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Measurement Report

Cleanroom suitability tests on PVC floorings produced by Tarkett AB

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Report No. **TA 1108-567**

Stuttgart, Germany, September 20, 2011

Index

1	**Introduction and objectives**	**3**
2	**Materials tested**	**4**
3	**Overview of results**	**5**
4	**Airborne particle emission tests on application of tribological stress**	**6**
4.1	Procedure for particle emission tests	6
4.1.1	Cleanroom-suitable material test bench	6
4.1.2	Test parameters	9
4.1.3	Cleanroom environment	10
4.1.4	Particle measuring technique	10
4.1.5	Test procedure	11
4.2	Material samples for particle emission tests	11
4.3	Particle emission results	12
4.3.1	Differential progression of particle emission	12
4.3.2	Size distribution of the emitted particles	14
4.3.3	Classification	16
5	**Outgassing tests under thermal stress**	**19**
5.1	Material samples	19
5.2	Outgassing test procedure	21
5.2.1	Sampling chamber	22
5.2.2	Analysis devices	24
5.3	Outgassing results	25
5.3.1	General explanation of chromatograms	27
5.3.2	iQ Optima	28
5.3.3	iQ Granit	35
6	**Comparison of Classifications of Airborne Particulate Contamination**	**42**

1 Introduction and objectives

Tarkett AB is an manufacturer of chemistry and industrial materials. The components are produced under high quality requirements and are successfully implemented in a wide range of industries.

To secure the market position of Tarkett AB in the sector of cleanroom technology, the aim is to identify optimization potentials for its products. The suitability of a product for use in clean areas is significantly influenced by the materials used in its manufacture.

The industrial alliance "Cleanroom Suitable Materials CSM" has developed procedures for determining the cleanroom suitability of materials. Depending on the area of implementation concerned, the behavior of materials with regard to particle emission and outgassing is taken into consideration. The tests are carried out in a standardized way in compliance with relevant national and international norms.

The results obtained provide an objective and substantiated basis for comparison and can be referred to when selecting suitable materials for specific manufacturing environments and areas of implementation. In consequence, this improves the cleanroom suitability of the respective products.

2 Materials tested

TESTED MATERIALS
NAME (FULL LENGTH)
iQ Optima Batch number: 1674741 Roll number: 3101050215 Production date: 2011-08-10
iQ Granit Batch number: 1672983 Roll number: 3201074923 Production date: 2011-08-10
PA6 Nylon

Figure 1 Overview of materials tested

3 Overview of results

TESTED MATERIALS	PERFORMED TESTS		
NAME (FULL LENGTH)	PARTICLE EMISSION	OUTGASSING	BIOLOGICAL RESISTANCE
iQ Optima	X	X	
iQ Granit	X	X	
PA6 Nylon	X		

Figure 2 Overview of materials tested

PARTICLE EMISSION (ACCORDING TO ISO 14644-1 AND GMP-GUIDELINE VOLUME 4, ANNEX 1)			
MATERIAL PAIRING			ISO CLASS
SPECIMEN	COUNTER SPECIMEN	LUBRICANT	GMP-CLASS
iQ Optima	PA6 Nylon	(none)	**4**
iQ Granit	PA6 Nylon	(none)	**4**

OUTGASSING (ACCORDING TO ISO 14644-8)			
MATERIAL	TEMPERATURE	TESTED FOR	ISO-AMC (or)
iQ Optima	23°C	TVOC	**-9.6**
	TEMPERATURE	TESTED FOR	OUTGASSING RATE
	90°C	Amines	**Not detectable**
		Organo-phosphates	**Not detectable**
		Siloxanes	**7.9 x 10^{-9} g/m²**
		Phthalates	**Not detectable**
MATERIAL	TEMPERATURE	TESTED FOR	ISO-AMC (or)
iQ Granit	23°C	TVOC	**-9.6**
	TEMPERATURE	TESTED FOR	OUTGASSING RATE
	90°C	Amines	**Not detectable**
		Organo-phosphates	**Not detectable**
		Siloxanes	**4.1 x 10^{-9} g/m²**
		Phthalates	**Not detectable**

Figure 3 Overview of results obtained

4 Airborne particle emission tests on application of tribological stress

4.1 Procedure for particle emission tests

4.1.1 Cleanroom-suitable material test bench

A special, cleanroom-suitable material test bench developed by Fraunhofer IPA and called Material Inspec is used for the tests. The test bench enables material pairings to be subjected to controlled tribological stress and permits the resulting particulate emissions to be measured without the influence of any cross-contamination.

Figure 4 Cleanroom-suitable material test bench "Material Inspec" developed by Fraunhofer IPA with module for ball on disk test

Tribological stress

The cleanroom-suitable material test bench "Material Inspec" enables tests to be carried out using the tribological methods known as **ball-on-disk** and **reel-on-disk** tests.

With the ball-on-disk test, a ball with a **radius r** is pressed onto the face of a disk with a **normal force F**. In the process, the disk rotates with a **frequency f** so that a **relative velocity v** results at the point of contact. The **single measurement track s** is calculated from the circumference of the circle with the radius r. The **number of revolutions N** is the number of rotations completed by the disk beneath the ball during the test.

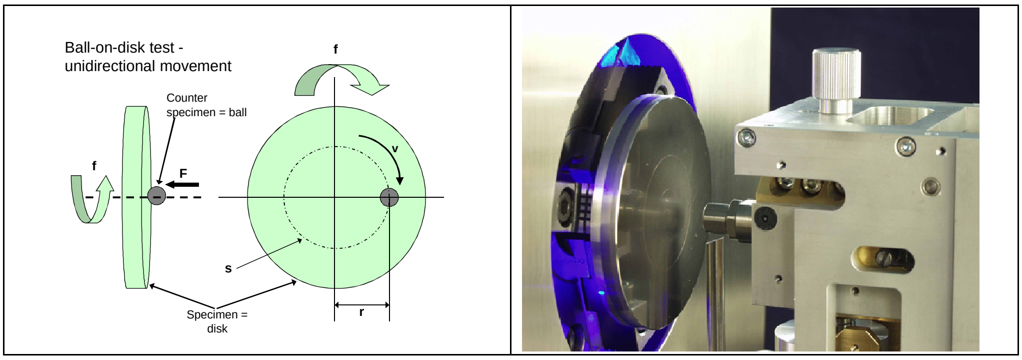

Figure 5 Tribological stress on material pairing – principle of **ball-on-disk test**

The ball-on-disk test simulates pure dynamic friction between two materials. The point of contact is punctiform; this fact needs to be taken into consideration when assessing the resulting local force applied.

All of the tests which are carried out are model tests. This means that the forces mentioned or applied are similar to but may not be exactly the same as those encountered in reality. This fact requires special consideration when interpreting the results and transferring them to real components.

4.1.1.1 Force transmission and measurement recordings

The normal force is applied using a force transmission unit. For the ball-on-disk test, dead weights are implemented. For the reel-on-disk test, steel springs are utilized because of the increased forces.

The **normal force** applied is recorded continuously during the test using a load cell based on the principle of the strain gauge.
With the ball-on-disk test, in addition to the normal force, the frictional force acting vertically downwards at the point of contact is also recorded synchronously. This enables the **progression of the friction coefficient** to be determined as the ratio between frictional force and normal force.

Particle measurement
Particulate emissions are measured directly beneath the point of contact of the material test specimen. In the case of the ball-on-disk test (punctiform contact of the test specimen), a chamfered particle probe tube is used. With the reel-on-disk test, because of the broader line-shaped contact, a cylindrical particle probe with an aperture of 35 mm in diameter is used.
The area of contact has been specially designed from an airflow point of view to ensure that the majority of particles emitted are detected.

4.1.2 Test parameters

For both the ball-on-disk and the reel-on-disk tests, the essential test parameters affecting particulate emission include the **single measuring track s**, the **relative velocity v**, the **normal force F** and the **number of revolutions N**. Standardized sets of stress parameters are formed using these values to facilitate the comparison of results obtained from the various tests.

SET OF PARAMETERS	s/mm	v/mm/s	F/N	N		SET OF PARAMETERS	s/mm	v/mm/s	F/N	N
A 01	70	50	1	1500		B 01	250	150	15	1500
A 02	90	50	3	1500		B 02	250	150	45	1500
A 03	110	50	5	1500		B 03	250	150	75	1500
A 04	130	100	6	1500		B 04	250	150	90	1500
A 05	150	100	8	1500		B 05	250	150	120	1500
A 06	170	100	10	1500		B 06	250	150	150	1500
A 07	200	100	11	1500		B 07	250	150	165	1500
A 08	220	100	13	1500		B 08	250	150	195	1500
A 09	240	100	15	1500		B 09	250	150	225	1500
A 10	260	150	16	1500		B 10	250	150	240	1500
A 11	280	150	18	1500		B 11	250	150	270	1500
A 12	300	150	20	1500		B 12	250	150	300	1500

Figure 6 Defined set of stress parameters; left: ball-on-disk test; right: reel-on-disk test

The amount of stress to be applied to each material pairing is decided upon individually by Fraunhofer IPA on taking into account the quantity of particles generated and the measuring range of the device used in the test.

The following table shows the degree of accuracy achieved when setting the test parameters as well as fluctuations in these parameters which are experienced during the tests.

