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**Building boards Determination of
formaldehyde emission—
Desiccator method**

ICS 13.040.99; 71.080.80; 79.060.10

Descriptors : wall coverings, formaldehyde, chemical hazards, pollutant gases

Reference number : JIS A 1460 : 2001 (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 in accordance with the Industrial Standardization Law:

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Building boards Determination of formaldehyde emission— Desiccator method

1 Scope This Japanese Industrial Standard specifies a method of test for determination of quantity of formaldehyde emitted from building boards.

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 K 0050 *General rules for chemical analysis*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 8001 *General rule for test methods of reagents*
- JIS K 8005 *Reference materials for volumetric analysis*
- JIS K 8027 *Acetylacetone*
- JIS K 8180 *Hydrochloric acid*
- JIS K 8355 *Acetic acid*
- JIS K 8359 *Ammonium acetate*
- JIS K 8576 *Sodium hydroxide*
- JIS K 8625 *Sodium carbonate*
- JIS K 8637 *Sodium thiosulfate pentahydrate*
- JIS K 8659 *Starch, soluble*
- JIS K 8872 *Formaldehyde solution*
- JIS K 8913 *Potassium iodide*
- JIS K 8920 *Iodine*
- JIS K 8951 *Sulfuric acid*
- JIS R 3503 *Glass apparatus for chemical analysis*
- JIS R 3505 *Volumetric glassware*
- JIS Z 8401 *Guide to the rounding of numbers*
- JIS Z 8703 *Standard atmospheric conditions for testing*

3 Principle of test The test for determination of quantity of formaldehyde emitted from building boards by desiccator method is carried out by using the glass desiccator as shown in Fig. 1. The emitted quantity of formaldehyde is obtained from the concentration of formaldehyde absorbed in distilled water or deionized water when the test pieces of specified surface area are placed in the desiccator filled with the specified amount of distilled water or deionized water and 24 h has elapsed.

The principle for determination of concentration of formaldehyde absorbed in distilled water or deionized water is based on the Hantzsch reaction in which the formaldehyde reacts with ammonium ions and acetylacetone to yield diacetyldihydrolutidine (DDL).

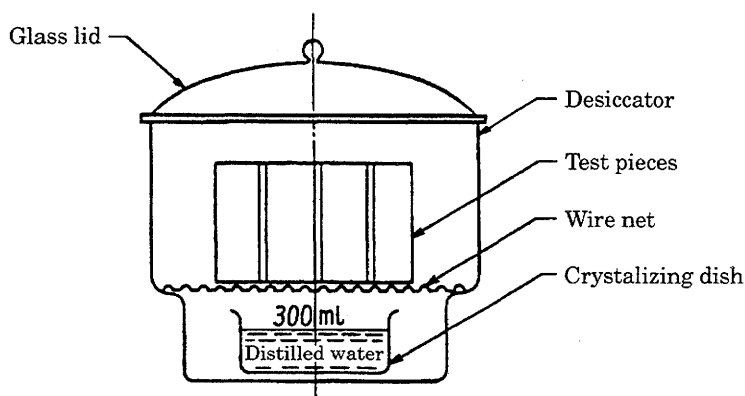
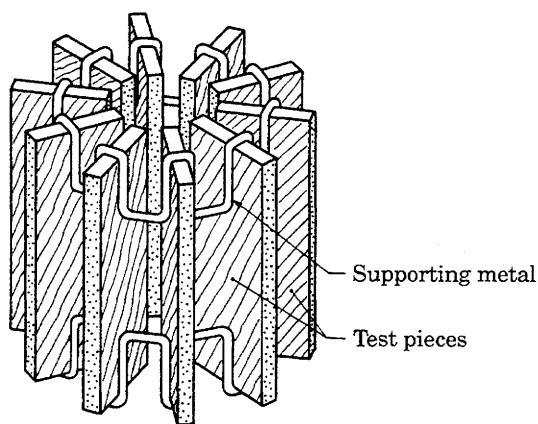
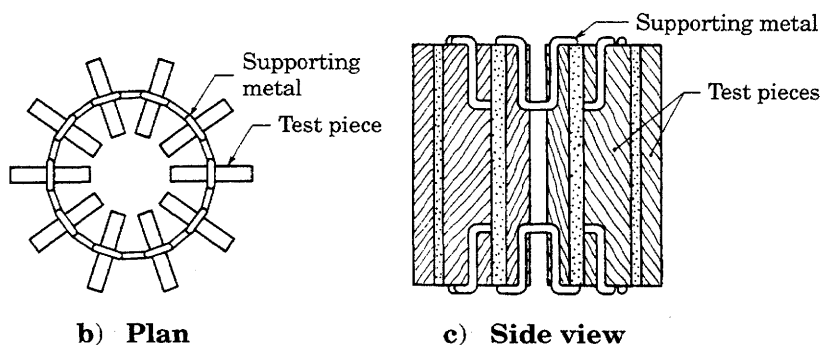


Fig. 1 Desiccator method (schematic diagram)



a) Sketch



b) Plan

c) Side view

Fig. 2 Specimen supporting metal

4 General conditions

4.1 Test atmosphere The conditions of test place shall be 20 °C temperature grade 0.5 (20 °C ± 0.5 °C) specified in **JIS Z 8703**.

