



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 1474 : 2014

(JICIA/JSA)

Test methods for activated carbon

ICS 71.060.10

Reference number : JIS K 1474 : 2014 (E)

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K 1474 : 2014

Date of Establishment: 1975-09-01

Date of Revision: 2014-02-20

Date of Public Notice in Official Gazette: 2014-02-20

Investigated by: Japanese Industrial Standards Committee
Standards Board
Technical Committee on Chemical Analysis

JIS K 1474:2014, First English edition published in 2014-12

Translated and published by: Japanese Standards Association
Mita MT Building, 3-13-12, Mita, Minato-ku, Tokyo, 108-0073 JAPAN

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Printed in Japan

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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 Inorganic Chemical Industry Association (JICIA)/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 1474:2007** is replaced with this Standard.

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Test methods for activated carbon

1 Scope

This Japanese Industrial Standard specifies the test methods for activated carbon for industrial use.

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 H 3100 *Copper and copper alloy sheets, plates and strips*
- JIS H 5120 *Copper and copper alloy castings*
- JIS K 0050 *General rules for chemical analysis*
- JIS K 0102 *Testing methods for industrial wastewater*
- JIS K 0115 *General rules for molecular absorptiometric analysis*
- JIS K 0116 *General rules for atomic emission spectrometry*
- JIS K 0121 *General rules for atomic absorption spectrometry*
- JIS K 0127 *General rules for ion chromatography*
- JIS K 0133 *General rules for high frequency plasma mass spectrometry*
- JIS K 0211 *Technical terms for analytical chemistry (General part)*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 0970 *Piston pipettes*
- JIS K 1105 *Argon*
- JIS K 8005 *Reference materials for volumetric analysis*
- JIS K 8012 *Zinc (Reagent)*
- JIS K 8102 *Ethanol (95) (Reagent)*
- JIS K 8121 *Potassium chloride (Reagent)*
- JIS K 8180 *Hydrochloric acid (Reagent)*
- JIS K 8201 *Hydroxylammonium chloride (Reagent)*
- JIS K 8202 *1,10-Phenanthroline chloride monohydrate (Reagent)*
- JIS K 8247 *Potassium permanganate (Reagent)*
- JIS K 8322 *Chloroform (Reagent)*
- JIS K 8355 *Acetic acid (Reagent)*
- JIS K 8356 *Zinc acetate dihydrate (Reagent)*
- JIS K 8359 *Ammonium acetate (Reagent)*
- JIS K 8383 *Sucrose (Reagent)*
- JIS K 8517 *Potassium dichromate (Reagent)*

- JIS K 8541 *Nitric acid (Reagent)*
JIS K 8548 *Potassium nitrate (Reagent)*
JIS K 8550 *Silver nitrate (Reagent)*
JIS K 8576 *Sodium hydroxide (Reagent)*
JIS K 8625 *Sodium carbonate (Reagent)*
JIS K 8637 *Sodium thiosulfate pentahydrate (Reagent)*
JIS K 8659 *Starch, soluble (Reagent)*
JIS K 8789 *1,10-Phenanthroline monohydrate (Reagent)*
JIS K 8897 *Methylene blue (Reagent)*
JIS K 8913 *Potassium iodide (Reagent)*
JIS K 8920 *Iodine (Reagent)*
JIS K 8951 *Sulfuric acid (Reagent)*
JIS K 8979 *Ammonium iron (II) sulfate hexahydrate (Reagent)*
JIS K 9007 *Potassium dihydrogen phosphate (Reagent)*
JIS K 9019 *Disodium hydrogenphosphate 12-water (Reagent)*
JIS M 8100 *Particulate materials—General rules for methods of sampling*
JIS M 8811 *Coal and coke—Sampling and sample preparation*
JIS P 3801 *Filter paper (for chemical analysis)*
JIS R 1301 *Porcelain crucibles for chemical analysis*
JIS R 3503 *Glass apparatus for chemical analysis*
JIS R 3505 *Volumetric glassware*
JIS Z 0701 *Silicagel desiccants for packaging*
JIS Z 8801-1 *Test sieves—Part 1: Test sieves of metal wire cloth*
JIS Z 8802 *Methods for determination of pH of aqueous solutions*

3 Terms and definitions

For the purposes of this Standard, the terms and definitions given in **JIS K 0211**, and the following apply.

3.1 granular sample

sample whose indicated particle size is not less than 150 μm

3.2 powder sample

sample whose indicated particle size is less than 150 μm

NOTE : In some samples, indicated particle size can be across 150 μm (e.g. 180 μm to 75 μm). Test methods for this type of samples shall be based on the agreements between the parties concerned with delivery.

4 Test items

Test items shall be as follows.

According to the intended use, test items may be selected from the following items between the parties concerned with delivery.

- a) Adsorption performance
 - 1) Determination of adsorption isotherm in liquid phase
 - 2) Determination of adsorption performance in gas phase
- b) Caramel decolouring performance
- c) Particle size
- d) Particle size distribution
- e) Effective diameter, uniformity coefficient and mean particle diameter
- f) Hardness
- g) Ignition temperature
- h) Bulk density
- i) Loss on drying
- j) Ignition residue
- k) pH value
- l) Chloride
- m) Iron
- n) Zinc
- o) Cadmium
- p) Lead
- q) Arsenic

5 Sampling method

Samples shall be taken so as to be representatives of the whole, in accordance with **JIS M 8100** and **JIS M 8811**.

6 General

General matters of test shall be in accordance with **JIS K 0050**, **JIS K 0115**, **JIS K 0121** and **JIS K 0127**.

