

Application of a novel reference material in an international round robin test on material emissions testing

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Abstract

Emission testing of products is currently a rapidly increasing field of measurement activity. Labeling procedures for construction products are based on such emission test chamber measurements, and hence, measurement performance should be verified. One possible route is to conduct testing of one material in different laboratories within a round robin test (RRT), ideally using homogeneous reference materials, which can be used within interlaboratory studies or as part of the quality management system to ensure comparable results. The applicability of a lacquer system with nine added VOCs (hexanal, styrene, n-decane, limonene, 2-ethyl-1-hexanol, N-methyl- α -pyrrolidone, 2-ethylhexyl acrylate, dimethyl phthalate, and n-hexadecane) was evaluated in an international RRT with 55 participating laboratories. An intralaboratory quality check confirmed the homogeneity and reproducibility of the lacquer material for most of the compounds (RSD 5%-6%), which was confirmed in the RRT. However, emissions varied for the polar compound N-methyl- α -pyrrolidone and the higher boiling compounds 1,2-dimethyl phthalate, and n-hexadecane which could be traced back to analytical issues. In the RRT, the interlaboratory relative standard deviations (RSDs) ranged from 30% to 65% for all participants but for reference laboratories the range was between 20% and 45%.

KEYWORDS

emission test chamber, QA/QC, reference material, round robin test, VOC

1 | INTRODUCTION

In Europe, the Construction Products Regulation (CPR, 2011/305/EU) sets basic requirements (BR) on how construction works must be designed and built. BR 3 "hygiene, health, and the environment" states low emissions of toxic gases, volatile organic compounds (VOCs), particles, etc. from building materials. Meanwhile, a worldwide network of professional commercial and non-commercial laboratories performing emission tests for the evaluation of products for interior use has been established. Therefore, comparability of test results must be ensured. The participation in round robin tests (RRTs) is a means to prove a laboratory's proficiency. For the measurement of emissions of VOCs from materials in emission test chambers, only few interlaboratory comparisons have been published so far.¹⁻⁶

It has become obvious that a major problem of such comparisons is the lack of reference materials with known emission rates of target substances. The homogeneity of the test material is of great importance and could not be assured in earlier comparison exercises. Over the last 10 years, a lot of work has been carried out to improve comparability. Moritz and Breuer⁷ implemented a VOC test gas generation line with a large number of sampling ports for the loading of test tubes. Every year, RRTs are offered for the control of the analytical performance of laboratories. Cox et al⁸ and Wei et al⁹ described reference emission materials that are suitable for loading into test chambers, but these materials emit only one specific compound (toluene or formaldehyde). Based on these materials, a first round robin test was conducted.¹⁰ However, due to the high variety of VOCs usually emitted from building materials, furniture and other products used indoors,

a broader spectrum of compounds should be covered by a reference material.

In recent years, BAM has organized RRTs on the quality control of the VOC emission testing procedure in test chambers and VOC analysis according to the established testing standards ISO 16000-9¹¹ and ISO 16000-6.^{5,12}

This study reports on the test carried out in 2014 (RR-VOC-G-BAM-2014). The participants had to identify and quantify VOC emissions from a test material within their own test facilities. For this purpose, a reference sample based on a lacquer system as described by Nohr et al^{13,14} was used. It consisted of a clear, glossy water-based lacquer (WGC-lacquer) proven to have inherently low VOC emissions. Common VOCs (in pure form) were added to this liquid lacquer, which was then homogenized, transferred to Petri dishes and cured under defined conditions.

For the RR-VOC-G-BAM-2014, some adaptations to the design of the reference sample had to be made. Questionnaires filled in by the 55 participants of the RRT revealed that the laboratories were equipped with a broad variety of emission test chambers with volumes ranging from 0.02 to 3 m³. Lacquer preparation had to be optimized for larger test chambers, and tests for reproducibility, homogeneity, and long-term stability of the material were carried out. It was required that chamber air concentrations should be quantifiable, and between 20 and 150 µg/m³.

