

# Round robin test for the implementation of odour measurements according to DIN ISO 16000-28 into the assessment of emissions from building products

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**Abstract** Odour measurements become increasingly important in view of the assessment of indoor air quality and the emissions of building products. Hence they need to be implemented into evaluation schemes such as the AgBB scheme. While there are already standards like DIN ISO 16000-28 and VDI 4302 Part 1 specifying the procedure of odour measurements using a comparison scale, no final validation of the method has been carried out so far. Therefore, a round robin test was conducted in 2012 with 12 participating laboratories that mainly consisted of the following two parts: 1) testing of the available comparison scales with respect to their operational performance and 2) execution of an odour assessment of an acrylic sealant using emission test chambers. Part one included the measurement of the air flow rates and the provided concentrations of the reference substance (acetone). Part two was conducted to test the performance of the laboratories and the applicability of DIN ISO 16000-28 and supplementary VDI 4302 Part 1 by assessing the perceived intensity and the hedonic tone of the air samples. Moreover, samples for VOC analysis were taken from the air in the emission test chamber on the 7<sup>th</sup> day of testing to allow for quality control. Resulting from this round robin test main deficits were detected and suggestions for the improvement of the odour measurement procedure were made.

## 1 Introduction

Nowadays, people, especially in cities, tend to spend most of their time indoors, e.g. at home, at workplaces, in public and using private transport. Therefore, indoor air quality has become increasingly important over the last decades. Along with the emissions of volatile organic compounds (VOC) and other air pollutants, odorants have become another aspect of interest and research during the past years. They are also under suspicion to directly affect human well-being and very often they are responsible for complaints of inhabitants. That is why the assessment of odour emissions from materials was already taken into account during the development of the AgBB (Ausschuss zur gesundheitlichen Bewertung von Bauprodukten – Committee for health-related evaluation of building products) scheme and therefore was fixed as a placeholder.

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## Ringversuch zur Einführung von Geruchsmessungen nach DIN ISO 16000-28 in die Bewertung von Emissionen aus Bauprodukten

**Zusammenfassung** Im Rahmen der Bewertung von Innenraumluftqualität und von Emissionen aus Bauprodukten gewinnt auch die Beurteilung entstehender Gerüche zunehmend an Bedeutung. Um der daraus resultierenden Forderung nach Geruchsbewertungen gerecht zu werden, müssen entsprechende Methoden in bestehende Bewertungssysteme, wie z. B. das AgBB-Schema, aufgenommen werden. Als Grundlage hierfür bieten sich die bereits bestehenden Regelwerke DIN ISO 16000-28 und VDI 4302 Blatt 1 an, die das Vorgehen bei Geruchsmessungen unter Verwendung eines Vergleichsmaßstabes vorgeben. Da für die beschriebene Methode bisher keine Validierung vorliegt, wurde 2012 ein Ringversuch mit zwölf Instituten durchgeführt, der sich hauptsächlich aus zwei Teilen zusammensetzte: 1) Überprüfung der technischen Funktionsfähigkeit der verwendeten Vergleichsmaßstäbe und 2) Durchführung einer Geruchsbewertung für eine Acryldichtmasse mithilfe von Emissionsprüfkammern. Im ersten Teil wurden sowohl die Volumenströme als auch die bereitgestellten Konzentrationen der Referenzsubstanz Aceton bestimmt. Der zweite Teil diente neben der Ermittlung der Leistungsfähigkeit der Laboratorien vor allem der Feststellung, inwieweit eine Bestimmung der empfundenen Intensität und der Hedonik nach DIN ISO 16000-28, ergänzt durch VDI 4302 Blatt 1, einheitlich möglich ist. Aus Gründen der Vergleichbarkeit und der Qualitätskontrolle wurden am siebten Tag der Messungen zusätzlich Proben der Prüfkammerluft zur VOC-Bestimmung genommen. Mithilfe des Ringversuchs konnten Defizite sowohl technischer als auch methodischer Art festgestellt und Vorschläge für die Verbesserung des Vorgehens bei Geruchsbewertungen erarbeitet werden.