7 Test methods

7.1 Adsorption performance

7.1.1 Summary

Adsorption by activated carbon is classified into liquid phase adsorption and gas phase adsorption. Liquid phase adsorption shall be assessed from the adsorption amount at the specific density, by preparing the adsorption isotherm, using iodine and methylene blue as representatives of the substances to be adsorbed. Gas phase adsorption shall be assessed by measuring the adsorption amount of solvent vapour.

7.1.2 Adsorption isotherm in liquid phase

7.1.2.1 Determination of adsorption isotherm in liquid phase

Adsorption isotherm in liquid phase shall be determined as follows.

- a) **Summary** The adsorption isotherm is intended to indicate the relation between the concentration and adsorption amount of the substance to be adsorbed, which is in equilibrium state at a constant temperature. A diagram is illustrated by plotting the residual concentration on the abscissa of the graph, and the adsorption amount per mass of activated carbon on the ordinate.
- b) **Sample preparation** For powder sample, dry as it is for 3 h in a constant temperature oven maintained at $(115 \pm 5) ^\circ\text{C}$, and allow to cool in a desiccator [use silica gel as a desiccant (hereafter applied unless otherwise specified)]. For granular sample, pulverize to such a degree that at least 90 % of the sample can pass through a 45 μm wire sieve specified in **JIS Z 8801-1** (dimension of sieve frame: internal diameter 75 mm above the sieve surface) and dry by the method mentioned above.

NOTE : When pulverizing granular samples, attention shall be paid to sample contamination caused by the wear of pulverizer, etc. Sample contamination, which can affect adsorption performance test or others, may be confirmed from the change of the sample before and after pulverization, by such methods as the ignition residue analysis and fluorescent X-ray analysis.

- c) **Test solution** Use a solution to be adsorbed for the test solution. Conditions shall be constant according to the test purpose, since adsorption performance can be influenced by such factors as the type of solvent, concentration of substance to be adsorbed, coexisting components, temperature, pH value and contact time.
- d) **Apparatus and implement** According to the operating conditions of activated carbon adsorption, the apparatus and implement shall be selected from the following items.
 - 1) **Constant temperature oven**, capable of adjusting the temperature within the range $(115 \pm 5) ^\circ\text{C}$.
 - 2) **Shaker**, capable of reciprocating horizontally 100 times/min to 250 times/min, at an amplitude 40 mm to 50 mm.
 - 3) **Centrifugal separator**, capable of rotating a 50 ml precipitation tube at 2 000 r/min to 4 000 r/min.
 - 4) **Spectrophotometer**, as specified in **JIS K 0115**.
 - 5) **Container** Erlenmeyer flask or beaker.
 - 6) **Filter paper**, complying with Class 5 C as specified in **JIS P 3801**, with diameter 110 mm.
 - 7) **Membrane filter**, with pore diameter 0.45 μm .
 - 8) **Vacuum filtration equipment**, capable of holding a membrane filter and filtering under reduced pressure.

- 9) **High speed vibration sample pulverizer**, capable of pulverizing a sample to such a degree that at least 90 % of the sample can pass through a 45 μm wire sieve specified in **JIS Z 8801-1**, with a container which hardly causes sample contamination by wear or others.

e) **Procedure** The test procedures shall be as follows.

- 1) Place a given amount of test solutions into a series of several containers, add samples, and bring into contact with the test solutions sufficiently using a shaker until the samples reach adsorption equilibrium, as maintained at the specified temperature.

Add at least three levels of samples within the range including the amount required to produce the expected effect of sample adsorption (e.g. add respectively 0.1 g, 0.2 g, 0.3 g, 0.5 g ... to each 100 ml of test solution), while the amount of sample to be added depends on the type, concentration and test purpose of the substance to be adsorbed.

- 2) Separate the solution and sample with a filter paper, membrane filter (pore diameter 0.45 μm), centrifugal separator, etc., without making leakage.
- 3) Determine the substance to be adsorbed in each separated solution and obtain each equilibrium concentration (residual concentration). Employ a method complying with the natures of the substance to be adsorbed. Coloured substance may be indicated by absorbance, etc.
- 4) Perform a blank test by carrying out procedures in 1) to 3) without containing samples.
- 5) Prepare the adsorption isotherm by plotting, on a logarithmic coordinate paper, the residual concentration of the substance to be adsorbed on the abscissa, and plotting the adsorption amount of the substance to be adsorbed per mass of sample on the ordinate.
- 6) When the isotherm within the targeted range cannot be obtained, repeat the test by increasing or decreasing the sample amount. Prior to retest, it shall be confirmed, by changing the adsorption time, the time required for the adsorption amount to become constant, that is, to reach the equilibrium.

7.1.2.2 Iodine adsorption performance

The iodine adsorption performance shall be as follows.

- a) **Summary** Add iodine solution to the sample, adsorb at room temperature (20 °C to 30 °C), then separate the supernatant liquid. Add starch solution as the indicator, and titrate with sodium thiosulfate solution. Obtain the adsorption amount per mass of sample from the iodine residual concentration to obtain the adsorption isotherm. Obtain the adsorption amount per mass of sample at 2.5 g/L iodine residual concentration by reference to the adsorption isotherm, to take as iodine adsorption performance.
- b) **Reagents** The following reagents shall be used.
 - 1) **Sulfuric acid (1+5)**, prepared by using sulfuric acid specified in **JIS K 8951**.

- 2) **Starch solution (10 g/L)** Add approximately 10 ml of water to 1 g of starch (soluble) specified in **JIS K 8659** and mix them. Add this solution to 90 ml of hot water while stirring, boil for approximately 1 min, and cool to use the supernatant liquid. Prepare this solution immediately before use.
- 3) **0.1 mol/L sodium thiosulfate solution** Dissolve approximately 25 g of sodium thiosulfate pentahydrate specified in **JIS K 8637** and approximately 0.2 g of sodium carbonate specified in **JIS K 8625** into 1 L of water, and shake to mix well. Apply a stopper, leave for two days, and standardize as follows.

