
Measurement and Classification Method regarding the Emission of Volatile Organic Compounds from Cleanroom Materials

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Abstract

In controlled environments and cleanroom technology, construction materials implemented in specific applications need to be assessed for emissions of volatile organic compounds (VOC) and classified in order to permit direct comparisons to be made. The paper presents a standardized procedure for testing and classifying materials with uniform sample preparation and defined storage times. It describes preparation of the samples, their storage in a VOC-reduced minienvironment, avoidance of cross-contamination, sampling using appropriate adsorbers in semi-automated micro-chambers and analysis via thermodesorption coupled with chromatography and mass spectrometry (TD-GC/MS). Once the active surface area, sampling time and VOC mass emitted are known, a specific emission rate can be determined which is then converted into a classification number. Knowledge of the emission class enables the VOC charge of a defined area at a specific point in time to be calculated in reality. The standardized procedure is laid down in the new guideline VDI 2083 part 17. The test and classification allow a fast material screening compared to existing large chamber measurements and other cleanroom relevant methods. The resulting material classification numbers are entered into the database www.cleanmanufacturing.fraunhofer.de and www.ipa.csm.com of the Fraunhofer industrial alliance CSM – cleanroom suitable materials to allow the tested products to be directly compared with one another. This enables a material selection already in the planning phase of a cleanroom or other environment. An easy-to-use simulation model enables the user to estimate the expected VOC-level and therefore the ISO-AMC-Classification according to ISO 14644-8 of the environment to be constructed.

Key words: material emission, VOC, micro-chamber, ISO-AMC-Classification, thermodesorption, VDI guideline 2083 part 17.

1. Introduction

The outgassing of volatile organic compounds (VOC) from materials considered suitable for usage in cleanroom settings is becoming an increasingly important factor. Several published outgassing studies regarding building materials already demonstrated the VOC emission potential from different materials (1), (2), (3), (4). Regarding the German AgBB-scheme using chamber measurements according to ISO 16000-9 (5), VOC-emissions from all relevant building materials are measured three and 28 days after application/production. The term VOC and total VOC (TVOC) refers to the definition given in ISO 16000-6 (6). Limit values for market release are 10 mg/m³ TVOC₃ after three days and 1 mg/m³ TVOC₂₈ after 28 days. SVOC and cancerogenic

substances are regarded as well (7). Firstly, the quantities of substances emitted from the materials deemed suitable may not exceed statutory maximal allowable workplace concentrations (MAC or LCI values) or recommended maximum total VOC limits. Secondly, sensitive manufacturing environments require a controlled low level of molecular organic contamination in the ambient air. Regarding the standard ISO 14644-8 (8), airborne molecular contamination (AMC) is defined as the presence in the atmosphere of a cleanroom or controlled environment of molecular (chemical, non-particulate) substances in the gaseous or vapour state that may have a deleterious effect on the product, process or equipment in the cleanroom setting or controlled environment. According to ISO 14644-8, AMCs can be categorized in the following substance groups:

- Acids (ac)
- Bases (ba)
- Biotoxin (bt)
- Condensable contaminants (cd)
- Corrosive contaminants (cr)
- Volatile organic compounds (or)
- Dopants (dp)

Comparable categorizations are described in standard JACA No. 35A (9). Organic compounds outgassed from building materials (softeners, solvents and other volatile constituents of materials) play a vital role regarding the amount of airborne organic contamination contained in a cleanroom setting (10).

Various damage scenarios are reported from industry (11): airborne organic contamination (referred as volatile organic compounds VOC. For definition, see ISO 16000-6 (6)) could settle on the lenses of lithographic units and significantly impair their performance. In the semiconductor industry, organophosphates or doping substances condensing on wafers could severely damage them due to wrong doping. The lifetime of lithographic masks is shortened dramatically if VOC deposition causing haze defects occurs (12), (13). Siloxanes could lead to the complete failure of electric contacts, e.g. when testing circuit connections on wafers after processing. Therefore, some classes of compounds are considered to be especially critical and may not be contained in the cleanroom atmosphere of a semiconductor factory above an internally defined detection limit. Such compounds include siloxanes, phthalates, amines, ammonia, acids, organophosphates, doping materials and other dangerous process-specific substances (14). These substances require particular attention when considering the outgassing behavior of cleanroom-suitable materials.

Different national labeling and/or ranking systems regarding VOC-emissions from building materials are established (3), but lack in comparable classification, harmonization and suitability for the selection of low VOC-emission cleanroom suitable materials. This situation did not change until today. Therefore, there is a need for an easy-to use method to determine surface-specific VOC-emission rate of materials using an adapted micro chamber sampling technique together with a comparable classification system.

