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**Determination of volatile organic
compounds in indoor and test
chamber air by active sampling on
Tenax TA[®] sorbent, thermal desorption
and gas chromatography using MS/FID**

ICS 13.040.20

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Land, Infrastructure and Transport, through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law.

This Standard has been made based on **ISO 16000-6: 2004** *Indoor air —Part 6 : Determination of volatic organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS/FID* for the purposes of making it easier to compare this Standard with International Standard; to prepare Japanese Industrial Standard conforming with International Standard; and to propose a draft of an International Standard which is based on Japanese Industrial Standard.

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In the event of any doubts arising as to the contents,
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Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS/FID

Introduction This Japanese Industrial Standard has been prepared based on the first edition of *ISO 16000-6 Indoor air—Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS/FID* published in 2004 with some modifications of the technical contents.

The portions underlined with dotted in this Standard are the matters modified from the original International Standard. A list of modifications with the explanations is given in Annex 1 (informative).

1 Scope This Standard specifies a method for determination of volatile organic compounds (hereafter referred to as “VOCs”) in indoor air and in air sampled for the determination of the emission of VOCs from building materials using test chamber and test cells.

The method is based on use of Tenax TA®⁽¹⁾ sorbent with subsequent thermal desorption and gas chromatographic analysis[1]. In addition, the method is applicable to the measurement of non-polar and slightly polar VOCs in a concentration range of 1 µg/m³ up to several mg/m³. Using the principles described in this Standard, some very volatile compounds (hereafter referred to as “VVOCs”) and semivolatile organic compounds (hereafter referred to as “SVOCs”) can also be analysed.

Note⁽¹⁾ “Tenax TA®” shown here is cited as a generally available product for the convenience of users of this Standard. Other equivalent products may be used if a certification showing the same effect is available.

NOTE : The International Standard corresponding to this Standard is as follows.

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are **IDT** (identical), **MOD** (modified) and **NEQ** (not equivalent) according to **ISO/IEC Guide 21**.

ISO 16000-6: 2004 *Indoor air—Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS/FID* (MOD)

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. If the indication of the year of publication is given to these referred standards, only the edition of indicated year constitutes the provisions of this Standard but the revision and amendment made thereafter do not apply. The normative references without the indication of the year of publication apply only to the most recent editions (including amendments).

JIS A 1901 *Determination of the emission of volatile organic compounds and aldehydes for building products—Small chamber method*

JIS A 1960 *Indoor air—General aspects of sampling strategy*

NOTE : ISO/DIS 16000-1: 2001, *Indoor air—Part 1: General aspects of sampling strategy* is equivalent to the said standard.

JIS A 1966 *Indoor air—Sampling and analysis of volatile organic compounds by sorbent tube/ thermal desorption/capillary gas chromatography—Pumped sampling*

NOTE : ISO 16017-1:2000, *Indoor, ambient and workplace air – Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography—Part 1: Pumped sampling* is equivalent to the said standard.

ISO/DIS 16000-10: 2004 *Indoor air—Part 10: Determination of the emission of volatile organic compounds—Emission test cell method*

3 Terms and definitions For the purposes of this documents, the following terms and definitions apply.

3.1 semi-volatile organic compound (SVOC) ⁽²⁾ organic compound whose boiling point is in the range from (240 °C to 260 °C) to (380 °C to 400 °C)⁽³⁾

Note ⁽²⁾ This classification has been defined by World Health Organization (WHO).

⁽³⁾ Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for classification of compound volatility that may be used for classification of organic compounds [3].

NOTE : SVOCs have vapour pressures between 10^{-2} kPa and 10^{-8} kPa.

3.2 volatile organic compound (VOC) ⁽²⁾ organic compound whose boiling point is in the range from (50 °C to 100 °C) to (240 °C to 260 °C) ⁽³⁾

NOTE : VOCs generally have saturated vapour pressures at 25 °C greater than 10^{-2} kPa

3.3 very volatile organic compound (VVOC) ⁽²⁾ organic compound whose boiling point is in the range from (0 °C to 5 °C) to 100 °C ⁽³⁾

NOTE : VVOCs typically have vapour pressures greater than 15 kPa [3].

3.4 total volatile organic compounds (TVOC) the sum of volatile organic compounds, sampled on Tenax TA®, which are eluted between and including *n*-hexane and *n*-hexadecane, are detected with a flame ionization detector (hereafter referred to as “FID”) or mass spectrometric analyzer (hereafter referred to as “MS”), and are quantified by converting the total area of the chromatogram in that analytical window to toluene equivalents and are shown as TVOC/FID and TVOC-MS

NOTE : While this Standard describes the determination of individual volatile organic compounds, it is common in practice to generate a single concentration value to characterize the total amount of VOCs present in the air. This value is called the values of total volatile organic compounds (hereafter referred to as “TVOCs”, see 12.2 and clause 14). It should be emphasized that the TVOC value so obtained depends on the sampling and analytical methods used, and therefore should be interpreted taking into account the full description of these methods.