Standardization Dry potassium iodate for reference material for volumetric analysis specified in **JIS K 8005** for 2 h in a constant temperature oven maintained at $(130 \pm 10) ^\circ\text{C}$, and allow to cool in a desiccator for 30 min to 60 min. After cooling, weigh 1 g to 1.5 g of potassium iodate to the nearest 1 mg, and dissolve in water. Transfer into a 250 ml volumetric flask and add water to the mark. Transfer 20 ml from this solution into a 300 ml Erlenmeyer flask with interchangeable ground joint with a volumetric pipette, and add 2 g of potassium iodide specified in **JIS K 8913** and 5 ml of sulfuric acid (1+5). Apply a stopper immediately, shake gently to mix, and leave for 5 min in a dark place. Add 100 ml of water and titrate the liberated iodine with this solution. When the yellow colour of iodine fades, add 1 ml of starch solution (10 g/L) as the indicator, and continue to titrate. Take the moment when the blue colour of the iodine starch disappears as the endpoint. Also, the blank test shall be carried out.

Calculate the factor by formula (1).

$$f = \frac{a \times \frac{b}{100} \times \frac{20}{250}}{(c - d) \times 3.567} \dots\dots\dots (1)$$

where, f : factor of 0.1 mol/L sodium thiosulfate solution
 a : sampling amount of potassium iodate (g)
 b : purity of potassium iodate (mass fraction %)
 c : amount of 0.1 mol/L sodium thiosulfate used for titration (ml)
 d : amount of 0.1 mol/L sodium thiosulfate used for blank test (ml)
 3.567: amount of potassium iodate corresponding to 1 ml of 0.1 mol/L sodium thiosulfate solution (mg/ml)

- 4) **0.05 mol/L iodine solution** Dissolve 25.0 g of potassium iodide specified in **JIS K 8913** in approximately 30 ml of water. Add approximately 13 g of iodine specified in **JIS K 8920** to dissolve, add water to make approximately 1 L, and standardize. This solution shall be stored in a dark place at approximately $10 ^\circ\text{C}$, and shall be newly standardized immediately before use.

Standardization Take 20 ml of this solution with a volumetric pipette, and titrate with 0.1 mol/L sodium thiosulfate solution. When the yellow colour of iodine fades, add 1 ml of starch solution (10 g/L) as the indicator, and continue to titrate. Take the moment when the blue colour of the iodine starch disappears as the endpoint. Calculate the factor of iodine solution by formula (2).

$$f' = K \times \frac{f}{20} \dots\dots\dots (2)$$

where, f' : factor of 0.05 mol/L iodine solution
 K : amount of 0.1 mol/L sodium thiosulfate solution used for titration (ml)
 f : factor of 0.1 mol/L sodium thiosulfate solution
20 : sampling amount of iodine solution (ml)

c) **Apparatus and implement** The apparatus and implement shall be as follows.

- 1) **Constant temperature oven**, capable of adjusting the temperature within the range $(115 \pm 5)^\circ\text{C}$ and the range $(130 \pm 10)^\circ\text{C}$.
- 2) **Shaker**, capable of reciprocating horizontally 100 times/min to 250 times/min, at an amplitude 40 mm to 50 mm.
- 3) **Centrifugal separator**, capable of rotating a 50 ml precipitation tube at 2 000 r/min to 4 000 r/min.
- 4) **Membrane filter**, with pore diameter 0.45 μm .
- 5) **Vacuum filtration equipment**, capable of holding a membrane filter and filtering under reduced pressure.

d) **Procedure** The test procedures shall be as follows.

- 1) Weigh a specified amount of sample to the nearest 1 mg and transfer into a 100 ml brown Erlenmeyer flask with interchangeable ground joint. Add 50 ml of 0.05 mol/L iodine solution with a volumetric pipette. Take a suitable amount of sample according to 7.1.2.1 e) 1) so that the residual concentration after adsorption can remain within the range including 2.5 g/L.
- 2) Shake the sample for 15 min with a shaker at room temperature (20°C to 30°C), transfer into to a 50 ml precipitation tube and precipitate the sample with a centrifugal separator. Alternatively, a membrane filter (pore diameter 0.45 μm) may be used to separate the activated carbon and the solution.
- 3) From this solution, place 10 ml of the supernatant solution with a volumetric pipette, and titrate with 0.1 mol/L sodium thiosulfate solution. When the yellow colour of iodine fades, add 1 ml of starch solution (10 g/L) as the indicator and continue to titrate. Take the moment when the blue colour of iodine starch disappears as the endpoint.
- 4) Calculate the iodine residual concentration by formula (3).

$$I_N = K \times f \times 12.69 \times \frac{1}{10} \dots\dots\dots (3)$$

where, I_N : iodine residual concentration (g/L)
 K : amount of 0.1 mol/L sodium thiosulfate solution used for titration (ml)
 f : factor of 0.1 mol/L sodium thiosulfate solution
12.69: amount of iodine corresponding to 1 ml of 0.1 mol/L sodium thiosulfate solution (mg/ml)
10: sampling amount of supernatant solution (ml)

- 5) Calculate the iodine absorption amount per mass of sample by formula (4). Express the adsorption amount with 10 mg/g increments.

