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Validation Study for Establishing a Standard Test Method for Volatile Organic Compounds in Indoor Air in Japan using Thermal Desorption

Maiko Tahara,[a,*] Masahiro Chiba,[b] Shiori Oizumi,[b] Aya Onuki,[c] Ikue Saito,[c] Reiko Tanaka,[d] Takashi Yamanouchi,[d] and Shinobu Sakai[a]

[a]*National Institute of Health Sciences, 3-25-26 Tonomachi, Kawasaki-ku, Kawasaki, Kanagawa 210-9501, Japan;* [b]*Hokkaido Institute of Public Health, Kita 19, Nishi 12, Kita-ku, Sapporo, Hokkaido 060-0819, Japan;* [c]*Tokyo Metropolitan Institute of Public Health, 3-24-1 Hyakunin-cho, Shinjuku-ku, Tokyo, 169-0073, Japan;* [d]*Yokohama City Institute of Public Health, 2-7-1, Tomiokahigashi, Kanazawa-ku, Yokohama, Kanagawa, 236-0051, Japan*

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The Committee on Sick House Syndrome: Indoor Air Pollution, established by the Ministry of Health, Labour and Welfare of Japan, is reviewing indoor air quality guidelines. A comprehensive exposure assessment is essential for pollutants with revised guideline values or newly developed candidate pollutants, necessitating the development of standardized test methods for an accurate evaluation. However, the available test methods that have been provided as a standard test method (measurement manual) were introduced over 20 years ago. Its applicability to pollutants for which guideline values have been established since then had not been examined. Therefore, we established a test method for six compounds based on the current guideline values and three candidate compounds that underwent initial risk assessment. This method considered the new guideline values established after 2001 using solid-phase adsorption-thermal desorption-gas chromatography/mass spectrometry, as indicated in the measurement manual for volatile organic compounds. This method was validated at four institutions using samples at approximately 1/10th the concentration of the current, revised, and newly proposed guideline values, as of 2017. Results revealed that the average recovery of the four laboratories ranged from 84.2 to 95.6%, the repeatability ranged from 0.43 to 16%, which was <20% for all nine compounds, and the reproducibility ranged from 4.4 to 16%, which was <20%, thereby effectively achieving the target evaluation criteria. Therefore, this method could be presented as a standard test method for nine volatile organic compounds.

Key words indoor air, volatile organic compounds, inter-laboratory validation, thermal desorption, standard test method

INTRODUCTION

Severe health problems (Sick House Syndrome) caused by indoor living environments with high airtightness and thermal insulation were observed in the 1990s. To address the indoor air pollution caused by chemical compounds, the Ministry of Health, Labour, and Welfare (MHLW) of Japan has established guideline values for indoor air concentrations. Current guidelines include 13 volatile organic compounds (VOC) and semi-volatile organic compounds (SVOC), and more than 20 years have passed since the last compound was added in 2002.[1)] As the possibility of new indoor air pollution from various household products as well as alternative compounds to those for which guideline values are established and compounds derived from building materials has increased,[2-4)] the Committee on Sick House Syndrome: Indoor Air Pollution (CIAP) of the MHLW is currently reviewing the chemical compound guideline values for indoor concentrations. A detailed exposure assessment requires accurate survey of concentrations of indoor pollution compounds in Japan. Therefore, developing standard test methods with verified reliability and validity for each target chemical compound is necessary. The previously provided "Manual for analysis of indoor air chemicals" (Measurement manual) was published in 2001.[5)] The compounds for which guideline values were subsequently set had not been comprehensively evaluated. Against this background, we evaluated and published the validity of the solvent extraction method for VOC and the solvent extraction method for SVOC as standard test methods for guideline values.[6-8)] As the next step, we focused on the thermal desorption (TD) method. Although the TD method is less versatile, it does not involve extraction and is highly sensitive. Therefore, in this study, an inter-laboratory validation of the solid-phase adsorption-TD-gas chromatography/mass spectrometry (GC/MS) method was conducted to determine a versatile standard test method for six VOC for which guideline values are established and three candidate compounds for which an initial risk assessment was conducted in 2016 [9)] and 2024.[10-13)]

