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# Sensory-based evaluation of building product emissions

Integration into the Blue Angel award criteria and assessment scheme of the Committee for Health Evaluation of Building Products



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# Sensory-based evaluation of building product emissions

Integration into the Blue Angel award criteria and assessment scheme of the Committee for Health Evaluation of Building Products

by

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On behalf of the Federal Environment Agency (Germany)

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# Preliminary note

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Some results of the study have already been published:

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# List of Abbreviations

Symbols:

L	loading factor [m <sup>2</sup> m <sup>-3</sup> ]	$\widehat{oldsymbol{\delta}}$	estimated population variance
n	air exchange rate [h <sup>-1</sup> ]	SE	standard error
П	perceived intensity [pi]	$t_{df, conf}$	t value for n-1 degrees of freedom
q	area-specific air flow rate [m <sup>3</sup> m <sup>-2</sup> h <sup>-1</sup> ]	n	random sample number
$\overline{x}$	random sample average		

Abbreviations and terms:

AirProbe	sample provision device for odour samples	LCI	lowest concentration of interest
AgBB	Committee for Health- related Evaluation of Building Products	4-PCH	4-phenylcyclohexene
DL	Determination limit	R. H.	relative humidity
CLIMPAQ	chamber for laboratory investigations of materials, pollution and air quality	SVOC	semivolatile organic compounds
DNPH	dinitro-phenylhydrazine	VOC	volatile organic compounds
GC	gas chromatograph	VVOC	very volatile organic compounds
HPLC	high performance liquid chromatography	TVOC	sum of all VOC in the retention range $C_6$ to $C_{16}$
C subst	carcinogenic substance	ΣSVOC	sum of all SVOC in the retention range $C_{16}$ to $C_{22}$
MS	mass spectrometer	TDS	thermodesorption system
МІТ	methylisothiazolinone	TENAX	polymer of 2,6-diphenyl-p- phenylene oxide
n.d.	non detected, non detectable	PD	percentage dissatisfied
ADAM	AgBB/DIBt assessment mask	R value	Risk factor in the AgBB scheme: sum of all quotients from the individual concentrations per LCI value of VOCs

# **1 INTRODUCTION AND AIM OF THE RESEARCH PROJECT**

Emissions from building products can substantially reduce indoor air quality. In this context, volatile organic compounds (VOC) emitted from building products can be evaluated using the AgBB scheme, while health assessment under this scheme requires measuring methods that are product specific. These are now available for a set of products and have been validated through the involvement of various research establishments and measurement institutes and integrated into the assessment criteria of the "Blue Angel" environmental label. However, the evaluation parameters have become stricter since lower threshold values are mostly used.

Since VOC emissions are frequently accompanied by odours, which can also lead to a decline in health, using the human nose to determine the acceptability of odours (sensory-based tests) is an important part of the assessment of building products and ought to be incorporated into the AgBB scheme. However, this aspect cannot currently be implemented into the actual assessment, since there are no generally agreed and acknowledged procedures available [1] as yet.

The objective of this research project is to test the odour measurement method we have developed in the project "Environmental and Health Provisions for Building Products – Determination and Assessment of VOC and Odour Emissions" (UFOPLAN number 202 62 320) [2] by practical application and to integrate it into the scheme of the Committee for Health-related Evaluation of Building Products (AgBB scheme).

The Blue Angel – as a voluntary mechanism for product-specific environmental protection – is ideally suited to the introduction of measurement and assessment of odour emissions for particular product groups. Based on these observations, if the compulsory introduction of odour measurements is implemented at a later date, then a decision must be made to see if it is possible to establish appropriate limits for the reasonableness of odour emissions from building products.

Reasonableness describes the level of acceptable discomfort that a person experiences from a smell. Reasonableness is used as a term in building law guidelines among other things, and is used within this research project to describe the sensory-based assessments. Reasonableness has previously not been used as an evaluation criterion for sensory-based investigations and as far as reasonableness

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is concerned, the panellists need only decide whether or not they consider the sample being evaluated to be a reasonable atmosphere for normal daily working. Reasonableness is not equivalent to acceptance which is used in the results of the assessment.

The introduction of odour evaluation into the Blue Angel assessment criteria will enable customers to actively seek harmless and low-odour materials so reducing health risks caused by odours emitted from building products. The results of this project will therefore directly help protect customers.

Indoor odours also have a major effect on how customers use ventilation and thus on the energy usage within a building. Since implementation of the Energy Saving Ordinance where energy performance calculations assume a minimum rate of air exchange, buildings have to be built with increasing air tightness. Standards such as DIN EN 15251 [3] stipulate ventilation rates for non-residential buildings in three categories (polluted, low pollution and very low pollution buildings) without specifying what is behind these categories. Introducing an odour test into the AgBB scheme and the Blue Angel criteria may contribute towards the selection of low-odour and lowemission building products. Customers behaviour towards building ventilation may also be influenced so that the building's energy needs are sustainably reduced.

# **2 OBJECTIVES**

Forty building products (e. g. floor covering adhesives, floor coverings, sealing materials) were initially subjected to sensory-based tests and their emission behaviour was appraised under the AgBB scheme. The tests were performed on the first, third, tenth and 28th day. The results and open issues of the "Environmental and health provisions for building products – Identification and evaluation of VOC emissions and odour exposure" research project (UFOPLAN project number 202 62 320) [2] were taken into account and the following particular aspects are discussed in Chapters 4.8 Sensory-based and analytical investigation of building products:

- Checking the sample container,
- Designing a simple comparative scale,
- Comparison of sample provision in emission chambers under DIN EN ISO 16000-9 [4] and Tedlar® sample containers using the CLIMPAQ (Chamber for Laboratory Investigations of Materials, Pollution and Air Quality) emission chambers widely used in Northern Europe,
- Checking whether the 1-m<sup>3</sup> and/or 5-m<sup>3</sup> emission chambers according to DIN EN ISO 16000-9 enable direct evaluation of the contaminated air flow,
- Specifying the minimum size of the panel.

The results of the sensory-based tests were used to specify a benchmark for intensity evaluation, a suggestion for defining a limit of reasonableness for the approval of building products and an evaluation method for Blue Angel (both for intensity and hedonics). Chapter 4.8 Sensory-based and analytical investigation of building products, Chapter 4.10 Specifying a comparative scale, Chapter 5.1 Sensory-based and analytical testing of building products and Chapter 5.2 Specifying a comparative scale show these terms. Since the evaluation of individual building products has so far failed to provide information about exposure to odour in a normal room, and because air pollution is composed of simultaneous emissions from several building products under conditions other than in an emission chamber, the combination of building products was also assessed in a test room. First, a material was placed in a 13-m<sup>3</sup> chamber under normal room conditions. Tests then showed whether the same chemical composition could be found as in the standardized chamber tests. Subsequently, combinations of building products were tested in

emission chambers. Tests were undertaken to see if the results would be transferable to existing rooms where conditions different to those in emission chambers prevail. Normally, a number of odours from various sources overlap, some odours may be absorbed and other products may act as sinks. Chapters 4.9 "Sensory-based and analytical testing of building products in a 13-m<sup>3</sup> chamber" and 5.1.6 "Sensory-based and analytical investigations of building products in the 13-m<sup>3</sup> chamber" discuss these tests and their results as well as the conclusions.

# **3 FUNDAMENTALS**

# 3.1 THE NOSE AND ODOUR PERCEPTION

The nose works by moistening and warming inhaled air. At the same time it serves as a reflex organ (e.g. sneezing if dust is inhaled) and houses the smell receptors. During breathing, air is inhaled and flows via the external nose into the internal nasal area. This is almost completely lined with mucous membrane. Air then passes through the nasopharyngeal cavity into the lower respiratory system. The olfactory region is responsible for smell perception (olfactory epithelium, olfactory bulb). This is an area of approx. 2 x 2.5 cm<sup>2</sup> (Deetjen, 1992 [5]) to 2 x 5 cm<sup>2</sup> (Schmidt, 2000 [6]) of the olfactory epithelium. The smell receptors, the so-called cilia, are on the olfactory epithelium, Figure 3-1 shows the structure. According to Schmidt, the olfactory epithelium comprises the olfactory receptor cells, supporting cells, cilia, apical knobs and some serous glands.



# Figure 3-1: Scheme of olfactory epithelium with connections to olfactory bulb

According to Schmidt the smell receptors (cilia) produce electrical impulses and pass them to the smell sensory nerve fibres. The smell information converges at the only synapse (external plexiform layer) between the receptors and the brain cortex (internal plexiform layer) and is transferred to a nerve cell of the olfactory bulb. From the olfactory bulb the smell information is then passed by nerve tracts (axons) to the brain. The brain recognises the odour through interaction with other brain regions (thalamus, limbic system). Existing memories are activated and the incoming stimulation is integrated. Various other functional circles are also activated in humans through the sensation of smell. Thus there is a highly emotional component of smell perception due to the close connection to the limbic system. Smell perception can very rapidly evoke pleasant or unpleasant feelings. These feelings are called hedonic components of smell perception.

Figure 3-2 shows the transformation of chemical smell stimuli into electrical signals (transduction). This begins with the contact of an odour molecule on a specific receptor protein in the cilia membrane.



# Figure 3-2: Signal pathway of an odour stimulus

When the odour molecule binds to the receptor protein, the membrane conductivity is increased by opening up ion channels and an ion flow is produced (Na pump). This ion flow causes a cell polarisation. A receptor potential develops in the cell body producing an action potential, which is passed on via the axon.

Schmidt shows the principle of signal transduction in the cilia membrane (see Figure 3-2, cilia). If an interaction occurs between an odorous substance and a receptor, the signal transduction mechanism is triggered. A stimulating guanylic nucleotide (regulatory protein) activates the adenylate cyclase enzyme, which in turn increases the concentration of the messenger substance cAMP (cyclic adenosine mono phosphate) in the cell. Odorous substances can generate thousands of these

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messengers. The cAMP molecules open cation channels in the cell membrane. Influx of cations (sodium, calcium) through this channel into the cells generates a receptor potential.

According to Deetjen et al. [5] the cilia are in a mucous layer which must be first penetrated by the odour molecules. For this purpose the odorous substances must be sufficiently volatile and sufficiently water-soluble and must also exhibit certain fatsolubility.

The influx of calcium and sodium ions into the cell increases their concentration. The ions may be bound to the channel, blocking it, so turning it off. Consequently, the odour will no longer be detected. This explains the process of adaptation at a molecular level. Adaptation is understood to be the process of getting used to odours and the associated reduction in perception strength. After a long period in the same environment, odours become much less noticeable or completely unnoticeable. If the receptor is no longer being activated, the original sodium and calcium concentration is restored.

#### 3.2 EMISSIONS FROM BUILDING PRODUCTS

Humans need an environment in which they can live healthily. Those living in Central Europe spend a considerable part of their time within buildings, therefore indoor air quality is important for human health and comfort. In order to preserve the air quality of the room, contamination should be as low as possible, therefore materials and objects used should be of low emission, i.e. they should give off as few pollutants as possible [7]. Building products play a major role here because many of them have a large surface area within the room and their selection frequently does not lie with the discretion of room users. Protecting the health of building users is undisputed, however it was still unclear how this protection could be achieved in detail [1]. The European Collaborative Action (ECA) "Indoor Air Quality and its Impact on Man" dealt with the evaluation of VOC emissions from building products. Specialist knowledge about the most diverse interior related topics available in Europe was compiled and summarised by ECA experts (European Union as well as Switzerland and Norway) in reports. They contain such specific data that they can be considered "pre-normative". ECA published report No. 18 "Evaluation of VOC Emission from Building Products" - a milestone in the evaluation of emissions from building products - in which an evaluation scheme for emissions from floor coverings was described as an example [8].

The Committee for Health Evaluation of Building Products (Ausschuss zur gesundheitlichen Bewertung von Bauprodukten - AgBB) produced the AgBB scheme initially for Germany in 2003. This scheme improves on the issues of the ECA Report No. 18, and is constantly being developed aimed at a Europe-wide application. AgBB was founded in 1997 by the States' Working Committee 'Environment-related Health Protection' (Länderarbeitsgruppe "Umweltbezogener Gesundheitsschutz", LAUG) of the Working Association of the Senior State Health Authorities (Arbeitsgemeinschaft der Obersten Landesgesundheitsbehörden, AOLG). The AgBB scheme (see Figure 3-3) describes a test and an evaluation concept for emissions of volatile organic compounds from building products [1, 9]. This concept establishes adequate requirements for health compatibility of building products which will enable reliable product selection in the future. These evaluation criteria were previously discussed fully with relevant manufacturers, specialist institutes and authorities.

#### 3.2.1 THE AGBB SCHEME

The assessment based on the AgBB scheme takes place using emission chamber tests on the building products. The relevant standards [4, 10, 11, 12] form the basis for the measurements. The measurement cycle begins as the chamber is

loaded. A sample is taken after 3 days and another after 28 days, both being used for the assessment. Figure 3-3 shows the assessment [1] flow chart.

Table 3-1 provides information about which groups the volatile organic components can be attributed to. The basis for the retention ranges of VOCs is the elution on a nonpolar GC column analogous to ISO 16000-6 [10]. In addition to the AgBB scheme, this standard also suggests using the ISO 16000-3 (DNPH<sup>\*</sup> method with HPLC analysis) for detecting aldehydes, in particular for the LCI (NIK) substances butenal, pentenal, pentanal and glutaraldehyde [11]. This method enables a selective verification of aldehydes and ketones which is usually fairly accurate for smaller components, up to C<sub>5</sub>. The first test takes place after 3 days which checks if the TVOC<sub>3</sub> value is smaller than or equal to 10 mg m<sup>-3</sup> and the concentration of carcinogenic compounds amounts to less than 0.01 mg m<sup>-3</sup>. If these two provisions are met, product testing is continued.

After 28 days, the second test evaluates many more parameters:  $TVOC_{28} \le 1.0 \text{ mg m}^{-3}$ ,  $\Sigma \text{ SVOC}_{28} \le 0.1 \text{ mg m}^{-3}$  and other VOCs, which are evaluated with the help of the LCI list of the AgBB scheme. The R value is determined (R  $\le$  1) with the LCI values by summing the quotients of concentration and LCI value of the respective substances. Further, the VOCs, for which no LCI value is available, is evaluated more precisely, with a sum value of VOC<sub>without LCI</sub>  $\le 0.1 \text{ mg m}^{-3}$ . The sum of carcinogenic VOCs must also satisfy the values of  $\le 0.001 \text{ mg m}^{-3}$ .

An odour test on the third and 28th day is provided in the AgBB scheme, but so far it cannot be implemented in the actual assessment. This research project proposes an odour measurement method to be integrated in the AgBB scheme and the Blue Angel award criteria.

<sup>&</sup>lt;sup>\*</sup> DNPH = dinitrophenylhydrazine

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# Figure 3-3: AgBB scheme

# Table 3-1: VOC definitions in the AgBB scheme based on ISO 16000-6

VOC	All substances in the retention range $C_6 - C_{16}$
TVOC	Sum of all substances $\ge$ 5 µg m <sup>-3</sup> in the retention range C <sub>6</sub> – C <sub>16</sub>
SVOC	All substances in the retention range > $C_{16} - C_{22}$
Σ SVOC	Sum of all substances $\ge 5 \ \mu g \ m^{-3}$ in the retention range $> C_{16} - C_{22}$

### 3.2.2 APPLICATION OF THE AGBB SCHEME

The German Institute for Building Technology (Deutsches Institut für Bautechnik, DIBt) has included this scheme in the approval criteria for floor coverings and it will also integrate it in the approval procedures for other products [13, 14, 15, 16, 17]. The Blue Angel voluntary environmental label has integrated emission tests into the award procedure for numerous building products and indoor installations. The requirements for the values to be met are noticeably sharper than within the AgBB scheme [18, 19, 20].

For textile floor coverings the European wall-to-wall carpet industry introduced the voluntary GUT inspection seal in 1990 (GUT: Carpets tested for a better living environment) [21]. The standards for the emissions from floor coverings were rapidly refined during the first 7 years and are now based on the AgBB scheme, however this also now applies stricter requirements for the product emissions to be met. From 2006 there has also been a Blue Angel environmental label (RAL-UZ 128) for textile floor coverings. Fifty products now carry this label (status: 03-2010). A further potential emitter from a floor is the floor covering adhesive [22]. Here industry has already made voluntary efforts to reduce emissions which has led to the introduction of the Emicode system [23, 24]. An environmental label has also been introduced for floor covering adhesives and other products for flooring installation (RAL-UZ 113). There are currently 29 products that have been distinguished in this way (status: 03-2010).

# 3.3 EVALUATION METHODS TO DETERMINE PERCEIVED AIR QUALITY

Since VOC emissions are frequently able to be smelt, and can also lead to a reduction in health, sensory-based testing was included as an important element in the AgBB scheme. Though many different smell measurement procedures exist - see Fischer et al. (1998) [25] and ECA (1999) [26] - so far no generally recognisable method for smell evaluation of building products is available. However work is currently on-going in standardization (ISO 16000-28 and VDI 4302 sheets 1 to 3) which will specify the first methods for sensory-based evaluation of building products.

Despite improving analysis, capabilities and the development of "artificial noses", replacing the human nose in the determination of perceived air quality has not yet been successful. Odours arise from a number of chemical substances but not all materials generate the perception of smell in humans. Many thousands of different

substances can be detected in the room air, but even a quantitative determination of each single material would not enable the combined smell effect be described.

Various methods for assessing perceived air quality have been established, some of them were investigated in the predecessor project [2] and a method has been selected for the assessment of building products: assessment of the perceived intensity using a comparative scale. This new project looks into the perceived intensity using a comparative scale and, in addition, into hedonics and acceptability. The methods and the accompanied questions are described in the following text. Chapter 4.6 "Sensory-based tests" deals with the way panellists perform the tests with or without using a comparative scale.