$$A_t = \frac{(10 \times f' - K \times f) \times 12.69 \times 5}{S} \dots\dots\dots (4)$$

where, A_t : iodine adsorption amount per mass of sample (mg/g)
 S : mass of sample (g)
 f' : factor of 0.05 mol/L iodine solution
 K : amount of 0.1 mol/L sodium thiosulfate solution used for titration (ml)
 f : factor of 0.1 mol/L sodium thiosulfate solution
12.69: amount of iodine corresponding to 1 ml of 0.1 mol/L sodium thiosulfate solution (mg/ml)

- 6) Plot the iodine residual concentration (C) on the abscissa of a logarithmic coordinate paper, and plot the iodine adsorption amount per mass of sample on the ordinate, by reference to the iodine residual concentration and iodine adsorption amount obtained in 4) and 5). Determine the adsorption isotherm by formula (6) obtained by converting formula (5) (Freundlich formula) to a logarithmic formula.

$$A_t = kC^{\frac{1}{n}} \dots\dots\dots (5)$$

$$\log(A_t) = \log k + \frac{1}{n} \log C \dots\dots\dots (6)$$

where, A_t : iodine adsorption amount per mass of sample (mg/g)
 C : iodine residual concentration in solution (g/L)
 k, n : constants to be determined by the sample and the adsorption series

k is the intercept of the straight line when $C = 1$, and the inclination of this straight line $\tan \theta$ is $1/n$.

- 7) By reference to the adsorption isotherm obtained in 6), obtain the iodine adsorption amount per mass of sample at the point of iodine residual concentration 2.5 g/L to take as iodine adsorption performance.

- e) **Example of obtaining method for iodine adsorption performance** Examples of preparing procedures for adsorption isotherm and obtaining method for iodine adsorption performance are shown in table 1 and figure 1.

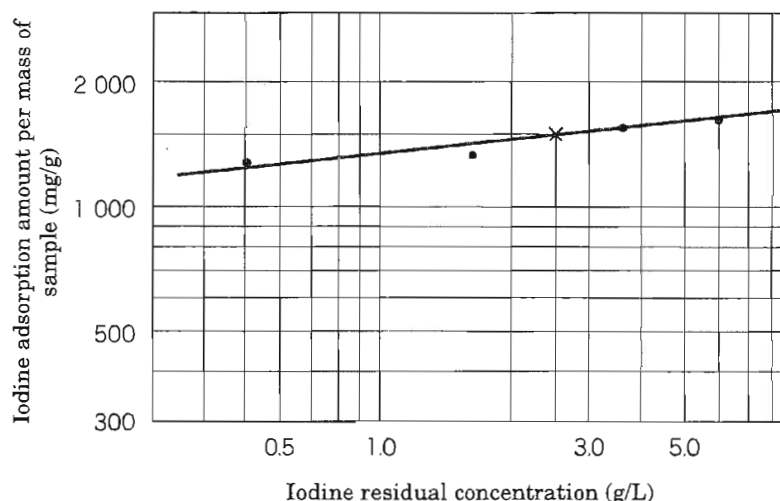
Table 1 Example of adsorption performance data

Amount of sample added (g/50 ml)	Iodine residual concentration (g/L)	Iodine adsorption amount per mass of sample (mg/g)
0	12.69	—
0.200	6.00	1 670
0.300	3.58	1 520
0.400	1.66	1 380
0.500	0.49	1 220

$$k = 1\,320$$

$$\frac{1}{n} = 0.12$$

$$A_t = 1\,320 \times C^{0.12}$$



The iodine adsorption performance of this solution is 1 470 mg/g.

Figure 1 Adsorption isotherm in iodine adsorption performance (room temperature) (example)

7.1.2.3 Methylene blue adsorption performance

The methylene blue adsorption performance shall be as follows.

- a) **Summary** Add methylene blue solution to the sample, adsorb at room temperature (20 °C to 30 °C) and filter. Determine the absorbance of the filtrate, obtain the adsorption amount per mass of sample from the residual concentration, and prepare the adsorption isotherm. By reference to the adsorption isotherm, obtain the adsorption amount per mass of sample at the time of methylene blue residual concentration 0.24 mg/L, and calculate methylene blue adsorption performance.

b) **Reagents** The following reagents shall be used.

- 1) **Potassium dihydrogen phosphate (1/15 mol/L)** Dissolve 9.08 g of potassium dihydrogen phosphate specified in **JIS K 9007** in water to make 1 000 ml.
- 2) **Disodium hydrogen phosphate (1/15 mol/L)** Dissolve 23.88 g of disodium hydrogen phosphate 12 hydrate specified in **JIS K 9019** in water to make 1 000 ml.
- 3) **Phosphate buffer solution (pH7)** Mix potassium dihydrogen phosphate solution (1/15 mol/L) and disodium hydrogen phosphate solution (1/15 mol/L) at the ratio of 4 : 6.
- 4) **Methylene blue solution** Weigh 1.2 g (converted to dry mass) of methylene blue specified in **JIS K 8897** into a 1 000 ml volumetric flask, and add phosphate buffer solution (pH7) to the mark. Methylene blue is subject to change its nature when dried. Therefore, previously weigh 1 g of methylene blue to the nearest 1 mg, dry for 4 h in a dryer at $(105 \pm 5) ^\circ\text{C}$, allow to cool in a desiccator, and obtain the drying loss of the methylene blue. From this value, calculate the sampling amount of undried methylene blue by formula (7).

$$S' = S \times \frac{1}{(100 - W)} \times 100 \% \dots\dots\dots (7)$$

where, S' : sampling amount of undried methylene blue (g)
 S : mass of methylene blue (converted to dry mass) (g)
 W : loss on drying (mass fraction %)

c) **Apparatus and implement** The apparatus and implement shall be as follows.

