



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 0129 : 2005
(JAIMA/JSA)

General rules for thermal analysis

ICS 71.040.50

Reference number : JIS K 0129 : 2005 (E)

PROTECTED BY COPYRIGHT

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee, as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Analytical Instruments Manufacturers' Association (JAIMA)/ Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14. Consequently **JIS K 0129 : 1994** is replaced with this Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

Date of Establishment: 1994-03-01

Date of Revision: 2005-05-20

Date of Public Notice in Official Gazette: 2005-05-20

Investigated by: Japanese Industrial Standards Committee

Standards Board

Technical Committee on Chemical Analysis

JIS K 0129:2005, First English edition published in 2005-11

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

© JSA 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

PROTECTED BY COPYRIGHT

Contents

	Page
1 Scope	1
2 Normative references	1
3 Definitions	1
4 Summary	6
5 Apparatus.....	6
5.1 Construction of apparatus	6
5.2 Example of construction of apparatus	7
5.3 Accessory device.....	12
5.4 Additional functions	13
6 Operation	14
6.1 Installing conditions of apparatus.....	14
6.2 Calibration of apparatus	15
6.3 Preparation of sample.....	16
6.4 Establishment of operational condition for apparatus	17
6.5 Measurement	17
7 Control of quality of data	18
8 Reading of measurement results	19
9 Matter to be mentioned in analytical results	19
9.1 Expression method of numerical value	19
9.2 Report	19
10 Matter to be specified in individual standard	20

General rules for thermal analysis

1 Scope This Japanese Industrial Standard specifies the general rules for thermal analysis carried out using differential thermal analyzer, differential scanning calorimeter, apparatus for thermogravimetry, apparatus for thermomechanical analysis and apparatus for dynamic mechanical analysis.

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0050 *General rules for chemical analysis*

JIS K 0215 *Technical terms for analytical chemistry (analytical instrument part)*

JIS K 7115 *Plastics—Determination of creep behaviour—Part 1: Tensile creep*

JIS K 7121 *Testing methods for transition temperatures of plastics*

JIS K 7161 *Plastics—Determination of tensile properties—Part 1: General principles*

JIS K 7244-1 *Plastics—Determination of dynamic mechanical properties—Part 1: General principles*

3 Definitions For the purpose of this Standard the definitions given in **JIS K 0050**, **JIS K 0215**, **JIS K 7115**, **JIS K 7121** and **JIS K 7244-1** and the following definitions apply.

- a) **thermal analysis: TA** a generic term for the series of techniques to measure the physical properties of a substance as the function of temperature, while the temperature of the substance including the reaction products is being changed in accordance with a specified program
- b) **differential thermal analysis: DTA** a method to measure the thermal difference between sample and reference material as the function of temperature, while the temperature of both the sample and reference material is being changed in accordance with a specified program
- c) **differential scanning calorimetry: DSC** a generic term for the following measuring methods:
 - 1) **input compensation type differential scanning calorimetry (input compensation type DSC)** a method to measure the input difference of heat energy, as the function of temperature, applied on sample and reference material per unit time so as to make their temperature equal, while the temperature of sample part, composed of the sample and reference material, is being changed in accordance with a specified program
 - 2) **heat flux type differential scanning calorimetry (heat flux type DSC)** a method to measure the temperature difference between sample and reference material, as the function of temperature, while the temperature of sample part, composed of the sample and reference material, is being changed in accordance with a specified program

This temperature difference is proportional to the input difference of thermal energy per unit time.