## 3.3.1 PERCEIVED INTENSITY

The perceived intensity  $\Pi$  can only be determined with trained panels using a comparative scale. Unlike the acceptability method with untrained panels (see Chapter 3.3.2 "Acceptance"), the intensity of odorous substances in the air is determined by comparison to different specified intensities of the reference material acetone.

The smelling capability varies from human to human. Use of comparative sources ensures that the influence of subjective perception of the test result is reduced since all panel members evaluate air quality based on the same scale.

In assessing the perceived intensity of unknown samples, the panellists rely on a comparative scale of acetone air mixtures. The comparative scale scheme is illustrated in Figure 3-4.

The comparative scale is in essence composed of three parts: sample air circulation, a source of acetone and a dosing device. The units in contact with air are almost wholly manufactured from stainless steel, glass or PTFE (polytetrafluoro-ethylene), which are all practically smell-neutral.

Sample air circulation is connected via a flange to a suitable smell-neutral air supply. The sample air circulation provides constant flow rates between 0.9 and 1.0 I/s per marker (5.4 to 6.0 I/s for six markers) which ensures an undisturbed operation. The constant source of acetone consists of a pressure-resistant wash bottle and a cooling device. The acetone-filled wash bottle has synthetic compressed air pumped through which is enriched with acetone. Cooling prevents an over saturation of the compressed air and consecutive condensation in the pipes. The acetone fog is effectively separated by a cellulose filter from the enriched air.



# Figure 3-4: Scheme of comparative scale

The six funnels are supplied with a constant air/acetone mixture via a distribution hose. A metering valve on each funnel regulates the amount of the acetone/air mixture added to the sample air within the range of 0 to 1150 mg m<sup>-3</sup>.

The design of the air supply ensures a homogeneous mixing of the acetone in the sample air. If the funnel air supply has a constant flow rate and constant pressure is ensured, the desired quantity of acetone can be adjusted by the metering valves. The adjusted concentrations have to be tested by a suitable measuring instrument.

The comparative scale consists of a range of acetone air mixtures, the graduation is linear in terms of the acetone concentration in the measurement range. The unit of the perceived intensity  $\Pi$  is pi. Six different, precisely specified mixes of acetone concentrations in the range between 20 mg m<sup>-3</sup> (0 pi) and 300 mg m<sup>-3</sup> (15 pi) help the panellists gain an ability to determine the perceived intensity of an unknown sample. It is also possible to adjust higher concentrations on the comparative scale. If samples with intensities noticeably higher than 15 pi occur, suitable alternative reference values should be made available. The values of  $\Pi$  are indicated at the funnels.

The comparative scale of intensity is specified by the following points:

- 0 pi = 20 mg acetone/ $m_{air}^3$ .
- 50% of the panel can notice an odour at 20 mg acetone/m<sup>3</sup><sub>air</sub>. It is the odour threshold for acetone.
- Concentrations for 1 to n pi follow a linear gradation of the acetone concentrations.

## 3.3.2 ACCEPTANCE

When the air quality is determined by questioning the acceptance, the panellists are asked whether they would be comfortable if they were exposed to this air over a longer period of time. For reasons of statistical safeguards a larger number of panellists (see also Chapter 5.1.5 "Requirements for a minimum panel size") is required for this question in order to obtain results with a low standard deviation and narrow confidence intervals (see the evaluation in Chapter 4.6.1 "Panel with comparative scale" and 4.6.2 "Panel without comparative scale"). The result is expected to reflect the average perception of the normal population, therefore a panel of about 40 people is needed. The question of acceptability of the presented air sample can be asked in two different ways. One version is a yes-no inquiry where the panellists need only decide whether they accept the air sample or not. From this information a PD value (percentage dissatisfied) can be calculated in %, which is the ratio of the number of dissatisfied people (who have answered 'no') to all people questioned (Formula 3-1).

# Formula 3-1: Calculation of the PD value

 $PD = \frac{number \ of \ dissatisfied \ people}{number \ of \ people \ questioned} \cdot 100 \ \%$ 

In addition to the direct determination of the PD value, the second version can take into account the degree of dissatisfaction through a more differentiated consideration. The panellists assess the acceptance from "clearly unacceptable" to "clearly acceptable".

The panellists give their assessment by a marking on a scale (Figure 3-5). The value obtained in such a way is called 'acceptance'. To determine the acceptance value, both parts of the scale are graduated into equal numbers of steps from 0 to 1 (-1: "fully unacceptable" and +1: "fully acceptable") or from 0 to 10 (-10: "fully unacceptable" and +10: "fully acceptable")). The panellists do not see the graduation of the scale and can move the slide of the scale at will (see Figure 4-20 in Chapter

4.6.2 "Panel without comparative scale"). The PD value is calculated as the ratio of the number of assessments in the negative acceptance range to the total number of assessments. Gunarssen [27] has developed another method to determine the PD value in % of the group when only the mean acceptance value ( $Acc_{arithm}$ ) is known (Formula 3-2).

# Formula 3-2: Calculation of the PD value

 $PD = \frac{\exp(-0.18 - 5.28 \times Acc_{arithm})}{(1 + \exp(-0.18 - 5.28 \times Acc_{arithm}))} \times 100 \%$ 



# Figure 3-5: Acceptance scale

# 3.3.3 HEDONICS

The hedonic note represents the emotional effect of the odour. It describes whether an odour impression is perceived as pleasant or unpleasant. The hedonic note of an odour represents the average assessment of a panel. The graduated scale shown in Figure 3-6 can be used to assess the hedonic smell effect. To avoid different interpretations, the terminal points and the middle of the bipolar scale are marked accordingly.



# Figure 3-6: Hedonic scale

The evaluation is carried out using the 9-step scale from "extremely unpleasant" (-4) to "extremely pleasant" (+4).

# 4 EXPERIMENTAL SET-UP AND TEST PROCEDURE

Experimental set-ups, methods and procedures are described for all tests in the following text (Chapter 4.1 "Test procedure" to 4.7 "Analytical measurements"). Subsequently, Chapter 4.8 "Sensory-based and analytical investigation of building products" are dealt with in test-specific set-ups and procedures.

# 4.1 TEST PROCEDURE

The measurements were simultaneously performed in "CLIMPAQ" type emission chambers in HRI's air quality laboratory and in BAM's 24-litre-chambers. Selected samples were also tested in BAM's CLIMPAQ chambers. The samples were prepared and stored according to DIN EN ISO 16000-11. Sensory-based and analytical tests were performed on the third (second), eighth (seventh), fifteenth (fourteenth) and twenty-ninth (twenty-eighth) days, and optionally on the first day. Moving the testing days from the original days, indicated in brackets, depends on the day cell loading took place and the fixed days when the panellists were available.

Two panels carried out sensory-based tests (with and without a comparative scale). In addition to acceptance and perceived intensity, hedonics and reasonableness of the samples were also determined. Figure 4-1 shows the procedure. Assessments by the panellists without a comparative scale were always performed on Wednesdays, i.e. on test days two, nine, sixteen and thirty.





#### Figure 4-1: Measurement flowchart

The same area-specific flow rate q was adjusted in the tests to ensure that the measurement results were compatible. The building products No 3900, 3901, 3915, 3916, 3948, 3949, 3950 and 3951 were tested using a flow rate of  $q = 1.25 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ . A loading factor of L = 0.8 m<sup>2</sup> m<sup>-3</sup> and a flow rate of 0.9 l s<sup>-1</sup> an area of 2.6 m<sup>2</sup>. This corresponds to 10 double-sided pieces of material with dimensions of 0.65 m x 0.2 m. The high material density in the CLIMPAQ caused problems during loading and additionally led to an inhomogeneous flow around the samples. Therefore, starting from building product No 3974, q = 2.00 m<sup>3</sup> m<sup>-2</sup> h<sup>-1</sup> was used. This corresponds to 6 dual plates of 0.65 m x 0.2 m with a total area of 1.6 m<sup>2</sup> and a flow rate of 0.85 l s<sup>-1</sup>. q = 1.56 m<sup>3</sup> m<sup>-2</sup> h<sup>-1</sup>, it was thus possible to test 8 dual plates in the CLIMPAQ.

Direct evaluation cannot be performed using the 24-litre chamber since the flow rate was too low to enable a sensory-based evaluation directly at the chamber. Besides, the ambient air of the 24-litre chambers was not suitable for sensory-based evaluation as the room in which the 24-litre chambers were placed exhibited an intense smell of the building products yet to be evaluated. Sample air was therefore transported in a 300-litre Tedlar® container made of polyvinyl fluoride<sup>\*</sup> (see Chapter 4.8.1 "Checking the sample container") from the BAM Federal Institute for Materials

<sup>\*</sup> Polyvinylfluoride container manufactured by DuPont, also denoted by the trade name "Tedlar"

Research and Testing to the air quality laboratory of the Hermann Rietschel Institute for Heating and Air Conditioning (HRI).



# Figure 4-2: Air circulation in CLIMPAQ

In the Hermann Rietschel Institute air quality laboratory, the prepared ambient air entered via a distribution box, from there it was distributed to the chambers. Flow rates were adjusted by orifices. Figure 4-2 shows an example of the distribution setup for one chamber.

The tests were performed on days 3 (2), 8 (7), 15 (14) and 29 (28) after loading the chamber. The following test sequence forms a standard test day:

- Shortly before filling, the Tedlar® containers are baked which removes their natural smell. Subsequently, the containers are transported to BAM.
- The TENAX® tubes are transported from BAM to HRI.
- In BAM, the Tedlar® containers are connected directly to the 24-litre chambers and filled up overnight (Figure 4-3).
- The filled Tedlar® containers are then transported to HRI's air quality laboratory within 3 hours.

- Simultaneously, VOC sampling is performed using TENAX® tubes in BAM and HRI. In BAM, it takes place in 24-litre chambers and, if necessary, in the CLIMPAQ, while at HRI in CLIMPAQ and Tedlar® containers.
- A panel evaluates the sample air from the Tedlar® containers and directly at HRI's CLIMPAQ with or without a comparative scale.



### Figure 4-3: Filling the containers

### 4.2 SAMPLE SELECTION

All products tested in this project were purchased in specialist shops or ordered from a catalogue. Thus the date of manufacture could not normally be identified. It was an advantage however, that the products were obtained and tested at the same time that the end-user would have acquired them.

Many products were tested in the project according to their environmental symbols or other labelling which emphasized their low-emission properties. Almost all floor covering adhesives tested carried the Emicode label with EC1 classification or the Blue Angel environmental label. This however only applied to some of the products from other product groups tested in the project. The key product types were textile floor coverings (wall-to-wall carpets), non-textile, elastic floor coverings (PVC, linoleum, rubber coverings), a hard covering (parquet floor), a pre-mixed screed, floor covering adhesives as well as a sealing compound and a levelling compound. Compatibility Labels (Ü Labels) of the German Institute of Building Technology (Deutsches Institut für Bautechnik, DIBt), the label of the Association of "Carpets tested for a better living environment" (Gemeinschaft umweltfreundlicher Teppichböden, GUT label) and the Blue Angel environmental label for the selection

of products were all considered for floor coverings. Floor coverings which carry the M1 voluntary Finnish label of low-emission building products or the Danish Indoor Climate Label (DICL) were also tested. The latter ones were selected because only these products can be identified as meeting smell requirements. Thus the product assessment based on the measuring method proposed in this project would be comparable to other assessments.

Table 4-1 lists the building products tested. All building products were assessed using sensory-based tests and, in addition, almost all of them were assessed analytically (see Chapter 5.1 "Sensory-based and analytical testing of building products").

Number	Building product	CLIMPAQ HRI	q HRI	24-litre chamber	CLIMPAQ BAM	q BAM
2008-3900	PVC floor covering	X	1.25	Х		1.25
2008-3901	Wall-to-wall carpet	Х	1.25	Х		1.25
2008-3915	Rubber floor covering	X	1.25	Х		1.25
2008-3916	Wall-to-wall carpet	X	1.25	Х		1.25
2008-3948	Rubber floor covering	Х	1.25	Х		1.25
2008-3949	Levelling compound	X	1.25	Х		1.25
2008-3950	Floor covering adhesive	Х	1.88	Х	Х	1.88
2008-3951	Wall-to-wall carpet	Х	1.25	Х		1.25
2008-3974	Wall-to-wall carpet	X	2			
2008-3975	Wall-to-wall carpet	Х	2			
2008-3976	Wall-to-wall carpet	Х	2	Х	Х	2
2008-3977	Floor covering adhesive	Х	2	Х		2
2008-3978	Floor covering adhesive	X	2	Х	X	2
2008-4003	Floor covering adhesive	Х	2			
2008-4004	PVC floor covering	Х	2			
2008-4005	Rubber floor covering	X	2			
2008-4014	Linoleum	X	2	Х		2

## Table 4-1:Tested building products

Number	Building product	CLIMPAQ HRI	q HRI	24-litre chamber	CLIMPAQ BAM	q BAM
2008-4015	PVC floor covering	X	2	Х	Х	2
2008-4026	Acrylic sealing compound	Х	44	Х	X	44
2009-4033	Floor covering adhesive	X	2	Х		2
2009-4039	Linoleum	X	2			
2009-4040	Floor covering adhesive	X	2			
2009-4041	Wall-to-wall carpet	Х	2			
2009-4061	Floor covering adhesive	X	2	Х		2
2009-4073	Screed	X	7.24	Х		
2009-4074	Wall-to-wall carpet	ххх	7.24 / 7.24 / 1.7	Х		7.24
2009-4101	PVC floor covering	Х	1.56	Х	X	1.56
2009-4141	Wall-to-wall carpet	X	1.56			
2009-4159	Parquet	Х	1.56	Х		1.56
2009-4161	PVC floor covering	X	1.56	Х		1.56
2009-4162	PVC floor covering			Х		1.56
2010-4199	PVC floor covering	X	1.56			
2010-4290	Floor covering adhesive			Х		1.25

# Table 4-1: Tested building products (continued)

# 4.3 EMISSION TEST CHAMBERS

Investigations into emissions from building products were performed using emission test chambers constructed mainly from glass or stainless steel and with a volume of approx. 24 litres, 44 litres, 5 m<sup>3</sup> and 13 m<sup>3</sup> (DIN EN ISO 16000-9). Tests complying with the specification of this standard and the AgBB scheme usually take 28 days. Samples must not be removed from the chamber until the test is complete.

In addition to specified parameters such as temperature, relative humidity, areaspecific air flow rate and air flow velocity, test chambers must meet other requirements to determine emissions:

- Inert emission test chamber walls [glass or high-grade steel (polished)] to minimize wall effects
- Thermally regulated shell to minimize time-based and spatial temperature gradients
- Minimise sealing compounds capable of causing intrinsic emissions and adsorption/desorption effects
- High-purity air supply (free of VOC and dust)
- High-purity water supply (free of VOC and particles)
- As large a source/sink ratio as possible (important for semivolatile compounds)

The emission test chambers were operated under standard climate conditions of T = 23 C and RH = 50 % in accordance with ISO 1600 0-9 [4] and 16000-11 [12] with the exception of the 13-m<sup>3</sup> chamber.

### 24-litre chambers:

The 24-litre emission test chambers were based on desiccators in accordance with DIN 55666 [28] with additional optimisation. They were equipped with inlet and exhaust connection points and a number of sampling connection points, some of which were arranged as tubes connecting directly into the desiccator, while others were placed in a plane-polished stainless steel ring between the bottom and lid of the desiccator (see Figure 4-4: 24-litre chamber). Air flow velocity was adjusted by a propeller which was connected through a magnetic clutch to the exterior speedcontrolled motor. Specially sealed ball bearings were used on the chamber side of the propeller shaft in the magnetic clutch which, after careful cleaning of the surfaces, do not exhibit any provable emission. The air exchange rate was adjusted with the aid of a needle valve and flowmeter and checked by flow-measuring tubes with continuous flow rate recording. The flange between the desiccator, stainless steel ring and lid was sealed using a plane ground joint fixed with two threaded metal rings. Some connection points were provided on the stainless steel ring for air sampling to which sampling tubes with 6 mm or 14 mm outer diameters could be connected. The chambers correspond to the requirements of DIN EN ISO 16000-9 [4]. In the following part of this report, these chambers will be called 24-litre chambers (or 24LC and EXSI\_BAM Tedlar® containers in the diagrams).





Figure 4-4:

24-litre chamber

Figure 4-5: CLIMPAQ (44-litre chamber)

#### CLIMPAQ (44-litre chamber):

The CLIMPAQ is a special test chamber which is being increasingly used for testing perceived air quality. The Name "CLIMPAQ" stands for 'Chamber for Laboratory Investigations of Materials, Pollution and Air Quality'. The CLIMPAQ was developed by Gunnarsen, Nielsen and Wolkoff [29] at the Technical University of Denmark in Copenhagen in 1994. As in all investigations of pollution sources, low-emission materials were used to build these test chambers.

In this project, HRI used four CLIMPAQs and BAM used one. The slightly modified schematic structure of a chamber is depicted in Figure 4-6. The volume of the chamber is 44 litres. The majority of the kinetic energy of the air supply was dissipated by an impact plate immediately after entering the chamber. This plate was followed by the first of two laminarisator plates. This provides for an even distribution of the air flow over the entire chamber cross-section. The actual test chamber area with the test material was the space between the two laminarisators. The air supply for the chambers at HRI was provided by a ventilation and air conditioning system equipped with a class F6 fine dust filter and an activated carbon filter at the air inlet before entering the chambers. Temperature regulation was not made within the air flow itself; instead heating capillary mats were applied around the air ducts. This was aimed at preventing the air from becoming contaminated with smell from the fittings. The air ducts were manufactured from glass, the casing of the equipment was of stainless steel. Temperature and humidity were controlled.



### Figure 4-6: Structure of a CLIMPAQ

The air was fully contaminated by the material to be tested by the time it reached the exit of the test chamber. The laden air was assessed directly by a trained panel. In addition, the emissions were determined analytically.