- 1) **Constant temperature oven**, capable of adjusting the temperature within the range $(105 \pm 5) ^\circ\text{C}$ and the range $(115 \pm 5) ^\circ\text{C}$.
- 2) **Shaker**, capable of reciprocating horizontally by 100 times/min to 250 times/min at an amplitude of 40 mm to 50 mm.
- 3) **Spectrophotometer**, as specified in **JIS K 0115**.
- 4) **Filter paper**, complying with Class 5 C as specified in **JIS P 3801**.
- 5) **Funnel with flat perforated plate** The diameter of perforated plate is 15 mm.

d) **Preparation of working curve** The working curve shall be prepared as follows.

- 1) Place 10 ml of methylene blue solution into a 500 ml volumetric flask with a volumetric pipette, and add phosphate buffer solution (pH7) to the mark. From this solution, place respectively 5 ml, 10 ml, 25 ml and 50 ml with a volumetric pipette and transfer into a series of 500 ml volumetric flasks. Add phosphate buffer solution (pH7) to the mark.
- 2) For these solutions, prepare the relation curve of the concentration of methylene blue solution 0.24 mg/L to 2.4 mg/L and the absorbance at a wavelength of 665 nm, to take as the working curve.

e) **Procedure** The test procedures shall be as follows.

- 1) Weigh a specified amount of sample to the nearest 1 mg, and transfer into a 100 ml Erlenmeyer flask with interchangeable ground joint. Add 25 ml of methylene blue solution with a volumetric pipette. Take a suitable amount of sample according to 7.1.2.1 e) 1) so that the residual concentration after adsorption can remain within the range including 0.24 mg/L.
- 2) Immediately after shaking for 30 min with a shaker at room temperature (20 °C to 30 °C), use a filter paper, filter by suction with a funnel with flat perforated plate, and separate the activated carbon and the solution. Prior to this procedure, allow the filter paper used for filtration to filter by sucking 20 ml of methylene blue solution diluted to the concentration of 0.24 mg/L with phosphate buffer solution (pH7) according to d) 1), so that it can contain adsorbed methylene blue and that the decrease of the methylene blue concentration caused by adsorption to the filter paper can be avoided.
- 3) Place a portion of the filtrate into an absorption cell of optical path 10 mm. Determine, with a spectrophotometer, the absorbance at a wavelength of 665 nm using the phosphate buffer solution (pH7) as the reference, and obtain the methylene blue residual concentration by reference to the working curve.
- 4) Calculate the adsorption amount of methylene blue by formula (8), using the methylene blue residual concentration obtained in 3).

$$A_M = \frac{(1\,200 - C) \times \frac{25}{1\,000}}{S} \dots\dots\dots (8)$$

where, A_M : adsorption amount of methylene blue per mass of sample (mg/g)

S : mass of sample (g)

1 200: concentration of methylene blue solution (mg/L)

C : methylene blue residual concentration (mg/L)

25: amount of methylene blue solution added (ml)

1 000: conversion factor of 1 L = 1 000 ml

- 5) Prepare the adsorption isotherm by plotting, on a logarithmic coordinate paper, the residual concentration of methylene blue solution obtained in 3) on the abscissa, and the adsorption amount of methylene blue per mass of sample obtained in 4) on the ordinate.
- 6) By reference to the adsorption isotherm obtained in 5), obtain the adsorption amount of methylene blue of the sample at a methylene blue residual concentration of 0.24 mg/L.

Convert this value to the adsorption amount of methylene blue solution by formula (9) to take as methylene blue adsorption performance. Express adsorption performance with 10 ml/g increments.

$$M = \frac{Q}{1.2} \dots\dots\dots (9)$$

where, M : methylene blue adsorption performance (ml/g)
 Q : adsorption amount of methylene blue at 0.24 mg/L residual concentration (mg/g)
 1.2 : concentration of methylene blue solution (g/L)

- f) **Example of obtaining methylene blue adsorption performance** Examples of procedures for preparing an adsorption isotherm and obtaining method for methylene blue adsorption performance are shown in table 2 and figure 2.

Table 2 Example of adsorption performance data

Amount of sample added (g/25 ml)	Methylene blue residual concentration (mg/L)	Adsorption amount of methylene blue per mass of sample (mg/g)
0	1 200	—
0.141	0.488	213
0.152	0.053	197
0.161	0.019	186

As shown in figure 2, the adsorption amount of methylene blue at a methylene blue residual concentration of 0.24 mg/L is 210 mg/g. Therefore, methylene blue adsorption performance is expressed as follows by formula (9).

$$M = \frac{210}{1.2} = 175 \div 180$$

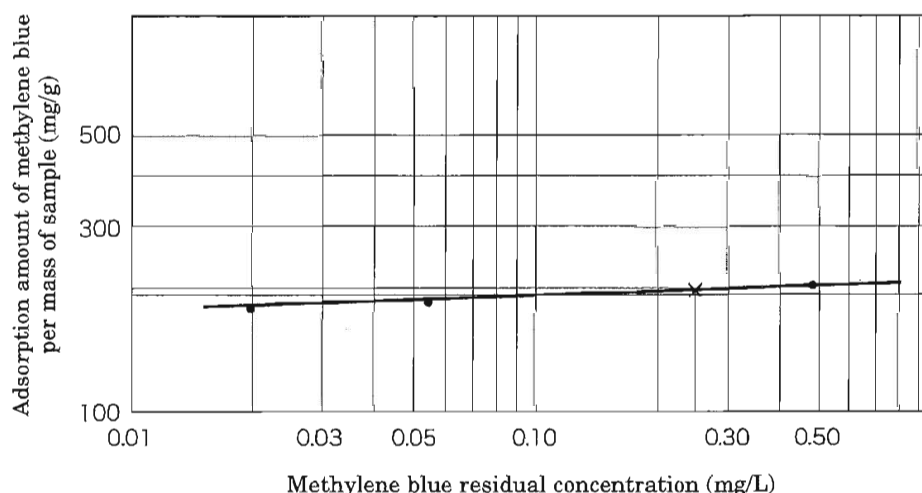


Figure 2 Adsorption isotherm in methylene blue adsorption performance (room temperature) (example)

7.1.3 Determination of adsorption performance in gas phase

Adsorption performance in gas phase shall be determined as follows.