*To whom correspondence should be addressed. e-mail: tahara@nihs.go.jp

MATERIALS AND METHODS

Target Compounds This study focused on the following six compounds (eight components) with currently established guideline values: toluene, xylene (*o*-xylene, *m*-xylene, *p*-xylene), ethyl benzene, styrene, *p*-dichlorobenzene, and tetradecane. Additionally, it included three candidate compounds (four components) for which initial risk assessment was conducted—2-ethyl-1-hexanol, 2,2,4-trimethyl-1,3-pentanediol monoisobutyrate (TMPD-MIB), (two isomers: 2,2,4-trimethyl-1,3-pentanediol 1-monoisobutyrate and 2,2,4-trimethyl-1,3-pentanediol 3-monoisobutyrate) and 2,2,4-trimethyl-1,3-pentanediol diisobutyrate (TMPD-DIB). This selection included nine compounds (Table 1).

Reagents and Sample Preparation Inert SafeLok™ stainless-steel tubes (Markes International Ltd., Bridgend, Wales, UK) filled with Tenax TA were used as collection tubes.

The standard materials were VOC mixed standard stock solution IV for indoor environmental measurements (a mixture of 10 types, 1,000 μg/mL each), toluene-d_8 standard solution from Kanto Chemical Co., Inc. (Tokyo, Japan), and tetradecane (Wako Special Grade) standard from Fujifilm Wako Pure Chemical Corporation (Osaka, Japan). Methanol 5,000 (for pesticide residue-PCB analysis) from Fujifilm Wako Pure Chemical Corporation was used as a dilution solvent for the standard stock solution.

A tetradecane standard stock solution (1,000 μg/mL) was prepared using methanol 5,000. A mixed standard solution of 500 μg/mL was prepared by mixing tetradecane and a VOC mixed standard stock solution IV in a ratio of 1:1. This mixed standard solution was used as a sample to be added to the collection tube and as a sample for calibration curves at each participating institution; it was stored in an airtight storage bottle.

Preliminary Experiments on VOC Stability for Validation The tubes were conditioned (cleaned) at 100°C for 1 h and 300°C for 2 h under high-purity helium ventilation using TC-20 (Markes International) before being tested. After adding mixed standard solution to the tube, high-purity helium gas was added at 50 mL/min for six minutes (approximately 300 mL). The sample tubes were then wrapped in an aluminum foil with a polyethylene cap and stored in an aluminum can containing activated carbon (at room temperature, $n = 3$). The recovery rates after 7 days were compared to the added amount.

Homogeneity Test for Distribution The homogeneity of the test samples was verified by the coordinator prior to distribution according to the procedure described in the International Harmonized Protocol for Proficiency Testing of (Chemical) Analytical Laboratories;[14] the only modification to this procedure was that the number of homogeneity test materials was five. Thirty tubes were filled with standard solutions for distribution, and five tubes were randomly selected for GC/MS analysis. The resulting values were compared to the added amount.

Inter-laboratory Validation of VOC Inter-laboratory validation of the analysis was conducted at the Hokkaido Institute of Public Health (Hokkaido, Japan), Tokyo Metropolitan Institute of Public Health (Tokyo, Japan), Yokohama City Institute of Public Health (Kanagawa, Japan), and the National Institute of Health Sciences (Kanagawa, Japan). These institutions are not listed in a specific order. The added amounts were blinded, and samples were analyzed within 7 days of addition.

A total of six sample tubes were sent—five with a mixed standard solution and one conditioned tube (travel blank). The tubes were fitted with Teflon caps at both ends, wrapped in an aluminum foil, and placed in aluminum cans with enclosed activated carbon. Six sample tubes were measured at each of the four institutions.

Analysis Method The analysis was performed using the internal standard method, with toluene-d_8 as the internal standard. As xylene is a mixture of three isomers and TMPD-MIB is a mixture of two isomers, three peaks for xylene and two peaks for TMPD-MIB were observed in the chromatogram. The isomeric peaks were combined and quantified. The concentration settings for the calibration curves and internal standard solutions, extraction methods, and analytical conditions for each validity evaluation were not specified, and the standard methods utilized at each institution were used (Table 2). Preliminary experiments and homogeneity test used the conditions in Table 2, D. The quantitative value was calculated for the analysis by subtracting the travel blank. Each institute reported the quantification values of travel blanks and added samples, and the signal-to-noise (S/N) ratio at the lowest concentration of the calibration curve.