2 | MATERIALS AND METHODS

2.1 | Chemicals

As typical indoor air pollutants, the following analytes were selected as follows: hexanal ("hex," CAS 66-25-1, ALDRICH, 98%), styrene ("styr," CAS 100-42-5, ALFA AESAR, 99.5%), n-decane ("C10," CAS 124-18-5, MERCK, >99%), (R)-(+)-limonene ("lim," CAS 138-86-3, ALDRICH, 97%), 2-ethyl-1-hexanol ("EHOH," CAS 104-76-7, ALDRICH, 99.6%), N-methyl- α -pyrrolidone ("NMP," CAS 872-50-4, FLUKA, >99.9%), 2-ethylhexyl acrylate ("EH-acr," CAS 103-11-7, ALDRICH, 98%), 1,2-dimethyl phthalate ("DMP," CAS 131-11-3, ALFA AESAR, 99%), and n-hexadecane ("C16," CAS 544-76-3, ALDRICH, 99%). These compounds were mixed into a clear, glossy water-based lacquer (Meffert AG Farbwerke, Bad Kreuznach, Germany).

2.2 | Sampling and analysis at BAM

Air sampling was carried out on Tenax[®] TA tubes followed by thermal desorption and GC/MS analysis on an Rxi-5 mseconds column (RESTEK GmbH, Bad Homburg a.d.H., Germany, 60 m, 25 mm, 0.25 µm). Analyte separation, identification, and quantification were performed based on the procedure described by Nohr et al^{13,14}.

2.3 | Emission test chambers used at BAM

For most of the preliminary emission tests, 24-L glass desiccators were used in accordance with ISO 16000-9.¹¹ Between the base part

Practical Implications

- For quality assurance/quality control (QA/QC) of emission test chamber measurements, reference materials with reproducible emission rates are essential. As long as it is thoroughly characterized, any commercially available product on the market might be used for this purpose. However, these materials mostly emit only a few, material-specific VOCs and often the homogeneity is not known. In this study, the application of a multi-VOC-emitting material in an international interlaboratory comparison with more than 50 participants is discussed. It is based on a lacquer system, which ensures homogeneous and reproducible sample preparation.

and lid, a plain stainless steel ring is mounted that provides sampling ports and connections for supply and exhaust air. Through the lid, a blade stirrer for the homogenization of test chamber air is installed. Its rotation is adjusted to obtain an air velocity above the surface of the test specimen between 0.1 and 0.3 m/s. Comparative tests with same test conditions were performed in a commercial 1 m³ emission test chamber (Vötsch Industrietechnik, Balingen, Germany) and in emission cells according to ISO 16000-10¹⁵ based on a flat flange lid with four standard ground necks with a total volume of 1 L and equipped with a blade stirrer. For sample conditioning of the high number of lacquer-filled Petri dishes for the RRT, a commercial 20 m³ chamber (WEISS TECHNIK, Balingen, Germany) was employed. Generally, the chambers were operated at a temperature of 23°C and a relative humidity (RH) of 50% in all steps. The supply airflow through the desiccators and cells was controlled periodically with an electronic soap film flow meter (Giliblator[®] 2, Sensidyne). In accordance with VDI 4300-7,¹⁶ the air change rates in the larger chambers were calculated by measuring decay curves of the tracer gas nitrous oxide (N₂O) using the gas analyser UNOR 6 N (SICK MAIHAK GmbH, Düsseldorf, Germany).

2.4 | Preliminary testing of lacquer system

Prior to the production of the RRT sample batch, the homogeneity between the samples and the reproducibility when put into different-sized test chambers needed to be investigated, as well as the required amount of added compounds. Therefore, a smaller amount of lacquer mixture was prepared and filled into 13 Petri dishes with a diameter of 94 mm (D_{94 mm}). The test specimens were cured for 3 days in a 1 m³ chamber at 23°C and 50% RH. Afterward, eight of them were loaded individually into 24 L chambers, while the remaining five samples were loaded into a 1 m³ chamber. To ensure comparable analyte air concentrations, the chamber flow rate was adjusted to a piece-specific airflow rate of 100 L/h per Petri dish. This resulted in an airflow rate of 100 L/h for the smaller chambers and 500 L/h for the larger one. Air sampling took place 7 days after loading.

2.5 | Sample adaptation for lower chamber flow rates

In the questionnaire, it turned out that some participants could only operate their chambers at airflows below 100 L/h. Therefore, smaller Petri dishes with a diameter of 34 mm ($D_{34\text{ mm}}$) were filled with lacquer mixture. The ratio of the two surface areas is approximately 8, making 2.5 g of lacquer in a small Petri dishes comparable. This resulted in a piece-specific airflow rate of 12.5 L/h per Petri dish. The comparability of these types of test materials was tested by loading four of them into four emission cells, operated at 12.5 L/h. Air sampling was carried out 7 days after loading.