- d) **thermogravimetry: TG** a method to measure the mass of sample as the function of temperature or time, while its temperature is being changed or kept in accordance with a specified program
 - 1) **simultaneous thermogravimetry and differential thermal analysis: TG-DTA** a method in which thermogravimetry and differential thermal analysis are combined and measured simultaneously by a single apparatus
 - 2) **thermogravimetry and mass spectrometry: TG/MS** a method in which the gas desorbed or produced from the sample in thermogravimetry is introduced to mass spectrometers (MS) and the gas component is identified
 - 3) **thermogravimetry and Fourier transform infrared spectrometry: TG/FTIR** a method in which the gas desorbed or produced from the sample in thermogravimetry is introduced to Fourier transform infrared spectrophotometer and the gas component is identified

Information : The symbol of “–” is used when there is not the timely order in the measurement, and the symbol of “/” is used when there is the timely order in the measurement.
- e) **dynamic mechanical analysis: DMA** a method in which the complex modulus of the sample is measured as the function of temperature or time while the temperature of sample is changed or maintained according to a definite program
- f) **thermomechanical analysis: TMA** a method to measure the deformation of sample, as the function of temperature or time, applying non-oscillatory load such as compression, tension, bending and torsion, while its temperature is being changed in accordance with a specified program
- g) **tensile-mode measurement** a method by using TMA apparatus to measure the displacement of a film type or fibrous sample of which upper end and lower end are fixed with small chucks supported by the probes and on which a tension load is applied
- h) **bending-mode measurement** a method by using TMA apparatus to measure the displacement caused by bending of a sample which is placed on the two supporting points and on the centre of which a compressive load is applied
- i) **penetration-mode measurement** a method by using TMA apparatus to measure the displacement caused by softening of a sample on which a compressive load is applied by the needle-shaped probe
- j) **thermodilatometry** a method by using TMA apparatus to measure the displacement caused by expansion of a sample on which a load so as not to give a deformation to sample is applied in the range of measuring temperature using a flat-pointed probe

When film type or fibrous sample is measured under above-mentioned tensile mode, this is a method to measure the displacement caused by thermal expansion

of the sample when a load is applied so as not to give deformation to the sample in the range of measuring temperature.

- k) **derivative differential thermal analysis** a technique to give the first derivative on the time or temperature of DTA curve
- l) **derivative differential scanning calorimetry** a technique to give the first derivative on the time or temperature of DSC curve
- m) **derivative thermogravimetry** a technique to give the first derivative on the time or temperature of TG curve
- n) **derivative thermomechanical analysis** a technique to give the first derivative on the time or temperature of TMA curve
- o) **differential scanning calorimeter** the apparatus employed for differential scanning calorimetry
- p) **reference materials (for thermal analysis)** in the thermal analysis, the materials which can be used for comparing its thermal change with that of sample
- q) **standard materials (for thermal analysis)** in the thermal analysis, the materials which can be used for calibrating temperature or other physical quantity
- r) **DTA curve** the curve plotted at differential thermal analysis by graduating the temperature difference on ordinate and temperature or time on abscissa
- s) **DSC curve** at differential scanning calorimetry, the curve plotted by graduating the input difference of heat energy, applied per unit time in sample and reference material so as to make their temperatures equal, on ordinate, and temperature or time on abscissa
- t) **TG curve** the curve plotted at thermogravimetry by graduating mass on ordinate and temperature or time on abscissa
- u) **TMA curve** the curve plotted at thermomechanical analysis by graduating the amount of deformation on ordinate and temperature or time on abscissa
- v) **stress-strain curve** the curve plotted at stress-strain test by graduating stress on ordinate and strain on abscissa
- w) **creep curve** the curve plotted at creep test by graduating the amount of deformation on ordinate and time on abscissa
- x) **stress relaxation curve** the curve plotted at stress relaxation test by graduating stress on ordinate and time on abscissa
- y) **DMA curve** a curve obtained in dynamic mechanical analysis in which complex modulus is taken on an ordinate and temperature or time is taken on an abscissa
- z) **baseline** the range of temperature or time of TG curve, DTA curve, DSC curve or TMA curve where no change is generated on the sample

In case of measuring specific heat capacity by DSC, it means the steady state of curve given at isothermal condition before or after raising temperature.