	ACCURACY; MAXIMUM VARIATION DURING TEST	
	BALL-ON-DISK-TEST	REEL-ON-DISC-TEST
NORMAL FORCE FN	0.01 N; +/- 3 %	0.01 N; +/- 3 %
SINGLE MEASURING TRACK S	0.1 mm; n.a.	0.1 mm; n.a.
RELATIVE VELOCITY V	0.5 mm/s; +/- 3 %	0.5 mm/s; +/- 3 %
NUMBER OF REVOLUTIONS N	+/- 1 %	+/- 1 %

Figure 7 Degree of accuracy achieved when setting the test parameters and fluctuations thereof during the test

4.1.3 Cleanroom environment

All tests are carried out at the Fraunhofer IPA test center for semiconductor equipment. Measurements are taken in a cleanroom fulfilling Class 1 specifications (in accordance with ISO 14644-1). A vertical, unidirectional airflow prevails in the cleanroom with a first air flow velocity of 0.45 m/s. Environmental conditions are kept constant with a room temperature of 22 °C ± 0.5 °C and a relative humidity of 45 % ± 5 %.

In compliance with ISO 14644-1, Cleanroom "Class 1" means that only two particles the size of 0.2 µm may be found in a reference volume of one cubic meter in the first air (filtered air introduced into the cleanroom). In practical operation, even fewer particles are found in this class.

4.1.4 Particle measuring technique

Optical particle counters are utilized to determine particle emission during the tests.

Optical particle counters function according to the theory of scattered light. Using a sampling probe, a defined volume of air of 1 cubic foot (1 cft = 28.3 liters) is sucked in per minute and guided into a measuring chamber via a tube connected to it. The air sucked in is illuminated by a laser beam. As soon as a particle carried by the airflow is hit by a light ray, the light is scattered and recorded by photo-detectors.

The amount of impulses registered equates to the number of particles found in the volume of air; the height of the impulse gives an indication of particle size.

Depending upon the size and amount of particles generated, 3 different measuring devices are used.

MODEL	COMPANY	PARTICLE SIZES DETECTED
LasAir II 525	PMT AG, Heimsheim	0.5 / 0.7 / 1.0 / 5.0 / 10.0 / 25.0 µm
LasAir II 110	PMT AG, Heimsheim	0.1 / 0.2 / 0.3 / 0.5 / 1.0 / 5.0 µm
LPSA 210	PMT AG, Heimsheim	0.2 / 0.3 / 0.5 / 5.0 µm

Figure 8 Optical particle counters used to record particle emissions

The volume of air sucked in by all devices is 1 cft/min = 28.3 l/min.
In order to obtain a chronological progression of the particles emitted, particle measurements are recorded every 6 seconds.

4.1.5 Test procedure

The test specimens are **introduced** into the cleanroom before the tests are commenced. In the process, the surfaces of the test pieces are cleaned to remove any sedimented particles or filmy contamination which may be present.

The **tribological reel-on-disk tests** are carried out using **1 different set of stress parameters**, taking into account the quantity of particles generated. To ensure reliability of the results, **10 repetition tests** are carried out for the chosen stress parameter.

4.2 Material samples for particle emission tests

Figure 9 Materials tested – left: iQ Optima; right: iQ Granit

Figure 10 Materials tested – left: PA6 Nylon

TESTED MATERIALS				LOAD
ID	SPECIMEN	COUNTER SPECIMEN	LUBRICANT	
IP Tarkett 01	iQ Optima	PA6 Nylon	(none)	Reel-on-disk-test
IP Tarkett 02	iQ Granit	PA6 Nylon	(none)	Reel-on-disk-test

Figure 11 Materials for the particle emission tests

The table also includes the codes used by the industrial alliance CSM to identify material pairings.

For the material pairing IP SIKA 10, a floor covering founded on a 15 mm thick and a diameter of 140 mm disk are used as a specimens.

A reel with a width of 60 mm and a diameter of 100 mm, made of PA6 Nylon, is used as counter specimen.

4.3 Particle emission results

4.3.1 Differential progression of particle emission

4.3.1.1 Method

Particle emission is measured every 6 seconds during the application of tribological stress. Depending upon the particle counter used, particle emission is classified into various **particle size channels**. The values measured are expressed **cumulatively**, i.e. the result for one size always includes all particles equal to or larger than the reference size for that channel. For example, the information obtained for the particle size 0.1 µm includes all particles with a diameter of 0.1 µm or larger.

Each diagram shows the progression of particle emission measured in the smallest particle size channel for the three repeated tests on application of one set of stress parameters. Where appropriate, the **scale of the y-axis** is adjusted, please note that the scale may vary from one graph to another!

4.3.1.2 IP Tarkett 01: iQ Optima

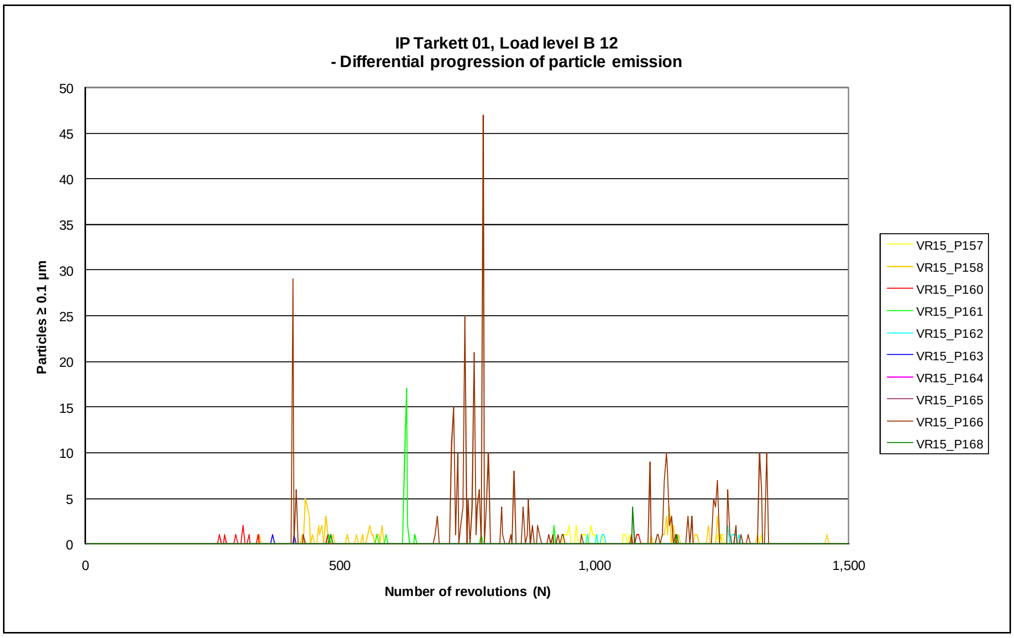

Figure 12 IP Tarkett 01 – progression of particle emission, particle size **0.1 µm**, set of stress parameters **B 12**

4.3.1.3 IP Tarkett 02: iQ Granit

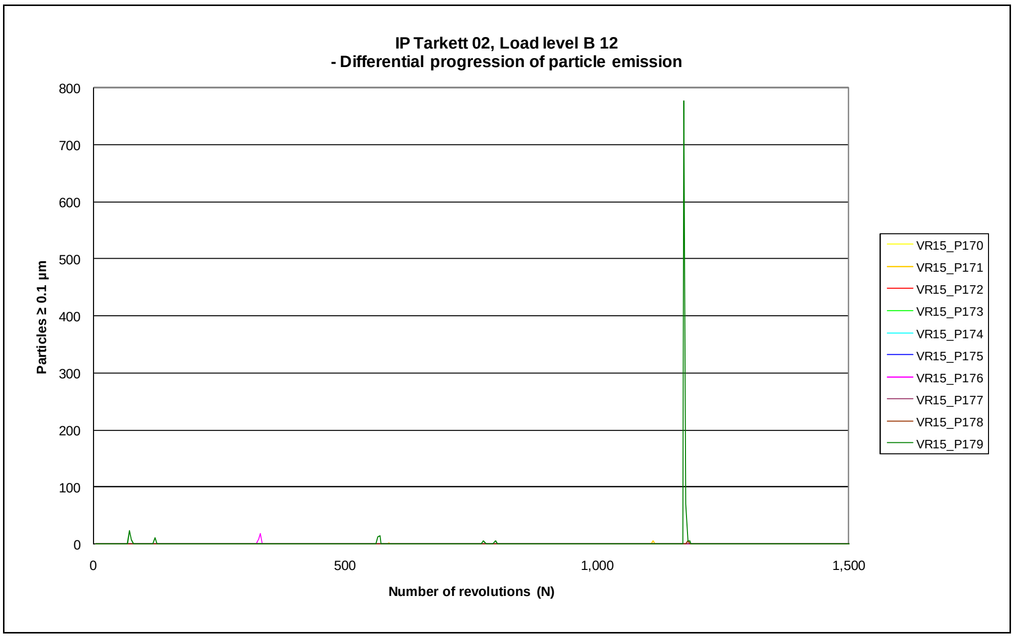

Figure 13 IP Tarkett 02 – progression of particle emission, particle size **0.1 µm**, set of stress parameters **B 12**

4.3.2 Size distribution of the emitted particles

4.3.2.1 Method

From the particle emission progression data, the percentage of each particle size in relation to the total count of emitted particles is calculated. If, for example, the particle sizes 0.1 µm, 0.2 µm, 0.3 µm, 0.5 µm, 1.0 µm and 5.0 µm are recorded by the optical particle counter, the percentage of the

- Particles in the size channel 0.1 µm relates to particles with a diameter of 0.1 µm to 0.2 µm,
- Particles in the size channel 0.2 µm relates to particles with a diameter of 0.2 µm to 0.3 µm,
- Particles in the size channel 0.3 µm relates to particles with a diameter of 0.3 µm to 0.5 µm,
- Particles in the size channel 0.5 µm relates to particles with a diameter of 0.5 µm to 1.0 µm,
- Particles in the size channel 0.5 µm relates to particles with a diameter of 0.5µm to 5.0µm,
- Particles in the size channel 5.0 µm relates to particles with a diameter equal to or greater than 5.0µm.