4.2 Common conditions

- a) **General matters in chemical analysis** The general matters in common to chemical analysis shall be in accordance with **JIS K 0050**.
- b) **Water** The water to be used in this Standard shall be the water of A1 to A4 specified in **JIS K 0557**.

5 Apparatus and instruments

- a) **Measuring device for temperature and humidity** The thermometer shall be able to measure the temperature of air by 0.1 °C in precision. The hygrometer shall be able to measure by 5 % of relative humidity in precision.
- b) **Spectrophotometer** The spectrophotometer shall be able to measure absorbance in the range 410 nm to 415 nm.
- c) **Thermostatic water bath** The thermostatic water bath shall be able to maintain a temperature of 65 °C ± 2 °C.
- d) **Chemical balance** The chemical balance shall be able to weigh the mass of 100 g to 200 g and to read a difference of 0.1 mg.
- e) **Desiccator** The desiccator shall have an airtight construction and 240 mm in nominal dimension specified in **JIS R 3505**.
- f) **Glass crystallizing dish** The glass crystallizing dish to be used for holding distilled water or deionized water shall be 120 mm in outside diameter, 115 mm ± 1 mm in inside diameter, 60 mm to 65 mm in depth with a pour spout at the edge.
- g) **Volumetric flask** The volumetric flasks shall be 100 ml and 1 000 ml in nominal capacity specified in **JIS R 3505**.
- h) **Volumetric pipette** The volumetric pipettes shall be 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml and 100 ml (calibrated at 20 °C) in nominal capacity specified in **JIS R 3505** or automatic pipette having the same quality.
- i) **Burette** The burette shall be as specified in **JIS R 3505** or an automatic metering device.
- j) **Flask with a ground stopper** The flask with a ground stopper shall be an Erlenmeyer flask with interchangeable ground joint of 100 ml in nominal capacity specified in **JIS R 3503**.
- k) **Specimen supporting metal** The specimen supporting metal to fix test pieces with desiccator as shown in Fig. 2 shall be stainless steel wire.
- l) **Stainless steel wire net** The wire net to place the specimen supporting metal fixed with test pieces in the desiccator shall be that of 240 mm in diameter in which the intervals of the mesh in the stainless steel wire part are made so as to be wider than 15 mm.

6 Preparation of reagents

- a) **Iodine solution (0.05 mol/l)** The solution shall be prepared so that 40 g of potassium iodide specified in **JIS K 8913** is dissolved in 25 ml of water, 13 g of iodine specified in **JIS K 8920** is also dissolved in it, and afterwards it is transferred into a 1 000 ml volumetric flask, 3 drops of industrial hydrochloric acid specified in **JIS K 8180** are added, and finally water is added up to the marked line.
- b) **Sodium thiosulfate solution (0.1 mol/l)** The solution shall be prepared so that 26 g of sodium thiosulfate pentahydrate specified in **JIS K 8637** and 0.2 g of sodium carbonate specified in **JIS K 8625** are dissolved in 1 000 ml of water free from dissolved oxygen. The mixture is allowed to stand for two days, and afterwards the standardization by **JIS K 8001, 4.5 (21.2)** 0.1 mol/l sodium thiosulfate solution is carried out by using potassium iodate specified in **JIS K 8005**.
- c) **Sodium hydroxide solution (1 mol/l)** The solution shall be prepared so that 40 g of sodium hydroxide specified in **JIS K 8576** is dissolved in 200 ml of water, it is transferred into a 1 000 ml volumetric flask, and then water is added up to the marked line.
- d) **Sulfuric acid solution (1 mol/l)** The solution shall be prepared so that 56 ml of sulfuric acid specified in **JIS K 8951** is dissolved in 200 ml of water, it is transferred into a 1 000 ml volumetric flask and water is added up to the marked line.
- e) **Starch solution** The solution shall be prepared so that 1 g of starch specified in **JIS K 8659** is thoroughly mixed with 10 ml of water, and it is added to 200 ml of hot water while agitating. It is boiled for about 1 min and cooled, then filtered.
- f) **Formaldehyde standard stock solution** The solution shall be prepared so that 1 ml of formaldehyde solution specified in **JIS K 8872** is placed in a 1 000 ml volumetric flask, and water is added up to the marked line.

