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Volatile organic compounds from building products—Results from six round robin tests with emission test chambers conducted between 2008 and 2018

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Abstract

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Emission testing of volatile organic compounds (VOC) from materials and products is commonly based on emission test chamber measurements. To ensure the comparability of results from different testing laboratories, their measurement performance must be verified. For this purpose, Bundesanstalt für Materialforschung und -prüfung (BAM) organizes an international proficiency test (round robin test, RRT) every two years using well-characterized test materials (one sealant, one furniture board, and four times a lacquer) with defined VOC emissions. The materials fulfilled the requirements of homogeneity, reproducibility, and stability. Altogether, 36 VOCs were included of which 33 gave test chamber air concentrations between 13 and 83 μ g/ m³. This is the typical concentration range to be expected and to be quantified when performing chamber tests. Three compounds had higher concentrations between 326 and 1105 μ g/m³. In this paper, the relative standard deviations (RSD) of BAM round robin tests since 2008 are compared and the improvement of the comparability of the emission chamber testing is shown by the decrease of the mean RSD down to 28% in 2018. In contrast, the first large European interlaboratory comparison in 1999 showed a mean RSD of 51%.

KEYWORDS

construction product, emission test chamber, interlaboratory comparison, proficiency testing, round robin test, VOC emission

Practical Implications

The comparability of measurements carried out by different testing laboratories is shown, and the state-of-the-art relative standard deviation (RSD) of the test method is stated. The observed improvement of the comparability leads to a better reliability of test results bringing about a higher degree of consumer protection. Moreover, manufacturers of regulated construction materials have a higher certainty when developing their products. Harmonization of the testing quality leads to a fair comparison of products tested at different test institutions.

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1 | INTRODUCTION

In 1999, de Bortoli et al. published a paper in Indoor Air¹ about the results of a European interlaboratory comparison with eighteen laboratories from 10 European countries. The paper showed results from the VOCEM project (1996–1998), the first study to investigate reasons for interlaboratory discrepancies in comparison experiments on material emission measurements. This is now more than 20 years ago, and the question is if and how the state of the art in the measurement of volatile organic compounds (VOC) emitted from building products has further improved since.

The labeling of construction products or building materials as "low-emitting" is based on measurements using third-party emission test chambers. In practice, it is difficult to validate the performance of such test chamber measurements. There always is a variation of results when testing one material at different laboratories. Therefore, it is important to know this variation and to keep it as small as possible. One way to get a value for the variation and to ensure comparable results is to conduct round robin tests (RRTs) as part of the quality management system.

In 2008, BAM started to organize such international comparisons by means of emission test chambers as proficiency tests for laboratories which are involved in the certification (labeling) or approval of building products, for example, CE marking.^{2,3} BAM has organized these RRTs on the quality control of the VOC emission testing procedure in test chambers and VOC analysis according to the established standards ISO 16000–9,⁴ ISO 16000–6,⁵ and EN 16516.⁶ EN 16516 was published in 2017 and is the latest horizontal standard for test chamber measurements. The most significant change regarding the analysis is the use of a slightly polar column.

One problem for the comparability of product emissions is the possible inhomogeneity of real samples taken from the production. In the first BAM-RRTs, test specimens from real products were selected and tested for their homogeneity. However, homogeneous material was often hard to find, and the source strengths of single VOCs were mostly not in a suitable range for a comparison exercise. To fulfill these requirements, BAM started to develop a suitable reference material for the use in test chamber measurements.⁷⁻¹⁰ In the RRTs in 2009, 2014, 2016, and 2018, a lacquer system added with a VOC mixture in suitable amounts was used as test material. It was easy to prepare, and homogeneity was ensured. In 2008, a sealant was used which was selected based on measurements in a research project about emission testing of building products. In 2012, an

industrial veneered and coated particleboard was employed which was characterized within contract testing.

The number of participating laboratories has increased from 29 from 10 European countries in 2008 to 51 participants from 14 European countries, the USA, Canada, Singapore, Japan, and China in 2018. Overall, 76 laboratories participated in the RRTs.

Some results for the individual round robin tests in 2008, 2009, and 2014 have been already published.^{2,11,12} This paper is a roundup of the RRTs organized in 2008, 2009, 2012, 2014, 2016, and 2018.

2 | MATERIALS AND METHODS

2.1 | Test design

The participants used their individual test chambers (volumes from 25 up to 3000 liters) and analytical equipment (Table 1). According to the requirements of the standards, test chambers were operated at a temperature of $23 \pm 1^{\circ}$ C and a relative humidity of $50\% \pm 5\%$. Sampling volume and sampling pumps were laboratory specific. The expected concentration range was communicated to the participants.