Values are obtained from all three repeated tests. The size channel stated is dependent upon the optical particle counter used in the tests.

In order to ensure reliability of the data, only those percentages of particles are calculated where a minimum of 100 particles was observed in the smallest size channel in the course of the entire test.

The following diagrams show the particle size distribution for the material pairings and the corresponding sets of stress parameters. If data is absent in the diagram, this means that the required minimum count of 100 particles was not recorded in the smallest size channel.

4.3.2.2 IP Tarkett 01: iQ Optima versus PA6 Nylon

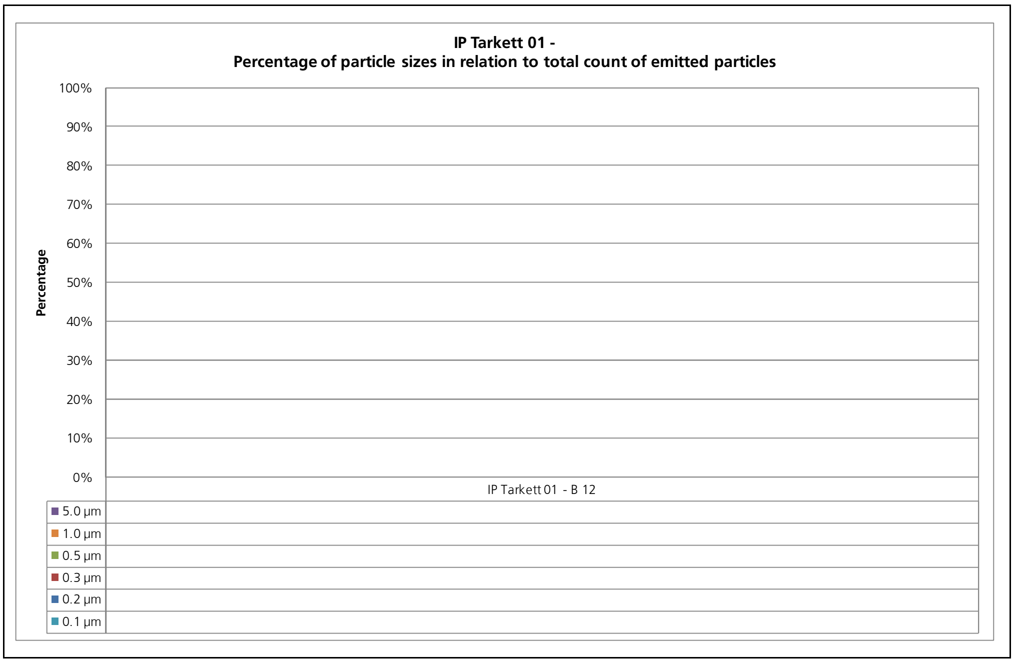

Figure 14 IP Tarkett 01 – required minimum count of 100 particles was not recorded

4.3.2.3 IP Tarkett 02: iQ Granit versus PA6 Nylon

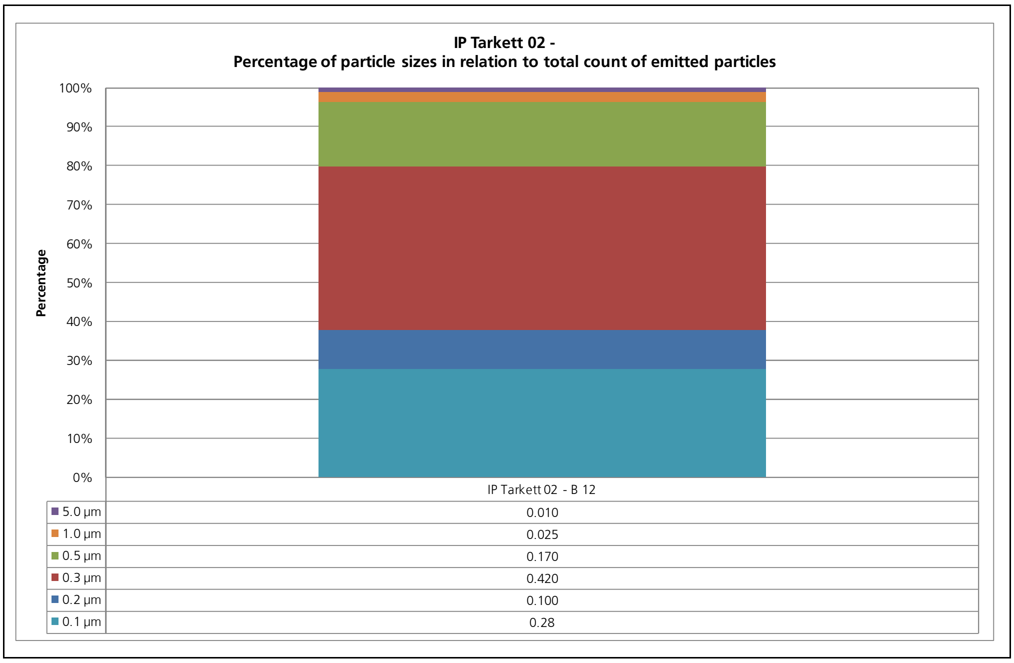

Figure 15 IP Tarkett 02– Size distribution of the emitted particles

Fraunhofer IPA

4.3.3 Classification

4.3.3.1 Method

In general, airborne particulate contamination is the main issue considered when assessing cleanroom suitability. The most important aspects of this are the size and concentration of airborne particles. Relevant standards state limiting values for the concentration of airborne particles in dependence upon particle size, as found in ISO 14644-1. This norm describes the quality of cleanrooms using Air Cleanliness Classes ranging from 1 to 9. The lowest class, Class 1, fulfills the highest requirements with regard to air cleanliness; the limiting value of particles permitted increases with each successive cleanroom class. Calculations can be made for limiting values of any particle size between 0.1 µm and 5.0 µm for all classes using the method for calculating permitted limiting values as described in ISO 14644-1. The norm states the maximum permitted number of particles of each size for the reference volume (in this case: 1 m^3).

The tests performed record particle emissions generated when tribological stress is applied to material pairings. The amounts of particles measured are dependent upon the material pairing concerned and the set of stress parameters applied. In order to better appreciate the differences, Fraunhofer IPA has developed a method which enables classifications to be made based on the measurement results obtained using the procedure stated in ISO 14644-1.

In accordance with the procedure laid down in ISO 14644-1 for determining the permitted particle concentration of different Air Cleanliness Classes, limiting values are ascertained for the given particle size classes taking the test conditions into consideration. The limiting value is obtained from the test volume of air (sampling time multiplied by the particle counter's constant volume flow of 28.3 l / min) and the permitted particle concentrations (particles / m^3) for the corresponding Air Cleanliness Class and particle size. A comparison of these limiting values with the total counts of emitted particles gives the classification figure for the test. The calculation method has been extended to include particles sized between 0.1 µm and 25.0 µm.
Care is to be taken when comparing the classification figures; consideration of the particle size in relation to the values and also of the set of parameters applied in the respective test.

Three repeat measurements are carried out on each material pairing for each set of parameters. The highest value classification figure obtained applies. This figure is used in the corresponding tables and diagrams.
The following tables show the classification figures obtained for the material pairing. The availability of classification figures for the various particle sizes depends upon the resolution of the optical particle counter used.

4.3.3.2 Overview of classification results

LOAD LEVEL	NORMAL FORCE	DETECTED PARTICLE SIZE					
		0.1 µm	0.2 µm	0.3 µm	0.5 µm	1.0 µm	5.0 µm
B 12	300 N	2.1	2.4	2.7	3.1	3.6	4.0
CLASSIFICATION RELEVANT TO DOCUMENTS							4

Figure 16 IP Tarkett 01: iQ Optima versus PA6 Nylon
Overview of classification value attained in accordance with ISO 14644-1

The level of particulate contamination emitted during application of tribological stress on the material pairing **iQ Optima versus PA6 Nylon** lies within the permissible values of the corresponding Air Cleanliness Classes
ISO Class 4 in accordance with ISO 14644-1.

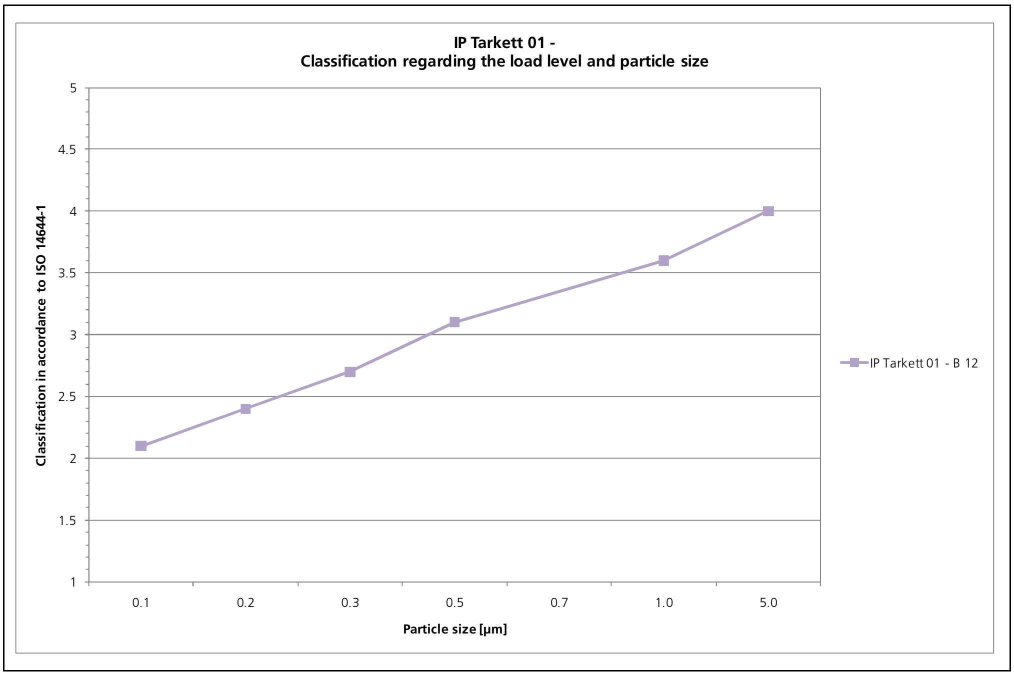

Figure 17 IP Tarkett 01: iQ Optima versus PA6 Nylon
Classification in accordance with ISO 14644-1 in dependence upon the particle size