The concentration of formaldehyde in this solution shall be obtained according to the following procedure:

Take an aliquot of 20 ml of formaldehyde standard stock solution mentioned above into a 100 ml Erlenmeyer flask with interchangeable ground joint, add 25 ml of 0.05 mol/l iodine solution prepared by using the iodine specified in **JIS K 8920** and 10 ml of 1 mol/l sodium hydroxide solution and leave it at room temperature for 15 min under shaded conditions as it is. Next, add 15 ml of 1 mol/l sulfuric acid solution and immediately titrate free iodine by using 0.1 mol/l sodium thiosulfate solution. After the colour of the solution has become pale yellow, add 1 ml of starch solution as an indicator, furthermore titrate it. Separately, carry out a blank test by using 20 ml of water, and obtain the concentration of formaldehyde according to the following formula:

$$C = 1.5 \times (V_0 - V) \times f \times 1\,000/20$$

where, C : formaldehyde concentration in formaldehyde standard stock solution (mg/l)

- V : titration amount of 0.1 mol/l sodium thiosulfate solution in formaldehyde standard stock solution (ml)
- V_0 : titration amount of 0.1 mol/l sodium thiosulfate solution in blank test (ml)
- f : factor of 0.1 mol/l sodium thiosulfate
- 1.5: amount of formaldehyde corresponding to 1 ml of 0.1 mol/l sodium thiosulfate solution (mg)

- g) **Formaldehyde standard solution** The solution shall be prepared so that a proper quantity of formaldehyde standard stock solution is taken into a 1 000 ml volumetric flask so as to contain 3 mg of formaldehyde in 1 000 ml of water and water is added to the marked line.
- h) **Acetylacetone-ammonium acetate solution** The solution shall be prepared so that 150 g of ammonium acetate specified in **JIS K 8359** is dissolved in 800 ml of water, 3 ml of glacial acetic acid specified in **JIS K 8355** and 2 ml of acetylacetone specified in **JIS K 8027** are added to it and mixed thoroughly in the solution, and furthermore water is added to make 1 000 ml. When the measurement will not be carried out immediately, it may be stored in a dark and cool place of 0 °C to 10 °C for the period not exceeding 3 days after preparation.

7 Test pieces

7.1 Cutting off of test piece The test pieces shall be cut off from boards selected by a reasonable sampling plan taking into consideration any necessary conditions for inspection of the characteristic of the building boards such as avoiding the end parts and the like.

7.2 Dimensions and number of test pieces

- a) The test piece shall be 150 mm $\pm$ 1 mm in length and 50 mm $\pm$ 1 mm in width.
- b) The number of test pieces shall be the amount which gives a total surface area, defined as the sum of the areas of the ends, sides and faces, as close as possible to 1 800 cm². Two sets of that shall be prepared.

7.3 Conditioning

- a) The test pieces shall be conditioned under the standard conditions at a temperature of 20 °C $\pm$ 2 °C and a relative humidity of (65 % $\pm$ 5 %) specified in **JIS Z 8703** until they have reached constant mass. The constant mass shall be considered to be reached when the results of two successive weighing operations, carried out at an interval of 24 h, do not differ by more than 0.1 % of the mass of test piece.

In addition, if one week has elapsed after starting of conditioning, it may be considered to have reached the constant mass.

- b) **Conditioning method** Every test piece shall be separated mutually by at least 25 mm and positioned so that all surfaces are freely exposed to air under the standard conditions as shown in a). The test piece with low formaldehyde emission level will absorb the formaldehyde in surrounding environment possibly, therefore care shall be taken when conditioning.

8 Test method

8.1 Preparation of test apparatus

- a) The desiccator and the glass crystallizing dishes (usually, three pieces) shall be prepared, and respectively rinsed thoroughly with water and dried before testing.
- b) Water of $300 \text{ ml} \pm 1 \text{ ml}$ shall be placed in each glass crystallizing dish, which is centrally located at the bottom of the desiccator.
- c) The stainless steel wire net shall be placed on the glass crystallizing dish inside the desiccator as shown in Fig. 1, and the specimen supporting metal shall be placed on it as shown in Fig. 2.
- d) The desiccators shall stand still in the test place maintained at $20^\circ\text{C} \pm 0.5^\circ\text{C}$ of the inside temperature.

8.2 Attaching of test pieces for determination

- a) The specified number of conditioned test pieces shall be attached to the specimen supporting metal.

However, to one desiccator, the test piece shall not be attached.

- b) The lid shall be put on the desiccator and the emission test shall be started.