The emission test chambers had to be loaded with the samples within one week after sample reception and in general 14 days after sample preparation at the latest.

Shipment of the samples was done by express service and reached most of the participants within two days. Test materials were sealed airtight in a special aluminum/polyethylene composite foil.

Sampling had to be conducted seven days after loading. The duration of seven days (in contrast to the normative testing of 28 days) was chosen for two reasons: On the one hand, the testing period for the participants should be as short as possible, and on the other hand, the emission profile at that time has a relatively consistent profile as shown by Nohr et al.^{7,8}

From one RRT to the next, some compounds were selected to increase the difficulty with regard to their physicochemical properties. Furthermore, since 2012 no information was given to the participants about the employed VOCs.

2.2 | Analysis

Sampling for VOCs was carried out by using Tenax TA tubes followed by thermal desorption and GC-MS analysis according to ISO

		2008	2009	2012	2014	2016	2018
Chamber size	≤100 L	25	32	29	33	13	7
	101-968 L	39	34	38	36	51	66
	≥1000 L	36	34	33	31	36	27
Column	DB-5	64	68	45	63	69	80
	DB-1	29	11	10	7	4	4
	Other	7	21	45	30	27	16

TABLE 1 Chamber volumes and GCcolumns used by the participant (in %)

Cug/m ⁴ KSD ⁴ D ⁴⁰ SSD ⁴ Cug/m ⁴ SSD ⁴ Cug/m ⁴ SSD ⁴ Cug/m ⁴ SSD ⁴ Cug/m ⁴ M ⁴
31/32 326 48/37 15 32 32/32 326 48/37 15 32 32/32 32/32 48/37 15 40 31/22 31/22 48/37 15 53 31/22 31/22 48/37 31/32 52 64 31/33 33 43/33 16 16 71 31/33 18 24/36 52 52 34/33 13 34/33 16 16 16 20/12 105 11 11 16 16 16 21/32 105 11 11 11 16 16 16 21/12 105 11 11 11 16 16 17 16 25/53 11 11 11 11 11 16 17 55/53 11 11 11 12 12 14 16 17 55/53 11 12 12 12 14 16 16 16 17
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63 71 72 73 73 74 74 74 74 74 74 74 74 74 74 74 74 74
71 30/20 51 22/17 39 24/25 52 42 33/33 33 33 33 31 34
42 33/33 33 43/40 18 34/33 13 13 14 26/22 17 27/24 86 21/12 26/22 30 21/12 30 55/53 71 55/53 71 22/20 37 55/54 31 22/21 45 22/19 31 52/21 45 22/19 31 22/22 88 23/22 88 23/22 88 23/22 64 23/22 88 23/22 88 23/28 88 23/2
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26/22 17 27/24 86 24/20 30 24/29 30 55/53 71 31/29 30 55/53 71 31/29 30 55/53 71 31/29 30 55/53 71 23/20 37 23/20 37 23/20 37 23/20 37 23/20 37 23/20 37 23/20 37 23/20 37 23/20 37 23/20 37 23/20 37 25/40 37 25/40 37 25/21 48 25/22 46 27/17 48 26/22 46 31/22 46 31/22 54 31/22 54 31/22 54 31/22 54 31/22 54 31/22 54
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30/21 45 22/19 31 28/27 38 22/17 48 22/17 48 22/17 48 25/25 83 25/25 83 31/22 65 31/22 65 31/
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36/32 54 28/27 31 30/28 54 28/27 31 30/28 54 31 30/28 54 31 39/36 17
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TABLE 2 Relative standard deviation (mean. in %) for evaluated VOCs (all laboratories/ref. laboratories)

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VOC				2008		2009		2012		2014		2016		2018		-W
	CAS	log K _{ow}	в. Р. /°С	RSD %	Сµg/m ³	RSD %	Cμg/m ³	RSD %	Cμg/ m ³	RSD %	C μg/m ³	RSD %	C µg/m ³	RSD %	C μg/ m ³	ILEY
Camphene	79-92-5	4.22	159											23/21	37	(—
Octanal	124-13-0	2.78	171											25/20	43	
p-Cymene	99-87-6	4.10	177											22/22	48	
Butylglycol acetate	112-07-2	1.57	192											25/25	52	
Dimethyl adipate	627-93-0	1.03	110											27/28	44	
Butyl hydroxy toluene	128-37-0	5.10	265											29/28	50	
mean relative standard deviati	ion (RSD)			22		36/29		34/32		39/31		29/28		28/27		
Percentage of compounds with RSD <=30%				80		33/67		38/38		33/56		63/75		83/83		
Number of participants				29		37/26		46/28		55/24		49/21		50/24		
K _{ow} : octanol-water coefficien	t															
B.P.: boiling point																
C: concentration																

16000–6⁵ or EN 16516. Quantification had to be carried out by using substance-specific response factors. Average results had to be reported in μ g/m³ from three to five replicate samples.