#### Figure 4-7: Photograph of a CLIMPAQ used at HRI

5-m<sup>3</sup> chamber:

This chamber was built according to DIN EN ISO 16000-9 [4] (see Figure 4-8). It consisted of an internal chamber of polished stainless steel and temperature regulation of the chamber was achieved by a thermally regulated blanket. Air movement in the internal chamber was achieved by a mobile fan. When baking the chamber (up to  $200^{\circ}$ ) this fan was replaced with a mobile baking system. The vapour humidification unit enabled a humidity range from about 10% to 90% relative humidity at 23 °C to be achieved. The parameters su ch as chamber air exchange rate, temperature and humidity were permanently checked, controlled and recorded. The chamber could be operated by applying air exchange rates within the range of about 0.2 to 5 h<sup>-1</sup>.



#### Figure 4-8: 5-m3 chamber with the door open

13-m<sup>3</sup> chamber:

The air quality laboratory of HRI has two chambers with a volume of 13 m<sup>3</sup> which served as comparison spaces. For this project the chambers were used both for the reproduction of a real floor and for the investigation of a combination of floor coverings. In the latter case the floor of the chamber was lined with a baked Tedlar® plastic sheet to provide an emission chamber. The layout of a 13-m<sup>3</sup> chamber is shown in Figure 4-9.

The wall structure consisted of an aluminium board on the outside, 4-cm thick insulation layer and a wallpapered plasterboard. A vapour barrier between the insulation and the inner wall provided an air-tight seal to the room. The door and window were built to be airtight. The chambers were not installed according to DIN EN ISO 16000-9, instead they were the same as a real "room". It was possible to equip them with any floor structure and with furniture as well if required.



#### Figure 4-9: Scheme of the 13-m3 chamber

The test rooms were supplied with fresh air via underfloor ventilation equipment equipped with an integrated heat exchanger. The ambient air was cleaned by a class F6 pocket filter and pumped by a central fan to the respective underfloor ventilation equipment in the test rooms. There was a second F6 filter installed in the underfloor ventilation equipment. A specified flow rate of the exhaust air was passed through stainless steel tubes into the air quality laboratory test cabin. Evaluation funnels made of glass were connected to the end of the tubes and the panellists evaluated the air from the test rooms at the glass funnels so that they did not directly enter the test rooms (blank test).

# 4.4 SAMPLING AND SAMPLE PROVISION USING TEDLAR<sup>®</sup> SAMPLE CONTAINERS

Müller tested [30] numerous plastic materials for their suitability for odour sampling and Tedlar was proved most suitable as a universal material. This thermoplastic material based on polyvinylfluoride is characterised by being highly inert and a minimum diffusion to VOC. 300-litre cushion-shaped containers were welded from this material which formed the core of the sampling and sample provision system called AirProbe (Figure 4-10). This was developed by Hermann Rietschel Institute. In order not to affect sample air, the material was baked before and after use for several hours at a minimum of 80 ℃.



### Figure 4-10: Sampling using AirProbe

It was possible to collect sample air over a long period of time using pre-treated Tedlar<sup>®</sup> containers (Figure 4-11). Sample air could be stored, transported and provided under controlled boundary conditions to a panel for sensory-based evaluation (smelling).

The AirProbe was equipped with the Tedlar<sup>®</sup> containers which could be rapidly exchanged on site, so that repeated sampling and sample collection could take place. The device was designed for sample provision in such a way that when emptying the Tedlar<sup>®</sup> container, sample air only ever came into contact with the odour-neutral materials stainless steel, glass and PTFE. A panel of 15 people could be supplied with sample air from a full container using a volumetric air flow of 0.7 to 0.9 l/s needed for the sensory-based evaluation.



#### Figure 4-11: Tedlar® container filled with sample air

All Tedlar® containers used to transport emission air were baked immediately after their use for three hours under a permanent air supply in a special baking cabinet built by HRI (Figure 4-12). This enabled the containers to be re-used since VOC adsorbed to the material could be removed. The containers were then stored in a closed empty state. Before the next sampling the containers were baked again for eight hours at 80 °C. Due to this intensive processing hardly any odour was perceptible (see Chapter 5.1.1 "Checking the sample container").

Sample containers could be used whenever the flow rate from the chambers was insufficient for a sensory-based evaluation. This was the common case as a flow rate of  $0.6 - 1 \, \mathrm{I \, s^{-1}}$  was needed for sensory-based investigations [31]. The use of containers was also beneficial when sensory-based evaluations were carried out in laboratories with a strong natural smell. Sample containers could also be used for the sensory-based evaluation of normal rooms since panellists may contaminate the air in the room and thus influence the measurement (they themselves may represent sources of pollution). It was also possible that the laboratory staff did not to want the panellist to enter their labs as they might disturb the work processes being undertaken.



Figure 4-12: Handling of Tedlar® containers and the baking cabinet

#### 4.5 SAMPLE PREPARATION

Before the actual tests, the building products were conditioned in air (23 C, 50 % RH) and stored in their original packaging. Depending upon product type, samples were prepared in different ways according to the emission tests as described in the following text.

According to the DIN EN ISO 1600-9 standard, floor coverings should be tested at an area-specific emission rate (q) of  $1.25 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$  [4]. However, this was not possible in this project as the CLIMPAQ type chambers used operate at a much higher air flow rate (about  $3.6 \text{ m}^3 \text{ h}^{-1}$ ) and have a relatively small chamber volume of 44 litres. The thickness of the samples was therefore limited to about 10 to 15 millimetres. In order to be able to compare the emission results of the various chambers, all chambers had to be operated at the same or somewhat higher emission rate.

#### Wall-to-wall carpets:

The narrow sides of the test specimens were masked with self adhesive aluminium foil tape for the chamber test. For the CLIMPAQ tests, they were arranged back to back, connected with a self adhesive aluminium foil at the edges and placed upright in the chamber. Normally, two sheets were placed into the 24-litre chambers for the tests and were laminated with an aluminium foil on the back and with self adhesive plastic strips at the edges.



#### Figure 4-13: Wall-to-wall carpet tailored for chamber tests

Wall-to-wall carpets are usually supplied as rolled goods. As many metres as possible were purchased for the project in order to fulfil the requirements for sampling this product according to DIN EN ISO 16000-11 [12]. The exact age of the samples was not known. The samples placed into the chamber were taken from the centre of the rolled goods where possible. The wall-to-wall carpets could not be tested in the CLIMPAQ using the required loading of  $1.25 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$  because they were 1 cm thick. Therefore 16 samples with an area of 60 x 20 cm<sup>2</sup> would have been necessary for a q =  $1.25 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$  at a flow rate of about 3.6 m<sup>3</sup> h<sup>-1</sup>. But this number of samples, each with a thickness of about 1 cm, could not be placed into the CLIMPAQ. Therefore an increased q was used (see 4.1 "Test procedure").

#### PVC und linoleum floor coverings:

These products were prepared similarly to the wall-to-wall carpets. Since they were mostly a few millimetres thick, more of them could be placed in the CLIMPAQ.



Figure 4-14: Two PVC samples prepared for the 24-litre chambers

### Parquet:

Sealed packages of parquet were purchased, which mostly contained one to two square metres of the material. The tests were carried out at  $q = 1.56 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ .

### Floor covering adhesives:

The adhesives tested were applied with a notched trowel on up to eight glass plates for the CLIMPAQ and on two plates for the 24-litre chambers. The spread plates were placed into the chamber after about one hour of drying.



# Figure 4-15: Application of a floor covering adhesive on a glass plate

#### 4.6 SENSORY-BASED TESTS

Sensory-based tests of the building products were performed by a panel with and without a comparative scale in HRI's air quality laboratory. The panellists' comparative scale test assessments (see Chapter 5.1.4 "Direct assessment at the 5- $m^3$  chamber") were carried out directly at the emission chambers in BAM's laboratory.

Two interconnected [32] glass rooms were available in HRI's air quality laboratory. One of them served as a break area for the panellists between the assessments and the other one was used to perform the assessments. The funnels were in the evaluation room and were connected to the emission test chambers by glass pipes. The panellists entered the room one by one to smell the funnel air and make an evaluation. They were not allowed to talk about the tests in the breaks between the tests. Both rooms were supplied with the same air via air conditioning equipment whose air-mixing components were made of glass or stainless steel. This equipment also supplied air to the CLIMPAQs. The panellists could not see the samples being assessed nor did they have any information on them. This meant that

each sample was unidentified to the panellists. The test sequences as performed by the panellists with and without a comparative scale are described in the following.

#### 4.6.1 PANEL WITH COMPARATIVE SCALE

The panel that used a comparative scale consisted of 15 people who were selected in an initial test and had to pass several training sessions during their time as panellists. Nine to 14 panellists from this group were available for each test.

The group used a reference scale – the comparative scale – (Chapter 3.3.1 "Perceived intensity") and evaluated the strength of the smell using "perceived intensity  $\Pi$ " with a unit pi (perceived intensity).

Each test day started with one evaluation session of different acetone concentrations. In this way, the panel became accustomed to the environment and test conditions. The test manager provided one acetone concentration from the comparative scale, which was within the available comparative concentrations of 1 to 15 pi.

Subsequently, the panellists evaluated the perceived intensity of the building products. They decided the value of pi using the comparative scale and entered it into data acquisition software (Figure 4-16). Unlike the acceptance and hedonics assessments, this could also go beyond 15 pi; the scale was not limited. If it turned out that the combined assessments of the products was noticeably higher than 15 pi, a higher comparison concentration was used.



Figure 4-16: Interface of the data acquisition software for perceived intensity evaluation by panellists using a comparative scale

Additionally, the hedonics were also assessed by the panellists using the comparative scale (Chapter 3.3.3 "Hedonics"). See Figure 4-17.



# Figure 4-17: Interface of the data acquisition software for hedonics evaluation by panellists using a comparative scale

The assessments were saved in a text file as raw data and then evaluated automatically.

Evaluation:

An arithmetic mean was calculated for each estimated value of perceived intensity and hedonics from the individual assessments by the panellists. In addition to the mean value, the standard deviation, median, 5%, 25%, 75% and 95% quantiles (Figure 4-18 shows the example of the evaluation for floor covering adhesive # 3978) and the 90% confidence interval was determined for each building product. The width of the ranges between the relevant quantiles represents a measure of the quality of the assessment. Figure 4-19 shows the distribution and the quantiles schematically. The illustrated results of the sensory-based evaluation indicate immediately whether the assessment is "good" or "poor" and whether the distribution is symmetrical or asymmetric. The grey area contains 50% of all assessments. The 90% confidence interval (Formula 4-3 explains the calculation of the upper and lower limits) is the range of the estimated mean value which includes the actual mean of a population (of panellists) at a 90% probability. It also represents a measure of the quality of the assessment. In order to calculate the 90% confidence interval, the random sample mean value ( $\bar{x}$ ), estimated population variance (Formula 4-1), standard error (Formula 4-2) and the t values for n-1 degrees of freedom  $(t_{df conf})$  are necessary and can be taken from charts [33].

#### Formula 4-1: Calculation of the estimated population variance

$$\hat{\delta} = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \overline{x})^2}$$

#### Formula 4-2: Calculation of the standard error

$$SE = \hat{\delta}_{\bar{x}} = \frac{\hat{\delta}}{\sqrt{n}}$$

# Formula 4-3: Calculation of the lower and upper limits of the confidence interval

Upper limit :  $\bar{x} - \hat{\delta}_{\bar{x}} \cdot t_{df,conf}$ Lower limit :  $\bar{x} + \hat{\delta}_{\bar{x}} \cdot t_{df,conf}$ 



Figure 4-18: Assessment of building product 3978 (floor covering adhesive)



Figure 4-19: Distribution of quantiles

#### 4.6.2 PANEL WITHOUT COMPARATIVE SCALE

HRI had a larger group of about 50 people available to act as a panel without a comparative scale. On the actual test days, 17 to 25 people were used.

Panellists who did not use a comparative scale evaluated acceptance, hedonics and reasonableness of the sample. Reasonableness is used as a term in building law guidelines among other things, this is why it is included in the sensory based assessments within this research project. Reasonableness has previously not been used as an evaluation criterion for sensory-based investigations. As far as reasonableness is concerned, the panellists need only decide whether or not they consider the presented sample as a reasonable atmosphere for normal daily working. Reasonableness is not equivalent to acceptance, which is used in the results of the assessment.

Panellists who do not use a comparative scale likewise input data via data acquisition software. Figure 4-20 shows the input dialogue interface visible to the panellists.



Figure 4-20: Interface of data acquisition software for panellists without comparative scale

After stating their name and sex, the panellists answered the following questions:

Question 2: What is your impression of the odour in the air sample?

Question 3: How acceptable do you think the air sample is if you assume you are to be exposed to it for a longer period of time?

Question 4: Do you consider the ambient air quality for daily work as unreasonable?

Evaluation:

A panel without the comparative scale usually evaluated acceptance, hedonics and reasonableness of the building products on days 2, 9, 16 and 30. Since the panel was only available at HRI for the assessments on a specific date, normally a weekday, the assessment days were different to those specified in the AgBB scheme. More accurate data on the days can be found in the annex. This panel was only included in the project from the ninth sample (building product # 3974) onwards because the proposal was originally planned only to include the panel with the comparative scale. However, due to practical experimental aspects it seemed pertinent to also include a panel without a comparative scale. The arithmetic mean, standard deviation and the 90% confidence interval of the group was determined from the individual assessments for acceptance and hedonics. The PD value in percent was then calculated from the mean of acceptance (see Formula 3-2 in Chapter 3.3.2 "Acceptance").

The reasonableness of a building product can be obtained as the quotient of the number of positive answers (i.e. air samples evaluated as reasonable) to the number of all answers. The result is given in percent (Formula 4-4).

### Formula 4-4: Calculation of reasonableness

 $Re \ sonableness = \frac{Number \ of \ positive \ answers}{Number \ of \ all \ answers} \cdot 100 \ \%$ 

# 4.7 ANALYTICAL MEASUREMENTS

# 4.7.1 ANALYSIS TO DETERMINE VOLATILE ORGANIC COMPOUNDS

The VOC sampling from the emission test chamber air took place according to ISO 16000-6 [10]. The air sample was drawn through a glass tube filled with Tenax® TA (similar to Figure 4-21). 20 ng cyclodecane in 1  $\mu$ l methanol as an internal standard was introduced in the Tenax® tubes before sampling. The sample volume was between 0.2 and 5 litres. The sample flow rate was 100 ml min<sup>-1</sup> and was maintained by a FLEC pump from the Chematec company or a pump trolley of our own design, consisting of a diaphragm pump and high-quality mass flow control devices (see Figure 4-22).



Figure 4-21: TENAX® tube for VOC adsorption



Figure 4-22: Pump trolley used for sampling at the emission test chamber

# Chromatography conditions:

### Injector (thermodesorption (TDS)):

•	TDS system	Gerstel TDS – 2, splitless
•	Start temperature	30 °C
•	Temperature programme	30 °C/min to 260 °C for 5 mi n; 30 °C/min to 300 °C for 10 min
•	Cold injection system	Gerstel KAS – 4, electronically controlled, splitless 1 min
•	Temperature programme	-120°C at 12 °C/s to 300°C; isothermal for 3 min
•	Liner	deactived glass tube with glass or quartz wool filling

# Gas chromatograph:

•	GC system	Agilent 6890
•	Column type	RXI 5 (dimethylpolysiloxane)
•	Column dimensions	60 m, 0.25 mm, 0.25 μm
•	Column flow rate	1.4 ml/min (constant flow)
•	Oven programme	40 °C 8 min, 10 °C/min to 150 °C f or 1 min at 8 °C/min to 300 °C for 5 min

### Detector:

•	MS system	Agilent MSD 5973
•	Temperature zones	Zone 1 (150°C/quadrupole), zone 2 (230°C/source)
•	MS conditions	Solvent delay: 5 min; mass range 25 – 550 u
•	Substance identification	Mass spectrum library NIST-02

The Tenax method enabled the most VOC to be collected and detected using the method described above. The determination limits were estimated on the basis of the smallest calibration standards. For most of the VOCs a determination limit of 1-2 ng  $\mu$ l<sup>-1</sup> could be achieved. By increasing the sample quantity for components with a higher determination limit, lower concentrations of around 1  $\mu$ g m<sup>-3</sup> were obtained. A few compounds, mostly those having stronger polar characteristics, exhibit higher determination limits in the method used.

# 4.7.2 DNPH ANALYSIS TO DETERMINE VERY VOLATILE ORGANIC COMPOUNDS

Aldehydes and ketones, among them formaldehyde, can be detected very sensitively using 2,4-dinitrophenylhydrazine (DNPH) (DIN ISO 16000-3 [11]). The resulting reaction products from aldehydes and ketones with DNPH can easily be quantified using high performance liquid chromatography (HPLC). Naturally, for this purpose, solid-phase collectors are used which are coated with the derivatization reagent (DNPH on silica gel). Commercial Supelco sampling cartridges were used for the tests in this project.

After sampling principally 30 or 60 litres of air by a FLEC pump or pump trolley at a collection rate of 500 ml/min, the loaded cartridges were stored in a refrigerator at approx. 6 - 8 °C and extracted with 1.5 ml acetonit rile for processing. The eluate was immediately measured using HPLC where analysis took place using the following devices and parameters:

 HPLC: HP1100 of Agilent Company (formerly Hewlett Packard) consisting of a binary pump, gas-sampling valve, columns, thermostat, vacuum degasifier and diode-array detector (DAD)

Column: ULTRASEP ES ALD 125 x 2.5 mm, 3 μm + pre-column

• Column temperature: 30 °C

•	Pump programme:	Solv. A	Solv. B	Flow	Time
				ml/min	min
	Start:	25 %	75 %	0.6	15
		30 %	70 %	0.6	20
		57 %	43 %	0.3	26
		52.4 %	47.6 %	0.3	32
		80 %	20 %	0.6	42
		25 %	75 %	0.6	50
		25 %	75 %	0.6	55

• Solvent: Solv. A = acetonitrile

Solv. B = 0.9 litre water + 0.1 litre tetrahydrofuran

- Flow: 0.200 ml/min
- Injection volume: 5 μl
- Wave length: 365 nm for quantification
- Assessment: HP Chem Station for LC Systems Rev.A.05.01



#### Figure 4-23: DNPH sampling cartridge for aldehydes and ketones

Substances calibrated using this method are:

Formaldehyde, acetaldehyde, acetone, propanal, butanal, benzaldehyde, pentanal, hexanal, heptanal, octanal, nonanal, decanal, pentenal, hexenal, heptenal, octenal, nonenal, decenal, undecenal, dodecenal, cyclohexanone and crotonaldehyde.

