

<b>STN</b>	<b>Tuhé biopalivá</b> <b>Stanovenie samozohrievania peletizovaných</b> <b>biopalív</b> <b>Časť 1: Izotermická kalorimetria (ISO 20049-1:</b> <b>2020)</b>	<b>STN</b> <b>EN ISO 20049-1</b>  65 7426
------------	---	--

Solid biofuels - Determination of self-heating of pelletized biofuels - Part 1: Isothermal calorimetry (ISO 20049-1:2020)

Táto norma obsahuje anglickú verziu európskej normy.  
This standard includes the English version of the European Standard.

Táto norma bola oznámená vo Vestníku ÚNMS SR č. 09/20

Obsahuje: EN ISO 20049-1:2020, ISO 20049-1:2020

**131385**

EUROPEAN STANDARD

EN ISO 20049-1

NORME EUROPÉENNE

EUROPÄISCHE NORM

May 2020

ICS 27.190; 75.160.40

English Version

## Solid biofuels - Determination of self-heating of pelletized biofuels - Part 1: Isothermal calorimetry (ISO 20049-1:2020)

Biocombustibles solides - Détermination de l'auto-échauffement des granulés de biocombustibles - Partie 1: Détermination calorimétrique isotherme (ISO 20049-1:2020)

Biogene Festbrennstoffe - Bestimmung der Selbsterhitzung von Pellets aus biogenen Brennstoffen - Teil 1: Isotherme Kalorimetrie (ISO 20049-1:2020)

This European Standard was approved by CEN on 1 May 2020.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

**CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels**

<b>Contents</b>	<b>Page</b>
<b>European foreword.....</b>	<b>3</b>

## **European foreword**

This document (EN ISO 20049-1:2020) has been prepared by Technical Committee ISO/TC 238 "Solid biofuels" in collaboration with Technical Committee CEN/TC 335 "Solid biofuels" the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2020, and conflicting national standards shall be withdrawn at the latest by November 2020.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## **Endorsement notice**

The text of ISO 20049-1:2020 has been approved by CEN as EN ISO 20049-1:2020 without any modification.

# INTERNATIONAL STANDARD

# ISO 20049-1

First edition  
2020-05

---

---

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

### **Part 1: Isothermal calorimetry**

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

*Partie 1: Détermination calorimétrique isotherme*



Reference number  
ISO 20049-1:2020(E)

© ISO 2020

**ISO 20049-1:2020(E)****COPYRIGHT PROTECTED DOCUMENT**

© ISO 2020

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Fax: +41 22 749 09 47  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Principle</b> .....	<b>2</b>
<b>5 Apparatus</b> .....	<b>3</b>
<b>6 Sample handling</b> .....	<b>4</b>
6.1 General.....	4
6.2 Sampling.....	4
6.3 Sample transport and storage.....	4
6.4 Sample preparation.....	4
<b>7 Test procedure</b> .....	<b>5</b>
7.1 Temperature stabilisation.....	5
7.2 Sample vial preparation.....	5
7.2.1 Preparation procedure.....	5
7.2.2 Procedure to find proper test portion mass in case of influence from oxygen deficiency.....	5
7.3 Reference vial preparation.....	5
7.4 Measurement.....	6
7.4.1 First baseline measurement.....	6
7.4.2 Sample measurement.....	6
7.4.3 Second baseline measurement.....	6
7.4.4 Measurement data file.....	6
<b>8 Results</b> .....	<b>7</b>
8.1 Test data.....	7
8.2 Reported data.....	7
<b>9 Test report</b> .....	<b>7</b>
<b>10 Repeatability and reproducibility</b> .....	<b>8</b>
<b>Annex A (normative) Calibration of the calorimeter</b> .....	<b>9</b>
<b>Annex B (informative) Examples of screening data</b> .....	<b>11</b>
<b>Annex C (informative) Determination of reaction kinetics</b> .....	<b>13</b>
<b>Annex D (informative) Information on the Interlaboratory study (ILS)</b> .....	<b>21</b>
<b>Bibliography</b> .....	<b>27</b>

## ISO 20049-1:2020(E)

### Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

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](http://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<sup>[1]</sup> 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 this document, the heat flow generated from the test portion is measured directly.
- b) In basket heating tests, 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, CATs are found for a 1 l sample portion in the range 150 °C to 200 °C.

The application of the test data should thus be identified before selecting the appropriate analytical method.

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<sup>[2]</sup>.

**ISO 20049-1:2020(E)**

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<sup>[3]</sup>.

Isothermal calorimetry is used for determination of the thermal activity or heat flow of chemical, physical and biological processes. The method described in this document is developed for the measurement of heat flow from the self-heating of solid biofuel pellets, but the technique is most commonly used in the fields of pharmaceuticals, energetic materials, and cement<sup>[3]</sup> to <sup>[Z]</sup>.

Data from the isothermal calorimetry screening test procedure included in this document is intended for comparison of the spontaneous heat generation (self-heating) of solid biofuel pellets ([Annex B](#)).

Guidance is additionally given on the use of isothermal calorimetry test data for the calculation of the overall reaction rate of the heat producing reactions ([Annex C](#)).

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

## Part 1: Isothermal calorimetry

### 1 Scope

This document:

- a) specifies a general test procedure for quantification of the spontaneous heat generation from solid biofuel pellets using isothermal calorimetry;
- b) specifies a screening test procedure for wood pellets using an instrument temperature of 60 °C;
- c) establishes procedures for sampling and sample handling of solid biofuel pellets prior to the analysis of spontaneous heat generation; and
- d) gives guidance on the applicability and use of isothermal calorimetry for calculation of the net reaction rate of the heat producing reactions of solid biofuel pellets.

The test procedure given in this document quantifies the thermal power (heat flow) of the sample during the test, it does not identify the source of self-heating in the test portion analysed.

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

This document is applicable to solid biofuel pellets only.

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 20049:2020.

### 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*

ISO 18846, *Solid biofuels — Determination of fines content in quantities of pellets*

**koniec náhľadu – text ďalej pokračuje v platenej verzii STN**