2.6 | Reference sample preparation and shipment

For the preparation of the RRT samples, 12 L of the lacquer system were prepared and mixed according to the scheme shown in Figure 1. Its composition was based on the findings published by Nohr et al^{13,14}. In summary, 20 g of the mixture was weighed into 500× large polystyrene (PS) Petri dishes ($D_{94\text{ mm}}$, surface area 69.4 cm²), while 2.5 g of the mixture was weighed into 40× small PS Petri dishes ($D_{34\text{ mm}}$, surface area 9.1 cm²). The ideal amount of added VOCs was determined in the preliminary tests.

After filling, the dishes were covered with the cap and transferred to a 20 m³ chamber for curing at 23°C and 50% RH at an air exchange rate of one per hour. After all samples were prepared and placed in the chamber, the caps were all removed at almost the same time and cured for 3 days. Then the Petri dishes were capped again, sealed with an aluminum-coated polyethylene (PE) composite-foil (CLIMAPAC 2810, METPRO Verpackungsservice GmbH) and shipped to the participants.

In each package, data loggers (Keytag KTL108, Keylog) were used to monitor the temperature conditions during shipment and storage at the laboratory prior to chamber loading.

Out of the RRT batch, four Petri dishes were randomly selected for quality control and loaded individually into 24 L chambers for checking

whether the results of homogeneity testing in the preliminary tests were reproducible. Additionally, to evaluate the quality of sealing of the samples in terms of possible losses of analyte, two sealed lacquer samples were stored at 23°C and 50% RH for 14 days (estimated maximum time for shipment and storage). Then, the foil was opened, and the samples were loaded directly into two 24 L chambers.

2.7 | Instructions to participants

Fifty-five participants took part in the RRT. In advance and parallel to the test, a questionnaire had to be filled in with testing details, such as chamber parameters (volume, air flow rate, temperature, relative humidity, air velocity above sample surface) and analytical parameters (type of sampling device, sampling tubes and columns, use of an internal standard, type of thermal desorption system, gas chromatograph, and detector). The emission test chambers had to be loaded in the same week of sample reception, which could be managed by nearly all participants. The sampling had to be conducted 7 days after loading. The number of 7 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 is not significantly affected by experimental parameters (e.g. air change rate, loading, air velocity above material surface), as was shown by Nohr et al^{13,14}.

For the VOC measurements, five air samples should be taken and the results reported in mass concentrations (µg/m³). The participants had the choice either to load only one chamber or two (some asked for more). Most of them decided to use two chambers in parallel.

2.8 | Data analysis

Statistical data analysis was carried out using the software PROLab Plus A (2014) and their customized statistical approaches (QuoData GmbH, Dresden, Germany).

3 | RESULTS AND DISCUSSION

3.1 | Preliminary testing of lacquer system

Table 1 summarizes the results of the preliminary intralaboratory test for homogeneity and reproducibility performed at BAM.

Quantifiable concentrations between 6 and 88 µg/m³ could be achieved, with the amount of VOCs spiked into the lacquer meeting the initial requirements of 20–150 µg/m³ with the exception of “hex.” The mean air concentrations in the 24 L chambers could be reproduced in the 1 m³ test chamber, indicating the independence of the test chamber volume. The RSDs between the individual air concentrations in the 24-L glass desiccators ranged from 5% to 19%, and for the majority of the compounds it was well below 10%. “hex,” “NMP,” “C16,” and “DMP” showed variations of 11%, 14%, 16%, and 19%, respectively.

Three main influencing factors need to be discussed as follows: homogeneity of emissions from the material, test chamber parameters, and analysis.

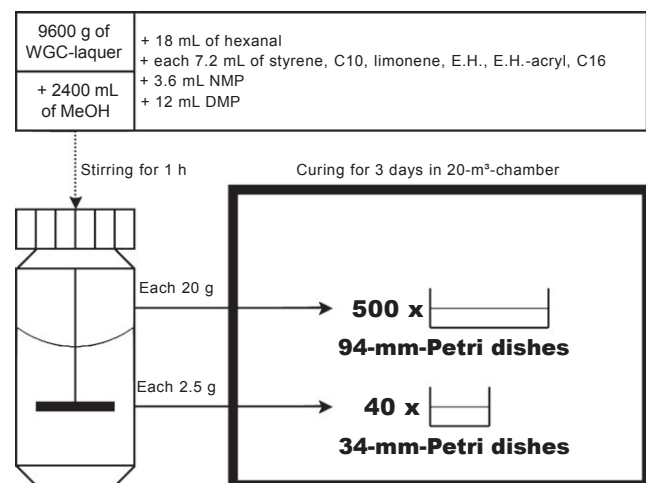
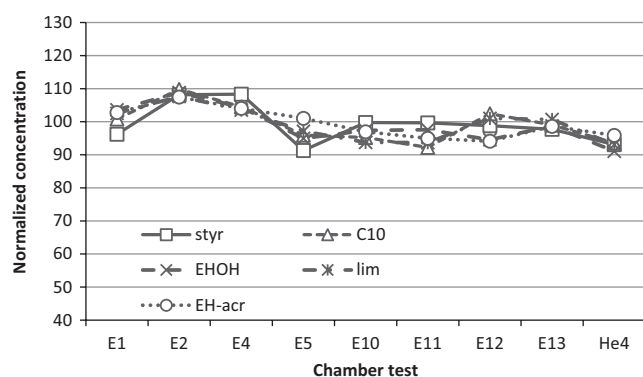