4 Principle A measured volume of sample air is aspirated and collected from room air or from an emission test chamber (JIS A 1901) or an emission test cell (ISO/DIS 16000-10:2004) by drawing through one (or more) sampler containing Tenax TA® sorbent. VOCs are retained by the sampler, and the compounds are subsequently analysed in the laboratory. The collected VOCs are desorbed by heat and transferred under inert carrier gas via cold trap/sorbent trap into a gas chromatograph system equipped with a capillary column and FID and/or MS. The principle shall be referred to JIS A 1966.

5 Reagents and raw materials

5.1 VOC for calibration including VOC, VVOC and/or SVOC to be determined, which shall be provided with suitable types and concentrations..

5.2 Dilution solvent Prepare blend solution for calibration by addition in a liquid. The dilution solvent shall be those of the highest grade or a suitable use grade specified in the relevant JIS, if any. If not, a solvent without any trouble for the test shall be used. It shall be free from compounds that may be eluted together with the single compound or the composite compound of interest (5.1).

NOTE : It is in most cases beneficial to use dilution solvent that is considerably more volatile than VOCs to be analysed. Methanol most commonly fulfills this criteria. Health and safety data for organic compounds is given in e.g. Material Safety Data Sheet (MSDS).

5.3 Tenax TA®, particle size 0.18 mm to 0.25 mm (60 mesh to 80 mesh). Tenax TA® is a porous polymer based on 2,6-diphenylene oxide. Manufactured Tenax TA® contains large quantities of impurities, which shall be removed before using it for VOC sampling. Perform cleaning by thermal conditioning the Tenax TA® under a flow of highly pure carrier gas. Select cleaning conditions so that no degradation of the sorbent occurs, e.g. at temperature of 330 °C for at least 18 h using a carrier gas at a flowrate of 100 ml/min. Pack precleaned Tenax TA® into sampler that are tightly sealed and store in a closed, emission-free container. Check the success of the cleaning procedure by performing an analysis of the cleaned sorbent.

5.4 Standard atmospheres Prepare standard atmospheres of known concentrations of the compound(s) of interest by a commonly recognized procedure. Methods described in ISO 6141 and the appropriate part of ISO 6145 are suitable. If the procedure is not applied under conditions that allow the establishment of full traceability of the generated concentration to primary standards of mass or volume, or if the chemical inertness

of the generation system cannot be guaranteed, the concentrations shall be confirmed using the independent procedure.

5.5 Standard atmosphere loaded standard sampler Prepare standard loaded sampler by passing an accurately known volume of the standard atmosphere through the sampler, e.g. by means of a pump. The volume of atmosphere sampled shall not exceed the breakthrough volume of the sorbent. After loading, disconnect and tightly seal the sampler. Prepare fresh standard loaded samplers with each batch of samples. For example, prepare the standard atmosphere so as to get 100 µg/m³ of component to be tested. For indoor air determination, load samplers with 100 ml, 200 ml, 400 ml, 1 L, 2 L, 4 L or 10 L of the 100 µg/m³ standard atmosphere selected.

5.6 Liquid loaded blend standard solutions for calibration The stability and safe storage time of blend standard solutions prepared above shall be determined. Thus, fresh standard solutions shall be prepared accordingly or if evidence of deterioration is noted.

5.6.1 Solution containing approximately 10 mg/ml of each liquid component Accurately weigh approximately 1 g of substance or substances of interest into a 100 ml volumetric flask, starting with the least volatile substance. Make up to 100 ml with methanol, stopper and shake to mix.

5.6.2 Solution containing approximately 1 mg/ml of each liquid component Introduce 50 ml of methanol into a 100 ml volumetric flask. Add 10 ml of solution prepared in 5.6.1. Make up to 100 ml with methanol, stopper and shake to mix.

5.6.3 Solution containing approximately 100 µg/ml of each liquid component Accurately weigh approximately 10 mg of substance of interest into a 100 ml volumetric flask starting with the least volatile substance. Make up to 100 ml with methanol, stopper and shake to mix.

5.6.4 Solution containing approximately 10 µg/ml of each liquid component Introduce 50 ml of methanol into a 100 ml volumetric flask. Add 10 ml of solution prepared in 5.6.3. Make up to 100 ml with methanol, stopper and shake to mix.

5.6.5 Solution containing approximately 1 µg/ml of each liquid component Introduce 50 ml of methanol into a 100 ml volumetric flask. Add 10 ml of solution prepared in 5.6.4. Make up to 100 ml with methanol, stopper and shake to mix.

5.7 Preparation of standard liquid loaded sampler Standard liquid loaded sampler shall be prepared by injecting aliquots of standard solutions into a clean standard sampler. The sampler is fitted to the injection unit (6.10) through which an inert purge gas is passed, and a 1 µl to 4 µl aliquot of an appropriate standard solution is injected through the septum. After a suitable period, the sampler is then disconnected and sealed. Prepare fresh sampler with each batch of samples.

Introducing standards solution onto samplers via a gas chromatograph injector is considered the optimum approach to standard solution introduction, as components reach the sorbent bed in the vapour phase. Alternatively, the standard solution may be introduced directly onto the sorbent bed using a syringe (6.3).