The procedure described here can provide determination limits from 0.2 to  $1 \text{ ng }\mu\text{I}^{-1}$ . Determination limits smaller than  $1 \text{ ng }\mu\text{I}^{-1}$  refer to the rather short-chain aldehydes and ketones such as formaldehyde, acetaldehyde and acetone. The determination limits are indicated for standard solutions, they can be somewhat higher for actual samples. Assuming a sample volume of 60 litres of air, these determination limits correspond to concentrations of 1 to 5  $\mu\text{g m}^{-3}$ .

### 4.8 SENSORY-BASED AND ANALYTICAL INVESTIGATION OF BUILDING PRODUCTS

The investigations are described as per Chapter 2 "Objectives" in the following text.

#### 4.8.1 CHECKING THE SAMPLE CONTAINER

The Tedlar® sample container has already been used in the first research project. The ambient smell of the container could not be excluded in all cases since it was not possible to guarantee uniform handling of the container material. The parameters for preparation of the sample container were specified in a new bake-out system. New plastic foil materials were checked.

Five new materials listed in Table 4-2 were tested for their permeation properties in order to check the sample containers.

1	PE foil
2	Tedlar <sup>®</sup> TR 20 TG
3	Tedlar <sup>®</sup> TR 10 GB
4	Kapton <sup>®</sup> polyimide foil 250 FN
5	Polyethylene foil with an
	aluminium layer

#### Table 4-2:Foils tested

Figure 4-24 shows the schematic layout of the equipment used to determine the permeation behaviour of polymer foils under environmental conditions. It can be seen that the equipment is separated into two parts by the plastic foil to be tested. The substances were placed into part (1) of the equipment. This part is called "before the plastic foil" (1) in the following text. That part of the equipment where no substance was placed, is called "behind the plastic foil" (2). High-purity air was supplied to both parts of the equipment and was mixed in order to maintain steady-state conditions. The internal propellers established a uniform concentration on either side of the foil. Contaminated air was removed from both parts of the chamber. A measuring point was provided in both chambers for air sampling. In practice the equipment consisted of two equal 150 mm dia plane flange lids with the necessary connections and a longish cylinder whose ends were also provided with a plane flange. The materials used for this equipment were glass, stainless steel or Teflon®. Cleaned and degreased copper and stainless steel pipes were used for transporting high-purity air.



Figure 4-24: Layout of the experimental setup to investigate the permeation behaviour of polymer foils

Zuluft	High-purity air supply (2 x)
Reinstluft (2 x)	
Probenahme (2 x)	Sampling (2 x)
Durchmischung (2 x)	Mixing (2 x)
Abluft (2 x)	Contaminated air (2 x)
9 Substanzen	9 substances
Folie	Foil



Figure 4-25: Test equipment to investigate the permeation behaviour of polymer foils

Figure 4-25 shows the test equipment as set up. The equipment consisted of two plane flange lids, which were attached to a 300-mm-long and 150-mm dia glass cylinder. The plane flange lids were connected to the glass cylinder at either end by

quick-action fasteners making an air-tight seal. The plastic foil to be tested was clamped by one of the quick-action fasteners on the right side of the container, see Figure 4-25. The nine substances (Table 4-3) were loaded individually into small glass vials, and placed into the holder in front of the plastic foil. Capillaries of various inside diameter and length passing through the lids of the vials produced homogenous concentrations of the individual substances in the chamber section in front of the plastic foil according to the components' vapour pressure. The nine substances in Table 4-3 were specially selected and represented a reasonable range of volatility and are all substances which occur frequently in building materials and must be frequently transported in the sample containers.

### Table 4-3:Substances used to test the foils

Non-polar	Xylene, α-pinene, decane
Polar	Methyl butanone, pentanol, hexanal, butyl acetate, butoxyethanol, benzyl alcohol

There were four openings in each plane flange lid. Propellers were installed in the centre on both sides (see Figure 4-26), which were driven by a magnetic coupling to mix the air in the chamber. The air exchange rate and thus the supply of high-purity air into both chamber parts was adjusted by needle valves. The flow rate of the high-purity air could be checked by a flow meter.



### Figure 4-26: Plane flange lid with its four openings

Figure 4-26 illustrates a plane flange lid. The high-purity air supply can be seen on the top right in Figure 4-26 (1). The contaminated air was removed through an

opening in the plane flange lid, on the bottom right (2). Air sampling was performed at an outlet (3) in the lid which could be opened for sampling but was otherwise firmly closed. It is open in Figure 4-26. A TENAX® tube was passed through this opening into the equipment and a specified amount of sample drawn through the TENAX® tube. The sample flow rate must be less than the high-purity air flow rate into the respective chamber.

Table 4-4 shows the test conditions as adjusted in the equipment. The area of the plastic foil was 0.0177 m<sup>2</sup>. The flow rates before and behind the plastic foil were set to about 20 l/h which produced a relatively high concentration of the tested components in the air of about 1 mg/m<sup>3</sup> on the side that contained the substances. Air on the side behind the plastic foil was regularly checked during a period of about seven days by TENAX® tube sampling and analysed by a GC/MS system.

	Index	Chamber volume	Flow rate	Temperature	Rel. humidity
Chamber side with substances	1	7.5 litre	20 l/h	23C	45-50 %
Chamber side without substances	2	1 litre	20 l/h	23°C	45-50 %

 Table 4-4:
 Test conditions adjusted in the permeation equipment

In order to check the ambient smell of the sample containers, the cleaned containers were filled with clean air, e.g. equipment air (HRI) or air from non-contaminated 24-litre chambers (BAM) and, as with the other samples, were evaluated by panellists after three hours.

#### 4.8.2 DESIGNING A SIMPLIFIED COMPARATIVE SCALE

A new design (Figure 4-27) has been developed for a simplified comparative scale in the project. Since the comparative scale needed to be easy to use and to handle, it was operated without metering valves. A nozzle (similar to a Laval nozzle) ensured a supersonic speed for the gas (synthetic air) flow. If the pressure was constant before the nozzle, a constant mass flow could be maintained independently of the pressure drop across the nozzle. The gas was enriched with the reference substance (acetone) in a wash bottle at a constant temperature, thus the same acetone-air mixture could be produced all the times. Afterwards, the mass flow was distributed into six linearly graded acetone air-mixtures using specified orifice

gauges. All six acetone air mixtures were diluted again with air to obtain the desired concentrations (pi values) before flowing into the funnels.



#### Figure 4-27: Layout of a simplified comparative scale

# 4.8.3 COMPARISON OF SAMPLE PROVISION USING CHAMBERS AND SAMPLE CONTAINERS FOR DIRECT ASSESSMENT AT THE CLIMPAQ

The building products were placed into the chambers at HRI and BAM at the same time and the same area-specific flow rate q was used to ensure comparability of the measurement results. The measurement procedure is explained in Chapter 4.8 "Sensory-based and analytical investigation of building products".

CLIMPAQ type chambers and 24-litre emission chambers were also included in the investigations. The same building products were tested at HRI and at BAM using the CLIMPAQ and the 24-litre chambers. This was repeated for six building products. The building products stayed in the emission chambers for 29 days and were then analysed according to the AgBB scheme.

In this test series the sensory-based assessments were carried out in HRI's air quality laboratory. The assessment of the air samples from CLIMPAQ and BAM's 24-litre chamber were performed using sample containers described in Chapter 4.4 "Sampling and sample provision using Tedlar<sup>®</sup> sample containers".

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In addition to the tests in the chambers, the flow profile of the chambers was measured since some assessments exhibited differences. The flow profile in the CLIMPAQ and the 24-litre chamber was investigated using a Laser Doppler Anemometer (LDA). Based on the results, the flow in the CLIMPAQ was optimized by modifying the laminarisators as a potential source of the assessment differences.

### 4.8.4 DIRECT ASSESSMENT AT THE 5-M<sup>3</sup> CHAMBER

A flow rate of 0.7 to 0.9 l/s was necessary to provide a sufficient volume of sample air. Direct assessment of the exhaust air flow at the 1  $m^3$  emission chamber was not tested in the preceding research project. Therefore it had to be tested to see whether an increased air exchange rate in the chamber could be compensated for by a larger load or if smaller flow rates of the sample air would be sufficient for odour assessment.

BAM examined whether a panel using a comparative scale was able to perform a direct sensory-based assessment at the  $5\text{-m}^3$  chamber. An assessment at the  $1\text{-m}^3$  chamber was not possible since the actual chamber technically does not make it feasible to provide air conditioning in terms of humidity and temperature for flow rates greater than  $0.5 \text{ I s}^{-1}$ .

The aim of these investigations was to check which of the emission test chambers was also suitable for odour assessment or what changes had to be made in the chambers in order to make them fit for the assessments. In addition, to what extent the odour assessments performed in HRI's air quality laboratory were comparable to those carried out at BAM was checked. This enabled conclusions to be drawn about the effect of different environments.

Since BAM has no smell-neutral lounge, which is common to other laboratories, an alternative had to be sought. The requirement was for the panellists to stay in a room with low ambient emissions and a high flow rate of fresh air. Therefore the corridor was selected. Before entering the laboratory which contained the emission chamber, each panelist had the opportunity to go outside into the fresh air. Since the lounge was not directly adjacent to the laboratory, the panellists had to pass through two other rooms.

A stainless steel hose with a glass funnel on the end was attached to the  $5\text{-m}^3$  chamber (see Figure 4-8 in Chapter 4.3 "Emission test chambers"), thus direct evaluation of sample air by the panellists was possible at the  $5\text{-m}^3$  chamber. A flow rate of about 0.7 I s<sup>-1</sup> could be maintained at the assessment funnel. The comparative

scale was set up in direct proximity to the evaluation station in a fume hood (see Figure 4-28) which was only opened for the period when an evaluation by the panellist was taking place. The room was equipped with air conditioning and thus an increased air exchange rate.

Product PVC (No. 4170) was placed into the 5-m<sup>3</sup> chamber, BAM's CLIMPAQ, HRI's CLIMPAQ and HRI's 13-m<sup>3</sup> chamber with the same loading factor. The samples were evaluated on the fourth day after placing them into the chambers. No 29-day test took place. In addition to the sensory-based evaluation at the 5-m<sup>3</sup> chamber by panellists using a comparative scale, the building product was also evaluated in BAM's CLIMPAQ.



# Figure 4-28: Mobile comparative scale and 5-m3 chamber with assessment funnels

### 4.8.5 REQUIREMENTS FOR A MINIMUM PANEL SIZE

The requirement for a minimum panel size to ensure a specified selectivity between low-smell and smell-intensive building products was based on a statistical analysis of the measured values.

The data obtained from Chapter 4.8 "Sensory-based and analytical investigation of building products" were statistically analyzed in these investigations. A new experimental set-up was not necessary.

# 4.9 SENSORY-BASED AND ANALYTICAL TESTING OF BUILDING PRODUCTS IN A 13-M<sup>3</sup> CHAMBER

# 4.9.1 INVESTIGATION OF BUILDING PRODUCTS IN THE 13-M<sup>3</sup> CHAMBER AND IN A "REAL ROOM"

Two actual building product combinations were placed into the 13-m<sup>3</sup> chamber. The first combination consisted of a pre-mixed screed, a floor covering adhesive and a wall-to-wall carpet (see Figure 4-29). The second combination contained a PVC floor covering instead of the wall-to-wall carpet as a floor covering component. Air

exchange rates lower than 3  $h^{-1}$  are not feasible in the 13-m<sup>3</sup> chamber due to technical reasons. The floor structure is thus exposed to q = 7.24 m<sup>3</sup> m<sup>-2</sup> h<sup>-1</sup> in the 13-m<sup>3</sup> chamber.



# Figure 4-29: Investigating a combination of products in the 13-m3 chamber

The first combination of pre-mixed screed, floor covering adhesive and wall-towall carpet was also tested in BAM's CLIMPAQ and 24-litre chamber applying the same q=7.24 m<sup>3</sup> m<sup>-2</sup> h<sup>-1</sup> as in the 13-m<sup>3</sup> chamber. Individual building products (premixed screed, wall-to-wall carpet) and the combination were also tested at the same q in other HRI CLIMPAQs. The wall-to-wall carpet was additionally investigated in the CLIMPAQ at q = 1.7 m<sup>3</sup> m<sup>-2</sup> h<sup>-1</sup>. Floor covering adhesive 3978 was tested in this project at q = 2.0 m<sup>3</sup> m<sup>-2</sup> h<sup>-1</sup>. Furniture (a desk, two chairs and an empty bookshelf) were placed into the chamber for the test in the first combination.

The second combination and the PVC covering (No. 4101) were also tested in various chambers at  $q = 1.56 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ . There was no furniture in the chamber while testing the second combination.

In addition to the tests in the 13-m<sup>3</sup> chamber, another test was performed in a real room. A PVC covering was placed on an existing floor covering (carpet) without fixing it permanently in one of HRI's offices (see Figure 4-30). This office was

supplied with about  $10 \text{ I s}^{-1}$  ambient air from a central air conditioning unit which provided an area-specific air flow rate of about  $q = 2.9 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ . Air samples for assessing the perceived air quality were taken from the room with the help of AirProbes (see Figure 4-10 in Chapter 4.4 "Sampling and sample provision using Tedlar<sup>®</sup> sample containers") and provided to the panelist in HRI's laboratory.





# Figure 4-30: Left: real room at HRI with a wall-to-wall carpet, right: the same room with a PVC floor covering

#### 4.9.2 INVESTIGATING COMBINATIONS OF BUILDING PRODUCTS

This test was aimed at assessing combinations of building products using a sensory-based assessment. For this purpose building products with various odour intensities and hedonic intensities were used. The investigation took place in CLIMPAQs. A combination of two unpleasant building products and another comprising an unpleasant and a pleasant product were tested over 29 days. A combination of two pleasant building products could not be tested due to the lack of building products perceived as pleasant.

The combination was tested at the same q used in the individual assessments. The half of the necessary emitting surface of both building products, i.e. unpleasant carpet (4200) and pleasant PVC (4201), were used and placed into the chambers. The building products were newly purchased for this test, i.e. they did not come from batches of building product that had already been through the individual tests.

Again, the same  $q = 1.56 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$  was used as in the individual assessments. The unpleasant smelling carpet (4200; as from the previous combination) and the PVC floor covering (4199) were tested.

#### 4.10 SPECIFYING A COMPARATIVE SCALE

The data generated in Chapter 3.7 (Analytical measurements) were evaluated and used to establish a proposal for the definition of the threshold of reasonableness for the approval of building products and an evaluation method for the Blue Angel according to the objectives set out in Chapter 2 about the requirements for a comparative scale concerning perceived intensity. A new experimental set-up was not necessary.

# 5 RESULTS AND DISCUSSION

## 5.1 SENSORY-BASED AND ANALYTICAL TESTING OF BUILDING PRODUCTS

All building products tested in this project underwent sensory-based tests and most of them underwent analytical tests as well. Table 4-1 (Tested building products) provides an overview of the tests. The sensory-based assessment is directed towards the description and testing of sensory-based quantities discussed in Chapter 3.3 "Evaluation methods to determine perceived air quality". They were evaluated according to the methods described in Chapters 4.6.1 "Panel with comparative scale" and 4.6.2 "Panel without comparative scale". The individual results of each building product that underwent sensory-based tests can be found in the Annex of this report. Figure 5-1 to Figure 5-4 show examples of the individual assessments.

Perceived intensity and hedonics were determined by a panel using a comparative scale for every building product tested in this project. Acceptance and reasonableness however were not determined because the larger panel without a comparative scale necessary for every test could not be organized and the decision to use such a group was only made when the project was already running. An overview of the building products additionally evaluated by the panel without a comparative scale is attached to this report as Annex 3.

#### Individual assessment using the example of two wall-to-wall carpets:

The perceived intensity of each building product basically changes over 29 days. This however does not exclude individual products which may behave similarly. As an example, the different decay behaviour of two wall-to-wall carpets is described here. Figure 5-1 shows the perceived intensity of two wall-to-wall carpets, Nos. 4141 and 4074, over all measurement days. Linear trends were established for both wall-to-wall carpets in order to be able to observe the change more clearly. The example of the wall-to-wall carpet 4141 shows an assessment of perceived intensity as being constant over the time. On the other hand, wall-to-wall carpet 4074 exhibits a diminishing perceived intensity.

In addition, Figure 5-2 to Figure 5-4 show a comparison of hedonics, acceptance and reasonableness for both wall-to-wall carpets (4141 and 4074) as functions of time.



# Figure 5-1: Perceived intensity of wall-to-wall carpets 4141 and 4074 as a function of time

Figure 5-2 illustrates the hedonics of wall-to-wall carpets 4141 and 4074 as a function of time. As typical for building products (also see Figure 5-7), hedonics is negative over the entire test period and relatively constant over time for wall-to-wall carpet 4141. Wall-to-wall carpet 4074 indicates a light increase which means that the panelists always evaluated wall-to-wall carpet 4074 as slightly more pleasant over the test period of 29 days. There are only a few building products which received a positive hedonic assessment (see Annex or summary of all building products in Figure 5-5).





# Figure 5-2: Hedonics of wall-to-wall carpets 4141 and 4074 as a function of time

Acceptance and reasonableness were assessed by a panel without a comparative scale. These assessments did not take place on the same test days as the assessment with a comparative scale, but near to the planned test days three (two), eight (seven), fifteen (fourteen) and twenty-nine (twenty-eight) and, optionally, on day one (see further details in Chapter 4.1 "Test procedure").

