SOLID MATERIALS:
SPONTANEOUS IGNITION TEMPERATURE
BY CONTINUOUS HEATING

Key words: solid materials, testing, ignition temperature

1. SCOPe

This test method specifies a procedure for determining the spontaneous ignition temperature $T_c$ of samples of radius $r$, and, from these temperatures, the material parameters $M$ and $P$ from the equation

$$\ln\left(\frac{\delta c T_c^2}{r^2}\right) = M - \frac{P}{T_c}$$

(1)

where $\delta_c$ is a Frank-Kamenetskii critical parameter. The theory is described more completely in Annex A. The method applies a linearly rising temperature in the test oven. The method is less time consuming and more practical than conventional methods utilizing constant ambient temperature.

2. FIELD OF APPLICATION

The test method is intended for any solid material consisting of grains, fibres, small particles or a single block. The thermal diffusivity of the material should not be smaller than $10^{-7}$ m$^2$/s. The porosity of the sample should be so high that oxygen depletion inside the sample remains minimal during the test. This can be checked after the test by inspection. Oxygen depleted samples ignite close to the surface. The influence of moisture content or other heavy mass transfer, microbiological activity, phase transitions or pyrophoric behaviour of the material are beyond the scope of this method.

3. REFERENCES

ISO 3261 Fire tests vocabulary.


4. DEFINITIONS

For the purposes of this test method, the definitions given in ISO 3261 and ISO/IEC Guide 52:1990 apply, together with the following:

4.1 Material
Solid substance which can be poured into or worked to a definite volume.

4.2 Specimen
A representative amount of the sample prepared to be tested.

5. SAMPLING

A representative portion of the material sufficient for three tests is collected. Care shall be taken that the sampling does not change the particle size distribution of the sampled material.

6 TESTING METHOD

6.1 Principle
The specimen made of the material to be tested is placed into an oven, where the temperature is raised continuously and linearly until spontaneous ignition occurs. In order to carry out the test quickly, the specimen should first be preheated close to but safely below the assumed spontaneous ignition temperature.
6.2 Apparatus

The test apparatus should consist of a computer-controlled, naturally ventilated oven. At least one K-type 0.25 mm thermocouple is connected below the oven ceiling for temperature measurement. Because temperature in the oven rises slowly, the heat capacity of the oven is not expected to have an influence on the test results.

Specimen holders are small baskets made of stainless steel gauze. At least three different sizes of the same shape are needed with linear dimensions increasing by a factor of two between steps. The specimen in a basket shall be placed in the middle of the furnace, preferably suspended from the ceiling of the furnace.

Two K-type 0.25 mm thermocouples are installed with the sample: one measures the temperature at the symmetrical midpoint of the basket and another is positioned halfway from the centre radially and at mid-height for recording internal temperature development.

The computer shall control heating according to the developed algorithm by using data measured by thermocouples. The temperatures shall be recorded as a function of time. The apparatus shall be equipped with a data acquisition system. Measurement arrangements are given schematically in Figure 1.

6.3 Preparation of test samples

The sample should represent the material to be tested as closely as practical. Therefore samples shall not be conditioned because this may result in changes in moisture content, grain size, density or escape of more volatile parts. If the sample is unisotropic, nonhomogenous, or specific ventilation conditions apply, the test should be carried out as a scaled down version of the actual situation. Therefore, unisotropic or nonhomogenous samples should be in the same position and be large enough to represent the real case.

The absolute size of the sample holders could vary according to applications. The shape of the basket should be a scaled down model of the shape for the container in the application. If this is not practical, a standard set could be cylinders with heights equal to diameters, and volumes starting from 0.1 litres, e.g. radii of 25, 50 and 100 mm.

The baskets are filled with the test materials to full volume, and the density of the porous sample shall correspond to that which prevails during practical application.

6.4 Procedure

The specimen should be preheated in an oven close to but clearly below the assumed spontaneous ignition temperature. The test oven can be used as a preheating oven. Thermal equilibrium with the environment should be reached within 30 K or less. The specimen shall then be transferred quickly into the test apparatus. Raising of the oven temperature may not exceed the condition

$$\beta \leq 1.413 \Delta T_n (\alpha / r_0^2)$$

where $\beta$, $\Delta T_n$, $\alpha$, $r_0$ are the temporal temperature slope in the oven, maximum allowable temperature and radius of the specimen, respectively. The measurement is carried out to satisfy the condition

$$|T_h - T_m| \leq 1/4 \Delta T_n$$

until a stationary temperature profile is reached within the sample. Here $T_h$ and $T_m$ are the half radius temperature and centre temperature of the sample, respectively. The fixed maximum temperature difference $\Delta T_n$ shall not exceed 5 K.