The criteria for evaluating the validity of this established test method were based on the organic matter section of the "Guidelines for the Validation of Testing Methods in Drinking Water." The targets were as follows: 70–130% recovery, ≤ 20% repeatability (relative standard deviation; RSD_r), and ≤ 25% reproducibility (RSD_R) by applying the more demanding intermediate precision conditions.[15]

Table 1. Summary of the Target Compounds.

		CAS No.	M.W.	b.p.
1	Toluene	108-88-6	92.14	111
2	Xylene			
	o-Xylene	95-47-6		144
	m-Xylene	108-38-3	106.17	139
	p-Xylene	106-42-3		138
3	Ethylbenzene	100-41-4	106.17	136
4	Styrene	100-42-5	104.15	145
5	*p*-Dichlorobenzene	106-46-7	146.99	174
6	Tetradecane	629-59-4	198.39	254
7	2-Ethyl-1-hexanol	104-76-7	130.23	185
8	2,2,4-Trimethyl-1,3-pentanediol monoisobutyrate (TMPD-MIB)	25265-77-4	216.32	253
9	2,2,4-Trimethyl-1,3-pentanediol diisobutyrate (TMPD-DIB)	6846-50-0	286.41	280

M.W.: Molecular weight, b.p.: Boiling point (°C)

RESULTS AND DISCUSSION

Establishment of the Evaluation Method We established a test method using Tenax TA single-layer tubes adopted according to international standards.[16] Before conducting validity evaluations at the four institutions, we set the amount to be added and confirmed the reproducibility of addition and stability after addition at the set amount.

Establishment of the Added Amount The added amount in this assessment was based on that of ethylbenzene (58 μg/m^3), which has the lowest concentration in the guideline values for indoor air concentrations and the amendment proposed in 2017.[17] In particular, assuming the measurement in a residential house (1–5 L sampling), the amount was set to 17 ng, less than one-tenth of the absolute amount when 3 L of air was collected (Table 3). For xylene, a compound with a revised guide-

Table 2. Analytical Conditions of Each Institution

	A	B	C	D
TD Conditions				
Instrument	Shimadzu, TD-20R	Markes, TD100	Perkin Elmer, TurboMatrix 650	Shimadzu, TD-30R
Tube desorption (°C)	280	280	250	280
Purge time (min)	10	10	3	8
Purge rate (mL/min)	60	50	50	50
Carrier gas	Helium	Helium	Helium	Helium
Cold trap temperature (°C)	2	0	–20	–20
Trap desorption (°C)	250	320	250	280
Line temperature (°C)	150	250	240	250
Valve temperature (°C)	180	210	240	250
GC/MS Conditions				
Instrument	Shimadzu, GCMS-QP2010	Agilent + JEOL, JMS-Q1500GC	Shimadzu, GCMS-2010 Plus	Shimadzu, GCMS-QP2020
Column	DB-1 (Agilent)	VF-1ms (Agilent)	Rtx-1 (RESTEK)	Rtx-1 (RESTEK)
	0.25 mm i.d. × 60 m, 1 μm	0.25 mm i.d. × 60 m, 1 μm	0.32 mm i.d. × 60 m, 1 μm	0.32 mm i.d. × 60 m, 1 μm
Column temperature	40°C (5 min) → 10°C/min → 300°C (3 min)	40°C (5 min) → 8°C/min → 240°C (5 min) → 20°C/min → 300°C (5 min)	40°C → 5°C /min→ 280°C (4 min)	40°C → 5°C/min → 250°C (3 min)
Interface temperature (°C)	250	250	250	250
Ion source temperature (°C)	200	150	200	200
Scan range (*m/z*)	45–450	29–350	35–450	35–450
Quantitative ion / Qualifying ion (*m/z*)				
Toluene	91 / 92	91 / 92	91 / 92, 65	91 / 92, 65
Xylene	91 / 106	91 / 106	91 / 106, 105	91 / 106, 105
Ethylbenzene	91 / 106	91 / 106	91 / 106, 65	91 / 106, 51
Styrene	104 / 105	104 / 103	104 / 78, 51	104 / 103, 78
p-Dichlorobenzene	146 / 148	146 / 148	146 / 148, 111	146 / 148, 111
Tetradecane	57 / 71	43 / 57	57 / 71, 43	57 / 71, 43
2-Ethyl-1-hexanol	70 / 57	57 / 41	57 / 41, 43	57 / 41, 43
TMPD-MIB	71 / 89	71 / 43	71 / 43, 56	71 / 43, 56
TMPD-DIB	71 / 43	71 / 43	71 / 43, 41	71 / 43, 56
Toluene-d_8	98 / 100	98 / 100	98 / 100, 70	98 / 100, 70