The aim of the method described is to define test criteria which enable materials to be evaluated and classified with regard to their VOC outgassing properties using the proposed micro chamber measurement setup (15), (16), (17), (18). The method permits the comparison of up to six different materials with one another from the point of view of emitting volatile organic compounds and allows them to be ranked in a list to facilitate the selection of appropriate materials. Using different emission chamber geometries and sizes, a variation of the determined SER of up to 60 % is reported (4). Variations in air exchange rate during chamber emission measurements can lead to different results depending on the sample material, too (19) (20). Therefore, it seems necessary to use a standardized emission chamber and test protocol for a worldwide comparable classification measurements described below.



Figure 1. Personal air sampling pump used for sampling of cleanroom air onto Tenax TA sorbent tubes for the determination of the ISO-AMC-Class (VOC) according ISO 14644-8.

The quantity of organic compounds outgassed from materials is dependent upon surface area, outgassing time, age and test temperature. The surface-specific emission rate SER for VOC is related to these parameters and is expressed as mass per surface area x time [g/m²h] at the corresponding ambient

temperature. In order to have a standardized comparable test procedure, measurements are carried out in a micro emission chamber in accordance to ASTM D5116-10 (21) and ISO 16000-9 (5). Outgassing is determined by collecting the emitted VOC from the micro-chamber on Tenax TA sorbent tubes and subsequently analyzing the sorbent tubes by way of thermodesorption with coupled gas chromatography and mass spectrometry (TD-GC/MS) according to the method described in VDA 278 (22).

The determination of the ISO-AMC class according to ISO 14644-8 is done by sampling onto suitable sorbent tubes (mostly Tenax TA) using personal air sampling pumps followed by laboratory analysis using TD-GC/MS, see figure 1.

2. Material and Methods

2.1. Preparing Samples

Samples have to be typical as far as their geometry and surface characteristics are concerned and need to be tested under the same conditions as those experienced by the material in the cleanroom. Ideally, flat samples with a homogenous surface should be used. In multiple layer applications, layer composition must correspond to the planned usage. The cut edges of solid samples which should not be considered as being part of the active surface area need to be covered appropriately, e.g. with an

aluminum cutting ring or with aluminum foil.

VOC-free carrier materials (glass dishes, stainless steel) are to be used for free-flowing samples (reactive hardening material samples during preparation, ex. epoxy-resins) and the active surface of the material samples must be quantifiable, see figure 1. Reactive hardening material samples need to be pre-conditioned over a period of 30 days under controlled climatic conditions (see also ISO 16000-11 (23) and VDI 2083 Part 9.1 (24)). Samples may not become contaminated during storage. This is achieved by storing samples in a VOC-reduced environment (minienvironment with filter-fan-unit, active charcoal VOC filtration and particulate HEPA-filtration, M+W Products GmbH, Stuttgart, Germany). The minienvironment is installed into an ISO-class 1 cleanroom with controlled climatic conditions (cleanroom temperature: 22 ± 1 °C, relative humidity: 45 %, see ISO 14644-1). Remark: The minienvironment has no own HVAC (heating, ventilation and air conditioning) -system), so the cleanroom climatic conditions have to be used, see figure 2. The VOC-reduced storage environment has to be at least one class better than the anticipated VOC assessment of the test piece. The achieved ISO-AMC class (VOC) of the storage environment has to be determined by VOC-sampling using e.g. Tenax-tubes and appropriate portable sampling pumps. The blank value of the measurement is determined using field blank samples. For these samples, closed conditioned tubes are kept in close proximity to the sample tubes from prior conditioning until final analysis.



Figure 2. Left: Different flooring material samples for the measurement of the material specific emission rate SER regarding volatile organic compounds VOC according VDI 2083 part 17. Right: Minienvironment with filter-fan-unit, active charcoal VOC filtration and particulate HEPA-filtration, M+W Products GmbH, Stuttgart, Germany.

