JIS

JAPANESE INDUSTRIAL STANDARD

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Japanese Standards Association

JIS Z 3198-1 : 2003

(JWES)

Test methods for lead-free solders—Part 1 : Methods for measuring of melting temperature ranges

ICS 25.160.50

Reference number : JIS Z 3198-1 : 2003 (E)

Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal of establishing a Japanese Industrial Standard from The Japan Welding Engineering Society (JWES), with a draft of Industrial Standard based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

Soldering is called HANDAZUKE (Japanese), a technique to be used for mounting electronic and electrical machinery and apparatus, communication equipment and the like and its use field is wide and expectation for high reliability of the connection is large.

Though there are standards inside and outside Japan as well as International Standards like **IEC** or **ISO**, this Standard uses results of "standardization of test methods and the like necessary for solder connection corresponding to the reduction of an environmental load" based on the research and development by contract of New Energy and Industrial Technology Development Organization.

This Standard concerns the methods for measuring a melting temperature range of lead-free solders which are environmentally friendly and is positioned as a standard for considering environment.

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.

JIS Z 3198 includes the following 7 parts with the general title *Test methods for lead-free solders*

Part 1 : Methods for measuring of melting temperature ranges
Part 2 : Methods for testing of mechanical characteristics—tensile test
Part 3 : Methods for spread test
Part 4 : Methods for solderability test by a wetting balance method and a contact angle method
Part 5 : Methods for tensile tests and shear tests on solder joints
Part 6 : Methods for 45° pull test of solder joints on QFP lead
Part 7 : Methods for shear strength of solder joints on chip components

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

Printed in Japan

Contents

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Introduction This Japanese Industrial Standard specifies the methods for measuring of melting temperature ranges of lead-free solders based on occupational results by contract of New Energy and Industrial Technology Development Organization concerning "the standardization of test methods and the like necessary for solder connection corresponding to the decrease of an environmental load" carried out in 2000 and 2001.

1 Scope This Standard specifies the methods for measuring of melting temperature ranges of lead-free solders used principally for connection of wiring, connection of parts and the like of electrical machinery and apparatus, electronic apparatus, communication equipment and the like.

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

JIS C 1601 *Indicating thermoelectric thermometers*

JIS C 1605 *Mineral insulated thermocouples*

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

JIS K 8034 *Acetone*

JIS R 1301 *Porcelain crucibles for chemical analysis*

JIS R 2701 *Graphite crucible and its accessories*

JIS Z 3001 *Welding terms*

JIS Z 8401 *Guide to the rounding of numbers*

JIS Z 8704 *Temperature measurement—Electrical methods*

3 Definitions For the purposes of this Standard, the definitions given in **JIS Z 3001** and the following definitions apply:

a) **lead-free solder** It is the generic term of "a tin based solder" not containing lead as an alloy component. It is a solder not containing lead for a use corresponding to that of "a tin-lead based solder", which is used for mounting an electrical, electronic, communication equipment and the like.

b) **fusion start temperature** A temperature at which a lead-free solder starts to melt. It is generally marked as a solidus temperature.

c) **solidification start temperature** A temperature at which solidification of lead-free solder starts. It is generally marked as a liquidus temperature.

4 Summary of test The fusion temperature range of lead-free solder (hereafter referred to as "solder") is marked by a fusion start temperature and a solidification start temperature. The fusion start temperature is measured by using a differential scanning calorimetry (**DSC**) or a differential thermal analysis (**DTA**) and further a solidification start temperature is measured by using the cooling curve of a fused solder.

5 Fusion start temperature

5.1 Test method A test method shall be in accordance with either of the following methods:

a) **Differential scanning calorimetry** (hereafter referred to as "DSC") For a differential scanning calorimetry, an input-compensating DSC and a heat flux DSC shall be adopted.

b) **Differential thermal analysis** (hereafter referred to as "DTA")

5.2 Apparatus and equipment The apparatus and equipment to be used for a test shall be principally in accordance with **JIS K 7121**, and shall be as follows:

5.2.1 DSC apparatus and DTA apparatus The apparatus shall have two container holders whose heat capacities are equal and have a structure capable of being heated and cooled by the same heat exchange conditions.