- a) **Summary** Pass the air containing $1/n$ solvent vapour through granular sample at the rate of 2 L/min. From the increase of sample at the point when the mass becomes constant, obtain the equilibrium adsorption performance. $1/n$ is the dilution

ratio, and n is the dilution multiple of solvent saturation concentration. The dilution ratio shall be agreed between the parties concerned with delivery according to the intended use.

- b) **Reagents** The solvent to be adsorbed (see Examples 1 to 3) shall be agreed between the parties concerned with delivery according to the intended use.

Example 1 Acetone specified in **JIS K 8034**.

Example 2 Cyclohexane specified in **JIS K 8464**.

Example 3 Toluene specified in **JIS K 8680**.

- c) **Apparatus and implement** The apparatus and implement shall be as follows.

- 1) **Constant temperature oven**, capable of adjusting the temperature within the range $(115 \pm 5) ^\circ\text{C}$.
- 2) **U-tube for adsorption test**, as shown in figure 3. For samples whose particle size is not more than 2 mm, use U-tube (B).

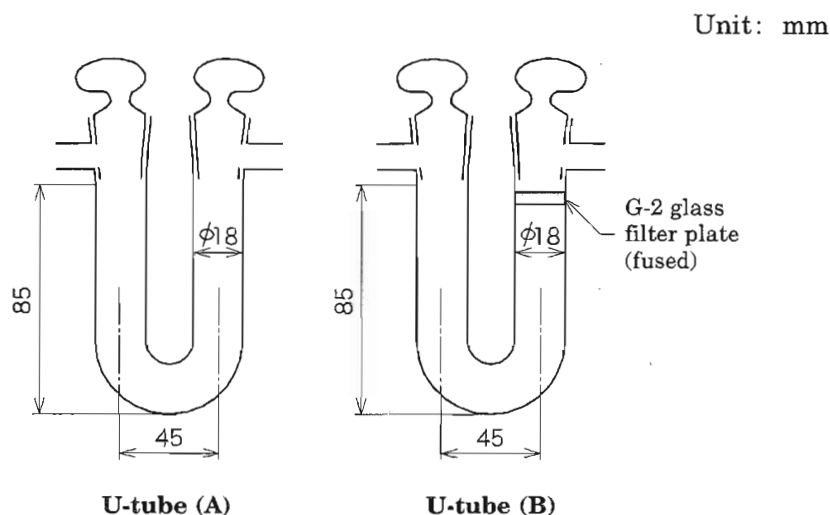
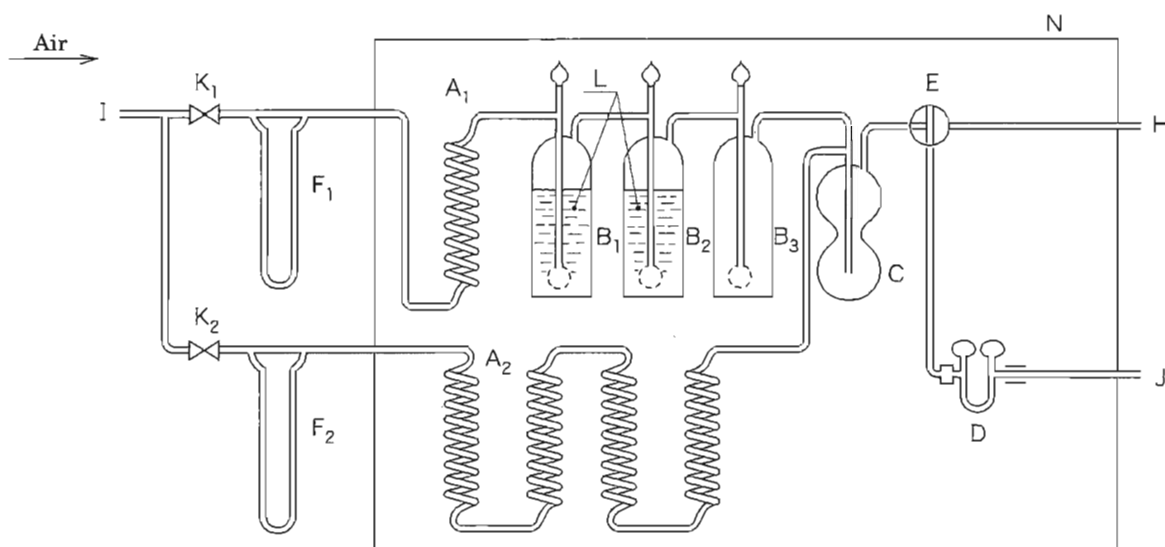


Figure 3 U-tube for adsorption test

- 3) **Constant temperature tank** A constant temperature tank or constant temperature water tank which is capable of adjusting the temperature within the range $(25 \pm 0.5) ^\circ\text{C}$.
- 4) **Tester for solvent vapour adsorption performance** An example is shown in figure 4.