2.2. Micro Chamber Measurements

After completion of the pre-conditioning period of 30 days in the storage environment beginning from the set-up of reactive materials, defined material samples with a surface area of 10 cm^2 are placed in a semi-automated micro test chamber (μ CTE, Markes International, Llantrisant, UK, see figure 3 and 4) with a diameter of $d = 45\text{ mm}$ and a volume of $V = 40\text{ ml}$ under atmospheric pressure at a standardized temperature and humidity of $22\text{ }^\circ\text{C} \pm 1\text{ }^\circ\text{C}$ and 45 \% rel. humidity (standardized room temperature according to VDI 2083 part 17 (25)). After an equilibration time of 15 min, the VOC-sampling takes place over a period of 60 min. As rinsing gas, ultrapure VOC-filtered nitrogen is used with a flow rate of 100 ml/min . The rinsing gas transports the volatile organic compounds (VOC) emitted from the material sample to a sorption tube containing as appropriate adsorbent Tenax TA® or Tenax GR® (Markes International, Llantrisant, UK) where they are absorbed (Purge and trap method). The polymer resin Tenax TA® is most commonly used for VOC-sampling from C6 to C16 and beyond. Tenax GR® is a graphitized (30 %) Tenax TA® for better thermal stability but with the same retention properties. The sampling time and flow rate form the basis of the subsequent quantitative classification. Samples can only be compared if pre-conditioning time, sampling temperature, and duration of VOC sampling are identical. Materials are classified solely based on tests performed at room temperature. A possible second analysis at the elevated temperature $T = 90\text{ }^\circ\text{C}$ is done to gain information about the total content of volatile organic contaminants and presence of critical substances in the material sample.

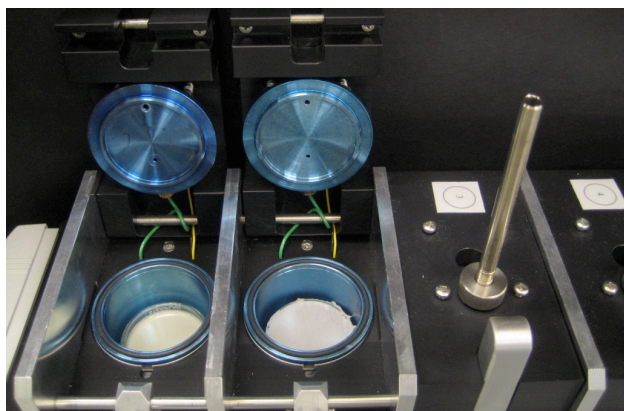


Figure 3. Material samples (epoxy flooring, left and silicone sealant, right) in micro-chamber μ CTE with two cover lid opened and other lids closed with an adsorption tube attached on the outlet of the closed chamber (right).



Figure 4. Semi-automated micro-chamber μ CTE using pneumatic cylinders for the time-controlled insertion and removal of sorbent tubes with appropriate diffusion lock caps from Markes International, Llantrissant, UK.

2.3. TD-GC/MS Analysis

Sorption tubes are analyzed by way of thermodesorption (ATD 650, PerkinElmer, Waltham, United States) with coupled gas chromatography and mass spectrometry (Clarus 600 GC with Clarus 600T, PerkinElmer, Waltham, United States). The thermodesorption process releases the VOCs held by the sorption tubes, enabling them to be subsequently analyzed. The analysis was carried out in compliance with VDA 278 (22). Calibration was performed using six-point calibration from 1 ng to $10\text{ }\mu\text{g}$ total mass of hexadecane in methanol. The solution from the dilution series were loaded onto the tubes using a Calibration Solution Loading Rig (Markes International, Llantrissant, UK) and a $5\text{ }\mu\text{l}$ glass syringe (Hamilton AG, Bonaduz, Switzerland) with a flow of 100 ml/min ultrapure air for 5 minutes. For each analyzed set of tubes, a standard tube dotted with hexadecane was analyzed as well to determine the response factor in regards to the six-point calibration.

2.4. Determining the specific emission rate and a standardized material classification number

The emission test initially only gives the mass of all volatile organic compounds m_{VOC} absorbed by the sorption tube after analysis in accordance to VDA 278. The mass is then related to both the surface area of the sample and the sampling time in order to enable the specific emission rate of the material per surface area to be calculated.

$$SER_m = \frac{m_{VOC}}{A_m \cdot t} \quad (1)$$

Here, SER_m means the specific emission rate of material per surface area at a room temperature of 22 ± 1 °C in $g/(m^2 \cdot s)$ using standardized SI units; m_{VOC} is the mass of all volatile organic compounds outgassed from material in g; A_m is the surface area of material m in m^2 and t means the duration of sampling time in s. The obtained surface specific emission rate is normalized to $1 m^2$ surface area, $1 m^3$ reference volume and a normed air exchange rate of $1 s^{-1}$.