The input-compensating DSC shall have a structure capable of measuring a difference of applied heat energy per unit time between a sample and a reference material so that a temperature of the sample and that of the reference materials become the same.

The heat flux DSC shall have a structure so that a temperature difference between the sample and the reference material is proportional to a difference between inputs in heat energy per time.

a) **Heating velocity** A heating velocity shall be 1 °C to 10 °C per min and its precision shall be within ±10 %.

b) **Gas inflow device** A gas inflow device shall have a structure capable of making a gas come around the sample.

c) **Container** A container shall be made of a material of a high thermal conductivity which is free from being eroded with the sample. The container made of aluminium is generally used.

d) **Recorder** A recorder shall be able to continuously record a DSC curve or a DTA curve.

e) **Noise level** A noise level shall be ≦ 1/10 of a peak height.

5.2.2 Appliance An appliance shall be as follows:

a) **Gas flow meter** A gas flow meter shall be able to measure a range of 10 ml to 50 ml per min.

b) **Scale** A scale shall be 0.1 mg and upward in sensitivity.

5.3 Calibration of temperature For calibration of a temperature, pure material, of purity not less than 99.99 %, given in Table 1 shall be used. Melting points of pure materials of two or more kinds near a temperature to be obtained, are measured by the same conditions as those for the sample, and temperature is calibrated by obtaining a calibration formula of a primary function from the obtained measured value and the melting point in Table 1.

Table 1 Melting point of pure material

Name of pure material	Melting point °C
Indium	156.6
Tin	231.9
Lead	327.4

5.4 Sample The mass of a sample shall be 5 mg to 50 mg. Unless otherwise specified, it shall be about 10 mg. The pretreatment of the sample shall be as agreed between the manufacturer and the purchaser.

5.5 Procedure A procedure shall be as follows.

a) **Installation of sample in container** Place a sample in an almost central part of a container, put a cover of the container and clamp them.

b) **Installation of container** Install a container in which a sample is packed to container holder on one side, and install a blank container to which the cover is clamped or the container in which alumina powder is packed to container holder on the other side.

c) **Atmosphere of sample** Set a flow rate of nitrogen gas to a suitable value within a range of 10 ml to 50 ml per min and make it flow in until the end of the test.

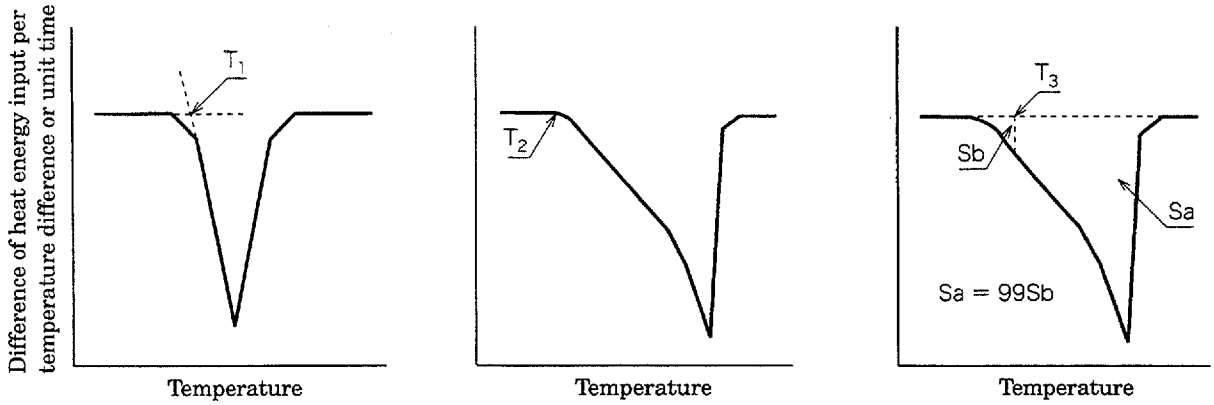
d) **Measurement** Make a heating velocity 1 °C to 10 °C per min and heat to a temperature higher by about 30 °C than that at the end of the melting peak. Unless otherwise specified, make the heating velocity about 2 °C per min.