LOAD LEVEL	NORMAL FORCE	DETECTED PARTICLE SIZE					
		0.1 µm	0.2 µm	0.3 µm	0.5 µm	1.0 µm	5.0 µm
B 12	300 N	2.5	3.0	3.3	3.3	3.1	3.9
CLASSIFICATION RELEVANT TO DOCUMENTS							4

Figure 18 IP Tarkett 02: iQ Granit versus PA6 Nylon
Overview of classification value attained in accordance with ISO 14644-1

The level of particulate contamination emitted during application of tribological stress on the material pairing **iQ Granit versus PA6 Nylon** lies within the permissible values of the corresponding Air Cleanliness Classes
ISO Class 4 in accordance with ISO 14644-1.

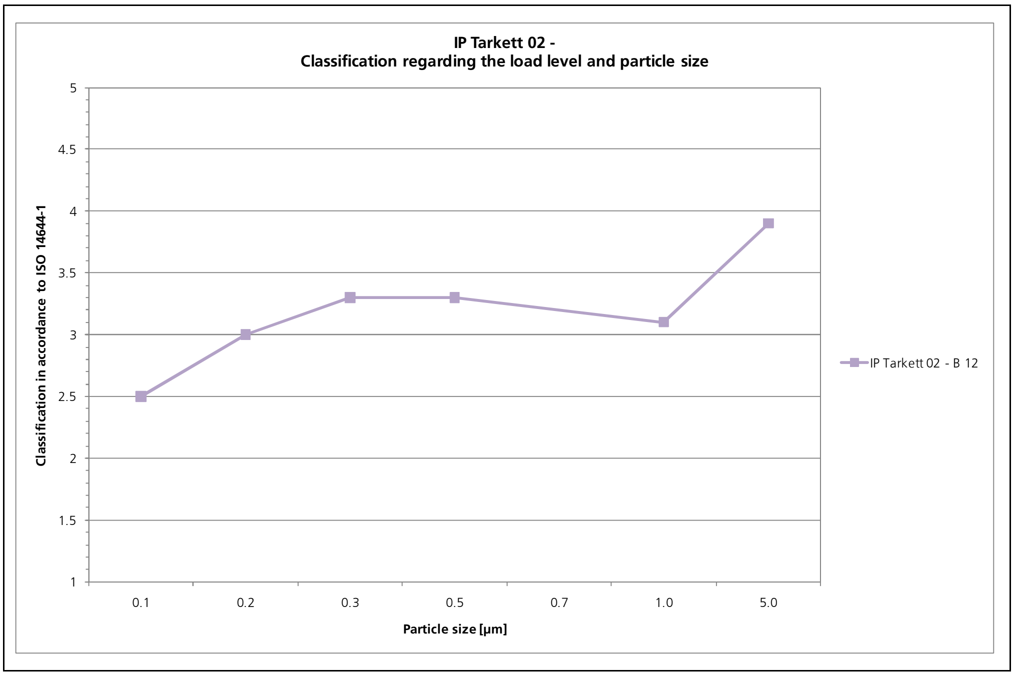

Figure 19 IP Tarkett 02: iQ Granit versus PA6 Nylon
Classification in accordance with ISO 14644-1 in dependence upon the particle size

5 Outgassing tests under thermal stress

5.1 Material samples

The material samples required for the test were supplied by the customer. Tests were carried out on the following materials.

TESTED MATERIALS OUTGASSING	TEMPERATURES TESTED		TESTED FOR
NAME	23°C/73 °F	90°C/194 °F	
iQ Optima	x	x	TVOC
iQ Granit	x	x	TVOC

Figure 20 Material samples tested for outgassing

Figure 21 Materials tested – (partial samples for outgassing measurements): iQ Optima

Figure 22 Materials tested – (partial samples for outgassing measurements): iQ Granit

Solid samples were wrapped in aluminum foil prior to dispatch and sealed in a polypropylene bag to prevent the samples from altering in any way during transport.

In accordance with their preparation or application, reactive samples were stored open in the cleanroom under constant climatic conditions (relative humidity, temperature) for a period of 30 days. This complies with the instructions laid down in VDI 2083 Part 14 for the airing time for reactive samples. This achieved a stable outgassing phase and enabled test results obtained from different samples to be compared with one another.

On arrival at the Fraunhofer IPA, the samples were prepared for the outgassing tests. The weight, volume and active surface area of the partial sample were then ascertained. One identical sample was taken for the otugassing test, first at 23 °C without thermal stress and afterwards at 90 °C. The samples were prepared in outgassing-free Petri dishes made of borosilicate glass.

DESCRIPTION OF PARTIAL SAMPLES		PROCEDURE			
NAME	TEST AT	MEASURED SURFACE	MASS	SAMPLE PREPARATION	TESTED ON
iQ Optima	23°C/73 °F	0.001 m²	3.39 g	10.08.2011	12.09.2011
	90°C/194 °F	0.001 m²	3.39 g	10.08.2011	12.09.2011
iQ Granit	23°C/73 °F	0.001 m²	3.69 g	10.08.2011	12.09.2011
	90°C/194 °F	0.001 m²	3.69 g	10.08.2011	12.09.2011

Figure 23 Partial samples for outgassing tests – determination of sample characteristics

5.2 Outgassing test procedure

The tests were carried out in compliance with the established procedure developed by the industrial alliance "Cleanroom Suitable Materials (CSM)".
The purge-and-trap thermodesorption method was implemented with subsequent analysis using gas chromatography and mass spectrometry (TD-GC/MS).
The various analysis steps are depicted in the following diagram.

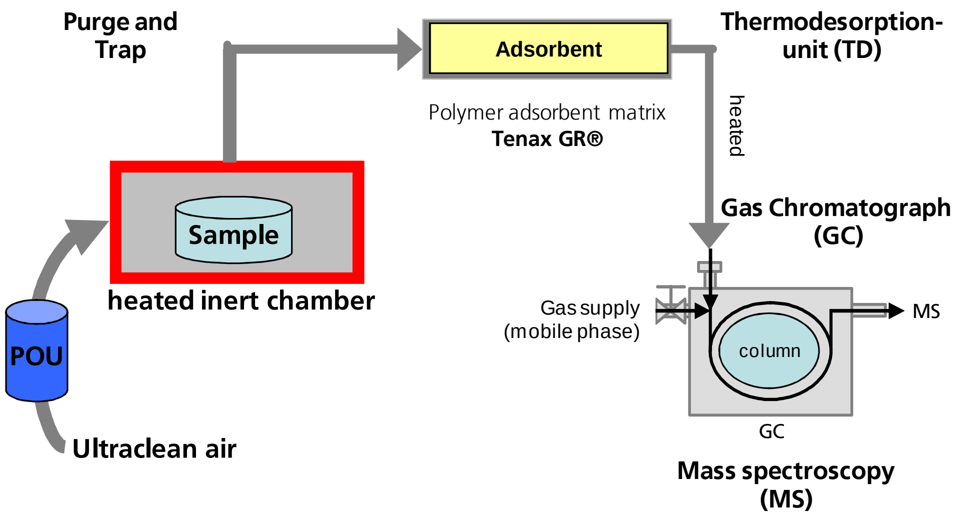

Figure 24 Outgassing tests – overview of analysis steps

This procedure is suitable for testing material samples for the outgassing of **V**olatile **O**rganic **C**ompounds (VOC).

5.2.1 Sampling chamber

Chamber used: Markes Micro Chamber/Thermal Extractor µCTE

Manufacturer: Markes International Limited, UK

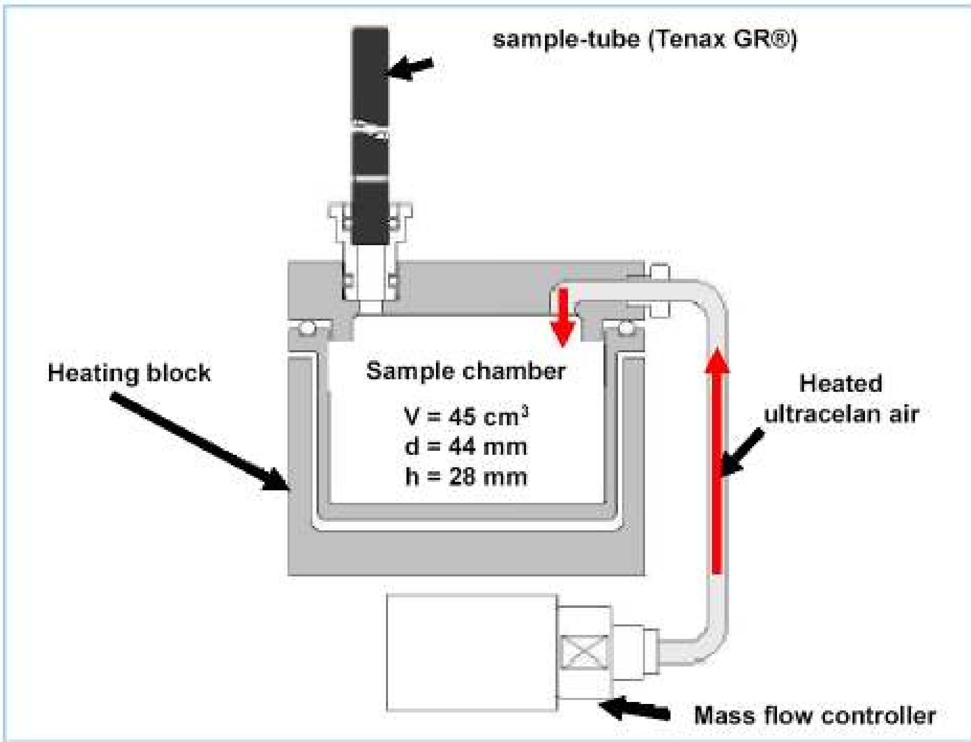

Figure 25 Outgassing test – diagram of sampling chamber used

The partial samples were placed in a heated chamber. The CSM method specifies test temperatures of **23 °C and 90 °C**. Tests were performed to determine **VOC emissions**. A cover ring guides the air onto the sample surface.