FIGURE 1 Preparation scheme of the RRT reference materials (MeOH: methanol)

TABLE 1 Test results of homogeneity and reproducibility investigations of lacquer system in comparison with analytical uncertainty given as RSD in %

Compound	Hex	Sty	C10	EHOH	Lim	NMP	EH-acr	DMP	C16
Spiked amount	0.85	0.45	0.36	0.41	0.41	0.26	0.44	1.18	0.38
AV(24 L, n = 8)	7	25	59	30	65	40	55	88	66
RSD (24 L)	11	6	6	5	5	14	5	19	16
1 m ³ (conc.)	6	23	55	28	60	40	53	85	67
RSD of standard solution (n = 6)	2.0	1.5	1.0	2.5	1.5	6.7	2.1	5.7	4.2

The amount of VOCs spiked into the lacquer is given in mg/g. AV, Average concentrations are given in µg/m³.

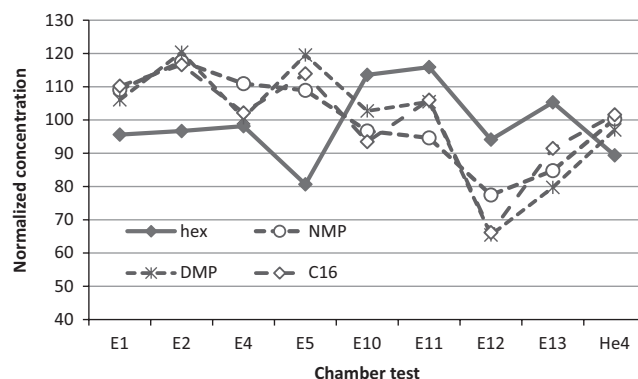
**FIGURE 2** Emissions of the compounds from lacquer samples with lowest RSD between nine different chambers [eight 24 L chambers (E1-E13); one 1 m³ chamber (He4)] with normalized data

The last line of Table 1 “RSD of standard solution” represents the results of six Tenax tubes spiked with a VOC standard solution (100 ng per tube). In this case, the results are only influenced by the thermal desorption unit and the GC, and not by sampling and chamber parameters. The resulting RSDs of these spiked tubes are significantly lower than those of the chamber measurements but show higher RSDs for “NMP,” “C16,” and “DMP” compared to the other compounds.

The graphs in Figures 2 and 3 show the results in more detail and are normalized based on the mean value of all chamber tests for each compound and grouped into those with lower and higher variations.

In addition to the three previously mentioned VOCs “NMP,” “C16,” and “DMP” shown in Figure 3, “hex” also has an enhanced RSD. The reasons might be the smaller chamber air concentration of “hex” in contrast to the concentrations of the other VOCs, or interaction of this polar compound with the chamber walls. The RSD for “hex” is much better when the liquid solution is spiked on the sampling tubes (Table 1). Degradation effects of “hex” on Tenax[®] TA, as reported by Brown et al.,¹⁷ can be excluded here because analysis took place shortly after sampling.

Chamber parameters and material inhomogeneity should only have a small impact in view of the good comparability of the measured concentrations in eight 24 L and one 1 m³ test chambers depicted in Figure 2. If this were not the case, the curve progression in Figure 2 would be similar to the ones in Figure 3. Therefore, the reference

**FIGURE 3** Emissions of the compounds from lacquer samples with highest RSD between nine different chambers [eight 24 L chambers (E1-E13); one 1 m³ chamber (He4)] with normalized data

sample was found to be suitable for its use in the RRT. As the “hex” emissions were too low, its amount in the reference lacquer batch was enhanced from 0.85 to 1.19 mg/g.