2.3 | Test materials

In 2008, a sealant was used which had to be filled in u-shape profiles by the participants. An area-specific airflow rate of 44 m^3/m^2 h had to be adjusted by the participants.

In 2009, a lacquer was used for the first time which was doped with VOCs and had to be filled in Petri dishes and cured by the participants. The chamber flow rates of the participant's chambers had to be adjusted to a piece-specific airflow rate of 100 l/h per Petri dish.

In 2012, an industrial veneered and coated particleboard was employed. The test pieces were cut by BAM according to the test chamber size of the participants and an area-specific airflow rate of $1.0 \text{ m}^3/\text{m}^2$ h.

In 2014, 2016, and 2018, the lacquer system was used again and doped with different VOCs. In contrast to 2009, the lacquer was filled in Petri dishes by BAM and dried in a 20 m^3 chamber at BAM until the day of shipping.

2.4 | Data analysis

Statistical data analysis was carried out by using the software PROLab Plus A and their customized statistical approaches (QuoData GmbH) based on ISO 13528 with robust statistics and z-scores. For successful participation, a standard deviation of 30% must not be exceeded for 80% of the results. Since 2009, the evaluation of the data based on the mean value and standard deviation of a group of reference laboratories. In the beginning, these were laboratories with long-time experience in emission testing. Later on, they were defined as those having successfully participated in three consecutive round robin tests organized by BAM.

3 | RESULTS AND DISCUSSION

Table 2 lists the relative standard deviations (RSD) and the mean concentrations for all VOCs used for the evaluations of these RRTs. The first number in the column "RSD %" represents the RSD of all laboratories, and the second number is the RSD of selected reference laboratories. Their RSDs were better than the average of all laboratories for almost all compounds and years.

The bottom line of Table 2 gives information on the development of the mean standard deviation of all evaluated compounds in each RRT. They also show the percentage of compounds with an RSD of lower or equal than 30%. There is an increase of this percentage rate, which in 2009 was only 33% and in 2018 was 83%. This



FIGURE 1 Development of the percentage of compounds with an RSD \leq 30% over the years



FIGURE 2 Progress of mean RSD over the years

increase indicates the improvement of the comparability of emission test chamber measurements.

Figures 1 and 2 show the development for all laboratories and for the reference laboratories with regard to this percentage and also for the mean RSD values since 2009. It becomes obvious that the laboratories could gain experience over the years and improve their performance. It is notable that there has been a decrease in the RSDs despite the fact that from 2014 onwards the choice of compounds included more analytical challenging substances (eg, methyl pyrrolidone, triethylamine) and also higher boiling VOCs (eg, dimethyl phthalate, hexadecane). Furthermore, since 2012 no information about the substances was given to the participants. As discussed later, this might be one reason for the comparatively low mean RSD in 2008.

Figures 3 and 4 depict the correlation of the octanol/water partition coefficient (K_{OW}) and the boiling point of the used compounds on the RSD. In general, analytical results of compounds with a log K_{OW} below 1.0 had higher RSDs in the range from 36% to 55% (with exception of butanol in 2008, 18%, and 2-propoxyethanol in 2018, 28%). This might be due to the higher polarity of the compounds because of analytical difficulties when using non-polar or slightly polar columns for the gas chromatography (Table 1). The highest RSD value (55/53%) is stated for



FIGURE 3 Correlation of K_{ow} and RSD (compounds with a boiling point above 280°C were not included)



FIGURE 4 Correlation of boiling point and RSD (compounds with a log K_{OW} below 1.0 were not included)

acetic acid, underpinning that reliable analysis of this compound by Tenax[®] TDS-GC-MS is not advisable.

RSDs of analytical results of compounds with a log K_{OW} above 1.0 were in the range from 14% to 34% (with exception of hexanal, up to 43%, and butyl acetate in 2016, 48%).