5.6 Method of obtaining fusion start temperature The method of obtaining a fusion start temperature shall be as follows:

a) In the case where melting is sudden, a fusion start temperature shall be a temperature T_1 at an intersecting point of the straight line elongated from a base line on the low temperature side to the base line on the high temperature side as given in Fig. 1 **a**) and a tangential line drawn along the curved line on the low temperature side of a melting peak so that the inclination becomes the maximum.

b) In the case where melting is gradual, a point T_2 wherein the curved line given in Fig. 1 **b**) starts to deviate from the base line is obtained. In this case, calibration is carried out by using a point wherein the curve of a pure material starts to deviate from the base line. Measurement is carried out several times and their mean shall be taken.

c) In the case where melting is gradual but a point starting to deviate from the base line is indistinct, instead of the fusion start temperature given in Fig. 1 **c**), a peak area from near a point wherein the curve starts to deviate from the base line to a point of returning to the base line on the high temperature side is obtained, and a temperature corresponding to 1 % of this area is obtained as 1 % fusion temperature (T_3) from the fusion start point. The measurement is carried out several times and their mean is taken.



a) In the case where melting is sudden **b) In the case where melting is gradual** **c) In the case where a point starting to deviate is indistinct**

Fig. 1 Method for obtaining of fusion start temperature

5.7 Rounding off of numerical value The temperature is obtained by calculating down to the first decimal place and rounded off in accordance with the method specified in **JIS Z 8401**.

6 Solidification start temperature

6.1 Test method For a test method, melt a sample in a container in an electric furnace and obtain a cooling curve when it is allowed to cool in the electric furnace while the temperature is recorded.

6.2 Apparatus and equipment The apparatus and equipment shall be as follows.

a) **Electric furnace** An electric furnace which can heat to ≧400 °C and is excellent in heat insulation properties, shall be used.

b) **Thermocouple** Among thermocouples of different classes specified in **JIS C 1602**, that conforming to the use temperature shall be used.

When a sheathed thermocouple is used, that conforming to the use temperature among those of different classes specified in **JIS C 1605** shall be used.

For a lead wire, that suitable for the thermocouple to be used, shall be used.

c) **Measuring instrument** A measuring instrument specified in **10.5** of **JIS Z 8704** which can measure at an interval of ≦1 s shall be used.

d) **Recorder** A recorder which can record a cooling curve and can read to the nearest 0.1 °C, shall be used.

e) **Container** A graphite crucible specified in **JIS R 2701** or a porcelain crucible specified in **JIS R 1301** shall be used.

6.3 Calibration of temperature Calibrate a temperature in the same way as that in **5.3**.

6.4 Pretreatment of sample Clean a sample by acetone specified in **JIS K 8034**.

6.5 Procedure The procedure shall be as follows:

a) **Mass of sample** The mass of a sample shall be 500 g or over.

b) **Melting of sample** Put a sample in a crucible and melt it by heating in an electric furnace.

c) **Installation of thermocouple** Install a temperature measuring contact part of a thermocouple at almost the central part of a molten sample.