Figure 26 Outgassing test – cover ring

During the test, each partial sample was heated to the test temperature which caused it to emit volatile substances (outgassing). By introducing an inert, i.e. chemically non-reactive rinsing gas (purging), the substances present in the chamber were displaced and guided into an **adsorber tube**. The substances were "trapped" by the adsorbent material present in the tube and the (now re-purified) rinsing gas exited at the outlet of the tube.

On completion of the test, all substances emitted by the sample were fixed in adsorbed form in the polymer matrix in the tube. The tube was then sealed and transferred for analysis. The adsorber tube utilized in the tests was a stainless steel tube 90 mm long and 6.35 mm in diameter containing 200 mg Tenax GR®. The polymer adsorber matrix Tenax GR® is suitable for trapping C6 to C16 hydrocarbons. This equates with TVOC tests = Total Volatile Organic Compounds = absolute quantity of volatile organic compounds.

Ultra-pure air was used as a **rinsing gas** and was additionally filtered by an active charcoal filter before entering the chamber. **Before commencing the tests**, the chamber **was rinsed for 15 min** in order to remove any reactive gases which may have been present. The rinsing gas was fed through the chamber at a rate of 100 ml/min, i.e. the complete chamber volume was replaced approx 2.2 times per minute. By rinsing the chamber continuously, a balance between substances still contained in the sample and substances already in the gas phase can be avoided. This equates with conditions experienced by components in a cleanroom in reality.

After the 15 min pre-rinse, the standard **sampling time** of 60 min at 23° C/1 min at 90 °C was used. The carrier gas exiting from the chamber which contained the VOCs to be detected was then fed into the adsorber tube.

To determine the **blank value of the system**, before commencing the tests the complete sampling procedure was carried out without the presence of a sample in the chamber. This meant that the only variable in the entire analysis was the introduction of a sample into the chamber. The blank values of the various substances detected were then subtracted from the subsequent sample values obtained in order to ascertain absolute values for the components outgassed.

General parameters used:

Temperature of the test chamber	[°C]	23	90
Duration of preconditioning	[min]	6	6
Duration of sampling	[min]	60	10
Flow rate of carrier gas	[ml/min]	100	100

Figure 27 General parameters used for the test chamber procedure

5.2.2 Analysis devices

The adsorber tube containing all the outgassed substances was then analyzed using a combined TD-GC/MS unit (thermodesorption gas chromatography in combination with mass spectroscopy).

The tubes were heated for a short period of time to enable the substances trapped in the adsorber matrix to be released (thermodesorption). These were then guided into the measuring unit. Gas chromatography was used to separate the substances present in the mixture. The individual substances reached the attached mass spectrometer at different moments in time and were detected.

The following measuring devices were implemented in the test:

GC/MS:	a) GC: PerkinElmer Clarus 600 b) MS: PerkinElmer Clarus 600T
Thermodesorber:	PerkinElmer Turbomatrix ATD 650
Column:	PerkinElmer Elite 5 - MS, l = 60 m, ID = 0.25 mm, film = 0.25 µm

5.3 Outgassing results

The following results were obtained from the tests performed.

- Determination of the qualitative VOC spectrum
- Quantitative determination of the components outgassed (based on hexadecane equivalents),
- Quantitative determination of TVOC values (based on hexadecane equivalents),
- Relevant comments concerning contaminants critical to cleanroom environments
- Classification in accordance with ISO 14644-8

The **qualitative VOC spectra** were ascertained based on the chromatograms obtained from the gas chromatography tests and the mass spectrograms from the mass spectrometer.
Each separate substance possesses a characteristic retention period in chromatographs as well as a characteristic fragmentation in the mass spectrometer. This information is filed in appropriate databases. Substances can be identified by comparing the retention times and fragments observed in the test with those listed in the databases. Each chromatogram shows the chronological order of each substance detected.

Note:
Due to test-related fluctuations, the information obtained concerning the identities of the substances detected is associated with a certain degree of uncertainty. As a rule, the substance named is the one which has the highest probability of being the substance which was outgassed. If a detected substance cannot be ascertained, the substance is labeled **"n. i."** (=not identifiable) in the table. If a substance cannot be identified with certainty, the substance is marked in the table with a question mark (**"?"**).

By using a reference substance (e.g. hexadecane) in a known concentration parallel to the tests, the concentration of each substance identified can be ascertained as a toluol equivalent (**quantitative analysis based on hexadecane equivalents**). The quantities of substances detected have been documented in relation to the mass, volume and weight of the partial samples.

Within the scope of the industrial alliance Cleanroom Suitable materials (CSM) founded by the Fraunhofer IPA, a procedure has been developed to **classify** materials in accordance with ISO 14644-8 based on their outgassing products at defined temperatures.
ISO 14644-8 enables cleanrooms to be classified based on the concentration of specific substance classes present in the cleanroom air (ISO-AMC classes). In the process, the classes are calculated as a logarithm of the corresponding sub-

stance class, e.g. organic contaminants, in units of grams/cubic meter cleanroom air. By using logarithmic calculus, the ISO-AMC classes feature all negative values with the result that permissible concentrations decrease as quantities increase. Therefore, ISO-AMC Class -12 equates to a limiting value 1000 times lower than ISO-AMC Class -9.
The purpose of the outgassing tests carried out on a material using the CSM procedure is firstly to identify and measure the quantity of outgassing substances emitted from a sample of a defined geometry over a defined period of time at a specific temperature. These values are then classified into certain substance classes as stated in ISO 14644-8. Via the known test parameters of sample surface area and testing time, the quantity of outgassing substances can be converted to the standard SI reference values of 1 m^2 and 1 s. The outgassing quantity is then related to the reference value of 1 m^3 as laid down in ISO 14644-8 and compared with the limiting value, enabling a classification figure to be awarded.
The classification figure obtained states that 1 m^2 of the test material emits a quantity of the given substance class over the space of 1 s at the test temperature which, when related to the reference volume of 1 m^3, is within the permissible concentration for this class as laid down in ISO 14644-8. To do this, the reference volume is to be viewed statistically and may not be exchanged, thus enabling the values obtained to be converted to real cleanrooms without any problem.

According to the detection limit of the measurement system of 1 ng absolute mass, the following minimal outgassing rate (specific emission rate SER) can be determined:

Measurement temperature	[°C]	23	90
Duration of sampling	[min]	60	10
SER Detection limit	[g/m²s]	2.8E-10	1.7E-09

Figure 28 General parameters used for the test chamber procedure

If no contaminant could be detected, it is marked **“not detectable”** according to the applied detection limit of the measurement system.

5.3.1 General explanation of chromatograms

The assessment of the test results is based on the chromatograms obtained. The most important elements of a chromatogram are explained again here by way of example.

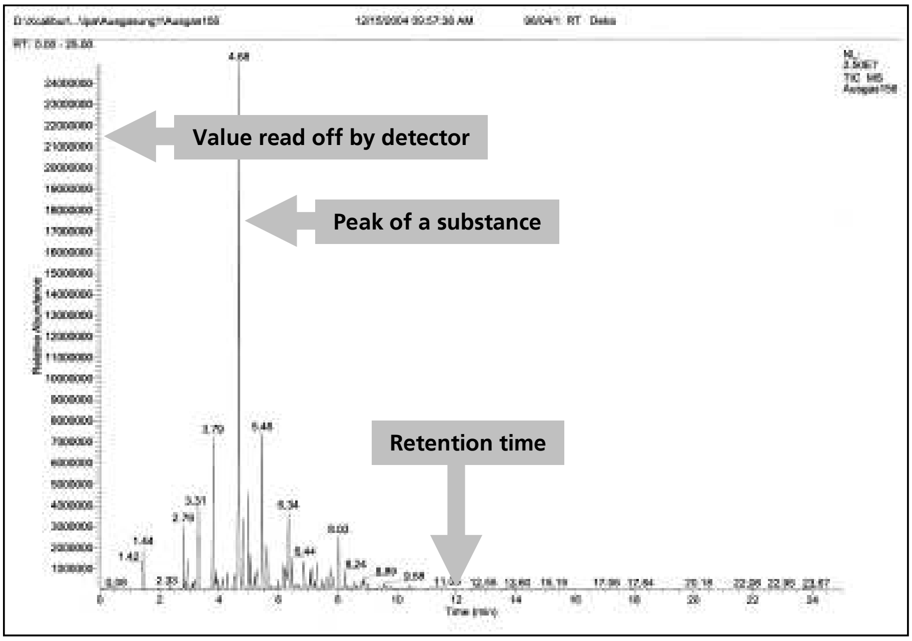

Figure 29 Example of a chromatogram – details of main values

- Peak: Indicates the detection of a substance
- Retention time: Characteristic time required by a substance to pass through the chromatograph
- Value read off: Signal strength detected

Note:
Due to the confidentiality of the test results, it is to be noted that it may be possible to reach conclusions from the following chromatograms about the substance composition ("recipe") of the test samples/materials.