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Figure 5-3 shows acceptance and Figure 5-4 reasonableness as functions of time for wall-to-wall carpets 4141 and 4074. Acceptance was evaluated negatively like hedonics, but it was found a little more acceptable around the end than at the beginning. Positive acceptance was achieved for wall-to-wall carpet 4074 around the end of test. Reasonableness of the odour from wall-to-wall carpet 4074 increased. While about 40% of the people questioned evaluated the sample as reasonable on the second day, around 70% did the test on day 30 and thereafter. Wall-to-wall carpet 4141 exhibited reasonableness around a relatively constant 45% over all test days.



Figure 5-3: Acceptance of wall-to-wall carpets 4141 and 4074 as a function of time





# Figure 5-4: Reasonableness of wall-to-wall carpets 4141 and 4074 as a function of time

#### Assessment of all tests:

The mean values of the panel's assessments were used for the diagrams in the following section showing the measurement values of all sensory-based assessments for single building products. Figure 5-5 shows the results of acceptance and perceived intensity (from the mean values of both panels) of all sensory-tested building products on all measurement days in HRI's CLIMPAQ.





# Figure 5-5: Relationship between perceived intensity and acceptance for building products (in CLIMPAQ) on all measurement days up to day 29

This figure shows that the panellists classified only a very few building products as acceptable in terms of odour. The floor covering adhesives were assessed as unacceptable even after 29 days (see Figure 5-6), although there are noticeable differences in this group as well. Since the floor covering adhesives – as all other samples – were tested separately and not in combination with any structure (e.g. floor covering), no negative conclusion can be drawn about the acceptance of the product group and its actual behaviour in a "real" room.

Chapter 5.1.7 "Investigation of building products in the 13-m<sup>3</sup> chamber and in a "real" room" describes some tests on building product combinations.





# Figure 5-6: Relationship between perceived intensity and acceptance for building products (in CLIMPAQ) on day 29

Figure 5-7 illustrates the relationship between perceived intensity and hedonics of the panel with a comparative scale for the tested building products on all measurement days.



# Figure 5-7: Relationship between perceived intensity and hedonics for building products on all measurement days up to day 29

Looking at the assessments of perceived intensity and hedonics, it is obvious that there is a range, typical for building products, in the intensity interval of about 3 pi to 16 pi and in the hedonics interval of about -3 to 1. The assessment on day 28 also shows this typical range (see Figure 5-8). A comparison of Figure 5-7 and Figure 5-9 clearly shows that it is a typical range. Figure 5-9 illustrates the results of the sensory-based assessments for the investigation of base odours<sup>\*</sup> [34]. Seven base odours were used following the proposal of Amoore 1964 [35] (see Table 5-1). The tests with base odours provided initial information about the relationships between the sensory-based quantities perceived intensity, acceptance and hedonics. The ether-like odours, their diluted versions and their mixtures indicate a noticeably larger scattering in the hedonics range over the same intensity range as in the assessment of the building products, which is primarily in the negative hedonics range. The quest for an odour which is assessed by the panelists as both intensive and positive at the same time led to a chocolate biscuit (see Figure 5-9).

<sup>\*</sup> There have been numerous trials to develop classification systems to subdivide the large number of odours into a manageable number of classes. Scientists have tried for decades to unambiguously specify the odour systems. Various approaches have been used so that both the number of odour classes and the type of the classes and their areas of application vary very strongly. None of the classification system developed have found general acceptance yet. One of the applied odour classification/base odours stems from Amoore 1964. This system is based on seven base odours, which were applied with minor changes in the tests on base odours at HRI.




Figure 5-8: Relationship between perceived intensity and hedonics for building products in CLIMPAQ on day 29

Table 5-1:	Characterization of odour classes (Amoore et al.
	in Schmidt [6])

Odour class	Chemical substance
Flowery	Phenylethyl-methyl-ethyl-carbinol
Ether-like	Ethylene dichloride
Musk deer-like	$\omega$ -hydroxy pentadecane acid lactone
Camphor-like	Camphor
Sweaty	Butyric acid
Putrid	Butyl mercaptan
Minty	Menthone



### Figure 5-9: Perceived intensity and hedonics for base odours and two other examples

In addition to the base odours, Figure 5-9 shows the assessment of an empty CLIMPAQ (intensity of about 1.5 pi and hedonics of about 1) and a foodstuff, i.e. chocolate biscuits (intensity of about 11.1 pi and hedonics of about 3.2). Building products failed to achieve a positive assessment similar to food, an empty chamber or pleasant base odours.

Figure 5-10 illustrates the sensory-based results of wood and timber materials (see the research project of the Federal Environment Agency [36]). Here the hedonics assessments are also in the negative range and the typical pattern of building products can be recognized. The increase in the typical pattern is less for wood products which means that the overall hedonics of wood and timber at the same intensity range is a little more positively assessed on all test days.







### Figure 5-10: Relationship between perceived intensity and hedonics for wood products [36] (in EXSI) over all test days up to day 29

The following text deals with the results of the individual product groups. Most of the products in Table 4-1 "Tested building products" were tested and assessed according to the specifications of emission tests for assessment according to the AgBB scheme and the relevant standards [4], [10], [11], [12]. All results of these tests are listed in Annex 3. An AgBB assessment was also performed for most of the test products whose result design corresponds to the ADAM assessment template of DIBt (ADAM: AgBB/DIBt assessment template) [37]. Wall-to-wall carpets No. 3974 and 4041, linoleum No. 4039 and floor covering adhesive No. 4040 were not analytically tested.

Typical emissions of the tested products or product groups will be illustrated in the following text. Product selection was focussed on textile floor coverings (wall-to-wall carpet) and flexible floor coverings (rubber, PVC and linoleum) as well as floor covering adhesives. Wall-to-wall carpets represent a product group which exhibits only a very low detectable emission in most cases within the range of a few  $\mu$ g m<sup>-3</sup> (see Table A-2 in the Annex). Nine wall-to-wall carpets were tested altogether, seven of them were also tested analytically (excluding 3974 and 4041), and all nine underwent sensory-based tests.

4-phenylcyclohexene (4-PCH) was detected in five out of seven carpets tested analytically. Figure 5-11 shows that the maximum detectable concentration of this substance after 29 days is 300  $\mu$ g m<sup>-3</sup> and the minimum detectable concentration 1  $\mu$ g m<sup>-3</sup>. To obtain the Blue Angel label, wall-to-wall carpets must not exceed a 4-PCH concentration of 5  $\mu$ g m<sup>-3</sup> after 28 days. Two carpets exhibited no detectable 4-PCH. One carpet contained considerably less after 28 days and another one, with 6  $\mu$ g m<sup>-3</sup>, just exceeded the threshold value. The other three carpets noticeably exceeded the threshold value for the environmental label.



# Figure 5-11: A: Concentration of 4-PCH as a function of time from five different textile floor coverings, B: Details for two low-emitting textile floor coverings

Figure 5-12 summarizes the sensory-based tests for wall-to-wall carpets. From amongst all carpets tested, carpets 3974 (4.8 pi) and 4074 (5.6 pi) exhibited the lowest perceived intensity after day 28. The highest perceived intensities were produced by carpets 3916 (10.1 pi), 3975 (8.6 pi), 3976 (8.3 pi) and 3951 (8.2 pi). If one compares the analytical and sensory-based results, it can be seen that carpet 4074 shows both a low 4-PCH concentration and a low perceived intensity after day 28. Carpet 3901 with 6  $\mu$ g m<sup>-3</sup> of 4-PCH concentration has an intensity of 7.1 pi. Those carpets which show very high 4-PCH concentrations after 28 days have higher perceived intensities as well: 4141 (7.6 pi), 3975 (8.6 pi) and 3976 (8.3 pi). Wall-to-wall carpets in which no 4-PCH concentrations were detected (3951 and 3916) likewise have high intensities after 28 days (8.2 pi and 10.1 pi). If the sensory-based limits for the Blue Angel (suggested in Chapter 5.2.3 "Establishing an evaluation method for the blue angel", Figure 5-53)) and the chemical analysis limits for awarding the environmental label were valid, only carpet 4074 and carpet 3901 (with 7.1 pi and 6  $\mu$ g m<sup>-3</sup>) would just qualify for the Blue Angel. Those carpets which did

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not undergo chemical tests (3974 and 4041) likewise have intensities under 7 pi and thus would fall below the limits for Blue Angel in terms of odour assessment.



#### Figure 5-12: Perceived intensity of floor coverings: results of sensorybased tests on various days of test

Numerous elastic floor coverings were tested in this study. Most of these products were PVC coverings since they often have a very distinctive, strong odour. Several of these products were simple goods from the builders merchants. Emissions from the PVC floor coverings do not contain any specific single compounds, like carpets, therefore TVOC and, for some components,  $\Sigma$ SVOC are illustrated in Figure 5-13 In the case of PVC 3900 especially, emissions are not exactly specified, rather they appear to be semi-volatile alkanes within the range of C<sub>14</sub> to C<sub>17</sub>, this is why the concentrations remain essentially constant during a long period of time as indicated in Figure 5-13. Products 4161 and 4162 are two various batches of the same PVC floor covering. A sample from each roll was tested in a 24-litre chamber. The emissions show a very good agreement. This comparable test was performed because [37] found considerable inhomogeneity in this floor covering. This project examined whether inhomogeneity in the same product leads to different emission behaviour.



Figure 5-13: TVOC concentration as a function of time for five different PVC floor coverings and SVOC cumulative values for three floor coverings (Diagram B shows details of some data from A)

Figure 5-14 shows an example of decay behaviour<sup>3</sup> in two substances as well as all unknown and non-specifiable VOCs and proves that there is a good agreement between the respective emissions and concentrations of both floor coverings. Furthermore, this comparison indicates that the investigations furnish very reliable values which are necessary for a fundamental emission assessment required for the approval or refusal of a product.

 $<sup>^{3}</sup>$  VOC tests for samples 4161 und 4162 were carried out up to day 24.



Figure 5-14: Concentration of various VOC from PVC floor coverings 4161 and 4162 over 24 days

Figure 5-15 illustrates the perceived intensities of sensory-based tests for PVC floor coverings. The perceived intensity is between 2.5 and 8 pi, while the assessments remain relatively constant over the test period. On day 28, three products (4015, 4101 and 4161) exhibited a perceived intensity of less than 6.5 pi. Products 3900 and 4199 showed 8 pi. Product 3900 had the highest TVOC concentration after day 28 and reached an intensity of 8.7 pi. In addition to the lowest intensity, i.e. 4.3 pi, PVC floor covering 4015 also had the lowest TVOC concentration from amongst all tested PVC floor coverings after day 28. In view of the sensory (Figure 5-53) and analytic limits for awarding the Blue Angel, PVC coverings 4015 and 4161 would qualify. Product No. 4101 only fulfils the requirements of the sensory-based limits, but not those of emissions. PVC coverings, however, are excluded from consideration for the environmental label because of their phthalate and chlorine-organic compound contents.



#### Figure 5-15: Perceived intensity of PVC floor coverings

Only a few tests were carried out on PVC floor covering 4004 at HRI's CLIMPAQ. This product exhibited very high diisobutyl phthalate (DiBP) emissions which were as high as 100 µg m<sup>-3</sup> even after 29 days. The behaviour of this SVOC was investigated in great detail within a masters thesis at BAM. Numerous repeat tests proved that the chamber concentrations of DiBP remain unchanged over a long period of time and considerable sink effects were observed and proved. A paper on the key results of phthalate emissions researched in this masters thesis was published in a journal [38]. PVC floor covering 4004 was evaluated by panelists with a comparative scale on days 13 and 27. The perceived intensity was 8.4 pi on day 27.



Figure 5-16: DiBP concentration from PVC floor covering 4004 as a function of time. 24-litre chamber test [38]

Rubber and linoleum floor coverings represent another major group of floor coverings which excelled in this project with mostly very low concentrations. One linoleum and three rubber floor coverings underwent both analytical and sensory-based tests. Both the linoleum and two of the three rubber floor coverings would meet the environmental label criteria. Rubber No. 3915 which failed the AgBB assessment stems from a batch which was considered critical in another investigation about five years ago, and this finding has now been proved in this study. Typical emissions from rubber such as benzothiazole were only detected in concentrations below 35  $\mu$ g m<sup>-3</sup> after 29 days. Although there is no LCI value available for benzothiazole and thus the 100  $\mu$ g/m<sup>3</sup> limit of the components without LCI is valid, the common emissions from rubber floor coverings do not have a negative effect on the assessment under AgBB.

With the exception of floor covering No. 3948 with 5.7 pi, rubber and linoleum floor coverings were between 8 and 11.2 pi and would not meet the Blue Angel

sensory-based prerequisites (see Figure 5-17). Rubber floor covering No. 3915, which would fail AgBB as has already been mentioned, exhibited 11.2 pi, i.e. one of the highest perceived intensities.



Figure 5-17: Perceived intensity of rubber floor coverings



Figure 5-18: Perceived intensity of linoleum floor coverings

An oak wood parquet was tested as a further floor covering, which exhibited an acetic acid emission higher than 100  $\mu$ g m<sup>-3</sup> after 29 days which is relatively high,

otherwise it emits hardly any other compounds apart from some aldehydes. It is surprising that despite acetic acid and aldehyde concentrations, odour assessment of the parquet was rather positive (see Figure 5-19).



#### Figure 5-19: Perceived intensity of parquet 4159

Numerous floor covering adhesives were tested in the project. This product group has the highest emission rates compared with other building products, as indicated by the respective sum values of the identified VOC (TVOC) in Figure 5-20. Only two of the floor covering adhesives achieved values under 1000 µg m<sup>-3</sup> by the third day and attained concentrations around 200 µg m<sup>-3</sup> after 29 days. Even these figures are still too high to qualify for the environmental label for floor covering adhesives. Components such as acetic acid or glycol ester are responsible for these high levels, are analytically accompanied with high measurement uncertainty and are more difficult to detect than other substances. Only one building adhesive (No. 4290/cartridge goods) meets the emission criteria of the environmental label. This adhesive, however, does not fall into the Blue Angel's sphere of legitimacy.



Figure 5-20: TVOC concentration as a function of time for floor covering adhesives tested in the project (Diagram B is an enlargement of an excerpt from A)



Figure 5-21: Perceived intensity of floor covering adhesives

Figure 5-21 shows the results for perceived intensity of the sensory-based assessments by panelists with a comparative scale. Only one of the floor covering adhesives, i.e. No. 3978, with an intensity of 7.1 pi and hedonics of - 1.3 is somewhat

below the proposed limits of the sensory-based criteria (Figure 5-53) for the environmental label. All other floor covering adhesives exhibit intensities in excess of 8 pi after day 28 and thus exceed the proposed limit of 7 pi.

Figure 5-22 shows the concentration of a few more typical components emitted from floor covering adhesives. For instance methylisothiazolinone (MIT) is a frequently used in-can preservative for floor covering adhesives. This prevents waterbased products from becoming unserviceable over time by fungal or bacterial growth in the packaging. MIT emissions from a floor covering adhesive were detected in concentrations of up to 40  $\mu$ g m<sup>-3</sup> for up to 29 days after spreading on the glass plate.



Figure 5-22: Concentration of some typical VOC as a function of time from floor covering adhesives

#### 5.1.1 CHECKING THE SAMPLE CONTAINER

The Tedlar® sample container had already been used in the previous research project. However, the natural smell of the container could not be safely excluded in all cases since it was not possible to guarantee a uniform handling of the container material. However, after HRI developed a new baking cabinet, the sample containers had to be tested for their natural smell and reusability in this project under the new baking procedure using both sensory-based and analytical methods. In addition, the permeation characteristics of different plastic foils (Table 4-2) had to be checked for their suitability as sample containers. The set-up for permeation tests has already been described in Chapter 4.8.1 "Checking the sample container" in detail.

Comparative and trial measurements of the natural smell of the Tedlar® containers indicate a low odour intensity of 1 to 2 pi and an almost neutral hedonic assessment of 0.1. Odour intensity and the hedonic assessment of the air supplied from the air conditioning (supplied air) indicated almost identical values directly at the funnel (Figure 5-23). Figure 5-23 illustrates smell assessments of the cleaned Tedlar® containers which were filled with air from the empty chamber, and as a comparison, the assessment of the supplied air and the empty CLIMPAQ. This supplied air was used within the CLIMPAQ and the air quality laboratory in HRI. Sample air in the sample container is denoted here by "bag". Fresh air means that a sample of air was drawn directly from the ambient air.



Figure 5-23: Sensory-based assessment of blank values

Air in the containers and in the chambers was also compared for VOC emissions and most components showed a high degree of agreement. Emissions of almost all components were low and considerably less than 50  $\mu$ g/m<sup>3</sup>. Thus only a few tests could be evaluated for a secure comparison since concentration uncertainties were around 10  $\mu$ g/m<sup>3</sup> for most VOC and thus exceeded the 50% deviation. Therefore utmost care should be exercised in considering concentrations as unambiguous in the range of a few  $\mu$ g/m<sup>3</sup> to about 20  $\mu$ g/m<sup>3</sup>. Above this order of magnitude, about a 20% deviation was found in the concentration between the different laboratory emission tests in interlaboratory comparisons [39]. In view of this deviation, one can say there is good agreement between the concentrations in this range. The maximum deviation between air from the containers and air from the chambers is between 10 and 20%. The component methylisothiazolinone alone exhibited noticeable deficits in the containers, but even semi-volatile components such as 4-phenylcyclohexane were detected in the respective containers at very high recovery rates. Figure 5-24 shows an example for a normalized comparison of air in the transport container (TrC) to the respective chamber air. The mean value comes from the CLIMPAQ and the 24-litre chamber 1 and the other values were accordingly converted. As can be seen, the chamber emissions correspond to each other quite well, but in particular, air in the relevant transport containers matches the respective chamber values very well.