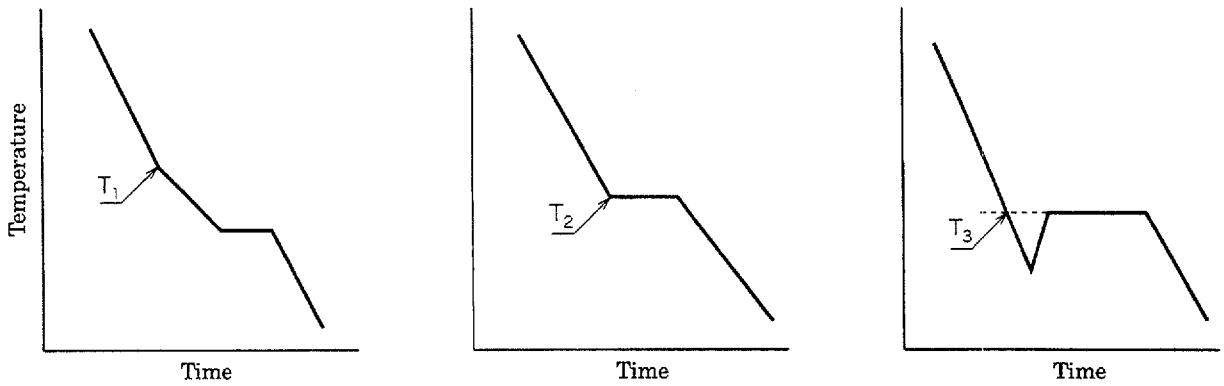
6.5.1 Reference contact point For a reference contact point, a freezing point type, a thermoelectric cooling type and a compensation type reference junction specified in **10.3** of **JIS Z 8704** shall be used.

6.5.2 Measurement The measurement shall be as follows:

a) Melt a sample completely in a crucible.

b) Turn off the power source of an electric furnace and measure a temperature in the cooling process.

6.6 Method for obtaining solidification start temperature For a solidification start temperature, the solidification start temperature (T_2) is obtained from the bend point (T_1) of a cooling curve (time—temperature curve) given in Fig. 2 **a**) and the horizontal part (T_2) given in Fig. 2 **b**). When not less than two positions of the bend point or the horizontal part appear, the firstly appeared part is made a solidification start temperature. Further, in the case where supercooling is appeared as given in Fig. 2 **c**), a point (T_3) wherein an elongated line to a low time side of the horizontal part crosses the cooling curve is made a solidification start temperature.



a) In the case where bend point appears **b) In the case where horizontal part appears** **c) In the case where supercooling appears**

Fig. 2 Method for obtaining solidification start temperature

6.7 Rounding off of numerical value Obtain the temperature to one decimal place and round off by the method specified in **JIS Z 8401**.

7 Recording

7.1 Recording of fusion start temperature Record the following items:

a) Classification of sample

b) Name and type of a test apparatus

c) Shape and material of a container

d) Mass of a sample

e) Pretreatment of a sample

f) Flowin velocity of nitrogen gas

g) Heating velocity, measurement starting temperature and end temperature

h) Classification of calibrating material, its read temperature and conversion formula

i) Read temperature and fusion start temperature or 1 % melting temperature of a sample

j) Measuring date

k) Items agreed between the manufacturer and the purchaser

l) Others

7.2 Recording of solidification start temperature Record the following items:

a) Classification of sample

b) Mass of sample

c) Classification of container

d) Classification of thermocouple

e) Heating temperature and retention time

f) Cooling velocity just before solidification start temperature

g) Classification of calibration material, its read temperature and conversion formula

h) Read temperature and solidification start temperature of sample

i) Date of measurement

j) Items agreed between the manufacturer and the purchaser

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c) **Container** A container shall be made of a material of a high thermal conductivity which is free from being eroded with the sample. The container made of aluminium is generally used.

d) **Recorder** A recorder shall be able to continuously record a DSC curve or a DTA curve.

e) **Noise level** A noise level shall be ≦ 1/10 of a peak height.

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b) **Installation of container** Install a container in which a sample is packed to container holder on one side, and install a blank container to which the cover is clamped or the container in which alumina powder is packed to container holder on the other side.

c) **Atmosphere of sample** Set a flow rate of nitrogen gas to a suitable value within a range of 10 ml to 50 ml per min and make it flow in until the end of the test.