Table 3. Establishment of Additive Amounts in Validation

	Guideline values for indoor air concentrations (μg/m^3)		Assuming a residential house (3 L sampling)		Addition (ng)
	Current [*1]	Revised / Newly proposed [*2]	Absolute amount [*3] (ng)	1/10th amount (ng)	
Toluene	260	-	780	78	17
Xylene	870	200	600	60	51
Ethylbenzene	3800	58	174	17	17
Styrene	220	-	660	66	17
p-Dichlorobenzene	240	-	720	72	17
Tetradecane	330	-	990	99	17
2-Ethyl-1-hexanol	-	130	390	39	17
TMPD-MIB	-	240	720	72	17
TMPD-DIB	-	100	300	30	17

[*1] as of 2017 [1)]
[*2] The values shown in this table were deliberated in CIAP in 2017 (21st meeting) [17,18)].
[*3] Xylene and ethylbenzene were calculated based on the proposed revised guideline values.

line value (200 μg/m^3),[17,19,20)] 17 ng of each of the three isomers was added, resulting in a total of 51 ng. Accordingly, 1.7 μL of the mixed standard solution (10 μg/mL) was added to the tubes as a sample.

Stability of VOC in the Sample Tubes The recovery rate of >79% for all compounds on the seventh day of storage at room temperature after the addition of the reference materials demonstrated that the Tenax TA tubes had no effect on the recovery at 7 days of storage at room temperature (Table 4(a)). Accordingly, the validation of the TD method was analyzed within 7 days of sample preparation.

Homogeneity Test for Distribution Random sampling analysis of the sample tubes from which standards were added showed that the reproducibility of the addition of standard

solutions was good, with recovery rates of >81% for all compounds and a concomitant accuracy of <6.5% (Table 4(b)). Therefore, five sample tubes were sent to each participating institution.

Inter-laboratory Validation Study Table 2 shows the analytical conditions for each institution, and Table 5 shows the calibration curves and S/N ratios measured under the conditions of Table 2. Although the equipment and analytical conditions differed among the institutes, the optimum conditions were set at each institute, and the analyzed calibration curves showed good correlation coefficient of 0.987-1.000. The variations (n = 5, RSD) in peak areas for toluene-d_8, the internal standard added to the five samples at each institution, were 1.1%, 0.54%, 6.7%, and 0.66% for institutions A, B, C, and D, respectively. Reproducibility was good, with <10% reproducibility at all institutions. Furthermore, the S/N ratio at the lowest concentration in the calibration curve were sufficient (Table 5), and the detections in the travel blank were sufficiently smaller than the peak at the lowest concentration in the calibration curve, indicating that the TD method is sufficient for quantifying low concentrations of VOC. The optimal conditions were established at each institution, and the sample tubes were analyzed.

The obtained quantitative values, repeatability (RSD_r), and reproducibility (RSD_R) are listed in Table 6. Compounds with high boiling points (long retention times) exhibited more significant variation in the TD method. However, the average recovery of the four laboratories ranged from 84.2% to 95.6%, RSD_r ranged from 0.43% to 16%, and RSD_R ranged from 4.4% to 16%, which was <20% for all the nine compounds, thus satisfying the target evaluation criteria. As good results were obtained for samples added at ≤1/10 of all concentrations of the current guideline values for indoor air concentrations and the revised and newly proposed guideline values as of 2017,[1,17,18] this method can be presented as a standard test method.