Figure 5-24: Normalized comparison (the mean value of the two chambers equals 1) of the emissions of the chambers compared to the emissions from the transport containers. (TrC: transport container 24-litre chamber; TrCC: transport container CLIMPAQ; CIB: CLIMPAQ BAM)

The results of the test on new foils are shown in the chromatograms Figure 5-25 and Figure 5-26. The black lines indicate the chemical substances where the samples are (i.e. before the foil (1); see Figure 4-24 in Chapter 4.8.1 "Checking the sample container"). The green line indicates those components which permeate through the foil (behind the plastic foil (2) see Figure 4-24 in Chapter 4.8.1). Apart from the simple PE foil (Figure 5-25), no permeation was detected through any of the foils during an exposure of at least one week. The two following figures (Figure 5-25)

and Figure 5-26) show the break-through behaviour for PE foil and, on the other hand, the polyimid foil as an example for the foils which do not show any break-through. With the exception of the PE foil, all other tested foils can be used as transport foils. Further tests on the natural smell should be performed before use.



Figure 5-25: Permeation behaviour of the PE foil (GC/MS chromatogram)



### Figure 5-26: Permeation behaviour of polyimide foil (GC/MS chromatogram)

The results indicate that the Tedlar® containers used are suitable as sample containers when the baking procedure described in Chapter 4.4 is applied and can

be used in future. It can be assumed that almost no substances will permeate through the foils even during longer storage (e.g. for transportation) of sample air in the containers, and the chemical composition will not change.

The natural smell of the reservoirs is so low that no difference can be detected in comparison to sensory-based assessment of supplied air.

#### 5.1.2 DESIGNING A SIMPLIFIED COMPARATIVE SCALE

As described in Chapter 3.3.1 "Perceived intensity", perceived intensity is determined by panellists using a comparative scale. Acetone-air mixture has been dosed by metering valves in the comparative scales used so far. The adjustment of the metering valves before each test is very time-consuming. In this project a comparative scale has been developed which is easy to commission and handle and does not include metering valves. For this purpose a comparative scale has been developed in which a nozzle (similar to the de Laval nozzle) ensures a constant dosage of the acetone-air mixture. Thus the time-consuming adjustment of the metering valves can be omitted.

A simplified mobile comparative scale has been developed (see Figure 5-27) and tested (see Chapter 4.8.2 "Designing a simplified comparative scale"). Measurements of the flow rates at the individual funnels show that the flow rate distribution exhibited a minor inhomogeneity. An adjustable fan helped improve homogeneity, but failed to provide complete consistency. An accurate adjustment of the acetone air mixture was therefore not possible. Measurements indicated that an aerodynamically improved air distribution would enable an accurate adjustment. Further solutions were sought and are being planned, for example the improvement of laminarisators in the distributor box. The development could not be completed within the project, but it is being further pursued and will be finished shortly.



#### Figure 5-27: Photograph of the simplified comparative scale

#### 5.1.3 COMPARISON OF SAMPLE PROVISION USING A CHAMBER AND SAMPLE CONTAINER FOR DIRECT ASSESSMENT AT THE CLIMPAQ

In this part of the project sensory-based and analytical assessment of building product emissions was tested for its reproducibility in different emission chambers and at different places. Altogether six building products were tested: No. 3950, No. 3976, No. 3978, No. 4015, No. 4026 and No. 4101. The building products were placed into the chambers at similar times in HRI (CLIMPAQ) and BAM (24-litre chamber and CLIMPAQ). The same surface-specific flow rate q was applied to ensure comparability of test results. The sensory-based assessment was performed in HRI's air quality laboratory. This means that the assessment of air samples from BAM's CLIMPAQ and BAM's 24-litre chamber was carried out using sample containers as described in Chapter 4.4 "Sampling and sample provision using Tedlar® sample containers". The building products remained in the emission chambers for 29 days and were also analytically tested according to the AgBB scheme.

The tests have already been described in Chapter 4.8.3 "Comparison of sample provision using chambers and sample containers for direct assessment at the CLIMPAQ".

Figure 5-28 and Figure 5-29 show the results obtained on a joint seal (No. 4026) and a PVC floor covering (No. 4101) over 29 days. The deviations of perceived intensity on the sampling days are about 2 pi, except for the test on the joint seal on day 15. On day 29 the results show a very good agreement. Floor covering adhesive 3978 exhibits similar results. The difference in the assessment of perceived intensity for the other three building products was approx. 4 pi on day 29.



Figure 5-28: Comparison of the results of intensity tests in different emission chambers (joint seal No. 4026)



Figure 5-29: Comparison of the results of intensity tests in different emission chambers (PVC floor covering No. 4101)



Figure 5-30: Comparison of ethanediol concentrations and up-down bars in different emission chambers (joint seal No. 4026) on days 15 and 29 for multiple tests (TrC: transport container; 24LC: 24-litre chamber; CIH: CLIMPAQ HRI; CIB: CLIMPAQ BAM)

Chemical comparison tests are only available for the joint seal on day 29. They are illustrated in Figure 5-30 for BAM's two chambers and HRI's CLIMPAQ using the example of ethanediol, being the main emission from a joint seal. A very good

agreement of the values can be seen, at least for day 29. No measurements are available for comparison with HRI's values for PVC floor coverings.

In order to obtain more information about the deviations in the assessment of perceived intensities from different emission chambers using Tedlar containers®, the repeatability of building product assessment was tested by repeating the assessment by the panellists of some of the building products (random samples) on the same day. Figure 5-31 shows the results of this test. It indicates that the repeat assessments of perceived intensity deviate from each other in the interval of approx. 0.2 to 1.6 pi which is within the limits of accuracy.



### Figure 5-31: Repeatability of the assessment of three individual building products and two combinations

In addition to checking the repeatability of an assessment, the extent to which an assessment of perceived intensity of emission air coming directly from CLIMPAQ deviates from one provided to the panellists from a Tedlar container® was also checked. In this test the panellists had to determine the perceived intensity of selected building products (two floor covering adhesives) twice on the same day. The first emission air sample came directly from the CLIMPAQ and then the same air was presented to the panellist from a Tedlar container®. The Tedlar container® was filled

with emission air from the CLIMPAQ directly before the tests. Figure 5-32 shows the results of this test which indicates that in the case of product 4061, on which this test was performed on two different days, intensity differences of up to 3 pi were found. In both cases emission air from the CLIMPAQ was assessed as more intensive than that from the Tedlar containers<sup>®</sup>. In the case of product No. 3978, the intensities agreed well with only minor deviations.



Figure 5-32: Comparison between assessments from CLIMPAQ and Tedlar containers®



Figure 5-33: Comparison between assessments from CLIMPAQ and Tedlar containers® for product PVC 4170

The above tests were carried out by randomly selecting days and products. Figure 5-33 shows an example of product 4170 (PVC, the same as 4199, but from a different batch). A comparison of the assessments from the sample container and the direct assessment from the CLIMPAQ indicates that the fluctuations of the assessments are within the range of accuracy for the assessment of perceived intensity. But there are greater differences between BAM's chambers and those of HRI. It can clearly be seen from the chemical analyses (Figure 5-38) that the concentrations of unknown VOCs have high values of up to about 15,000  $\mu$ g m<sup>-3</sup>. Differences of up to 3,000 – 4,000  $\mu$ g m<sup>-3</sup> were measured in the various chambers on the test days. In the case of smell-active substances this can lead to significant differences in the perceived intensity.

The differences of perceived intensities in the tests over 29 days can also have been influenced by other factors. For example the layout of the *building products*, product differences (inhomogeneity), flow pattern in the chambers or even the processing of supply air in the chambers may have had an effect. Processing fresh air for the supply to the emission chambers is different at the two institutes (BAM and HRI). In addition, other flow patterns prevail in the 24-litre chamber than in the CLIMPAQ. In order to obtain more information about the flow and the dominant

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velocities in the emission chambers, the flow profile was measured in the CLIMPAQ and in the 24-litre chamber with the help of a Laser Doppler Anemometer (LDA).

The differences discussed so far can only arise through a combined effect of all parameters since no assessment version can permanently provide higher values than another.

ISO 16000-9 requires chamber flow velocities to be in the range of 0.1 to 0.3 m/s. The results of the tests in the 24-litre chamber indicate that these velocities were indeed achieved (Figure 5-34). The results in the CLIMPAQ however clearly show that the flow is inhomogeneous and that the velocities are in the range of 0 to 0.4 m/s (Figure 5-35).



Figure 5-34: Flow profile in the 24-litre chamber

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### Figure 5-35: Flow profile in the CLIMPAQ at a distance of 50 cm and 100 cm from the laminarisator

Based on this finding, work is ongoing to optimize the laminarisator and develop a better version of the CLIMPAQ with an improved flow pattern.

The flow pattern in the CLIMPAQ can be improved by placing a laminarisator of different shape into the entry section. The measurement results indicate very uniform flow profiles after this first optimization measure was taken since the laminarisator distributes the supply air very uniformly. It can be seen that the velocities are within the range of 0.09 to 0.1 m s<sup>-1</sup> in the optimized CLIMPAQ (Figure 5-36). These values were measured in the empty chamber. If the CLIMPAQ is loaded, the velocity in the chamber increases and the ISO 16000-9 requirements can be met.

Six of these optimized CLIMPAQs were built at HRI and used for further tests on building products.

Even if the differences could not be fully clarified, the improved CLIMPAQs provided much better agreement with the 24-litre emission chamber.



Figure 5-36: Flow profile in the CLIMPAQ at distances of 50 cm and 100 cm from the optimized laminarisator

#### 5.1.4 DIRECT ASSESSMENT AT THE 5-M<sup>3</sup> CHAMBER

The feasibility of a direct sensory-based assessment by panellists using a comparative scale at BAM's 5-m<sup>3</sup> emission chamber was tested in this project. Also, the comparability of smell assessments in HRI's air quality laboratory with those performed directly at BAM was checked. HRI's air quality laboratory was provided with a smell-neutral lounge for the panellists and a smell-neutral assessment cabin. BAM's laboratories do not have these smell-neutral conditions for the panellists (see Chapter 4.8.4 "Direct assessment at the 5-m<sup>3</sup> chamber"). This test enables conclusions to be drawn about the effect of various environments. Sensory-based and analytic investigations took place directly at the 5-m<sup>3</sup> emission chamber and at the CLIMPAQ in BAM's laboratory and at the same time at the CLIMPAQ and the 13m<sup>3</sup> chamber in HRI's air quality laboratory. A building product (No. 4170) was placed into the aforementioned emission chambers with the same area-specific flow rate q applied. This investigation did not last for the full 28 days - the panellists carried out the sensory-based assessment only on day 6 after the product had been placed in the chamber. Product 4170 is a PVC floor covering which was only used for this investigation. However, the tests found that this PVC floor covering was very unpleasant, therefore another batch was bought and tested for 29 days as product No. 4199 and as a component of product combinations.

In order to decide whether the environment (in this case BAM's laboratory) and its odour intensity could have an effect on the assessment by the panellists in BAM's laboratory, the panellists also assessed the perceived intensity of the laboratory in which the 5-m<sup>3</sup> chamber was set up. The perceived odour intensity of the laboratory determined by the panellists was 3.7 pi.

Figure 5-37 illustrates a comparison of perceived intensities of the PVC floor covering (No. 4170) in the 5-m<sup>3</sup> chamber and other emission chambers. The deviation of the intensities of the direct sensory-based assessment at the emission chambers is in the range of about 2 pi. The intensity determined at the 5-m<sup>3</sup> chamber is the highest among all assessments.

The small deviation in the assessment of the same building product in different emission chambers and laboratories indicates that the sensory-based assessment with panellists is also feasible in laboratories which are not equipped with the special configuration of HRI's air quality laboratory.



Figure 5-37: Comparison of perceived intensity tests on a PVC floor covering (No. 4170) in different emission chambers using direct assessment

Similar to different basic products of this type floor covering, the VOC emission spectrum of PVC floor covering No. 4170 has high emissions of non-specific alkanes within the retention range of  $C_{12}$  to  $C_{14}$ . Figure 5-38 illustrates a comparison of the concentrations of non-identifiable VOC (as total unknown VOC) in various chambers. The tests indicate good agreement of VOC emissions between the chambers. The odour components of these VOC mixtures cannot be analytically identified accurately since they are contained in the non-specified amount of VOC and decrease over time similar to inconspicuous substances in terms of odour.

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Figure 5-38: Total concentration of unknown VOC (Σ VOC). Measurements simultaneous to direct sensory-based assessments of PVC 4170 at different chamber types

#### 5.1.5 REQUIREMENTS FOR A MINIMUM PANEL SIZE

Sensory quantities, which frequently play a role when determining air quality, include acceptance, hedonics and perceived intensity. Panel size is one of the variables in application which is required for determining the stated sensory quantities for statistical reasons. Thus a larger panel is used for determining acceptance while a smaller group is sufficient when the panellists determine the perceived intensity using a comparative scale. Statistics and costs of panel size play a key role in determining the most suitable sensory-based quantity, which will later represent the limits of sensory-based testing of building products.

Figure 5-39 shows the standard deviations related to the scale sizes of the three sensory quantities (perceived intensity, hedonics and acceptance) for a panel size of 11 people. The calculation of the mean value of the standard deviation for the three sensory-based quantities was exclusively based on those data where 11 panellists participated in the tests. It is clear that perceived intensity has the smallest standard deviation and acceptance exhibits the largest one. This comparison shows the dispersion magnitude and thus the reliability of measurement data from sensory-based quantities when the panel consists of 11 persons.



### Figure 5-39: Standard deviation of sensory-based quantities for a panel of 11 panellists

Allowing for a deviation of 10% of the actual scale size and a confidence interval of 90% for the sensory-based quantities, a panel size of at least eight people is required to determine the perceived intensity (with the smallest standard deviation). However, over 50 panellists are needed for determining the acceptance under the same conditions.

The evaluation of the data from the panel with a comparative scale related to the panel size and a confidence interval of 90% is shown in Figure 5-40. It is clear from the diagram that the evaluations of the perceived intensity are predominantly within an interval from 0 to + 2 from a panel size of eight people upwards, while it lies between 0 and 0.8 - 1 for hedonics (see Figure 5-41).



Figure 5-40: Number of panellists and 90 % confidence interval from the evaluation of perceived intensity



Figure 5-41: Number of panellists and 90 % confidence interval from the evaluation of hedonics by panellists using a comparative scale

#### 5.1.6 SENSORY-BASED AND ANALYTICAL INVESTIGATIONS OF BUILDING PRODUCTS IN THE 13-M<sup>3</sup> CHAMBER

A number of sensory-based and analytical tests were focused on the comparison of individual building materials and their combination in a real structure in the 13-m<sup>3</sup> chamber.

## 5.1.7 INVESTIGATION OF BUILDING PRODUCTS IN THE 13-M<sup>3</sup> CHAMBER AND IN A "REAL" ROOM

As already described in Chapter 4.9.1 "Investigation of building products in the 13-m<sup>3</sup> chamber and in a "real room", two real floors were laid in the 13-m<sup>3</sup> chamber. The first was a real floor consisting of a pre-mixed screed, a floor covering adhesive and a wall-to-wall carpet. The second likewise consisted of a dry screed and a floor covering adhesive, but the third layer was a PVC floor covering. These combinations (actual construction) were also placed into emission chambers. HRI's CLIMPAQ, BAM's 24-litre chamber and BAM's CLIMPAQ were also used here. All these building product combinations had already been investigated separately in earlier tests within this project. These investigations provided the first indication of whether the evaluation results from individual building products can be replicated by an evaluation in combination.

Figure 5-42 displays the results of the combination of a pre-mixed screed, floor covering adhesive and wall-to-wall carpet in a real floor in the  $13 \text{-m}^3$  chamber, compared to the same construction at the same loading (q =  $7.4 \text{ m}^3 \text{m}^{-2} \text{h}^{-1}$ ) in HRI's CLIMPAQ and BAM's CLIMPAQ and desiccator. The results for the individual building products used in the combination are also shown in this figure.

From the test on individual building products, floor covering adhesive No. 3978 (q =  $2.0 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ ) had an intensity of 12.5 pi after 29 days. This is the highest intensity of the tested building products of this combination. Wall-to-wall carpet No. 4074 (q =  $1.7 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ ) reached a perceived intensity of 5.6 pi. The same carpet at q =  $7.4 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$  (not shown in the figure) was evaluated to produce 2.3 pi on day 29. Pre-mixed screed No. 4073 (q =  $7.4 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ ) had a very low intensity from the 3rd day of the test onwards. The test was terminated on day 13 and the result of this day (4.6 pi) is indicated in the figure.

A comparison of intensities of individual building products with those of combinations shows that, although emissions from the adhesive are the most

intensive in the individual test, its intensity is not reflected in the combination. This may be explained by the fact that the floor covering adhesive is applied under the floor covering within the construction and therefore there is no direct contact with the room air. Contact with the screed may also have an effect. This however, does not mean that emissions from the floor covering adhesive are completely absorbed by the wall-to-wall carpet or screed. It is just an example showing that the results of tests on real combinations may be different to the results of individual building products. In the latter case the odours are mixed and the perceived intensity of the combination of building products is less than that of the most intensive individual product. What should be considered are the reactions which may occur between adhesives and floor coverings. This issue is however beyond the scope of this project.

It is important to emphasise that the q applied to the combination was different to the individual tests on the wall-to-wall carpet and floor covering adhesive. The intensity of the combination in other chambers was between 5 and 6 pi, which matches the intensity of the wall-to-wall carpet at  $q = 1.7 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ . The combination set up in a room with furniture (13-m<sup>3</sup> chamber with wallpapered walls) had an intensity of 9.7 pi on day 29 and only 6.8 pi on day 31.