- aa) **peak** the part of DTA curve or DSC curve from the instant when the curve leaves the baseline to the instant when it touches the line again
- ab) **extrapolated onset temperature** a temperature of intersection at which, in heating (cooling) measurement, the straight line, in which the base line at the side of low temperature (high temperature) is extended to the side of high temperature (low temperature), is intersected to the tangent drawn at the point where the gradient comes in maximum to the curve at the side of low temperature (high temperature) of peak
- ac) **mid-point temperature** a temperature at which the change of physical quantity reaches 50 % of the total amount of change
- ad) **peak temperature** a temperature at the top of peak
- ae) **extrapolated end temperature** a temperature of intersection at which, in heating (cooling) measurement, the straight line, in which the base line at the side of high temperature (low temperature) is extended to the side of low temperature (high temperature), is intersected to the tangent drawn at the point where the gradient comes in maximum to the curve at the side of high temperature (low temperature) of peak
- af) **transition enthalpy** the quantity of heat which is absorbed or released caused by transition
 That is obtained according to the comparison method of transition peak area so that the quantity of heat of reference material [the heat of fusion of indium (In), typically] measured using the same apparatus is made to be standard.
- ag) **Curie temperature** transition temperature at which magnetic property changes from ferromagnetism to paramagnetism
- ah) **glass transition temperature** temperature at which liquid state changes into noncrystalline solid (glass state) or noncrystalline solid (glass state) changes into liquid state
- ai) **linear (thermal) expansion** a value in which when the length in one axial direction is supposed to change from l_1 to l_2 at the time of changing of the temperature of sample from T_1 to T_2 , the changed length ($l_2 - l_1$) is divided by the length (l_0) of sample at room temperature or 0 °C

$$e = \frac{l_2 - l_1}{l_0} \dots\dots\dots (1)$$

where, e : linear thermal expansion
 l_0 : length of sample at room temperature or 0 °C
 l_1 : length of sample at T_1 in temperature
 l_2 : length of sample at T_2 in temperature

- aj) **coefficient of linear (thermal) expansion** when sample length in one axis direction changes by linear thermal expansion, the coefficient $[dl/dT]/l_0$ of its ratio to the temperature change

It is also called factor of thermal expansion, coefficient of expansion, or factor of linear expansion

$$\alpha = \frac{\left(\frac{dl}{dT} \right)}{l_0} \dots\dots\dots (2)$$

where, α : coefficient of liner thermal expansion
 dl : change of length of sample
 dT : change of temperature
 l_0 : length of sample at room temperature or 0 °C

ak) **mean coefficient of linear (thermal) expansion** the value $\{(l_2 - l_1)/[l_0(T_2 - T_1)]\}$ obtained by dividing the length difference $(l_2 - l_1)$ by both the sample length (l_0) at room temperature or 0 °C and temperature difference $(T_2 - T_1)$ when sample length changes from l_1 to l_2 in one axis direction owing to the temperature change of sample from T_1 to T_2 .

It is also called mean coefficient of thermal expansion, coefficient of linear thermal expansion, or mean factor of linear expansion.

$$\alpha_{T_2-T_1} = \frac{l_2 - l_1}{l_0(T_2 - T_1)} \dots\dots\dots (3)$$

where, $\alpha_{T_2-T_1}$: mean coefficient of linear thermal expansion
 T_1, T_2 : temperature of sample
 l_0 : length of sample at room temperature or 0 °C
 l_1 : length of sample at T_1 in temperature
 l_2 : length of sample at T_2 in temperature

al) **complex modulus** the ratio of complex stress amplitude to complex strain amplitude when sinusoidal vibration is added to sample

am) **storage modulus** the part of real number of complex modulus

It is the index to express the degree of difficulty of deformation from vibration load, and is proportional to the maximum energy accumulated in sample throughout the vibration period

an) **loss modulus** the part of imaginary number of complex modulus

It is proportional to the energy dissipated as heat in sample during one cycle of vibration

ao) **loss factor** the ratio of loss modulus to storage modulus

When the delay of vibration strain phase to vibration stress is supported to be δ , it is expressed by $\tan \delta$ and also called the loss tangent

ap) **elasticity** the property in which strain is proportional to stress

aq) **viscosity** the property in which strain rate is proportional to stress

ar) **viscoelasticity** the property having viscosity and elasticity together

as) **modulus of elasticity** the ratio of stress to strain and the index to express the degree of difficulty of deformation when a definite external force is added to the prescribed shape of material