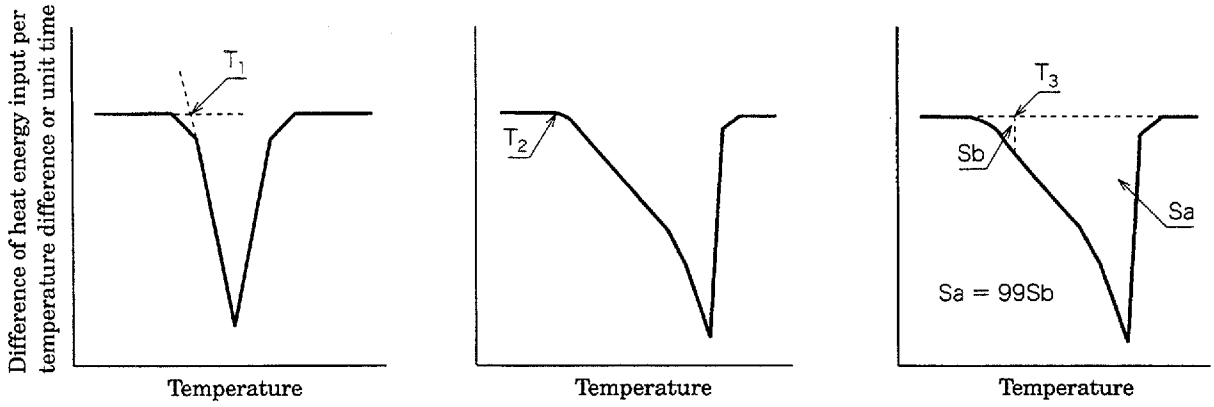
d) **Measurement** Make a heating velocity 1 °C to 10 °C per min and heat to a temperature higher by about 30 °C than that at the end of the melting peak. Unless otherwise specified, make the heating velocity about 2 °C per min.

5.6 Method of obtaining fusion start temperature The method of obtaining a fusion start temperature shall be as follows:

a) In the case where melting is sudden, a fusion start temperature shall be a temperature T_1 at an intersecting point of the straight line elongated from a base line on the low temperature side to the base line on the high temperature side as given in Fig. 1 **a**) and a tangential line drawn along the curved line on the low temperature side of a melting peak so that the inclination becomes the maximum.

b) In the case where melting is gradual, a point T_2 wherein the curved line given in Fig. 1 **b**) starts to deviate from the base line is obtained. In this case, calibration is carried out by using a point wherein the curve of a pure material starts to deviate from the base line. Measurement is carried out several times and their mean shall be taken.

c) In the case where melting is gradual but a point starting to deviate from the base line is indistinct, instead of the fusion start temperature given in Fig. 1 **c**), a peak area from near a point wherein the curve starts to deviate from the base line to a point of returning to the base line on the high temperature side is obtained, and a temperature corresponding to 1 % of this area is obtained as 1 % fusion temperature (T_3) from the fusion start point. The measurement is carried out several times and their mean is taken.



a) In the case where melting is sudden **b) In the case where melting is gradual** **c) In the case where a point starting to deviate is indistinct**

Fig. 1 Method for obtaining of fusion start temperature

5.7 Rounding off of numerical value The temperature is obtained by calculating down to the first decimal place and rounded off in accordance with the method specified in **JIS Z 8401**.

6 Solidification start temperature

6.1 Test method For a test method, melt a sample in a container in an electric furnace and obtain a cooling curve when it is allowed to cool in the electric furnace while the temperature is recorded.

6.2 Apparatus and equipment The apparatus and equipment shall be as follows.

a) **Electric furnace** An electric furnace which can heat to ≧400 °C and is excellent in heat insulation properties, shall be used.

b) **Thermocouple** Among thermocouples of different classes specified in **JIS C 1602**, that conforming to the use temperature shall be used.

When a sheathed thermocouple is used, that conforming to the use temperature among those of different classes specified in **JIS C 1605** shall be used.

For a lead wire, that suitable for the thermocouple to be used, shall be used.

c) **Measuring instrument** A measuring instrument specified in **10.5** of **JIS Z 8704** which can measure at an interval of ≦1 s shall be used.

d) **Recorder** A recorder which can record a cooling curve and can read to the nearest 0.1 °C, shall be used.

e) **Container** A graphite crucible specified in **JIS R 2701** or a porcelain crucible specified in **JIS R 1301** shall be used.