Figure 5-42: Test results for a combination of building products (premixed screed, floor covering adhesive, wall-to-wall carpet)

The second combination (pre-mixed screed, floor covering adhesive and PVC floor covering) was also tested in the 13-m<sup>3</sup> chamber (q = 7.4 m<sup>3</sup> m<sup>-2</sup> h<sup>-1</sup>). This time no furniture was placed into the chamber after the floor had been completed. This combination was also tested in HRI's CLIMPAQ (q =  $1.56 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ ) and BAM's 24-liter chamber (q =  $1.56 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ ). Only PVC No. 4101 was separately tested from among the individual building products in HRI's CLIMPAQ (q =  $1.56 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ ) since the other individual building products had already been tested. The results of the combination and the comparison with other chambers and individual building products are shown in Figure 5-43. They indicate that the intensity of the real construction in the  $13\text{-m}^3$  chamber is very similar to the intensity of PVC in the individual test, even if q differs. Similar results were obtained from a comparison of the various chambers.



Figure 5-43: Test results for a combination of building products (premixed screed, floor covering adhesive, PVC)

The emission potential of these combinations was not high enough to perform a comparison of chemical emissions. Those few unambiguously detectable components were mostly present in concentrations much less than 50  $\mu$ g m<sup>-3</sup>. At this concentration range, common measurement errors for the comparison of completely different chambers are typically around 50% even in interlaboratory comparisons [39]. Our tests also provided similar values. Thus the direct comparison often leads to ambiguous representations which are difficult to interpret.

Apart from the tests in the 13-m<sup>3</sup> chambers, a test was also performed in one of HRI's physical offices, as described in Chapter 4.9.1 "Investigation of building products in the 13-m<sup>3</sup> chamber and in a "real room". A PVC floor covering (No. 4199) was installed into one of HRI's furnished offices – without fixing the floor covering with adhesive. Analytical and sensory-based tests were carried out on the same building product in a CLIMPAQ. The quantity of PVC floor covering placed into the CLIMPAQ did not correspond to the amount of material in the physical room. The aim was to obtain preliminary information on whether and to what extent chamber tests can be transferred to a physical (also furnished) room i.e. investigations of a building product area-specific flow rate in emission at an q an
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#### chamber.

Figure 5-44 shows the results of this test. The intensity of office air with the floor covering and furniture having been in place (before installing the PVC floor covering) was evaluated to be 3.6 pi. The intensity rose to 4.7 pi over 28 days after having installed the PVC. The perceived intensity of PVC floor covering No. 4199, which was tested in HRI's CLIMPAQ, was 9.1 pi. Hedonics, on the right in Figure 5-44, deteriorates noticeably from about zero to -1.5 after installing the PVC floor covering into the room. It can be seen that the results of the chamber tests in this example do not correspond directly to the results of the physical room into which the building product was placed. This, however, was to be expected in a fully furnished room in which only the floor covering was replaced. In order to be able to better understand the transfer of results from the chamber tests onto a physical room, further investigations are necessary.

The comparison of the chemical analysis for PVC floor covering No. 4199 in HRI's CLIMPAQ and HRI's office room can be seen in Figure 5-48 in Chapter 5.1.8 "Investigation of combinations of building products".



Figure 5-44: Comparison of perceived intensity and hedonics in a real office

#### 5.1.8 INVESTIGATION OF COMBINATIONS OF BUILDING PRODUCTS

Combinations of two unpleasant building products and one unpleasant building product with a pleasant (low-odour) one were tested over 29 days. The building products were selected for this test from data (primarily hedonics) obtained earlier from product tests. The combination was tested by applying the same area-specific flow rate q as in the individual tests. Half of the required emitting surface of each building product, in this case unpleasant carpet No. 4200 and pleasant PVC No. 4201 was tested. Building products were freshly purchased for this test, i.e. it was not the same batch of building products which had already been tested individually. The combination of two unpleasant building products was also tested applying q =  $1.56 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$  as in the individual tests. The unpleasant building products carpet No. 4200 (as in the previous combination) and PVC floor covering No. 4199 were used.

As described in Chapter 4.9.2 "Investigating combinations of building products ", CLIMPAQs were used for testing combinations of building products.

Figure 5-45 illustrates the perceived intensities and hedonics of a combination of two unpleasant building products after 28 days. The wall-to-wall carpet and the PVC floor covering were evaluated as unpleasant in the individual tests. The PVC floor covering exhibited greater intensity than the wall-to-wall carpet. The intensity of the combination is between the intensity of the individual building products. Hedonics is negative and similar to the hedonics evaluation of the wall-to-wall carpet.



# Figure 5-45: Perceived intensity and hedonics of a combination of two building products evaluated as unpleasant

Figure 5-46 shows the perceived intensities and hedonics of a combination of one building product evaluated as unpleasant and another evaluated as pleasant. The intensity of the combination is less than the intensities of the individual values of the building products and hedonics is positive. The reason for this may be that only half of the materials were used in the tests, as indicated in Figure 5-46.



Figure 5-46: Perceived intensity and hedonics of a combination of one building product evaluated as unpleasant and another evaluated as pleasant

For the chemical analytic comparison of the combinations No. 4199 & No. 4200 and No. 4200 & No. 4201 only TVOC values were compared within the current investigation. This seems to be reasonable as the chromatogram of the respective

chamber tests consists of rather non-specific alkane peaks, as shown in Figure 5-47. It can be assumed that the individual components from this mix contribute to a noticeable odour of the floor covering without being able to be unmistakeably identified.



Figure 5-47: Chromatogram of PVC floor covering No. 4199 in HRI's CLIMPAQ on day 29

The TVOC evaluations using the calculation based on the toluene equivalents (TE) method are illustrated in Figure 5-48. The solid dots fully filled with colour show the mean value of the respective dual determinations and the pastel-filled dots give their individual values. Using the TE determination, the chromatogram is evaluated in such a way that all detectable components are calculated through the toluene response. This figure clearly indicates that the very intensively smelling floor covering 4199 has the highest TVOC values too. The combination of 4199 with textile floor covering 4200, which also emits an intensive smell however, results in considerably reduced VOC emissions, which are essentially due to a lower loading with PVC 4199. In the second combination, which is compared here, the strongly smelling wallto-wall carpet (No. 4200) with the moderately smelling PVC floor covering (No. 4201) only exhibits very low TVOC emissions. However, the component 4-PCH is present in a large quantity and dominates the intensive wall-to-wall carpet smell (see Figure 5-49). Thus substantially lower concentrations of this compound are sufficient to produce a sizeable odour. In combination with the strongly smelling PVC (No. 4199), the individual smell of 4-PCH is much less conspicuous. Figure 5-49 clearly shows that approximately the same volume of 4-PCH was present in the CLIMPAQ in both wall-to-wall carpet tests in spite of the presence of another product in the CLIMPAQ.

It is obvious that the topic 'material combinations' represents a large challenge. The first evaluations of material combinations presented here do not yet allow final conclusions to be drawn.



Figure 5-48: Results of combination tests in HRI's CLIMPAQs. Floor covering No. 4199 was placed into an office. TVOC values are given as toluene equivalents (TE).



Figure 5-49: 4-PCH concentrations for product combinations in HRI's CLIMPAQs

#### 5.2 SPECIFYING A COMPARATIVE SCALE

### 5.2.1 PRECONDITIONS FOR THE EVALUATION METHOD FOR PERCEIVED INTENSITY

The evaluation of the data gathered in this project clearly shows that perceived intensity is best suited as a quantity to characterize odour in the sensory-based evaluation of building products. Perceived intensity does not say anything about the hedonic character of odour. That which smells intensively, is not necessarily unpleasant. Therefore, in addition to perceived intensity, hedonics is also included as a sensory-based quantity in the evaluation of building products.

It can be easily seen that hedonics decreases with increasing intensity for building products. This means that panellists perceive building product emissions as more unpleasant if the intensity increases (see Figure 5-7 in Chapter 5.1 "Sensory-based and analytical testing of building products"). This is different to the investigations into base smells described in Chapter 5.1 It has been found that hedonics does not decrease for all base smells with increasing intensity (see Figure 5-9 in Chapter 5.1).

As described in the previous chapter, perceived intensity related to the scale size has the smallest standard deviation. Therefore, compared to the determination of acceptance, a smaller panel suffices to determine perceived intensity. This group uses a reference, in this case the comparative scale, which enables smaller standard deviations.

When specifying the threshold values for sensory quantities, upper and lower limits of the interval should be used and not one fixed individual value. As the data indicate, the actual value of the perceived intensity of an air sample (building product) is within the interval of -2 to +2 pi about the mean value determined by the panellists.

The limit of perceived intensity for sensory-based testing of building products was established via the relationship of perceived intensity and reasonableness, assuming 90 % confidence interval (see Chapter 5.2.2 "Proposal for specifying the reasonableness threshold for the approval of building products ").

### 5.2.2 PROPOSAL FOR SPECIFYING THE REASONABLENESS THRESHOLD FOR THE APPROVAL OF BUILDING PRODUCTS

A proposal for specifying a reasonableness threshold for sensory-based evaluation is the stipulation of a PD value (see Chapter 3.3.2 "Acceptance"). PD values are also used in regulations as criteria or limits. Thus for example one uses

PD values in the evaluation of thermal comfort in rooms according to DIN EN ISO 7730 [40]. In thermal comfort the highest occurring PD value is 30% according to the categorization used.

As described in Chapter 4.6.2 "Panel without comparative scale", the panel without a comparative scale evaluates acceptance, reasonableness and hedonics of an air sample. In this project reasonableness was used for the first time in sensory-based investigation of building products.

Figure 5-50 illustrates the relationship between reasonableness and PD value, which is calculated from acceptance (see Formula 3-2 in Chapter 3.3.2). If one puts a linear trend through all gathered data, it can be seen that the PD value yields another evaluation other than the question about reasonableness. Thus at a PD value of 30% for example (i.e. 70% of the panellists accept the air sample), about 80% of the panellists interviewed declare that the air sample is reasonable as a daily work environment. This minor displacement clearly indicates that German language usage makes a distinction between acceptance and reasonableness. Reasonableness is a harder criterion.



Figure 5-50: Relationship between reasonableness and percentage dissatisfied (PD value)



# Figure 5-51: Relationship between reasonableness and perceived intensity

Figure 5-51 elucidates the relationship between reasonableness and perceived intensity. It can be seen that in the case of a reasonableness of 80%, which corresponds to the PD value of 30%, a value of about 3 pi results for the perceived intensity. The intensity of about 3 pi however is achieved by only one building product.

#### 5.2.3 ESTABLISHING AN EVALUATION METHOD FOR THE BLUE ANGEL

In order to integrate sensory-based testing into the AgBB scheme and into the award criteria of the Blue Angel, the determination of perceived intensity in pi and hedonics is proposed. Hedonics correlates very well with perceived intensity in the cases investigated (see Figure 5-52) and a linear relationship can be detected.

There could be different limits for perceived intensity and hedonics for the Blue Angel according to product groups. A first proposal for possible limits, initially for all product groups, is 7 pi for perceived intensity (5 pi + 2 pi as safety, Chapter 5.1.5 "Requirements for a minimum panel size ") and -1 for hedonics (0 +/-0.8, rounded to -1) (see Figure 5-53). The values can be established from the assumption that a reasonableness of 70% is set for this proposal. This value differs from the PD values used in air conditioning because the evaluation of the building products is performed directly at the emission chambers and not in the room, as in air conditioning. The behaviour of the building products in a room and their combinations must be investigated in greater detail. Opening investigations into this theme can be found in

Chapter 5.1.6 "Sensory-based and analytical investigations of building products in the 13-m<sup>3</sup> chamber".



Figure 5-52: Relationship between perceived intensity and hedonics for building products on day 28



Figure 5-53: Proposal for the sensory-based limits for Blue Engel criteria

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50% reasonableness has been set as the integration point for sensory-based testing into the AgBB scheme. This results in higher perceived intensity and lower hedonics since the Blue Angel environmental label is primarily used to distinguish above-average products. These limits can be taken from Figure 5-54 and they are 9 pi (+/- 2 pi) for perceived intensity and -1.2 +/- 0.8 for hedonics.



Figure 5-54: Suggestion for the sensory-based limits for the AgBB scheme

The approval criteria are listed in Table 5-5 to Table 5-8 both for analysis (DIBt, Blue Angel environmental label (UZ) and AgBB) and for sensory-based evaluations of all tested building products sorted according to materials classes. A building product which passed or failed the test is shown accordingly. The requirements for AgBB, DIBt and environmental label (UZ) for the values to be met after 3 and 28 days are listed in Figure 5-2. Falling below the values of the third day is required in order to be able to carry on with the test. Falling below the values of day 28 is necessary for successfully passing the tests.

A premature termination of the test is possible if the criteria of the Ü label (DIBt) and also of some UZs are successfully met. Table 5-3 lists the respective criteria. Emission test for the Ü label and UZ 113, 120 and 128 can be prematurely terminated after 7 days when the conditions listed are adhered to and the emissions fail to increase from day 3 to day 7. A special regulation is valid for textile floor

coverings which permits a premature termination of the tests after a three-day period both for Ü label and for UZ 128 when the relevant threshold values are met.

Table 5-2:	Threshold values after 3 and 28 days for DIBt (AgBB) and the
	product-related environmental label. All data in mg/m <sup>3</sup> .

Day		TVOC (C <sub>6</sub> - C <sub>16</sub> )	Σ SVOC (C <sub>16</sub> - C <sub>22</sub> )	R	Σ VOC without LCI	C subst.
	AgBB (DIBt)	≤ 10				≤ 0.01
3	UZ-113 carpet	≤ 1				≤ 0.01
	UZ-120 floor covering	≤ 1.2				≤ 0.01
	AgBB (DIBt)	≤ 1	≤ 0.1	≤ 1	≤ 0.1	≤ 0.001
	UZ-38 wood	≤ 0.3	≤ 0.1			≤ 0.001 <sup>#</sup>
28	UZ-113 adhesive	≤ 0.1	≤ 0.05	≤ 1	≤ 0.04	≤ 0.001
	UZ-120 floor covering	≤ 0.36	≤ 0.04	≤ 1	≤ 0.1	≤ 0.001
	UZ-128 carpet	≤ 0.1	≤ 0.03	≤ 1	≤ 0.05	≤ 0.001

<sup>#</sup>: Value applies to CMT substances

Table 5-3:Threshold values for a successful termination after 3 or 7<br/>days at DIBt and product-related environmental label. All<br/>data in mg/m³.

Day		TVOC (C <sub>6</sub> - C <sub>16</sub> )	Σ SVOC (C <sub>16</sub> - C <sub>22</sub> )	R	Σ VOC without LCI	C subst.	
3	DIBt	≤ 0.3	≤ 0.03	≤ 0.3	≤ 0.05	≤ 0.001	
	UZ-128 carpet	≤ 0.25	≤ 0.03	≤ 1	≤ 0.05	≤ 0.001	
	DIBt	≤ 0.5	≤ 0.05	≤ 0.5	≤ 0.05	≤ 0.001	
7	UZ-113 adhesive	≤ 0.1	≤ 0.05	≤ 1	≤ 0.04	≤ 0.001	
	UZ-120 floor coverings	≤ 0.36	≤ 0.04	≤ 1	≤ 0.1	≤ 0.001	
	UZ-128 carpet	≤ 0.1	≤ 0.03	≤ 1	≤ 0.05	≤ 0.001	
	Additional requirement: no concentration increase between day 3 and 7						

The legend for sensory-based testing and chemical analyzes can be found in Table 5-4. The colours and symbols of the test results for the three time periods i.e. days 3, 7 and 28 have the following meaning: a green tick on day three for the evaluation according to AgBB means that the product meets the requirements for day three, but the test must be carried on; a red cross indicates that the criterion is not fulfilled etc. In Table 5-6 for PVC a line is inserted for UZ: strictly speaking this is only valid for the measured concentrations, PVC coverings are not regulated in UZ 120. A

premature termination criterion is provided both for DIBt tests and for some UZs. If a premature termination is possible, this is indicated in the last two lines of the tables showing the possible day of termination. "7 pi  $\Lambda$ -1" and "11 pi  $\Lambda$ -2", respectively, are indicated in the last two lines for day 28. These are the proposed sensory-based evaluations in intensity (pi) and hedonics ( $\Lambda$ ) according to AgBB or the awarding criteria of the UZs. Detailed information on the awarding criteria of the environmental label is available on the Blue Angel website at www.blauer-engel.de. In the following Table 5-5 to Table 5-8 the criteria described here are applied to the products tested.

Symbol	Sensory-based test	Sensory-based test	Chemical analysis
V	Intensity (PI) – yes Hedonics (H) – yes	passed	passed
×	PI – no H – no	failed	failed
××	PI – no H – yes	failed	
X	PI – no H – yes	failed	
$ abla^{\#}$			Passed UZ criteria, but products not regulated by UZ
×			Just failed
×*			Special threshold value 4-PCH
Ν			Premature termination is not possible

 Table 5-4:
 Symbols for sensory-based tests and chemical analysis

#### Table 5-5:Summary of the results for wall-to-wall carpets

		08-3901 carpet	08-3916 carpet	08-3951 carpet	08-3975 carpet	08-3976 carpet	09-4074 carpet	09-4141 carpet
Day 3	AgBB	M	M					
	AgBB							
Day 28	UZ 128	*		V	*	*		*
	7 pi ∧ −1	×	×	×	×	×		×
	11 pi Л –2							
Day of premature termination	DIBt	3	Ν	3	3	3	3	3
	UZ 128	Ν	N	3	Ν	N	Ν	Ν

		08-3900 PVC	08-4015 PVC	09-4101 PVC	09-4161 PVC	09-4162 PVC	09-4199 PVC
Day 3	AgBB					M	
	AgBB	×	V	×	Ø	V	×
Day 28	UZ 120#	×	$\blacksquare^{\#}$	×	$\checkmark^{\#}$	$\blacksquare^{\#}$	×
	7 pi ∧ −1	×	Ø	Ø	Ø	-	×
	11 pi Л –2	Ø	Ø	Ø	Ø	-	Ø
Day of	DIBt	N	7	N	7	7	N
termination	UZ 120#	Ν	7	Ν	7	7	N

## Table 5-6:Summary of the results for PVCs

# UZ 120 does not apply to PVC, it is only indicated here for comparison!

# Table 5-7:Summary of the results for rubber, levelling compound,<br/>acrylic sealant and parquet

		08-4014 Linoleum	08-3915 Rubber	08-3948 Rubber	08-4005 Rubber	08-3949 Level. comp.	08-4026 Acryl	09-4159 Parquet
Day 3	AgBB	M	V	M	M	Ø		M
	AgBB	M	×	M	M	Ø	×	V
Day 28	UZ XXX#	Ø	×	Ø	Ø		×	Ø
	7 pi Λ −1	×	×	Ø	×	×		Ø
	11 pi Л –2	Ø	×	Ø	Ø	×		Ø
Day of premature termination	DIBt	7	Ν	7	7	Ν	N	Ν
	UZ XXX#	7	N	7	7		N	7

# UZ 38 for linoleum and parquet; UZ 120 for rubber; UZ 123 for acrylic sealant

Table	5-8:
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### Summary of the results for floor covering adhesives

		08-3950 Adhesive	08-3977 Adhesive	08-3978 Adhesive	08-4003 Adhesive	09-4033 Adhesive	09-4061 Adhesive	09-4290 Adhesive
Day 3	AgBB	V		V	Ø	$\mathbf{\nabla}$		Ø
	AgBB	N	×	×	Ø	×		
Day 28	UZ 113	×	×	×	×	×	×	
	7 pi Λ –1	×	×	×	×	××^	×	×
	11 pi Л –2				×		××	×
Day of premature termination	DIBt	Ν	Ν	Ν	Ν	Ν	Ν	N
	UZ 113	Ν	Ν	Ν	Ν	Ν	Ν	N

## 6 SUMMARY AND OUTLOOK

Emissions from building products considerably impair the quality of indoor air. The AgBB scheme has been used to evaluate the emissions of volatile organic compounds (VOC) from building products. The hygienic evaluation according to the AgBB scheme requires product-specific measurement methods which are already available for a number of products. They have been validated in co-operation with several research and testing institutes and have been included in the award criteria for the Blue Angel environmental label. The evaluation is, however, more strict and generally has lower limits than those in the AgBB scheme.