For an unknown sample follow the procedure:

1. Estimate the value of $\beta$ from earlier experience.
2. Continue measurement for some 15-30 minutes to suppress initial transients.
3. Raise the value $\beta$ as high as allowed by the condition

$$|T_h - T_m| \leq 1/4 \Delta T_n$$

4. For optimal time use start the test using the smallest sample.
5. The application defines the size of samples (see 6.3). The values below are defaults. Three different sizes of samples are needed for determination of parameters for one material. Use cylinders with radii of 25, 50 and 100 mm. The heights should be equal to the diameters of the cylinders.
6. After the smallest sample measure the next larger size utilizing the information gained so far.
7. If at least two determinations are available, the spontaneous ignition temperature of the third sample can be estimated using a computer program (see Annex B). Use this information to start the third run as close to the ignition temperature as is thought to be safe taking into account the measurement uncertainties. Use a preheating oven for the specimen to reach the initial temperature of the oven.
8. After the third run use the computer program (see Annex B) to calculate the material parameters of the specimen with their error estimates.

6.5 Expression of results

The results shall be expressed in SI-units. The main results to be expressed are the spontaneous ignition temperatures $T_c$ of the test samples. At least three measurements corresponding to different volumes of samples are necessary for determining from the Arrhenius plot of Equation (1) the parameters $M$ and $P$. They and their estimated error are obtained using least squares fitting and Students $t$-distribution at 67% confidence level. This can be calculated using the computer code presented in Annex B. As inputs, the code requires first the number of data pairs, and the $\delta_c$-parameter of the measured geometry (see Table A1 in Annex A). Next the radius of the each sample $r$ and corresponding ignition temperature $T_c$ are needed. The program outputs the fitted coefficients.

When $M$ and $P$ are known the spontaneous ignition temperature of a specimen of any size can be calculated from Equation (1) by using calculated $\delta_c$ values from Table A1 in Annex A or from literature by using the computer code presented in Annex B.

6.6 Accuracy

The heating conditions are defined to better than 5% accuracy. The general accuracy in measurements should be better than 10%.

6.7 Test report

The test report shall include the following information if relevant:

a) Name and address of the testing laboratory
b) Identification number of the test report
c) Name and address of the organisation or the person who ordered the test
d) Purpose of the test
e) Method of sampling and other circumstances (date and person responsible for sampling)
f) Name and address of the manufacturer or the supplier of the tested object
g) Name and other identification marks of the tested object
h) Description of the tested object
i) Date of supply of the tested object
j) Date of the test
k) Test method
l) Handling of the test specimens from time of sampling to testing
m) Identification of the test equipment and instruments used
n) Any deviations from the test method
o) Test results (use SI units)
p) Inaccuracy or uncertainty of the test result
q) Date and signature
THEORY OF SELF-IGNITION TEMPERATURE TESTS

Self-ignition temperature measurements yield ignition temperatures for specimens of different radii of the tested material. When critical temperature is reached the Frank-Kamenetskii theory yields the following relation between the radius of the specimen and the self-ignition temperature in a defined geometry

\[ \ln \left( \frac{\delta_c T_c^2}{r^2} \right) = M - \frac{P}{T_c} \]  

(A1)

where \( \delta_c, T_c, r \) are the Frank-Kamenetskii critical delta-parameter, critical ignition temperature and the radius of the specimen, respectively. The constants M and P are material parameters of the specimen determined from experimental data. The theory applies for large Biot numbers (\( Bi > 10 \)). The function (A1) describes a line in semi-logarithmic presentation when the abscissa is the inverse value of the absolute temperature (Arrhenius plot). A computer code was written in GWBASIC that fits a line on data points and determines coefficients M and P by linear regression. Once the material parameters are known, the correlation can also be used to calculate self-ignition temperatures corresponding to specimens of different radii of a measured material and vice versa.

The listing of the GWBASIC code is presented in Annex B.

As inputs, the code requires first the number of data pairs, and the \( \delta_c \)-parameter of the measured geometry. Next the radius of the specimen \( r \) and the corresponding ignition temperature \( T_c \) are needed. The code calculates coefficients M and P of the fitted line by linear regression. The code also calculates the standard deviation of coefficients M and P by applying Student’s t-test. The program outputs the fitted coefficients.

The gained function can be used to calculate self-ignition temperatures for specimens of other geometries that have the same material parameters. The code requires the \( \delta_c \)-parameter and radius of the new geometry \( r \), and finds, by iteration, the self ignition temperature of the new specimen. The self-ignition temperature with error estimates appears on the screen. The code also repeats the temperature calculation as many times as needed by asking for a new possible geometry.