VOCs with a higher boiling point were introduced in the RRT 2014 for the first time to check the performance of test chambers also for VOCs with a lower volatility. High RSDs were observed for dimethyl phthalate (63%) and hexadecane (55%) with boiling points of 284 and 287°C, respectively (Figure 4). The RSDs for higher boiling VOCs used in the RRTs 2016 (dimethyl adipate, 227°C and butyl hydroxy toluene, 265°C) and 2018 (texanol, 244°C) were lower than 31% and thus in the same range as other VOCs.

In 1999, de Bortoli et al.¹ considered a value of 40% for the RSD as acceptable. In his paper, this value was met by 7 out of 15 target compounds. From all the BAM round robin tests, 39 out of 48 results for 36 compounds showed an RSD of better than 40% and 27 out of 48 had an RSD of better than or equal to 30%. In the 2018 RRT, 10 out of 12 substances reached an RSD of better than or equal to 30%.

According to Horwitz,¹³ a value for the standard deviation of 30% can be expected for the analysis of compounds in the concentration range of $10-100 \ \mu g/m^3$.

TABLE 3 RSDs for compounds from other published data (RRTs 1999-2017)

				de Bo 1999	rtoli	Windh 2005	övel	Kirchne	er 2007	Yrieix	2010	Oppl 20)17
VOC	CAS	log K _{ow}	В. Р. /°С	RSD %	Cμg/ m ³	RSD %	Cμg/ m ³	RSD %	C μg/ m ³	RSD %	Cμg/ m ³	RSD %	C μg/ m ³
Acetic acid	64-19-7	-0.17	118			n/a	19					64	480
Ethandiol	107-21-1	-1.36	197			n/a	188						
Propandiol	57-55-6	-0.92	188			n/a	15					39	77
2-Ethyl-1-hexanol	104-76-7	2.86	184			n/a	58					21/27	21/65
2-Ethylhexyl acetate	103-09-3	3.60	199			n/a	16						
Borneol	464-45-9	2.73	210			n/a	9						
alpha-Terpineol	20126- 76-5	1.95	209			n/a	17						
Nopol	128-50-7	2.88	235			n/a	17						
Longicyclene	1137-12-8	n/a	253			n/a	12						
Longifolen	475-20-7	5.48	258			n/a	19						
Styrene	100-42-5	3.05	145					22	43				
Cyclohexanone	108-94-1	0.81	155					22/87	14/195				
Benzaldehyde	100-52-7	1.48	178					34	16				
Benzothiazole	95-16-9	2.02	230					43	78				
Butyl hydroxy toluene (BHT)	128-37-0	3.20	144					38	5			42	15
2-Methoxy-1- methylethtyl acetate	108-65-6	0.54	146					88	69				
alpha-Pinene	80-56-8	4.48	155							30	32		
beta-Pinene	127-91-3	4.16	163							28	6		
Pentanal	110-62-3	1.31	103							44	7		
Hexanal	66-25-1	1.78	131							46	17		
1-Butanol	71-36-3	0.84	118									59	30
Triethylamine	121-44-8	1.45	89									49	390
Dipropylene glycol mono methyl ether	34590- 94-8	-0.35	198									61	210
Phenol	108-95-2	1.50	181	33									
Texanol	25265- 77-4	3.00	244	27									
C7 alkylbenzene				37									
C8 alkylbenzene-1				63									
C8 alkylbenzene-2				52									
C9 alkylbenzene				70									
TXIB	6846- 50-0	3.53	280	86									
Tetradecane	629-50-5	8.38	253	44									
2-(2-butoxyethoxy) ethanol (PVC)	112-34-5	0.47	231	29				64	58				
2-(2-butoxyethoxy) ethanol (paint)	112-34-5	0.47	231	75									
Decane	124-18-5	5.01	174	74									
Undecane	1120- 21-4	5.74	196	23									
Dodecane	112-40-3	6.01	215	23									

TABLE 3 (Continued)

				de Boi 1999	rtoli	Windhö 2005	övel	Kirchne	r 2007	Yrieix	2010	Oppl 20)17
voc	CAS	log K _{ow}	B. P. /°C	RSD %	Cμg/ m ³	RSD %	Cμg/ m ³	RSD %	Cμg/ m ³	RSD %	Cμg/ m ³	RSD %	C μg/ m ³
Tridecane	629-50-5	7.60	235	33									
2-Phenoxyethanol	122-99-6	0.56	246	91									
mean relative standard de	viation (RSD)			51		40-50		32/80		37		45	
Percentage of compounds with RSD<=30%				25		n/a		40/0		50		25	
Number of participants				18		20		11		6		33	
K _{OW} : octanol-water coeffi	cient												
B.P.: boiling point													
C: concentration													
n/a: not applicable													

Note: Compounds in bold were also used in the BAM round robin tests (table 2).