To achieve the implementation of odour measurement into the AgBB scheme, a reliable method was developed which by now is standardized in DIN ISO 16000-28 [1]. The standard includes amongst others the general structure of a technical device providing defined concentrations of a vaporized reference compound (here: acetone). The device provides different acetone concentrations as a comparison scale for the grading of air samples regarding their odorous intensities. During a pilot phase the method should be tested for reliable applicability. As a suitable pre-test, a round robin test was initiated. First the existing comparison scales used by the different laboratories were investigated in terms of their applicability within the procedure of odour measurement. Thereafter, an emitting material (acrylic sealant) was shipped to the participants of the round robin test to be assessed under defined parameters. In this paper the methodology and results of the round robin test conducted at the end of 2012 shall be discussed. This was aiming at the implementation of the method given by DIN ISO 16000-28 into the AgBB scheme.

Grading of the perceived intensity.

| pi-grade | concentration in mg/m <sup>3</sup> |
|----------|------------------------------------|
| 0        | 20                                 |
| 3        | 80                                 |
| 6        | 140                                |
| 9        | 200                                |
| 12       | 260                                |
| 15       | 320                                |

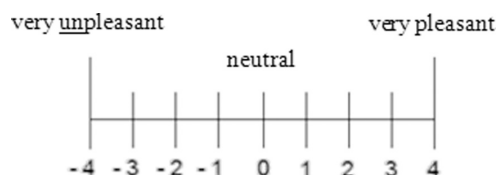


Figure 1. Hedonic tone – nine point scale.

## 2 Method

### 2.1 Preface

The basic tool for the odour assessment regarding the perceived intensity is a comparison scale that provides defined concentrations of acetone given in the unit “pi” which is the abbreviation of perceived intensity. Acetone was chosen as reference substance because the correlation between concentration and perceived intensity has proven to be approximately linear [2].

While acetone is known for generating an odour that activates not only the olfactory but also the trigeminal nerve [3; 4], a common air sample might only activate one of the nerves or also both at a time. That is why the assessors have to be trained thoroughly especially in view of the assessment of actual air samples from materials or from ambient air.

The device providing the acetone to the assessors generally consists of three parts:

- provision of the main/dilution gas flow,
- provision of a highly concentrated gaseous acetone/air mixture,
- addition of the acetone flow to the dilution gas flow.

Following the instructions of the ISO Standard, different institutes interested in odour assessment engineered a variety of comparison scales. Common to most devices is the provision of the acetone-air-mixture to the assessors' noses via glass cones. Thus, the process of smelling becomes better comparable among the institutes, particularly while taking into account a consistent way of smelling at the cones.

After all, the main aim of the method given by DIN ISO 16000-28 is to make the subjective perception of odour objectively assessable. The comparison scale is a prerequisite to allow for such an appropriate assessment.

### 2.2 Perceived intensity

The perceived intensity describes the strength of an odour experienced by the assessor. To assess the perceived intensity of an air sample a sufficient comparison scale is needed. Therefore, acetone is presented in suitable concentrations that cover the whole range of potential odour intensities for the assessment of air samples. Exemplary the comparison scale could be graduated as follows in the Table. It has no fixed upper limit.

As described in DIN ISO 16000-28, the perceived intensity as subjective stimulus has to be measured with a panel of at least eight well trained assessors. The training on the one hand consists of the grading of “random” acetone concentrations (known only by the panel leader) and on the other hand of the assessment of actual air samples (see DIN ISO 16000-28). The basis for both assessments or rather gradings is the acetone comparison scale. Since the assessment of random acetone samples should be relatively easy because identical odours are compared with each other, the transfer of other sample odours (e.g. from building materials) to the characteristic acetone odour is more difficult and requires a more intensive training of the assessment panel. Moreover, an untrained assessor might be more influenced by the hedonic tone (section 2.3) of an air sample than a trained one and thus, especially regarding off odours [5], tends to overestimate the sample.

To measure air and acetone samples the assessors have to:

- take a smell at the sample (first impression),
- take a smell at the comparison scale,
- if needed take another smell at the sample and/or comparison scale,
- assess the perceived intensity of the sample.

To allow for an objective and reproducible odour measurement the panel has to keep to the behavioural rules given in DIN ISO 16000-28 that are designed to decrease avoidable impacts on the odour assessment. Exemplary rules are the following:

- wearing of preferably odourless clothes and antiperspirants,
- no eating and drinking (except flavourless water) and smoking from at least 30 min before taking the first smell and during the whole odour assessment
- avoidance of distracting actions such as reading exciting newspaper articles, books etc. or listening to music.