In conclusion, a TD method was established for measuring VOC, and a validity evaluation was conducted using six compounds for which current guideline values have been established and three candidate compounds for which an initial risk assessment was conducted in 2016. Good results were obtained even for samples added at concentrations of approxi-

Table 4. Preliminary Experimental Results in Validation
(a) Stability of VOC in the collection tube, and (b) recovery testing of samples for distribution.

	(a) Stability		(b) Distribution	
	Average	RSD	Average	RSD
	(n = 3, %)		(n = 5, %)	
Toluene	95.5	0.39	98.9	0.36
Xylene	93.4	0.45	98.5	0.33
Ethylbenzene	93.5	0.41	98.5	0.35
Styrene	88.5	0.65	95.8	2.3
p-Dichlorobenzene	95.5	0.89	98.1	1.8
Tetradecane	89.0	2.0	93.3	3.8
2-Ethyl-1-hexanol	79.3	3.7	81.3	2.6
TMPD-MIB	89.4	7.8	106	1.6
TMPD-DIB	94.5	6.8	103	6.5

Table 5. Calibration Information and S/N Ratio for Four Institutions in the Validation

	A	B	C	D
Curve range (mg/L)	5–100	2–50	5–75	1–100
Correlation coefficient	0.999–1.000	0.987–0.995	0.987–0.999	0.995–1.000
Signal-Noise (S/N) ratio*				
Toluene	409	111	531	2001
o-Xylene	206	174	159	1793
m-Xylene *p*-Xylene	299	nr	187	1933
Ethylbenzene	310	308	844	1762
Styrene	120	82	179	1407
p-Dichlorobenzene	48	190	148	933
Tetradecane	183	14	228	851
2-Ethyl-1-hexanol	50	96	18	266
TMPD-MIB-1	38	2	18	np
TMPD-MIB-2	61		18	
TMPD-DIB	1167	130	228	138

nr: not reported, np: no peak
*The lowest concentration of each calibration curve

Table 6. Recovery, Repeatability (RSD_r), and Reproducibility (RSD_R) of the Four Institutions in the Validation

	Additive amount (ng)	Quantitative value (n = 5 average, ng)				RSD_r (n = 5, %)				Four institutions		
		A	B	C	D	A	B	C	D	Average (ng)	Recovery (%)	RSD_R (%)
Toluene	17	17.2	15.6	15.8	16.4	1.4	1.1	1.3	0.80	16.2	95.6	4.4
Xylene	51	52.9	46.3	46.4	48.4	1.5	1.3	1.3	1.1	48.5	95.1	6.4
Ethylbenzene	17	17.7	15.5	15.5	16.0	1.7	1.1	1.0	1.0	16.2	95.1	6.7
Styrene	17	16.7	14.4	14.4	15.3	1.5	1.0	2.3	0.43	15.2	89.3	7.0
p-Dichlorobenzene	17	17.2	15.5	13.7	16.4	1.5	1.0	2.8	2.0	15.7	92.4	9.5
Tetradecane	17	13.4	15.2	17.7	16.2	2.8	5.2	3.4	3.6	15.6	91.9	11
2-Ethyl-1-hexanol	17	16.3	14.2	17.9	14.1	12	1.8	5.7	1.1	15.6	91.9	12
TMPD-MIB	17	12.5	12.5	15.1	17.2	6.9	13	2.6	5.2	14.3	84.2	16
TMPD-DIB	17	16.4	13.6	17.2	15.9	8.6	16	14	5.0	15.8	92.8	9.9

mately 1/10 of the current, revised, and newly proposed guideline values, and this method can be presented as a standard test method. We have evaluated and published the validity of the solvent extraction method for VOC and the solvent extraction method for SVOC as standard test methods for compounds for which guideline values for indoor air concentrations have been established.[6-8] All of these methods were included in the "Measurement Manual of Chemical Substances in Indoor Air (Unified Edition)" which was proposed on August 19, 2024.[21] These methods were established as the standard test method (official method) for CIAP on January 17, 2025.[22] We will continue our efforts to develop and improve standard test methods in Japan.

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Conflict of interest The authors declare no conflict of interest.

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