Table A1. Approximate values of Frank-Kamenetskii \( \delta_c \)-parameters for some common geometries. Exact values are denoted by an asterisk(*).

<table>
<thead>
<tr>
<th>Geometry</th>
<th>Dimensions</th>
<th>( \delta_c )-parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Finite cylinder</td>
<td>Diameter 2r, height 2h</td>
<td>2.00 + 0.841 ( \frac{r^2}{h^2} )</td>
</tr>
<tr>
<td>Infinitely long cylinder</td>
<td>Radius r</td>
<td>2.00*</td>
</tr>
<tr>
<td>Equicylinder</td>
<td>Radius r, height 2r</td>
<td>2.76*</td>
</tr>
<tr>
<td>Sphere</td>
<td>Radius r</td>
<td>3.32*</td>
</tr>
<tr>
<td>Infinite slab</td>
<td>Thickness 2r</td>
<td>0.878*</td>
</tr>
<tr>
<td>Rectangular box</td>
<td>Sides 2r, 2l, 2m; r &gt; l,m</td>
<td>10.873(1+ ( \frac{r^2}{l^2} + \frac{r^2}{m^2} ))</td>
</tr>
<tr>
<td>Cube</td>
<td>Side 2r</td>
<td>2.52*</td>
</tr>
</tbody>
</table>
COMPUTER CODE FOR DATA REDUCTION OF SELF-IGNITION TEMPERATURE TESTS

10 REM COMPUTER PROGRAM FOR ANALYZING THE RESULTS OF SELFIGNITION EXPERIMENTS
20 REM FITS A CURVE ON DATAPoints ACCORDING TO FRANK-KAMENITSKII'S THEORY
30 REM THE THEORY HOLDS FOR LARGE BIOT NUMBERS hL/k > 10.
40 REM EG. FOR WOOD Bi ≈ 5, WHEN R = 25 mm. ACCORDINGLY, THOMAS'S THEORY IS
50 REM REQUIRED ONLY IF THE ANALYSE CONCERNS SMALL WELL CONDUCTING SAMPLES
60 REM SEE DRYSDALE, AN INTRODUCTION TO FIRE DYNAMICS, S. 260 - 261.
70 REM CALCULATES ESTIMATES OF CRITICAL TEMPERATURE FOR A GIVEN GEOMETRY
80 REM O. KESKI-RAHKONEN, 5.7.1990
90 DIM X(50), Y(50), S(50), R(30), TA(30), T(30)
100 REM STUDENT'S T-DISTRIBUTION VALUES FOR DOUBLE SIDED TEST AT 68 %
110 REM SIGNIFICANCE LEVEL (± STANDARD DEVIATION)
120 S(1)=1.97:S(2)=1.315:S(3)=1.172:S(4)=1.111:S(5)=1.077:S(6)=1.055
130 S(7)=1.04:S(8)=1.028:S(9)=1.02:S(10)=1.014:S(11)=1!:S(12)=1!
140 REM NUMBER OF MEASURED DATAPoints N
150 INPUT "NUMBER OF DATAPoints";N
160 IF N>14 THEN S(N-2) = 1
170 REM CRITICAL VALUE OF DELTA-C ACCORDING TO APPLIED GEOMETRY
180 INPUT "DELTA";D
190 REM INPUT OF MEASURED DATAPoints AND CALCULATION OF VARIABLES
200 REM THAT INPUT YIELDS
210 XM=O
220 YM=O
230 FOR I=1 TO N
240 REM INPUT THE RADIUS AND IGNITION TEMPERATURE OF THE SPECIMEN
250 INPUT "R [m],TA [°C]";R(I),TA
260 REM CHANGES TEMPERATURES TO KELVINS FROM CENTIGRADES
270 T(I)=TA+273.16
280 REM CALCULATES THE INVERSE VALUES OF ABSOLUTE TEMPERATURES
290 Y(I)=1/T(I)
300 REM CALCULATES ABSCISSAE USING DELTA, TEMPERATURE AND RADIUS
310 REM NOTE, THAT I/T IS NOW Y IN CONTRAST TO CONVENTIONAL
320 REM PRESENTATIONS. THE CHANGE IS DONE TO REDUCE THE FITTING ERROR
330 X(I)=LOG(D)+2*LOG(T(I)/R(I))
340 REM SUMS THE VALUES OF ABSCISSAE AND ORDINATES OVER THE GROUP OF FITTED
350 XM=XM+X(I)
360 REM XK JA YK ARE THE AVERAGES OF VARIABLES OVER THE DATAPoints
370 XK = XM/N
380 YM=YM+Y(I)
390 YK=YM/N
400 NEXT I
410 REM REGRESSION ANALYSIS OF DATA
420 REM FITS A LINE BY LEAST SQUARES TECHNIQUE
430 REM ERROR ANALYSIS IS GIVEN IN: OLLI LOKKI
440 REM STATISTICAL ANALYSIS AND USE OF RESEARCH DATA, INSINORITIETO
NORDTEST METHOD