### 2.3 Hedonic tone

The hedonic tone describes the impression or rather the emotional sensation of the received odour. Generally it is linked to learning processes. A panel of at least eight untrained<sup>1)</sup> assessors is needed to get reasonable results. The grading is based on a nine point scale ranging from -4 for “very unpleasant” over 0 for “neutral” to +4 for “very pleasant” as illustrated in Figure 1.

### 2.4 Interpretation of the collected data

Before the actual measurement of the sample's intensity the assessors have to graduate at least two different acetone concentrations. The maximum deviation from the set point of acetone concentrations is 2 pi [6]. The assessors whose assessments lie within this range are classified as sufficiently qualified.

Therefore, the first step of the interpretation was to eliminate all data that was determined by assessors whose measurements differed by more than 2 pi from the set point.

The remaining values for perceived intensity were averaged as arithmetic mean of every single institute. From those mean values the standard deviation and the 90% confidence interval were derived. According to DIN ISO 16000-28 the 90% confidence intervals need to be less than 2 pi to achieve

<sup>1)</sup> According to VDI 4302 Part 1 it is also possible to use the assessment panel that was trained to measure the perceived intensity for the assessment of the hedonic tone.

a sufficient precision regarding the measurement of the assessed sample.

With the standard deviation and the 90% confidence interval calculated, the results can be graphically presented. The mean values are displayed in form of bar charts including the 90% confidence as overlapping bars and the standard deviation as vertical lines (see chapt. 3.3.).

## 2.5. Round robin test for the odour measurement with comparison scale (2012)

### 2.5.1 Organizational background

On behalf of the Federal Environmental Agency (UBA), the Federal Institute for Materials Research and Testing (BAM) requested different test laboratories that implemented odour assessment, to participate in the round robin test 2012. Altogether 14 institutes took part. Due to technical difficulties the institutes 004 and 007 were not able to conduct the odour measurements.

Basically a sample as homogeneous as possible was needed for the test. According to experience, this criterion might best be met by acrylic sealant. Therefore, a selection of different sealants was tested to identify the most suitable one, which was a white acrylic sealant. Before shipment it was analysed in three parallel emission chamber tests at the BAM with regard to:

- the odour emissions (perceived intensity and hedonic tone),
- homogeneity (e.g. emission profile, reproducibility etc.),
- VOC-concentration (to quantify and qualify possible differences).

Subsequent to this preliminary enquiry the participants' comparison scales were inspected by BAM staff on-site. The focus of the inspection was on the basic design, the determination of the air flow rate as well as the acetone concentration at the devices' outlet (glass cones).

The constructional variety of the comparison scales ranged from versions with only one cone to versions with six to seven cones, from purchasable to self-developed and use of gaseous (certified acetone/air mixture in compressed gas cylinders) or liquid acetone as well as use of dry or humidified dilution gas flow. Summing up, the constructional main differences between currently tested comparison scales are:

- the number of (glass) cones,
- the generation of the dilution gas flow,
- the generation and addition of the acetone into the dilution gas flow.

The air flow rate was determined using a hot-wire anemometer<sup>2)</sup> (Co. Ahlborn) that was attached to the upper end of a 40 cm tube (ID = 3.5 cm). The acetone concentration was measured using a portable flame ionisation detector (FID, MicroFID™ of Photovac, Inc.) device.

### 2.5.2 Execution of the odour measurement

The test procedure was executed considering the requirements given by DIN ISO 16000-28 supplemented partially by VDI 4302 Part 1. The acrylic sealant was shipped in its original packaging (cartridge) along with aluminium U-profiles for the loading of the emission test chambers that were operated according to ISO 16000-9 ( $25 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  RH). A manual about how to prepare the samples and how to load and operate the emission test chambers was also attached.