$$TVOC_{norm} = \frac{SER_m \cdot A_{norm}}{V_{norm} \cdot n_{norm}} \quad (2)$$

Here, V_{norm} means the normed reference volume of $1 m^3$; A_{norm} is the normed surface area of material of $1 m^2$; n_{norm} equals the normed air exchange of $1/s$ and $TVOC_{norm}$ is the normed concentration of total products outgassed from material in g/m^3 . The value $TVOC_{norm}$ is the logarithmized in a decadal way to obtain a material-specific normed ISO-AMC_m class N (VOC) according to VDI 2083 part 17.

$$ISO - AMC_m \text{ class } N(VOC) = \log(TVOC_{norm}) \quad (3)$$

The material classification is expressed as follows: ISO-AMC_m class N (VOC) with a value lying between 0 and -12. Interim classification values may also be stated. In the process, 0.1 is the lowest permissible increment of N. The term “VOC” defines the volatile organic compounds as a contaminant group. The classification procedure can be done for all other AMC contaminant groups as well. The described standardized material classification results in an easy-to-use comparable and communicable classification number. Due to its logarithmic nature, the whole outgassing spectra from metallic materials up to high VOC-emitting materials can be expressed. This classification number can be later the basis for the calculation of

expected TVOC values of real cleanroom environments.

2.5. Using the ISO-AMC_m class to estimate the ISO-AMC class of a real cleanroom environments

The described calculation model enables a first rough estimation about the TVOC charge of the cleanroom environment regarded as defined cavity with corresponding air intake and exhaust. Regarding all relevant major VOC-emitting materials in a cleanroom $\sum(SER_m \cdot A_m)$, and the VOC-concentration of the intake air, the resulting steady-state ISO-AMC class (VOC) in the cavity can be calculated as follows:

$$TVOC_{CR} = \frac{\sum(SER_m \cdot A_{CR})}{V_{CR} \cdot n_{CR}} + c_{supply} \quad (4)$$

Here, $TVOC_{CR}$ equals the calculated total concentration of VOC emitted from the material into the cleanroom in g/m^3 ; A_{CR} is the surface area of the material in the cleanroom in m^2 ; V_{CR} resembles the cleanroom volume in m^3 ; n_{CR} is the rate of fresh air introduced into cleanroom in $1/s$ and c_{supply} means the VOC-concentration of intake air in g/m^3 . The rate of fresh air n_{CR} is calculated as follows:

$$n_{CR} = AER_{CR} \cdot f_{CR} \quad (5)$$

Here, n_{CR} resembles the rate of fresh air introduced into cleanroom; AER_{CR} is the air exchange rate in the cleanroom and f_{CR} equals the fraction of fresh air rate into the cleanroom. The decadal logarithm of $TVOC_{CR}$ gives the ISO-AMC class of the cleanroom and is expressed in the following way: ISO-AMC class N (VOC).

$$ISO - AMC \text{ class } N(VOC) = \log(TVOC_{CR}) \quad (6)$$

Regarding the relevant materials for the ISO-AMC class estimation, the proportionate material surface area and materials with a high TVOC emission play the major relevance regarding the SER_m -values. Furthermore, the applied room temperature and humidity play a significant role, too. But as the temperature and humidity inside a cleanroom mostly equals the defined chamber measurement parameters with 23 °C and 45 % rel. humidity, these parameters are comparable. Most notably relevant are for large surface areas flooring and wall

systems, ceiling, filter systems including the filter media and air conditioning technology. Possible high VOC-sources are sealants, adhesives and lubricants.

The ISO-AMC classification as referred in VDI 2083 part 17 is made in accordance with ISO 14644-8, see figure 5 (8).

ISO-AMC Class N	Concentration [g/m ³]
0	10 ⁰
-1	10 ⁻¹
-2	10 ⁻²
-3	10 ⁻³
-4	10 ⁻⁴
-5	10 ⁻⁵
-6	10 ⁻⁶
-7	10 ⁻⁷
-8	10 ⁻⁸
-9	10 ⁻⁹
-10	10 ⁻¹⁰
-11	10 ⁻¹¹
-12	10 ⁻¹²

Figure5: ISO-AMC air cleanliness classes in accordance with ISO 14644-8.

2.6. Theoretic background: mass flow equilibrium at the stationary phase

Regarding the air flow design of a cleanroom, the following simplification can be done. The recirculated air from the air-conditioning system can be regarded as part of the cleanroom and has not to be considered for the calculation of the mass flows as long as no VOC-filtration is installed. Only the incoming fresh air with a specific mass concentration of the regarded contamination (mass flow_{inlet}), the exhaust air with the mass concentration of the cleanroom (mass flow_{outlet}), both and the mass removal due to VOC-filtration (mass flow_{AMC-Filtration}) have to be regarded for the calculation of the stationary phase of the mass flow equilibrium (figure 6).