3.2 | Quality control of reference sample preparation, comparability between sample sizes and storage test

In Figure 4, the emissions from D_{34 mm} and D_{94 mm} samples of the RRT batch (picked out randomly for an intralaboratory quality check) are given as normalized concentrations based on the mean value of measurement results in the 24 L test chambers for each compound. The results of the measurements of the samples stored for 14 days are also compared. The RSD for all compounds released from the D_{94 mm} samples range from 6 to 15%, which again proves the homogeneity and reproducibility of the reference material already shown in the preliminary tests (Figures 2 and 3).

The same applies to the smaller D_{34 mm} samples, which performed with a slightly better RSD between 5% and 10%. However, compared to the compounds emissions from the D_{94 mm} samples the obtained test chamber air concentrations were significantly lower for “sty,” “C10,” and “NMP”. Also, the other compounds reveal significant variations around the 100% mark. Consequently, the results reported by the participants in the RRT using D_{34 mm} samples were not taken into consideration for the evaluation of the RRT.

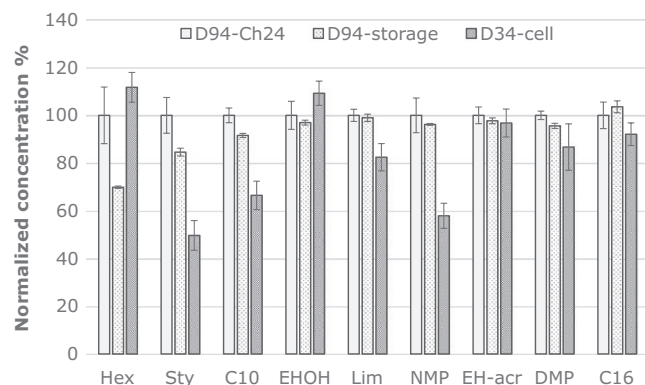


FIGURE 4 Normalized mean chamber air concentrations (based on 24 L chamber results) obtained from reference samples from the RRT batch intralaboratory quality check—four freshly produced $D_{94\text{ mm}}$ (D94-Ch24) and six $D_{34\text{ mm}}$ samples (D34-cell), and two samples stored for 14 days (D94-storage). Error bars represent standard deviation or differences (storage test)

The storage test showed that, except for the higher volatiles “hex,” “styr,” and “C10,” all compounds were in good agreement with the freshly produced samples.

3.3 | Temperature during transportation

An overview of the mean and median temperatures for transport and storage is given in Figure 5, and more details are provided in the Data S1. Apart from a few exceptions, the differences between the institutes are low and mostly between 20 and 25°C. Therefore, it can be assumed that the impact of temperature during transportation on the results is negligible. Some institutes stored the samples at very low temperatures (about or below 10°C), and here too there was no indication that test results were affected.

3.4 | Evaluation

The RRT results were evaluated using a robust statistical method according to DIN 38402-45¹⁸ and Müller & Uhlig.¹⁹ The mean value,

TABLE 2 Mean, RSD, relative repeatability (rr), and median of complete data set compared to mean reference data

Compound	Data of all participants (55)				Data of reference labs (24)	
	Mean	RSD	rr	Median	Mean	RSD
	$\mu\text{g}/\text{m}^3$	%	%	$\mu\text{g}/\text{m}^3$	$\mu\text{g}/\text{m}^3$	%
styr	50.7	30	4.6	53.0	55.1	19
C10	45.2	30	5.6	47.8	48.6	19
EHOH	29.8	31	6.9	29.7	28.7	25
lim	42.9	31	5.8	46.1	46.4	20
EH-acr	37.0	29	6.1	37.4	37.0	21
NMP	29.8	43	8.3	31.2	30.2	32
hex	17.7	43	8.8	18.0	18.1	40
DMP	55.1	63	5.5	51.0	63.1	44
C16	36.9	55	5.8	38.5	43.6	45

the RSD of the mean, the relative repeatability, and the median were calculated from the results of all participants for each compound (Table 2). In addition, reference mean and standard deviation values for the further fail or pass evaluation of the individual laboratories were calculated from the results of 24 expert laboratories. The criterion for having been selected as an expert laboratory was the successful participation in at least three former RRTs organized by BAM. Detailed data are available in the Data S1.

Individual measurement results that are quantified by use of the response factor for toluene (toluene equivalent) or with the small Petri dishes were excluded from the determination of the reference values. The exclusion of the $D_{34\text{ mm}}$ reference samples was due to the differences of the release of some compounds as reported above.