6.3 Calibration of temperature Calibrate a temperature in the same way as that in **5.3**.

6.4 Pretreatment of sample Clean a sample by acetone specified in **JIS K 8034**.

6.5 Procedure The procedure shall be as follows:

a) **Mass of sample** The mass of a sample shall be 500 g or over.

b) **Melting of sample** Put a sample in a crucible and melt it by heating in an electric furnace.

c) **Installation of thermocouple** Install a temperature measuring contact part of a thermocouple at almost the central part of a molten sample.

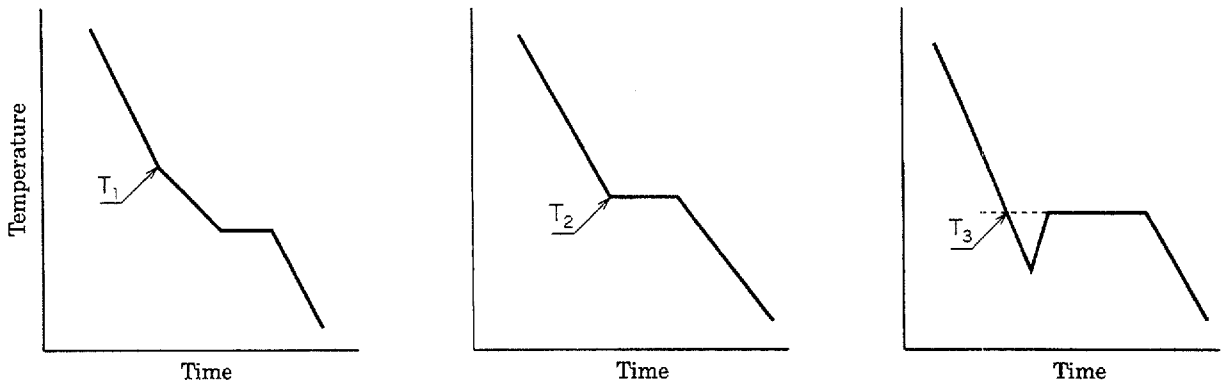
6.5.1 Reference contact point For a reference contact point, a freezing point type, a thermoelectric cooling type and a compensation type reference junction specified in **10.3** of **JIS Z 8704** shall be used.

6.5.2 Measurement The measurement shall be as follows:

a) Melt a sample completely in a crucible.

b) Turn off the power source of an electric furnace and measure a temperature in the cooling process.

6.6 Method for obtaining solidification start temperature For a solidification start temperature, the solidification start temperature (T_2) is obtained from the bend point (T_1) of a cooling curve (time—temperature curve) given in Fig. 2 **a**) and the horizontal part (T_2) given in Fig. 2 **b**). When not less than two positions of the bend point or the horizontal part appear, the firstly appeared part is made a solidification start temperature. Further, in the case where supercooling is appeared as given in Fig. 2 **c**), a point (T_3) wherein an elongated line to a low time side of the horizontal part crosses the cooling curve is made a solidification start temperature.



a) In the case where bend point appears **b) In the case where horizontal part appears** **c) In the case where supercooling appears**

Fig. 2 Method for obtaining solidification start temperature

6.7 Rounding off of numerical value Obtain the temperature to one decimal place and round off by the method specified in **JIS Z 8401**.

7 Recording

7.1 Recording of fusion start temperature Record the following items:

a) Classification of sample

b) Name and type of a test apparatus

c) Shape and material of a container

d) Mass of a sample

e) Pretreatment of a sample

f) Flowin velocity of nitrogen gas

g) Heating velocity, measurement starting temperature and end temperature

h) Classification of calibrating material, its read temperature and conversion formula

i) Read temperature and fusion start temperature or 1 % melting temperature of a sample

j) Measuring date

k) Items agreed between the manufacturer and the purchaser

l) Others

7.2 Recording of solidification start temperature Record the following items:

a) Classification of sample

b) Mass of sample

c) Classification of container

d) Classification of thermocouple

e) Heating temperature and retention time

f) Cooling velocity just before solidification start temperature

g) Classification of calibration material, its read temperature and conversion formula

h) Read temperature and solidification start temperature of sample

i) Date of measurement

j) Items agreed between the manufacturer and the purchaser

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