NOTE : Standard blend solution for calibration shall be prepared in controlled environmental temperature conditions. Before use the solution temperature shall be adjusted, if necessary.

5.8 Commercial, preloaded standard sampler Preloaded commercial standard samplers are available. They may be used for establishing analytical accuracy control and for routine preparation of calibration curve.

5.9 Carrier gas (inert gas such as helium, argon or nitrogen) The purity of the carrier gas should permit the detection of an injection of 0.5 ng of toluene.

NOTE : The quality of the carrier gas is of great importance, as contaminants possibly contained in the gases are enriched in the cold trap together with the substances to be analysed.

6 Apparatus and devices Ordinary laboratory apparatus and the following shall be applied.

6.1 Sampler This samplers are made of stainless steel or glass, containing at least 200 mg of Tenax TA® sorbent (5.3), and equipped with metal screw caps and polytetrafluoroethene (PTFE) ferrules. For example, tubes with outside diameters of 6 mm, inside diameter of 5 mm, and length of 90 mm satisfy the requirement and are used in many commercial thermal desorbers. Use deactivated glass wool or other suitable mechanism, e.g. stainless steel frit to retain the sorbent in the tube.

Precleaned samplers containing Tenax TA® are available commercially, or weigh a suitable amount of sorbent in the laboratory and fill it into the tube to take as a sampler. Use no less than 200 mg of sorbent per tube to maintain the adsorption capacity.

To pack the sorbent in the tube, insert a plug of deactivated glass wool or a stainless steel gauze into one end of the tube. Transfer the adsorbent into the tube, assisted by suction if desired. Place a glass wool plug or a stainless steel net after the sorbent to retain it in the tube.

NOTE : A description of the determination of breakthrough volume is given in **JIS A 1966**, Annex B. Breakthrough volumes are proportional to the dimensions of the sampling tube and quantity of sorbent. As an approximate measure, doubling the bed-length of sorbent while tube diameter is kept constant doubles the SSV (safe sampling volume).

6.2 Unions of sampler For sampling, two samplers may be connected in series using metal screw-cap couplings with PTFE ferrules.

6.3 Precision syringes The precision syringes shall be readable to at least 0.1 µl.

6.4 Sampling pump The pump should refer to the requirements of **EN 1232** or **ASTM D 3686**.

6.5 Tubing Polyethylene (hereafter referred to as “PE”) or PTFE tubing of appropriate diameter is used to ensure a leak-proof fit to both pump and sampler. Plastic tubing shall not be used at the upstream of sampler. Interfering substance from the tubing may introduce contaminants.

6.6 Flow meter This means a soap bubble meter or other suitable device for gas flow determination.

6.7 Gas chromatograph (GC) system A gas chromatographic system is fitted with FID and/or MS detector capable of detecting an injection of at least 1 ng of toluene with a signal-to-noise (SN) ratio of at least 5 to 1.

6.8 Capillary column A suitable capillary column is selected for separation of analyte in the sample. Chemically bonded 100 % dimethylpolysiloxane columns of 30 m to 60 m in length, internal diameter 0.25 mm to 0.32 mm and phase thickness 0.25 μm to 0.33 μm are examples of columns proven to be suitable for VOC analysis of indoor air and emission test chamber air.

6.9 Thermal desorption apparatus An apparatus for two-stage thermal desorption of the sampler and transfer of desorbed vapours via an inert gas flow into a GC is required.

A typical apparatus has a mechanism for holding the sampler to be desorbed whilst they are heated and purged simultaneously with inert carrier gas. The desorption temperature and time are adjustable, as is the carrier gas flowrate. The apparatus may also incorporate additional features, such as automatic sampler loading, leak testing and a cold trap or other suitable function to concentrate the desorbed sample. The desorbed sample contained in the purge gas is transferred to the capillary column in gas chromatograph via a heated transfer line.

6.10 Injection facility for preparing standards liquid loaded sampler (optional) A conventional gas chromatographic injection unit may be used for preparing a standard for calibration curve. This can be used *in situ*, or it can be mounted separately.

The carrier gas line to the injector shall be retained. The back of the injection unit shall preferably be adapted, if necessary, to fit the sampler. This can be done conveniently by means of compression coupling with an O-ring seal.

7 Conditioning and storage of sampler

7.1 Conditioning Prior to each sampling use, condition the precleaned sampler at 300 °C for 10 min under inert carrier gas at a flowrate of 50 ml/min to 100 ml/min, to remove residual VOC possibly trapped in the sampler. Analyse a representative number of conditioned sampler for blank value confirmation, using routine analytical parameters, to ensure that the thermal desorption blank is sufficiently small. The sampler blank level is acceptable if artifact peaks are less than 10 % of the typical areas of the analytes of interest. If the blank is unacceptable, recondition the samplers by repeating the conditioning procedure.

If the blank is still unacceptable even after repeated conditioning, the samplers shall be refilled (6.1).

7.2 Storage of conditioned sampler before sampling Seal conditioned sampler with metal screw-cap fitted with PTFE ferules and store in an emission-free container at room temperature. Use conditioned sampler within four weeks. Samplers stored for four weeks or longer shall be reconditioned before sampling.