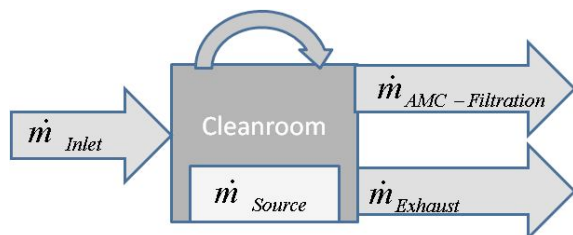


Figure 6. Simplification of the relevant mass flows for setting up the equation for the mass flow equilibrium in a cleanroom.

The stationary phase in a laminar cleanroom with an appropriate air exchange rate will settle in a very short period of time when all summed up relevant mass flows do not change any more. For the determination of the total mass flow in the cleanroom, the single mass flows from all relevant materials $SER_{m1} \cdot A_{CR1}$; $SER_{m2} \cdot A_{CR2}$; ..., the inlet mass flow, the outlet mass flow and the AMC filtration removal mass flow have to be summed-up. Taking the Volume V , the initial concentration of VOC in the cleanroom c_0 and the fresh air volume flow from the outdoor intake of the HVAC system, the mass flow equilibrium and therefore the concentration of the regarded contaminant group $x = \text{VOC}$ in the cleanroom is calculated as follows:

$$ISO-AMC(VOC) = \log \left(c_0 - \frac{\dot{m}_{total}}{F} \right) e^{\left(\frac{F}{V} t \right)} + \frac{\dot{m}_{total}}{F} \quad (6)$$

4. Results and Discussion

Different materials were measured and classified with the described method. The displayed selection of investigated flooring and sealant materials categorized in different material categories shows a huge difference in classification (see figure 7). Therefore, depending on the aimed ISO-AMC-class of the cleanroom setting, material selection has to be done very carefully. The actual detection limit of the TD-GC/MS is 1 ng absolute mass of VOC. With the described chamber measurement method, this equals a best determinable ISO-AMC_m class = -9.6 (VOC).

Using equation 6, the calculated value ISO-AMC class N (VOC) reflects the situation 30 days after introducing the material into the cleanroom environment. As VOC outgassing from materials reduces over time, the TVOC-level in the cleanroom will lower accordingly. Therefore, this represents a good estimation of the ISO-AMC class N (VOC) and gives not the very exact TVOC-value of the cleanroom environment as mainly the major VOC emitting materials are considered as relevant for calculation and not entirely all utilized materials. But together with the described material classification, this calculation tool enables the user a fast and effective material selection according to the needs for the later application.

Material	Type	ISO-AMC _m class N (VOC)
StoPox BB OS	E	-6.6
StoPox WHG Deck 100	E	-6.6
StoPox KU 601	E	-7.8
StoPox WL 100	E	-9.6
Sikaflex AT Connection	S	-4.6
Sikaflex Pro 3 (i-Cure)	S	-6.8
Sikaflex PRO 3 WF	S	-4.8
Sikaflex Wallcoat N	E	-9.6
Sikaflex-221 RLT uniwhite	S	-4.7
Sikafloor-235 ESD	E	-6.8
Sikafloor-263 SL	E	-6.5
Sikafloor-264	E	-6.5
Sikafloor-266 CR	E	-7.8
Sikafloor-266 ECF CR	E	-7.7
Sikafloor-269 CR	E	-9.6
Sikafloor-269 ECF CR	E	-9.6
Sikafloor-325	E	-7.1
Sikafloor-381	E	-9.6
Sikafloor-381 AS	E	-9.6
Sikafloor-390	E	-9.6
Sikafloor-390 AS	E	-9.6
Sikagard-183 W CR	E	-9.6

Figure 7: ISO-AMC_m class N (VOC) of different flooring and sealant materials. E = epoxy flooring; S = sealant.

4. Conclusions

The use of a suitable test chamber method and corresponding analytics enables the surface specific emission rate of volatile organic compounds to be determined from a material sample. Through appropriate standardization, a material class can be calculated based on this value. The standardized material classification permits a direct comparison of the tested materials to be made with regard to the emission of volatile organic compounds. The tested and classified materials are awarded a corresponding official test seal and are entered into the data base www.ipa-csm.com. The procedure presented in this article is developed by the Fraunhofer IPA industrial alliance for cleanroom suitable materials (CSM) and implemented in the VDI guideline 2083-17 (25), (26).

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