- A₁, A₂ Coiled tube for temperature adjustment
 B₁, B₂, B₃ Solvent vapour generating bottle
 C Mixing bottle (bulb internal diameter 60 mm, double sphere continued type)
 D U-tube for adsorption test
 E Three-way cock
 F₁ Flowmeter for solvent vapour generating air
 F₂ Flowmeter for diluted air
 N Constant temperature tank or constant temperature water tank
 H Outlet for excessive gas
 I Inlet for dry air
 J Exhaust opening
 K₁, K₂ Adjusting cock of gas flow rate
 L Solvent

Figure 4 Example of tester for solvent vapour adsorption performance

- d) **Preparation of air containing $1/n$ solvent vapour** Adjust the constant temperature tank at $(25 \pm 0.5) ^\circ\text{C}$. Place the solvent in solvent vapour generating bottles B₁ and B₂ shown in figure 4, and set the cock E so as to flow in direction H. Pass the calculated amount of dry air at the flow rate V_1 (L/min) while adjusting cock K₁. For air to be diluted, pass the calculated amount of dry air V_2 (L/min) while adjusting cock K₂.

Calculate the flow rates of dry air V_1 and V_2 by formulae (10) and (11).

$$V_1 = 2 \times \frac{1}{n} \times \left(1 - \frac{P_1}{101.325} \right) \dots\dots\dots (10)$$

$$V_2 = 2 \times \left(1 - \frac{1}{n} \right) \dots\dots\dots (11)$$

where, V_1 : flow rate of dry air passing through solvent vapour generating bottle (L/min)
 2: flow rate of air containing $1/n$ solvent vapour (L/min)

n : dilution multiple of solvent

P_1 : saturated vapour pressure of solvent at the specified temperature (kPa)

101.325 : standard atmospheric pressure (kPa)

V_2 : flow rate of dry air to be diluted (l/min)

Examples of calculating acetone, cyclohexane and toluene of 1/10 dilution rate at 25 °C are as follows.

Example 1 Acetone

The saturated vapour pressure of acetone at 25 °C is 30.8 kPa.

$$V_1 = 2 \times \frac{1}{10} \times \left(1 - \frac{30.8}{101.325} \right) = 0.139$$

$$V_2 = 2 \times \left(1 - \frac{1}{10} \right) = 1.80$$

If dry air is adjusted at cock K_1 and is sent to flowmeter F_1 at 0.139 L/min, and if dry air is adjusted at cock K_2 and is sent to flowmeter F_2 at 1.80 L/min, the mixed air of 1/10 dilution rate at 2.0 L/min can be obtained.

Example 2 Cyclohexane

The saturated vapour pressure of cyclohexane at 25 °C is 13.0 kPa.

$$V_1 = 2 \times \frac{1}{10} \times \left(1 - \frac{13.0}{101.325} \right) = 0.174$$

$$V_2 = 2 \times \left(1 - \frac{1}{10} \right) = 1.80$$

If dry air is adjusted at cock K_1 and is sent to flowmeter F_1 at 0.174 L/min, and if dry air is adjusted at cock K_2 and is sent to flowmeter F_2 at 1.80 L/min, the mixed air of 1/10 dilution rate at 2.0 L/min can be obtained.

Example 3 Toluene

The saturated vapour pressure of toluene at 25 °C is 3.79 kPa.

$$V_1 = 2 \times \frac{1}{10} \times \left(1 - \frac{3.79}{101.325} \right) = 0.193$$

$$V_2 = 2 \times \left(1 - \frac{1}{10} \right) = 1.80$$

If dry air is adjusted at cock K_1 and is sent to flowmeter F_1 at 0.193 L/min, and if dry air is adjusted at cock K_2 and is sent to flowmeter F_2 at 1.80 L/min, the mixed air of 1/10 dilution rate at 2.0 L/min can be obtained.

e) **Procedure** The test procedures shall be as follows.

- 1) Weigh the mass of U-tube for adsorption test to the nearest 1 mg.
- 2) Place 5 g to 10 g of the sample into U-tube and weigh to the nearest 1 mg. Previously, dry the sample for 3 h in a constant temperature oven maintained at $(115 \pm 5) ^\circ\text{C}$, and allow to cool to room temperature in a desiccator.
- 3) Pass a specified amount of air to the tester for solvent vapour adsorption performance maintained at $(25 \pm 0.5) ^\circ\text{C}$, and prepare the solvent mixed air diluted by n times.
- 4) Attach the U-tube to the specified position of the tester for solvent vapour adsorption performance. Switch cock E so as to flow in direction J, and pass the solvent vapour mixed air through the U-tube to adsorb the solvent. Adjust a fluctuation of flow rate at this time, if any.
- 5) After 1 h or later, remove the U-tube, wipe well with dry paper or cloth, and measure the mass immediately.
- 6) Reattach the U-tube to the tester for solvent vapour adsorption performance. Pass solvent vapour mixed air for 30 min, and remove the U-tube. Wipe well with dry paper or cloth, and measure the mass immediately.
- 7) Repeat the procedure in 6) until the increased amount in the U-tube is not more than 5 mg, then stop the adsorption procedure to obtain the increased amount of sample.

f) **Calculation** Calculate the adsorption performance of solvent vapour by formula (12), and round the value to the first decimal place.

$$Z = \frac{P}{S} \times 100 \dots\dots\dots (12)$$

where, Z : equilibrium adsorption performance of solvent vapour of sample (mass fraction %)

P : increased amount of sample (g)

S : mass of sample (g)

7.2 Caramel decolouring performance

The caramel decolouring performance shall be as follows.

a) **Summary** Add a caramel test solution to the sample to adsorb, and filter the solution. Obtain the caramel decolouring performance from the absorbance of the filtrate and that of caramel test solution.

NOTE: The caramel decolouring performance is mainly used for powder samples and is hardly used for granular samples.

b) **Reagents** The following reagents shall be used.

- 1) **Sulfuric acid (1+4)**, prepared by using sulfuric acid specified in **JIS K 8951**.
- 2) **Sulfuric acid (1+10)**, prepared by using sulfuric acid specified in **JIS K 8951**.