Since VOC emissions are often accompanied by odours which can cause health problems, sensory-based testing (nasal based testing using the sense of smell) is an important element in the evaluation of building products and, as a precaution, has been established in the AgBB scheme. So far this aspect cannot be integrated into the actual evaluation since no coordinated and generally approved procedure is yet available [1].

The objective of this project was to test the smell measurement method developed in the "Environmental and Health Provisions for Building Products – Identification and evaluation of VOC emissions and odour exposure" research project (UFOPLAN Project No 202 62 320) [2] via practical application and integration into the scheme of the Committee for Health-related Evaluation of Building Products (AgBB scheme).

The Blue Angel – as a voluntary instrument of product-related environmental protection – is well suited for introducing the measurement and evaluation of odour emissions for a product group.

The main goal of the project was to establish a sensory-based evaluation method and threshold values for awarding the Blue Angel and for the AgBB scheme. 33 individual building products were subjected to sensory-based tests and most of them to chemical analyses as well (see Chapter 5.1 "Sensory-based and analytical testing of building products"). Furthermore some combinations were tested both in the smaller emission chambers and in the 13-m<sup>3</sup> chamber (see Chapter 5.1.6 "Sensorybased and analytical investigations of building products in the 13-m<sup>3</sup> chamber"). Numerous rounds of discussions with experts from the Federal Environment Agency, with the experts' group and focus groups from within the experts' group resulted in the integration of a questionnaire for a panel without a comparative scale and the inclusion of the issues on acceptance and reasonableness. Building products selected for the tests and experimental methodologies were discussed and agreed upon with the experts.

Based on the research conducted (see Chapter 5 "Results and Discussion"), perceived intensity and hedonics were suggested as a suitable evaluation method. The limits for the sensory-based evaluation on day 28 were established in the present study through the additional questioning of panellists about the reasonableness of a sample (see Chapter 4.6.2 "Panel without comparative scale" and Chapter 5.2.2 "Proposal for specifying the reasonableness threshold for the approval of building products").

Day 28 has been selected as the day of evaluation because odour evaluation is supposed to run at the same time as the emission measurement and on this day a steady emission already prevails. Adhesives or paints for example are applied on to glass plates and must first dry or harden, thus emission is dominated by evaporation for a while and becomes diffusion-driven only after hardening.



Figure 6-1: Relationship between reasonableness and perceived intensity (blue: limits for Blue Angel, green: limits for the AgBB scheme)

Different limits of perceived intensity and hedonics may be feasible for each product group in view of the Blue Angel. A preliminary suggestion for possible

thresholds – for all product groups for the time being – is a perceived intensity of 7 pi (5 pi + 2 pi as safety margin, Chapter 5.1.5. "Requirements for a minimum panel size") and a hedonic value of -1 (0 +/-0.8, rounded to -1) (see Figure 6-1 blue lines and Figure 6-2). A perceived intensity of 7 pi corresponds to an acetone concentration of 160 mg/m<sup>3</sup>.



Figure 6-2: Suggestion for the sensory-based limits for Blue Engel criteria

Higher perceived intensity and lower hedonics are suggested for the integration of sensory-based testing into the AgBB scheme since the Blue Angel environmental label is primarily awarded to above-average products. These limits can be taken from Figure 6-3 and they are 9 pi (+/- 2 pi) for perceived intensity (9 pi corresponds to an acetone concentration of 200 mg/m<sup>3</sup>) and -1.2 +/- 0.8 for hedonics.

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Figure 6-3: Suggestion for the sensory-based limits for the AgBB scheme

The panel size has a major effect on the quality of the results in the smell tests, similarly, a large group requires extensive organization and represents a substantial cost factor. The methods for awarding quality labels or approval tests must not become unworkable because of the determination of odour parameters. Therefore the number of panellists required for the various methods to guarantee sufficient safety of the data was analysed. Figure 6-4 shows the standard deviations related to the scale sizes of the three sensory quantities (perceived intensity, hedonics and acceptance) for a panel size of 11 people. The panel's mean standard deviation of the perceived intensity, hedonics and acceptance was calculated and related to the respective scale size. Those data were used exclusively to calculate the mean of the standard deviation for these three sensory-based quantities where the panels consisted of 11 people. It can be seen that perceived intensity exhibits the smallest standard deviation and acceptance has the largest one. This example shows the extent of dispersion and degree of safety of the sensory-based quantities for measurement data obtained by 11-member panels.





Perceived intensity has the smallest standard deviation for a panel size of 11 compared to other sensory-based quantities. Permitting a deviation of 10% of the actual scale size and a confidence interval of 90% for the sensory-based quantities, a minimum panel size of eight people is obtained to determine perceived intensity. However, over 50 panellists are needed for determining acceptance under the same conditions.

One of the key objectives of the investigations was comparing the use of various emission chambers and performing suitability tests and/or upgrading certain chambers for smell evaluation.

The standard method of collaboration in conjunction with BAM was the use of a 24-litre chamber and filling sample containers with air to be tested. This variant showed good practicality. However, the instructions of use must be very precisely observed when sample containers are being used (the baking temperature of 80  $^{\circ}$ C must be maintained etc.) in order to avoid errors in the sensory-based evaluation.

In Denmark the CLIMPAQ is widely used. The advantage of this piece of equipment is that direct sensory-based evaluations at the chamber are feasible. However, extensive investigations suggest that the flow pattern is not very homogeneous. Therefore the CLIMPAQ's laminarisator was optimized in the project (see Chapter 5.1.3 "Comparison of sample provision using a chamber and sample container for direct assessment at the CLIMPAQ"). One disadvantage of the

CLIMPAQ is that the standard size is only 44 litres. It was reported in this project that the loading q of the material to be tested had to be adjusted several times in order to generate a flow at all. Therefore, in future, CLIMPAQs of various sizes shall be developed for different product groups which will enable the required area-specific emission rate q to be produced.

Direct sensory evaluations at emission chambers require a specified flow rate  $(0.6 - 1 \text{ I s}^{-1})$ . Since normal emission chambers have a maximum volume of 1 m<sup>3</sup>, which can normally only be operated by an air exchange rate of 2 h<sup>-1</sup>, they are not suited to direct sensory-based evaluations due to the limited air exchange. The strict limits as a rule stem from the cost of supplying highly purified and post moisturised air to the emission chambers. The adaptation of these chambers to odour measurements is thus mostly only conceivable after a re-fitting. Since this could not have been performed within this project, the influence of these parameter changes on the sensory-based evaluation cannot be finally clarified. The CLIMPAQ is an emission chamber well suited to direct sensory-based evaluations if the chamber size is adapted to the actual product group. For the analytical tests the 'background' odour of supply air must normally be taken into account in the evaluation.

The results of this project indicate that sensory-based tests can also be performed in laboratories not suitable for this purpose (e.g. BAM, see Chapter 5.1.3 "Comparison of sample provision using a chamber and sample container for direct assessment at the CLIMPAQ"). A comparison of different emission chambers reveals good agreement both in sensory-based and chemical tests.

The results of this project are being integrated into the standardisation work in the field of perceived air quality all the time. The sensory-based evaluation methods of perceived intensity and hedonics have been implemented in ISO 16000 – 28 "Determination of odour emissions from building products using test chambers". Experience from the project has also been included in the guidelines of VDI 4302 Sheets 1 and 2. In addition, another ISO to evaluate indoor air (ISO 16000 – 30) has been requested and headed by the coordinator of the project acting as chairman.

Important further steps are the integration of the threshold values into the Blue Angel environmental label and the AgBB scheme and testing these same thresholds. Since no odour-tested products so far exist in Germany, it is difficult to make the use of materials of low perceived intensity mandatory. Integration into the performance catalogue can only take place after gaining experience. Smell tests can be easily introduced and quickly implemented in laboratories which have already performed emission tests. A panel with a comparative scale of about 14 to 20 people is needed and approx. 10 people are necessary for a test. Also, a comparative scale must be acquired or built and the accuracy should be +/- 0.5 pi (10 mg/m<sup>3</sup> acetone). Furthermore, it is necessary to provide a sufficiently clean lounge for the panellists (ISO 16000-28). The room in which the smell tests are performed must meet the criteria of ISO 16000-28.

Further research into combinations of building products is needed; this could only be partially investigated in this project. Only a small amount of research has been done on both sensory-based and chemical behaviour of combinations of building products. In addition to the combinations of building materials, both chemical and sensory-based behaviour in rooms is of great importance, since odour may increase the energy requirement in the building due to intensification of the ventilation behaviour by the users.

The effect of odorous emissions from building materials on the energy requirement was discussed in a study by E.ON ERC and HRI (DKV 2010 publication, lecture at the final meeting of the 8.3.2010 project). The new European ventilation standards (DIN EN 15251, DIN EN 13779) already specify air quality classes which must be included into the design methods of air conditioning equipment. Ventilation rates are specified in view of the impacts of pollution in buildings. The calculation of energy requirement, however, fails to take into account ventilation heat losses which result from ventilation due to the impact of pollution. Equally, buildings of low pollution and low odour are mentioned, but no relevant limits are specified in the standards.

Each building material can be characterized by a dilution characteristic which gives the volume-specific area load (Aq) as a function of perceived intensity for the building material (Figure 6-5). Perceived intensity can be established from this as a function of flow rate taking into account room size, material surface and number of room users.

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Figure 6-5: Dilution characteristic of a building product (carpet); volumespecific area load (Aq) as a function of perceived intensity of the building material

Figure 6-5 gives the calculated energy requirement for a specified 'example' room contaminated by the carpet characterized in Figure 6-6. Figure 6-6 shows the energy requirement for the 'example' room using window ventilation and controlled ventilation. The proportions of energy requirement for ventilation and transmission are also illustrated in the figure. In this example, air quality of 13 pi would develop. Better air quality within an acceptable range of e.g. 6.5 pi could be provided by additional ventilation. The surplus energy requirement is indicated in red on the right in Figure 6-6. A surplus energy requirement of 100 % is expected in this example.

The results suggest that an energy-efficient building requires low-emission and low-odour building materials.

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Figure 6-6: Surplus energy requirement due to increased air exchange caused by odorous substances in the room

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## 8. GLOSSARY

ADAM AgBB/DIBt evaluation template based on Microsoft Excel. It is a helpful means for both metrological institutes and DIBt in evaluating product emissions. This evaluation is based on the criteria of the AgBB scheme and emissions from floor coverings and other materials are its primarily field of application. It can be acquired via DIBt.

- AgBB Committee for Health-related Evaluation of Building Products. It was founded by the Federal States Working Group for Environmental Health Protection (Länderarbeitsgruppe Umweltbezogener Gesundheitsschutz, LAUG) of the Working Committee of the Federal States Senior Health Authorities (Arbeitsgemeinschaft der Obersten Landesgesundheitsbehörden, AOLG) in 1997; with the Federal Environment Agency (Umweltbundesamt, UBA) acting as its agent.
- AgBB scheme Health-related Evaluation Procedure for Volatile Organic Compounds Emissions (VOC and SVOC) from Building Products; the notification took place in 2005. It is part of the "Fundamentals for the health-related evaluation of building products in indoor spaces", which is the basis for approval of building products by the responsible DIBt. Approved products obtain the Ü label with the additional remark "Emissions tested according to DIBt principles"
- AirProbe Sampling and sample provision device for odour samples using Tedlar<sup>®</sup> containers in odour sample tests

C substance Carcinogenic substance. Within the AgBB scheme the substances are considered as those belonging to category 1 and 2 of the EU's C substance list.

Category 1 substances are notoriously carcinogenic for humans

- Category 2 substances should be considered as carcinogenic for humans since sufficient reference is available from appropriate animal tests and other relevant information
- Category 3 substances give rise to concern because of possible carcinogenic effects for humans; some reference is available from appropriate animal tests, which however is not sufficient to classify the substance into Category 2

- CLIMPAQ Chamber for Laboratory Investigations of Materials, Pollution and Air Quality
- Determination limit (DL) The lowest concentration of a substance that can be quantitatively determined by a chemical analysis with high precision. Above the determination limit, quantitative analysis results are presented.
- Desiccator 24-litre emission chamber
- DIBt German Institute for Building Technology (Deutsches Institut für Bautechnik) is a common institute of the German State and the Federal States (Länder) with the task of uniformly performing building projects if the field of public law.
- Emission Discharge or release of solid, liquid, gaseous substances or noise from stationary or mobile sources also called emittents. Emissions can impair or pollute air, water or soil.
- Emission chamber Device to measure emissions under steady conditions, especially temperature, relative humidity and air exchange rate since these parameters have a key influence on the emission behaviour. A pipe continuously supplies cleaned air into the chamber, into which the material being tested emits its emissions. Air contaminated in such a way is then available for chemical and sensory-based tests. Air samples are taken at connection points and contaminated air not used for measurements is removed as exhaust air.
- Gas chromatograph (GC) The sample is placed using a suitable device (syringe or thermal desorption) into the separation column, which is inside a controlled temperature oven. The stationary phase is in the separation column and the mobile phase is usually helium gas. The mixture of substances is split into individual compounds by the different interactions with the stationary phase as a function of temperature. When a substance leaves the column, it triggers a signal which undergoes computer aided processing in the connected detector system. The measured substances appear as peaks in the resulting chromatogram.
- High-performance liquid chromatograph (HPLC). Unlike GC, HPLC uses a liquid as the mobile phase – usually an organic solvent. It transports the sample through the column at high pressure. The mixture of

substances is split into individual compounds as a function of the strength of the interactions with the stationary phase. At the end of the separating column they are detected by a suitable detector, e.g. a UV or diode array detector (a detector with a continuous spectrum in the UV wavelength range).

- LCI Lowest Concentration of Interest (NIK in German). The annex of the AgBB scheme lists health-related auxiliary parameters, so-called LCI values for a number of indoor-relevant VOCs. They are based on exposure values such as the MAK or AGW values. (MAK: maximum workplace concentration; AGW: workplace threshold value)
- Mass spectrometer (MS) consists of an ion source, in which the gaseous molecules from the sample are fragmented and ionized by electron bombardment in a typical way; of a mass analyzer, which splits up the ions according to their mass to charge number ratio; and of a detector, which measures the intensity of the ion hits. Thus a mass spectrum with several mass peaks of various intensity is created which is typical of each compound, to a certain extent like a "fingerprint". The combination of an MS with a GC is an important instrument in modern analytical techniques.
- PD Percentage dissatisfied
- pi Perceived intensity
- q Area-specific flow rate,  $m^3 m^{-2} h^{-1}$
- R value The sum of the ratios of all substance concentrations Ci to the respective LCI value LCI<sub>i</sub> (R<sub>i</sub> = C<sub>i</sub>/ LCI<sub>i</sub>). It is assumed in the NIK concept that no effect occurs if R is less than 1. If several compounds are detected with concentrations  $\ge 5 \ \mu g/m^3$ , it is assumed that the effects are additive and R, i.e. the sum of all Ri must not exceed 1. R =  $\Sigma R_i = \Sigma (C_i / LCI_i) \le 1$
- VOC Volatile organic compounds; their boiling range is between  $50 100 \ C$  and  $240 260 \ C$
- VVOCVery volatile organic compounds; their boiling range is between< 0  $\C$  and 50 100  $\C$
- TVOC Sum of all detected VOCs in the retention range of n-alkanes from  $C_6$  to  $C_{16}$
$\label{eq:stocc} \Sigma SVOC \qquad \qquad \mbox{Sum of all detected SVOCs in the retention range of n-alkanes} \\ \mbox{from $C_{16}$ to $C_{22}$}$ 

Tedlar<sup>®</sup> container Pillow-shaped container made of Tedlar<sup>®</sup> material with a volume of approx. 300 litres used to transport air samples to be tested

- Thermodesorption system (TDS) is a special injection system for GC. Substances previously collected on adsorbent tubes are desorbed again using careful and slow heating, then trapped in a cold trap and rapidly placed on the chromatographic column (comparably to a syringe injection). The benefits of this method are high sensitivity and low sample volume.
- TENAX A polymer of 2,6-diphenyl-p-phenylene oxide, frequently used in TDS adsorption tubes to detect VOCs.