<sup>2)</sup> calibrated using a gasmeter

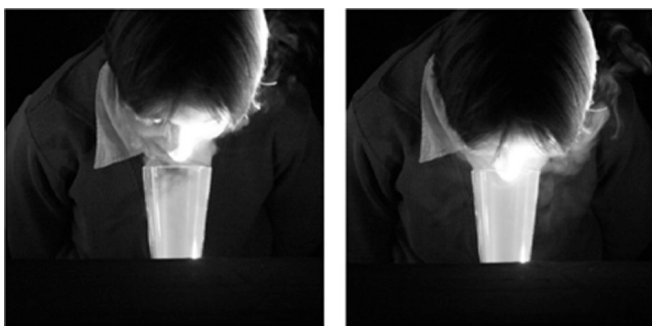


Figure 2. Wrong (left side) and correct position (right side) to take a smell – to the left the ambient air that the assessor is inhaling is visible as black spots within the white fume – to the right no black spots are visible and only sample air is inhaled.

Photos: ©Johannes Kasche

The sampling and odour measurements of the air samples were carried out on days 3 and 7 after loading the emission test chambers with the acrylic sealant. Additionally on the 7<sup>th</sup> day BAM-Tenax tubes were sampled by the participants, shipped to and analysed at the BAM.

During the odour measurement, among other behavioural rules [1; 6], it is essential that the assessors take the smells with a correct position of head and nose in order to avoid the additional inhalation of ambient air instead of solely the sample air. An example for a wrong and correct position is given in Figure 2.

## 3 Results and discussion

### 3.1 Comparison scales

The inspection of the comparison scales showed that the air flow rate ranged from 0.6 to 1.0 l/s and thus is within the pre-setting of DIN ISO 16000-28. Considering also the relative standard deviation of the air flow rate, at least two scales did not work as accurately as demanded by the standard.

The acetone concentration however differed in at least four cases by more than 20% from the reference value. In three cases the wrong concentrations might have been caused by inaccurately calibrated photo ion detectors (PIDs). Concentrations controlled by FID or dinitrophenylhydrazine checked PID showed much better results also regarding standard deviations of the determined values.

### 3.2 Emission chamber tests

Due to the provided manual and the long experience of the participants in chamber testing the loading of the emission chambers did not cause any difficulties. The standard requirements regarding temperature and humidity however could not be met by at least three institutes. How far this significantly affected the overall results of the VOC emission tests and of the odour measurements could not be determined within the round robin test. Further research should be carried out to determine this influence on the air samples.

### 3.3 Odour measurement

The round robin test showed that especially the state of experience of the assessment panel as well as the number of assessors has a significant impact on the results of the odour measurement. The more experienced the assessors were and the higher their number was, the lower the standard deviation was and the better the 90% confidence interval. With both influencing factors the confidence level and the precision of the received values were directly affected.

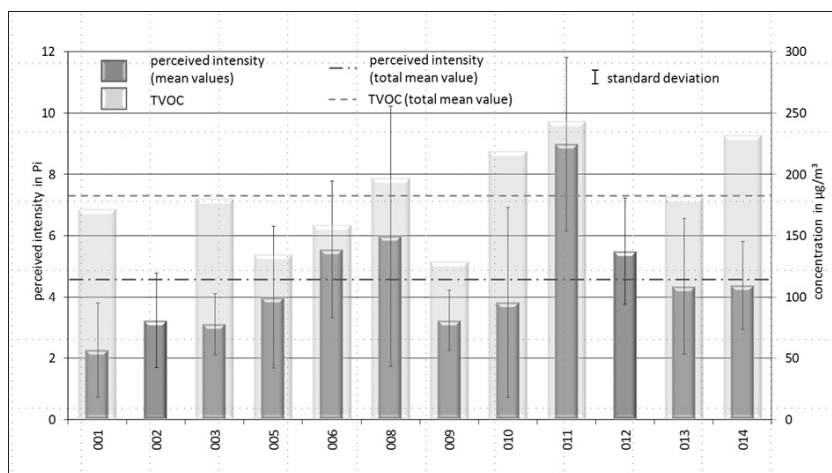


Figure 3. Perceived intensity of the round robin test's participants and total volatile organic compounds (TVOC) on the 7<sup>th</sup> day.

