~ a 25-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 m³; or~~
2	~~b)~~ 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 cube at 140~~ °C, a~~ °C and the substance or mixture is packed in packages with a volume of more than 3 m³; or b) A positive result is obtained in a test using a 100-mm ~~cube~~ sample cube at 140 °C, a negative result is obtained in a test using a 25-mm sample cube at 140 °C, a positive result is obtained in a test using a 100-mm sample cube at 120 °C and the substance or mixture is ~~to be~~ packed in packages with a volume of more than 450 ~~litres~~ l; or c) A positive result is obtained in a test using a 100-mm sample cube at 140 °C, a negative result is obtained in a test using a 25-mm sample cube at 140 °C and a positive result is obtained in a test using a 100-mm sample cube at 100 °C.
	~~c) A positive result is obtained in a test using 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~~packing group classification is prescribed depending on flammability characteristics of the material, see GHS, Table 32.1~~.~~.[11]

6.2 Classification criteria ~~-~~— IMO

Handling guidelines and hazard classifications for all cargoes, including ~~solid recovered fuels~~SRFs, transported onboard ocean vessels are specified by the International Maritime Organization (IMO) in the International Maritime Solid Bulk Cargoes (IMSBC) Code~~. The Code is stipulating UN~~ [13]. This stipulates the MTC Test N.4 to be used for testing but ~~has added~~includes additional criteria for solid possessing hazards compared to the GHS criteria in Table 1 ~~above~~, as follows:

Deleted Cells

Split Cells

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~~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 if temperature increase is 10 °C or more.

If yes, ~~Material Hazardous~~material hazardous in ~~Bulk~~bulk (MHB) applies.

If no, neither ~~Class~~ class 4.2 nor MHB applies.

6.3 Applicability of ~~UN~~MTC Test N.4 for solid recovered fuels

~~UN~~MTC Test N.4 ~~might~~will possibly be unsuitable for ~~solid recovered fuels~~SRFs.

Experience from testing several SRF samples indicates that the CAT for this type of material in 1,0 ~~L~~ l basket heating tests can be lower than 140 °C, especially when various materials, including inert ones, are present in the mixture, see Annex A~~, .~~ and References [14] and [15].

The reasons that this test ~~might~~will possibly be unsuitable as a general test method for ~~solid recovered fuels~~SRFs are ~~the following: 1~~as follows: i) the criteria in ~~UN~~MTC Test N.4 is based on ~~fix~~ the fixed-reaction kinetics of coal, which ~~is~~are not directly transferable to ~~solid recovered fuels, 2~~SRFs; ii) there is no published information on the selectivity and the correlation to large-scale storage of this ~~tests~~test for ~~solid recovered fuels, 3~~SRFs; iii) the self-heating process of ~~solid recovered fuels~~SRFs can undergo multi-step reactions at different temperature ranges, including phase transitions (melting). Low-temperature reactions and phase transitions are not covered by tests according to ~~UN~~the MTC N.4 method.

In the case of testing melting materials, sample preparation can be necessary. Corresponding instructions are given in ~~section~~ 8.4.

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 ~~below~~in this clause.

7.2 Isoperibolic test methods

7.2.1 General

The original basket heating test method was developed ~~at~~in 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[16] 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~~.~~[17],[18]. Lab-specific differences have been identified as possible reasons for the deviations, ~~e.g.:~~such as:

a) oven ventilation (enforced, natural convection~~),~~);

b) oven size~~,~~;