8.3 Monitoring of test conditions

8.3.1 Temperature A desiccator with no test pieces attached shall be used. The temperature inside the desiccator shall be measured continuously or at intervals not exceeding 15 min, and the temperature during the test period shall be recorded.

Remarks : Alternatively, the temperature may be measured by locating the thermocouple in the test environment adjacent to the desiccator.

8.3.2 Measurement for concentration of background formaldehyde (Blank test) The concentration of background formaldehyde shall be measured by using the desiccator with no test piece attached.

8.4 Test duration The duration required for one emission test shall be $24 \text{ h} \pm 5 \text{ min}$.

8.5 Collection of test solution The water absorbing the formaldehyde in the glass crystallizing dish shall be the test solution. After 24 h has elapsed, firstly this solution shall be thoroughly mixed. After a 100 ml Erlenmeyer flask with interchangeable ground joint is rinsed with this test solution, it shall be filled with this solution and sealed by using a glass ground stopper. If the concentration of formaldehyde in the test solution is not to be measured immediately, it may be stored at between 0°C and 5°C for a maximum of 30 h before measuring of the test solution concerned.

8.6 The concentration of formaldehyde in the solution for quantitative test shall be measured by acetylacetone molecular absorption spectrophotometry.

Pipette 25 ml of the test solution of **8.5** into an Erlenmeyer flask with interchangeable ground joint. Next, add 25 ml of acetylacetone-ammonium acetate solution and mix them lightly with the stopper on. Heat this Erlenmeyer flask with interchangeable ground joint in a water bath at $65\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 10 min and then allow this solution to stand under shaded conditions until it becomes room temperature. Take this solution into an absorption cell and measure the absorbance at 412 nm of wavelength against water as a control using a spectrophotometer.

Separately, the measurement shall be carried out on the background formaldehyde in the same way.

Remarks : When the maximum absorbance appears at other wavelengths than 412 nm, all measurements including that for preparing calibration curve may be carried out at this wavelength.

8.7 Preparation of calibration curve For preparation of the calibration curve, take 0 ml, 5 ml, 10 ml, 20 ml, 50 ml and 100 ml of formaldehyde standard solution using pipette, place each into a 100 ml volumetric flask respectively, and afterwards add water up to the marked line, and make it the formaldehyde solution for preparation of the calibration curve. Take an aliquot of 25 ml from the respective solutions for preparation of the calibration curve, carry out the operation of **8.6** and plot correlation between the quantity of formaldehyde (0 mg to 3 mg) and the absorbance. The slope (F) shall be obtained according to either graphical method or by calculation.

8.8 Calculation The concentration of formaldehyde from the test piece absorbed into the water in glass crystallizing dish inside desiccator shall be calculated according to the following formula:

$$G = F \times (A_d - A_b) 1\,800/S$$

where, G : concentration of formaldehyde from the test piece (mg/l)

A_d : absorbance of solution inside desiccator containing test piece

A_b : absorbance of background formaldehyde

F : slope of calibration curve on the standard solution of formaldehyde (mg/l)

S : surface area of test piece (cm^2)

The concentration of formaldehyde shall be calculated from two sets of test piece respectively, expressed by (mg/l) and rounded-off to one decimal place according to **JIS Z 8401**. In such a case, however, the test results of the two sets shall not deviate by more than 20 % from the mean value.

8.9 Expression of test result The test result shall be expressed by the mean value of two sets of measurements obtained.

9 Report The test report shall include the following information:

a) Type of board, its thickness (mm) and density (kg/m^3)

8.
A 1460 : 2001

- b) Location on the board from which the test piece was cut off (For example, illustration is used for the indication)
- c) Number of test pieces
- d) Emission quantity of formaldehyde (mean value of formaldehyde concentration obtained in 8.9), including the individual values of each measurement, and the value of the background formaldehyde.
- e) Date of test
- f) Temperature and humidity at test place
- g) Name of test body
- h) Name of person in charge of testing

The following information shall be included in the test report in principle:

- i) Name of manufacturer, place of manufacture, month and year of manufacture or lot number of manufacture.
- j) Storage conditions up to inspection after manufacture, especially matters that affect greatly the emission of formaldehyde into air, that is, temperature, humidity, sealing condition of material, storage condition and so on.
- k) Sampling method and date of sampling
- l) Place⁽¹⁾ and conditions⁽²⁾ for sampling of test pieces from factory or building or taking from building, furniture and so on.
- m) Temperature, humidity and time of conditioning condition of test pieces.

Moreover, in the case of other test methods, all matters related to the test (adjustment, temperature and the like) shall be included.

Notes (1) For example, factory, or the location such as ceilings, floors, walls, etc. in the case of the board used for construction.

(2) For example, moisture content, surface painting, finishing.

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