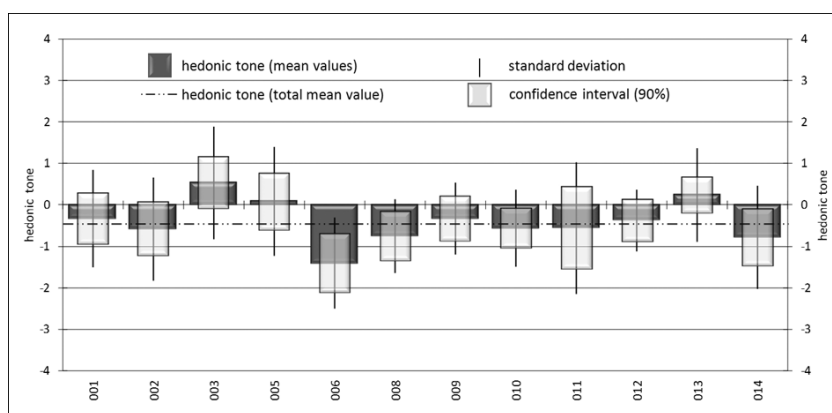


Figure 4. Hedonic tone of the round robin test's participants on the 7<sup>th</sup> day.

By measuring not only the odour but also the VOC concentrations on the 7<sup>th</sup> day of the round robin test it could be confirmed that there is no direct correlation between VOC concentrations and the perceived intensity of an air sample. This conclusion is also visually confirmed by Figure 3. For institute 002 no VOC concentrations were determined while the VOC measurements of institute 012 were not evaluable and such are not visualized in Figure 3.

The perceived intensities' mean value of all participating institutes amounts to 5.2 pi on the 3<sup>rd</sup> and 4.6 pi on the 7<sup>th</sup> day while the mean relative standard deviation of the odour measurements is about 46%. In comparison the standard deviation of round robin tests regarding VOC emissions mainly range between 20 and 30% [7 to 9]. For the VOC measurement of the current round robin test the relative standard deviations range between 23 and 35%.

Keeping in mind that the odour measurement strongly depends on the individual olfactory sense of each assessor<sup>3)</sup>, the relative standard deviation of about 46% seems acceptable and might be considered as good result.

In addition to the perceived intensity the hedonic tone of the samples was measured but was not the focus of the study.

<sup>3)</sup> e.g. a considerable amount of people are afflicted with at least partially anosmia; statistically 40% of the population are not able to smell androsterone (anosmia for urine), 36% isobutyraldehyde (anosmia for malt), 33% 1,8-cineole (anosmia for camphor), 20% 1-pyrroline (anosmia for sperm); [10] according to Hanns Hatt.

Nonetheless, statistic values were determined and are visualized in Figure 4. The hedonic tone's mean value on the 3<sup>rd</sup> day is -0.6 while it rises to -0.4 on the 7<sup>th</sup> day.

With one exception all participating institutes were able to determine the hedonic tone within the required 90% confidence interval. The averaged values of the institutes range between -1.6 and -0.2 on the 3<sup>rd</sup> day and -1.4 and 0.5 on the 7<sup>th</sup> day while the assessment of individual assessors ranges from -4.0 to 4.0 on the 3<sup>rd</sup> day and -3.0 to 4.0 on the 7<sup>th</sup> day.

## 5 Conclusion

The basic technical components of the odour measurement such as the emission test chambers and the comparison scales for the most part work sufficiently accurate. The main influencing factors lie within the operation of those two procedures. While the operation of emission test chambers was substantially tested and should not be error-prone, the odour measurement with comparison scale has not yet completed its set-up period. Therefore, further testing and validation of the method given by DIN ISO 16000-28 is needed. For example the provision of the samples should be executed as standardized as possible. For instance VDI 4302 Part 1 recommends the use of glass cones with a defined opening angle of about 12° that should be taken into account by the test laboratory.

The deficits regarding the technical aspects of the comparison scales such as acetone concentrations deviating too much from the set values have to be eliminated or at least reduced to an acceptable level according to DIN ISO 16000-28. For all the differences, nonetheless, the comparison scales allowed for relatively well comparable odour assessments among the different institutes. However, there is still room for improvement of the technical aspects but especially also regarding the state of training and experience of the assessment panel.

For several institutes an improvement could be achieved for example by a continuously supervision of the assessment panel. That way it could be guaranteed that the assessors keep to the given behavioural rules and the control could be retained over the correct way of taking smells at the cones to avoid the inhalation of ambient air and thus avoid a dilution of the actual air sample.

Concluding the method given in DIN ISO 16000-28 is potentially applicable as long as certain requirements are taken into account for the execution of odour measurements.

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