- 3) **Sodium hydroxide solution (200 g/L)**, prepared by using sodium hydroxide specified in **JIS K 8576**.
 - 4) **Sodium hydroxide solution (100 g/L)**, prepared by using sodium hydroxide specified in **JIS K 8576**.
 - 5) **Chromaticity standard solution** Crush potassium dichromate specified in **JIS K 8517** in an agate mortar, and dry for 3 h to 4 h in a constant temperature oven maintained at $(105 \pm 5) ^\circ\text{C}$. Allow to cool in a desiccator, weigh 0.310 g, and add water to dissolve. Transfer into a 1 000 ml volumetric flask and add water to the mark to take as chromaticity standard solution. Determine the absorbance of this solution at a wavelength of 430 nm with a photometer.
 - 6) **Caramel stock solution** Dry the saccharose specified in **JIS K 8383** for 24 h or longer in a desiccator, weigh 60 g and transfer into a 500 ml Erlenmeyer flask. Add 240 ml of water and mix to dissolve at room temperature ($20 ^\circ\text{C}$ to $30 ^\circ\text{C}$). Add 25 ml of sulfuric acid (1+4) with a volumetric pipette and immerse in a water bath maintained at $(80 \pm 1) ^\circ\text{C}$. While stirring the solution in the Erlenmeyer flask, raise the temperature up around $80 ^\circ\text{C}$ and maintain at $(80 \pm 1) ^\circ\text{C}$ for 30 min. Remove the Erlenmeyer flask from the water bath and immediately add 50 ml of sodium hydroxide solution (200 g/L) with a volumetric pipette. Immediately transfer the Erlenmeyer flask in the boiling water bath and heat for 15 min. Remove the flask from the boiling water bath, leave over night at room temperature, regulate the pH to the value of 7.0 ± 0.1 by adding sulfuric acid (1+10) or sodium hydroxide solution (100 g/L) with a pH meter. Transfer into a 500 ml volumetric flask and add water to the mark. This solution shall be stored in a cold and dark place, and shall not be used if it has elapsed 5 days or longer since prepared.
 - 7) **Caramel test solution** Dilute the caramel stock solution with water to the volume ratio 1 : 20, leave for 30 min, and determine the absorbance at a wavelength of 430 nm with a photometer. Obtain such dilution ratio that the caramel test solution coincides with the absorbance of chromaticity standard solution obtained in 5). The dilution ratio outside the range 1 : 20 to 1 : 25 shall not be used. In this case, prepare a new solution by using the caramel stock solution.
- c) **Apparatus and implement** The apparatus and implement shall be as follows.
- 1) **Constant temperature oven**, capable of adjusting the temperature within the range $(105 \pm 5) ^\circ\text{C}$ and the range $(115 \pm 5) ^\circ\text{C}$.
 - 2) **Shaker**, capable of reciprocating horizontally 100 times/min to 250 times/min at an amplitude of 40 mm to 50 mm.
 - 3) **Spectrophotometer**, as specified in **JIS K 0115**.
 - 4) **pH meter**, of Type 0 to II as specified in **JIS Z 8802**.
 - 5) **Filter paper**, complying with Class 5 C as specified in **JIS P 3801**, with diameter 110 mm.
- d) **Procedure** The test procedures shall be as follows.

- 1) Weigh 150 mg of the sample to the nearest 1 mg, and transfer into a 100 ml Erlenmeyer flask with interchangeable ground joint. Add 50 ml of caramel test solution with a volumetric pipette. Previously, dry the sample for 3 h in a constant temperature oven maintained at $(115 \pm 5) ^\circ\text{C}$ and allow to cool to room temperature in a desiccator.
 - 2) Shake for 30 min with a shaker at room temperature ($20 ^\circ\text{C}$ to $30 ^\circ\text{C}$).
 - 3) Filter with a filter paper.
 - 4) Discard approximately 20 ml of the initial filtrate and immediately determine the absorbance of the filtrate obtained thereafter at a wavelength of 430 nm.
 - 5) Perform a blank test by carrying out procedures in 2) to 4) on 50 ml of caramel test solution.
- e) **Calculation** Calculate the caramel decolouring performance by formula (13), and round the value to the first decimal place.

$$D = \left(1 - \frac{E'}{E}\right) \times 100 \% \dots\dots\dots (13)$$

where, D : caramel decolouring performance (%)
 E' : absorbance of solution decoloured by sample
 E : absorbance of blank test

7.3 Particle size

The particle size shall be as follows.

- a) **Summary** Screen the sample and obtain the particle size from the mass of sample retained on each sieve.
- b) **Apparatus and implement** The apparatus and implement shall be as follows.
 - 1) **Constant temperature oven**, capable of adjusting the temperature within the range $(115 \pm 5) ^\circ\text{C}$.
 - 2) **Sieve** The test sieve of metal wire cloth specified in **JIS Z 8801-1**, with a frame of internal diameter 200 mm above the sieve surface.
 - 3) **Sieve shaker** Tyler type sieve shaker or a shaker of equivalent quality which can tap 130 times/min to 165 times/min, and rotate 240 r/min to 295 r/min.
- c) **Procedure** The test procedures shall be as follows.
 - 1) Dry the sample for 3 h in a constant temperature oven maintained at $(115 \pm 5) ^\circ\text{C}$, and allow to cool to room temperature in a desiccator. Weigh approximately 100 g of the sample to the nearest 0.1 g.

NOTE : For samples which cannot be dried e.g. impregnated carbon, drying procedures may be omitted. Impregnated carbon means the activated carbon impregnated with acid, base or metallic salt because of its nature to increase the adsorption performance to the specific materials.