The following figures show the chromatograms obtained from the various materials at the specified test temperatures and also the substances identified.

5.3.2 iQ Optima

5.3.2.1 Test at 23 °C

The following chromatograms were obtained:

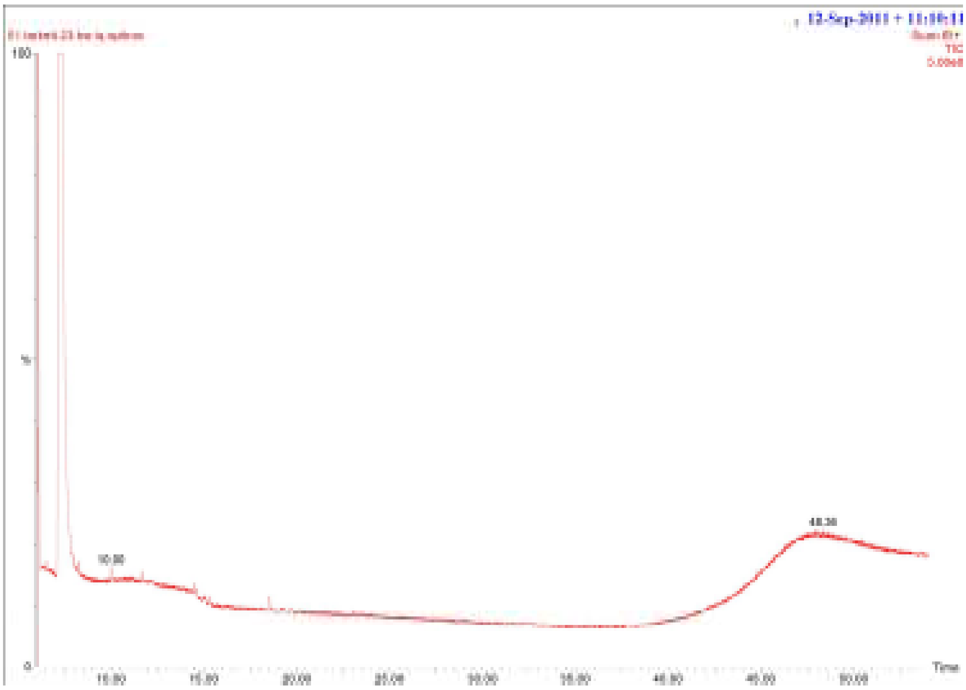

Figure 30 Outgassing test: iQ Optima: blank value chromatogram at 23 °C

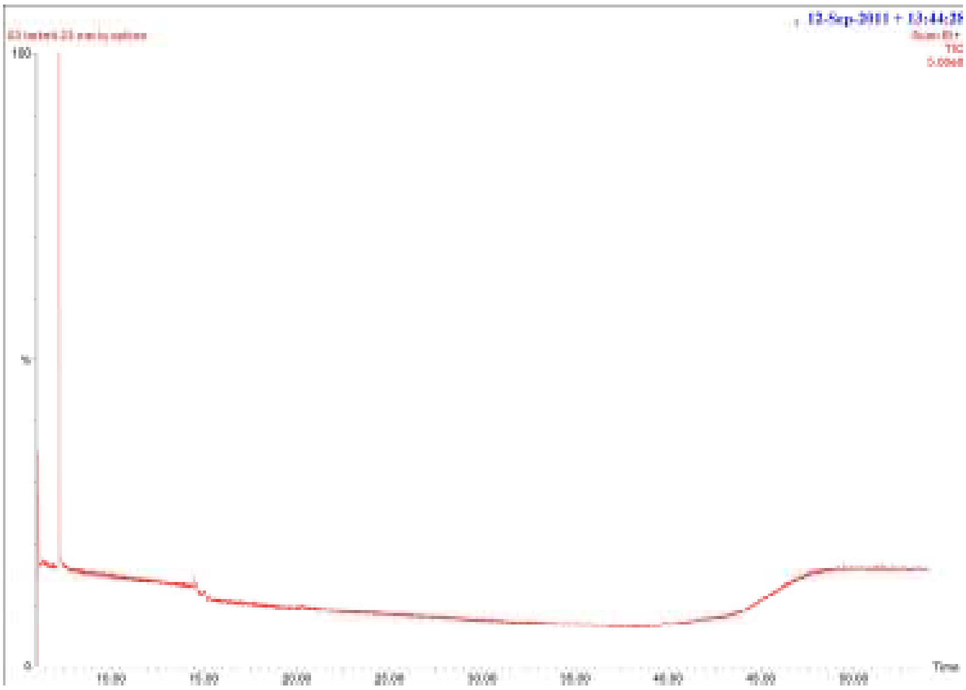

Figure 31 Outgassing test: iQ Optima: chromatogram of sample at 23 °C

No peaks could be identified which indicated the outgassing of volatile compounds.

Remarks about contaminant groups relevant to cleanrooms:
No outgassing of the following groups of components could be ascertained at 23 °C:

- Amines
- Organophosphates
- Siloxanes
- Phthalates
- SVOC

5.3.2.2 Test at 90 °C

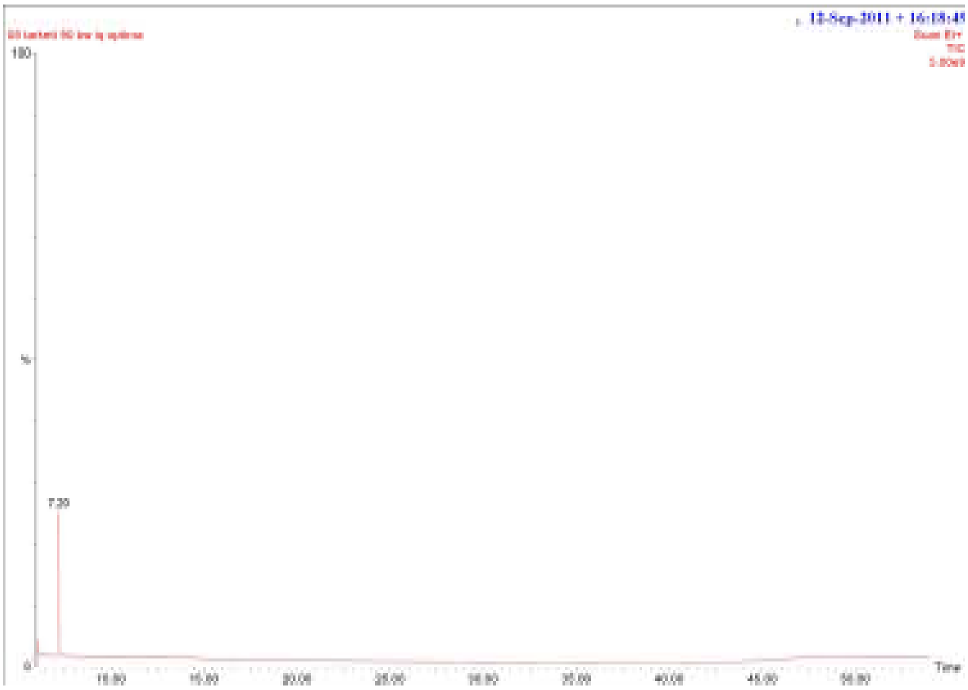

Figure 32 Outgassing test: iQ Optima: blank value chromatogram at 90 °C

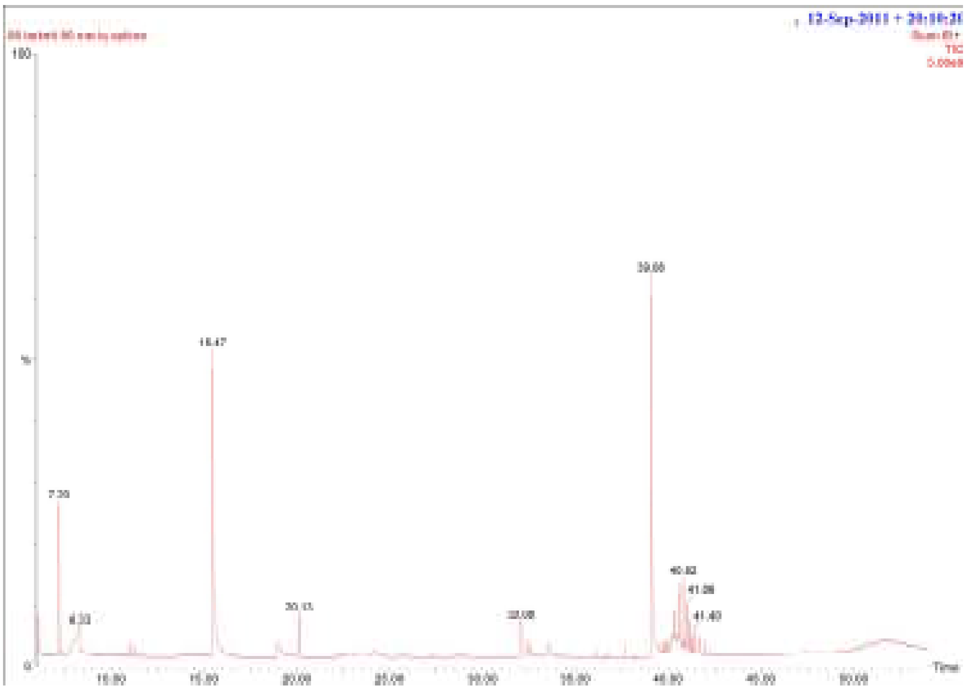

Figure 33 Outgassing test: iQ Optima: chromatogram of sample at 90 °C

Various peaks could be identified which indicated the outgassing of volatile compounds. The following substances could be identified. The blank value of the system is already subtracted if present.