However, the first international BAM RRT in 2008² showed even lower standard deviations of less than 20% for some compounds and a mean RSD of 22%. This superior performance can be explained with its structure as it was divided into three parts. Prior to the emission test chamber measurement (part 3), there were two parts to check and calibrate the analytical equipment which might have had a training effect for the participating laboratories. Additionally, the compounds were disclosed to them.

The intralaboratory variability was between 5 and 8% in the 2018 RRT, and with few exceptions it was below 10% in the earlier RRTs. These values are much smaller than the interlaboratory variability and indicate that sampling and analysis are well-controlled.

When looking at other round robin tests which were conducted in the first decade of this millennium, the results were as follows: Windhövel and Oppl¹⁴ reported relative standard deviations between 40 and 50% from the testing of adhesives as state of the art in 2005. In 2008, Oppl¹⁵ gave a variability of 50% as mean RSD. In 2007, Kirchner¹⁶ published results of an RRT using two flooring materials (rubber and PVC) as test materials. The RSDs for the compounds emitted from the rubber flooring (styrene, cyclohexanone, benzaldehyde, benzothiazole, BHT) were in the range from 22% to 43% with a mean of 32%. Testing of the PVC flooring (2-methoxy-1-methylethyl acetate, diethylene glycol mono butyl ether, cyclohexanone) resulted in very high RSDs between 64 and 88% and a mean of 80%. The high RSDs were probably due to surface damage of the samples caused by very tight rolling for shipment. In 2010, Yrieix et al.¹⁷ reported RSDs between 28 and 46% (mean 37%) for VOC measurements of the main compounds (alpha and beta Pinene, pentanal, hexanal) from a wood-based panel.

Recently, Oppl et al.¹⁸ published results of a round robin test from 2017 with two flooring adhesives and one parquet lacquer. The testing included the preparation of the test materials by the participants. The RSDs for individual substances, calibrated substance-specifically, ranged from 21% (2-ethyl-1-hexanol) to 64% (acetic acid) giving a mean of 45%.

Moritz and Breuer¹⁹ gave results for VOC measurements at a test gas generation system. Their laboratory (IFA) offers round robin tests for the measurement of VOC from air every year. These RRTs do not include a chamber test but are a good practice for sampling and analysis of VOCs from air. The RSDs for the VOCs are in general less than 10% showing that the operation of a test chamber (climate, sample preparation, loading factor, air change rate, size, material) constitutes an important source of the overall measurement uncertainty.

This literature overview (Table 3) reveals higher standard deviations for the emission test chamber-based RRTs than the BAM round robin tests and fewer compounds with a standard deviation of less than 30%.

One reason for the better results of the BAM round robin tests is probably the use of a homogeneous test material with defined compounds and appropriate emission rates. The spiked and pre-cured lacquer, which was used as test material in 2014, 2016, and 2018 turned out to be useful for such emission test chamber measurements.

4 | CONCLUSIONS

A value of less than 30% for the mean relative standard deviation is today's state of the art for the measurement of VOC-emissions by means of emission test chambers when comparing the results of laboratories worldwide. However, the results for some compounds show that it is possible to reach individual RSDs of less than 25%. To achieve this, it is necessary to have a continuous control of test chamber conditions and analytical performance. When testing real samples from a production, possible inhomogeneity and sample preparation have to be considered, additionally. The RRT of de Bortoli et al.¹ in 1999 resulted in a mean RSD of 51% and a value of 40% for the RSD was met by 7 out of 15 target compounds. In the 2018 BAM RRT for 10 out of 12 substances, an RSD of better than or equal to 30% was reached.

The BAM data show that the comparability of test chamber measurements could be improved within the last decade. During this time, the number of emission test laboratories increased as well as their proficiency. For some compounds which have been analyzed in more than one RRT (styrene, limonene, toluene, decane, and 2-propoxyethanol), no decrease in RSDs was observed, because an RSD of 30% seems to be the optimum value at present.

BAM will continue to organize round robin tests to ensure comparability and improvement of emission test chamber measurements in the future. At the same time, the reference material for emission chamber testing shall be further enhanced.

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