The trend for the individual compounds emissions stated in Figures 2 and 3 are mirrored by the results of the RRT. The RSDs of the reference data for “styr,” “C10,” “EHOH,” “lim,” and “EH-acr” were about 20%, a typical value for RRTs of material emissions including the complete procedure of loading, sampling, and quantification.⁵

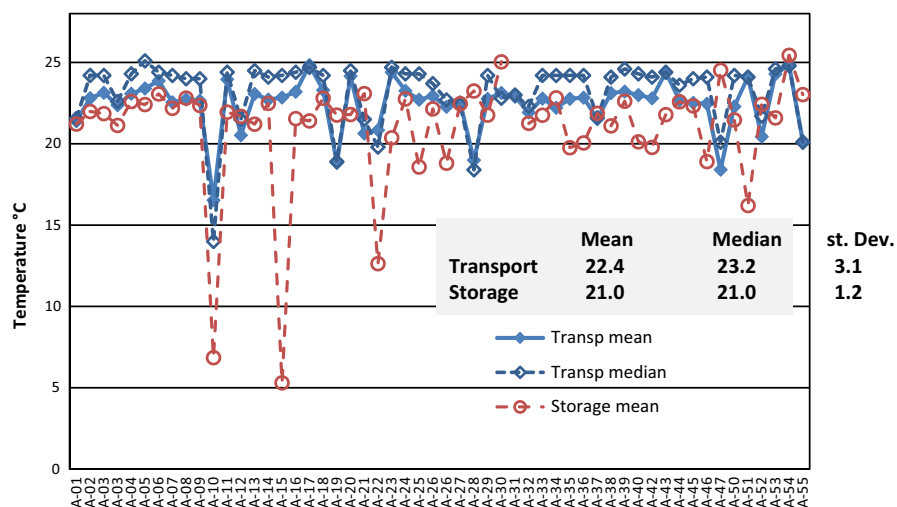


FIGURE 5 Mean and median temperatures during transportation and laboratory storage of the samples; gray table gives the overall mean of the institutes' data

The other compounds showed much higher standard deviations, which are most likely caused by interaction of the polar and higher boiling compounds with chamber walls and general analytical effects, as discussed above (Table 1). The overall results indicate the homogeneity of the RRT samples and therefore eligibility for application.

4 | CONCLUSIONS

In this study, the applicability and performance of a novel type of reference material in an international round robin test on material emissions testing with more than 50 participants are discussed. The reference material is based on a water-based, glossy lacquer with negligible inherent emissions, to which a mixture of nine VOCs was added and cured under well-defined conditions.

In preliminary chamber tests with two sample sizes ($D_{94\text{ mm}}$ and $D_{34\text{ mm}}$ Petri dishes), it turned out that the volatile compounds styrene, n-decane, 2-ethyl-1-hexanol, (R)-(+)-limonene, and 2-ethylhexyl acrylate produced highly comparable emissions regardless of the chamber type and sample amount with RSDs between 5% and 6%. In contrast, the polar compounds N-methyl- α -pyrrolidone and hexanal and those with higher boiling points (dimethyl phthalate and n-hexadecane) showed higher variations in results. However, it should also be noted that some increase in variability was even seen when these compounds were simply spiked onto sorbent tubes so material inhomogeneity is unlikely to be the major contributor to this.

For the round robin test, more than 500 samples were produced and cured for 3 days under the same conditions and wrapped in aluminum-coated PE-foil for shipment. The QA/QC tests with unpacked and packed samples picked out at random confirmed the homogeneity and reproducibility of the $D_{94\text{ mm}}$ samples. Different to the pre-tests, the emissions from $D_{34\text{ mm}}$ samples were significantly lower, and the reported results obtained with them not taken into consideration for the evaluation of the RRT. After a storage time of 14 days, the emissions from the packed samples agreed with the freshly produced ones, except for the VOCs hexanal, styrene, and n-decane. Evaluating the results of data loggers added to each package indicated no significant effect of temperature during transportation.

In general, the overall results of the RRT mirrored the observations made in the pre-tests in terms of homogeneous and reproducible emission rates for most of the doped VOCs. It is also the first approach with a mixture of VOCs covering a broad range of volatility. This is beneficial compared to other procedures in which only one compound is tackled, because in those cases effects related to sampling or the reactions of compounds with chamber walls become rather obvious.

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SUPPORTING INFORMATION

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