450 REM HELSINKI 1980, P. 188 - 192 (IN FINNISH). THE SYMBOLS OF REGRESSION
460 REM VARIABLES ARE ALSO THE SAME AS IN THE BOOK.
470 REM SETS THE SUMMING VAMABLES TO ZERO
480 C00=C01=C11=0
490 REM SUMS THE VAMABLES NEEDED FOR FITTING
500 FOR I=1 TO N
510 REM GENERATES THE CROSS CORRELATION SUM
520 CO1 = CO1 + (X(I) - XK)*Y(I)
530 REM GENERATES THE SUM OF SQUARES OF ORDINATES
540 C00 = C00 +(Y(I) - YK)^2
550 REM FORMS THE SUM OF SQUARES OF ABSCISSAE
560 C11 = C11 t (X(I) - XK)^2
570 NEXT I
580 REM CALCULATES THE COEFFICIENTS M AND P OF LINE Y = M - P*X
590 REM CALCULATES THE DIVIDING DETERMINANT OF THE COEFFICIENTS
600 BO = YK
610 B1 = C01/C11
620 REM JUMPS OVER THE ERROR CALCULATION IF ONLY TWO DATAPPOINTS ARE AVAILABLE
630 IF N <= 2 THEN 720
640 REM CALCULATES THE STANDARD DEVIATION
650 SO = SQR((C00 - B1*C11)/(N - 2))
660 REM CALCULATES THE ACCURACY OF BO AND B1 BY USING STUDENT'S TEST
670 DBO = S0*S(N-2)/SQR(N)
680 DB1 = S0*S(N-2)/SQR(C11)
690 REM CALCULATES THE ACCURARY OF M AS SUMS OF SQUARES FROM INDEPENDENT
695 REM VARIABLES
700 DM = SQR(DB0*DB0 + (XK*DB1)^2)
710 DP = DB1
720 REM OUTPUTS THE FITTED COEFFICIENTS
730 M = BO - XK*B1
740 P = -B1
750 PRINT "M =";M;
760 PRINT USING"#.###^#####";M;
770 PRINT "±";
780 PRINT USING"#.###^#####";DM
790 PRINT "P =";
800 PRINT USING"#.###^#####";P;
810 PRINT "±";
820 PRINT USING"#.###^#####";DB1
830 REM CALCULATES CRITICAL TEMPERATURES FOR GIVEN NEW GEOMETRICAL CASES
840 REM THAT HAVE THE SAME MATERIAL PARAMETERS AS THOSE IN THE MEASURED CASE
850 REM ASKS FOR THE CRITICAL DELTA-C VALUE OF THE NEW GEOMETRY
860 INPUT"INPUT DC";DC
870 REM ASKS THE RADIUS IN THE NEW GEOMETRY
880 INPUT"INPUT R[M]";R
890 REM ITERATES THE CRITICAL TEMPERATURE VALUE FOR A GIVEN SAMPLE
900 REM INITIAL TEMPERATURE DEFAULT
910 T1=300
920 REM ITERATING LOOP
930 \[ T = \frac{1}{P} \left( \frac{M}{P} - \log(DC^2(T1/R)^2) \right) \]
940 REM CONDITION FOR CONVERGENCE
950 IF ABS(T-T1) < .01 THEN 960
960 T1=T
970 GOTO 930
980 REM ERROR ASSESSMENT OF CALCULATED TEMPERATURE
990 DY = S0*S(N-2)*SQR(1+1/N+(LOG(DC^2*T*T/(R*R))-XK)^2/C11)
1000 DT= T*T*DY
1010 REM OUTPUTS THE CALCULATED TEMPERATURE WITH ERROR LIMITS
1020 PRINT "CRITICAL TEMPERATURE =";
1030 PRINT USING"####.##";T-273.16;
1040 PRINT "±";
1050 PRINT USING"####.##";DT;
1060 PRINT " °C"
1070 REM REPEATS THE TEMPERATURE CALCULATION WHEN REQUIRED
1080 INPUT "WILL YOU HAVE A NEW GEOMETRY? K/E";VASTAU$
1090 IF VASTAU$="K" THEN GOTO 860
1100 END