Substance	CAS-No.	RT	Value
TVOC (C6 to C 16)		[min]	Mass [ng]
2-Propeonic Acid	79-10-7	8,28	299
Propanoic acid, 2-methyl-, 1-methylethyl ester	617-50-5	11,07	13
Hexanal	66-25-1	11,28	13
Cyclotrisiloxane, hexamethyl-	541-05-9	11,73	5
Cyclohexanone	108-94-1	15,48	735
Benzaldehyde	100-52-7	18,99	92
Alkane		20,13	61
n.i.		24,21	19
Nonanal	124-19-6	25,48	13
Hexanoic acid, 2-ethyl-	149-57-5	25,85	17
Decanal	112-31-2	29,00	9
cis-Hexahydrophthalide?	6939-71-5	32,09	79
Alkane		32,43	18
Alkane		32,58	10
Alkanone		33,57	49
1-Oxaspiro(4,5)decan-2-one	699-61-6	34,42	12
Alkane		36,14	14
Butylated Hydroxytoluene	128-37-0	36,80	9
n.i.		37,67	14
		Total	**1482**

Figure 34 iQ Optima
Quantitative analysis of volatile organic compounds VOC at 90 °C and 10 min sampling time

Fraunhofer IPA

In relation to the surface area, volume and mass of the test sample at a temperature of 90 °C and a normed outgassing duration of 1 s, the following values were obtained for the outgassing substances detected:

	Thickness [m]	Surface [m^2]	Volume [m^3]	Mass [g]
	2,00E-03	1,00E-03	2,00E-06	3,39E+00
		Concentration with regards to		
Substance	CAS-No.	Surface	Volume	Mass
TVOC (C6 to C 16)		[g/m2s]	[g/m3s]	[g/gs]
2-Propeonic Acid	79-10-7	5,0E-07	2,5E-04	1,5E-10
Propanoic acid, 2-methyl-, 1-methylethyl ester	617-50-5	2,2E-08	1,1E-05	6,6E-12
Hexanal	66-25-1	2,1E-08	1,1E-05	6,3E-12
Cyclotrisiloxane, hexamethyl-	541-05-9	7,9E-09	4,0E-06	2,3E-12
Cyclohexanone	108-94-1	1,2E-06	6,1E-04	3,6E-10
Benzaldehyde	100-52-7	1,5E-07	7,7E-05	4,5E-11
Alkane		1,0E-07	5,1E-05	3,0E-11
n.i.		3,1E-08	1,6E-05	9,2E-12
Nonanal	124-19-6	2,2E-08	1,1E-05	6,6E-12
Hexanoic acid, 2-ethyl-	149-57-5	2,9E-08	1,5E-05	8,6E-12
Decanal	112-31-2	1,5E-08	7,7E-06	4,6E-12
cis-Hexahydrophthalide?	6939-71-5	1,3E-07	6,5E-05	3,9E-11
Alkane		3,0E-08	1,5E-05	8,7E-12
Alkane		1,6E-08	8,0E-06	4,7E-12
Alkanone		8,2E-08	4,1E-05	2,4E-11
1-Oxaspiro(4,5)decan-2-one	699-61-6	2,0E-08	9,9E-06	5,8E-12
Alkane		2,4E-08	1,2E-05	7,0E-12
Butylated Hydroxytoluene	128-37-0	1,6E-08	7,8E-06	4,6E-12
n.i.		2,4E-08	1,2E-05	7,1E-12
	Total:	**2,5E-06**	**1,2E-03**	**7,3E-10**

Figure 35 iQ Optima
Mass of outgassing components in relation to the surface area and mass of the partial sample at 90 °C and a normed outgassing duration of 1 s

Remarks about contaminant groups especially relevant to cleanrooms:
No outgassings of the following groups of components could be ascertained at 90 °:

- Amines
- Organophosphates
- Phthalates

In relation to the surface area, volume and mass of the test sample at a temperature of 90 °C and a normed outgassing duration of 1 s, the following values were obtained for critical contaminant groups:

SVOC:

		Concentration with regards to		
Substance	CAS-No.	Surface	Volume	Mass
SVOC (>C16)		[g/m2s]	[g/m3s]	[g/gs]
Benzophenone	119-61-9	1,5E-06	7,4E-04	4,4E-10
Heptadecane	629-78-7	1,8E-08	9,2E-06	5,4E-12
1-Decanol, 2-methyl-	18675-24-6	1,3E-08	6,3E-06	3,7E-12
Alkane		1,2E-08	5,9E-06	3,5E-12
2,3-Dimethyldecane	17312-44-6	1,7E-07	8,7E-05	5,2E-11
Alkanol		1,8E-07	9,2E-05	5,4E-11
Dichloroacetic acid, nonyl ester	83004-99-3	1,1E-07	5,3E-05	3,1E-11
Trichloroacetic acid, tridecyl ester	74339-51-8	6,3E-08	3,2E-05	1,9E-11
Dichloroacetic acid, nonyl ester??	83004-99-3	4,4E-08	2,2E-05	1,3E-11
Alkane		5,5E-08	2,8E-05	1,6E-11
Alkanole		4,0E-08	2,0E-05	1,2E-11
Alkanole		1,7E-08	8,5E-06	5,0E-12
Alkanole		1,2E-08	6,2E-06	3,6E-12
	Total:	**2,2E-06**	**1,1E-03**	**6,5E-10**

Figure 36 iQ Optima
Sum of relevant critical contaminant groups at 90 °C and a normed outgassing duration of 1 s

Siloxanes:

		Concentration with regards to		
Substance	CAS-No.	Surface	Volume	Mass
Siloxanes		[g/m2s]	[g/m3s]	[g/gs]
Cyclotrisiloxane, hexamethyl-	541-05-9	7,9E-09	4,0E-06	2,3E-12
	Total:	**7,9E-09**	**4,0E-06**	**2,3E-12**

Figure 37 iQ Optima
Sum of relevant critical contaminant groups at 90 °C and a normed outgassing duration of 1 s

5.3.2.3 Material Classification

PARAMETER			SUBSTANCE GROUP TESTED	OUTGASSING MASS		CLASSIFICATION IN ACCORDANCE TO ISO 14644-8
SAMPLE AREA	TESTING TIME	TEMPERATURE		DETECTED	NORMALIZED	
A/m²	*t*/s	*T*/°C		*m*/g	g/m²s	ISO-AMC (or)
0.001	3600	23	TVOC	<1E-09	<2.8E-10	-9.6

Figure 38 iQ Optima
Classification results in accordance with ISO 14644-8 at the test temperatures

At a temperature of **23 °C** and an assumed sample surface area of 1 m^2, IQ Optima emitted a quantity of TVOC over a period of 1 s which did not exceed the stated limiting value for **Class -9.6** according to ISO 14644-8 in a reference volume of 1 m^3.

5.3.3 iQ Granit

5.3.3.1 Test at 23 °C

The following chromatograms were obtained:

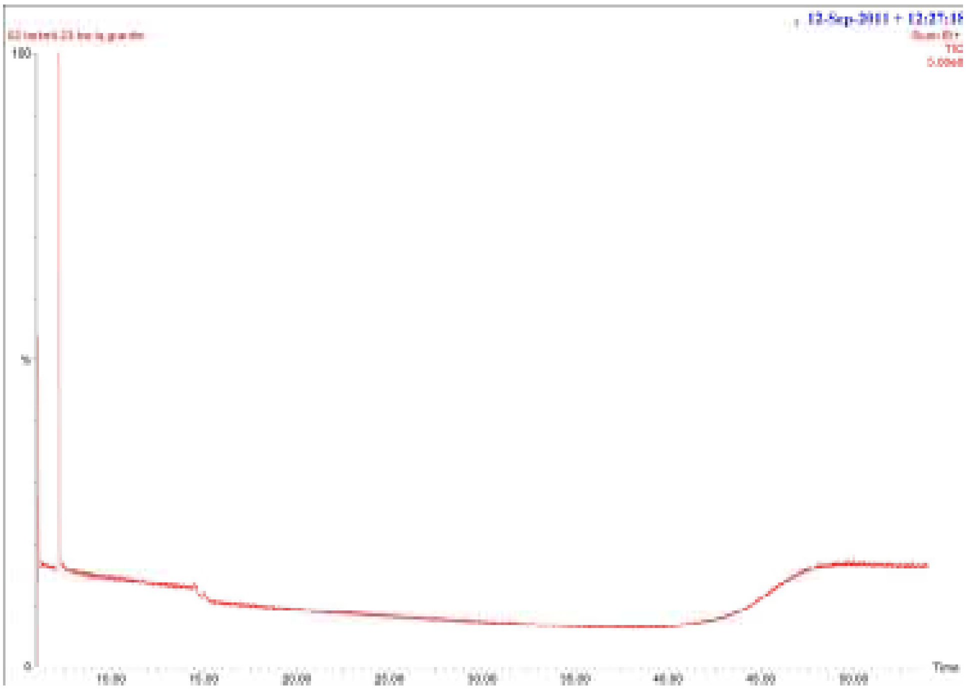

Figure 39 Outgassing test: iQ Granit: blank value chromatogram at 23 °C

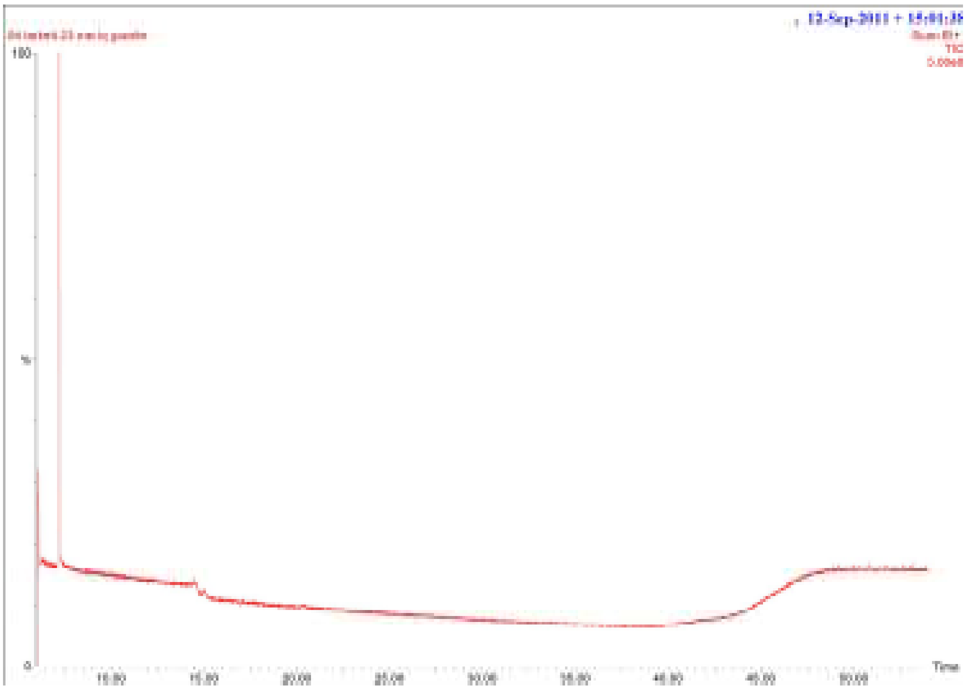

Figure 40 Outgassing test: iQ Granit: chromatogram of sample at 23 °C

No peaks could be identified which indicated the outgassing of volatile compounds.

Remarks about contaminant groups relevant to cleanrooms:
No outgassing of the following groups of components could be ascertained at 23 °C:

- Amines
- Organophosphates
- Siloxanes
- Phthalates
- SVOC

5.3.3.2 Test at 90 °C

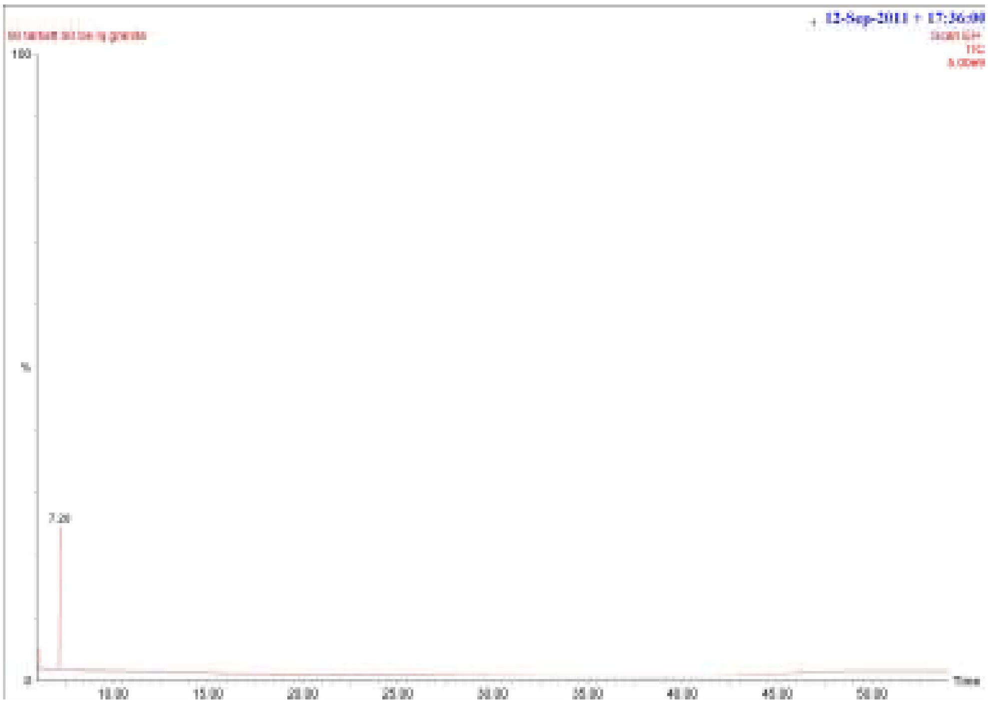

Figure 41 Outgassing test: iQ Granit: blank value chromatogram at 90 °C

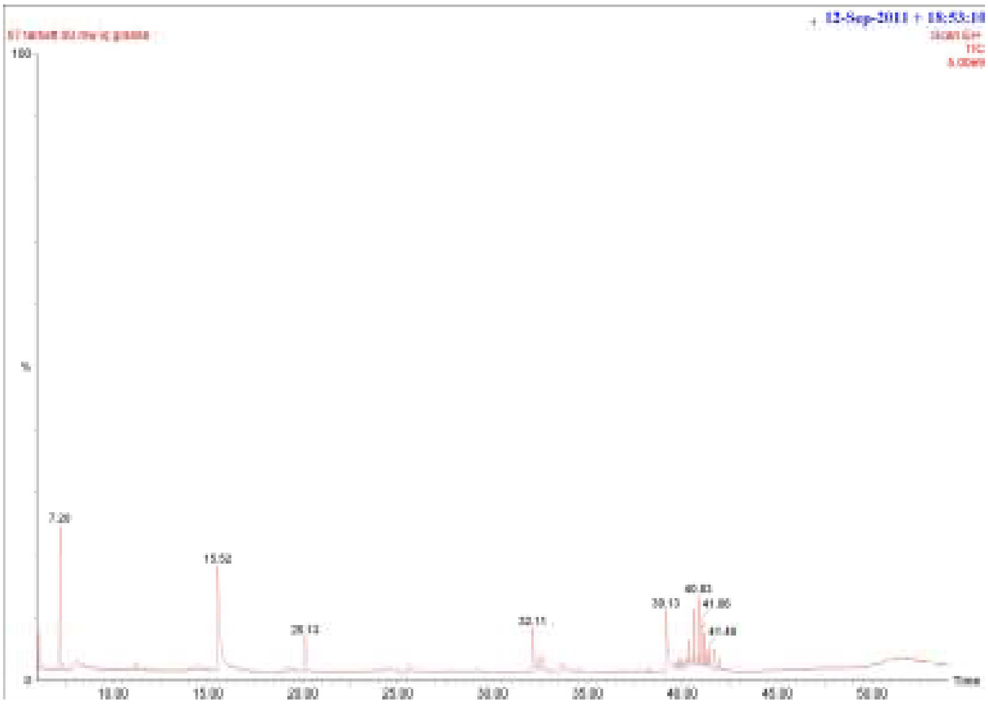

Figure 42 Outgassing test: iQ Granit: chromatogram of sample at 90 °C

Various peaks could be identified which indicated the outgassing of volatile compounds. The following substances could be identified. The blank value of the system is already subtracted if present.

Substance	CAS-No.	RT	Value
TVOC (C6 to C 16)		[min]	Mass [ng]
2-Propeonic Acid	79-10-7	8,12	102
Propanoic acid, 2-methyl-, 1-methylethyl ester	617-50-5	11,13	10
Hexanal	66-25-1	11,36	7
Cyclotrisiloxane, hexamethyl-	541-05-9	11,78	2
Cyclohexanone	108-94-1	15,54	512
Benzaldehyde	100-52-7	19,17	15
Alkane		20,13	47
n.i.		24,41	9
Nonanal	124-19-6	25,60	17
Decanal	112-31-2	29,06	9
cis-Hexahydrophthalide?	6939-71-5	32,12	96
Alkane		32,44	22
Alkane		32,58	21
Alkanone		33,63	34
1-Oxaspiro(4,5)decan-2-one	699-61-6	34,51	9
n.i.		37,72	9
n.i.		38,17	10
		Total	**932**

Figure 43 iQ Granit
Quantitative analysis of volatile organic compounds VOC at 90 °C and 10 min sampling time

In relation to the surface area, volume and mass of the test sample at a temperature of 90 °C and a normed outgassing duration of 1 s, the following values were obtained for the outgassing substances detected:

	Thickness [m]	Surface [m^2]	Volume [m^3]	Mass [g]
	2,00E-03	1,00E-03	2,00E-06	3,69E+00
		Concentration with regards to		
Substance	CAS-No.	Surface	Volume	Mass
TVOC (C6 to C 16)		[g/m2s]	[g/m3s]	[g/gs]
2-Propeonic Acid	79-10-7	1,7E-07	8,5E-05	4,6E-11
Propanoic acid, 2-methyl-, 1-methylethyl ester	617-50-5	1,6E-08	8,0E-06	4,3E-12
Hexanal	66-25-1	1,2E-08	5,9E-06	3,2E-12
Cyclotrisiloxane, hexamethyl-	541-05-9	4,1E-09	2,0E-06	1,1E-12
Cyclohexanone	108-94-1	8,5E-07	4,3E-04	2,3E-10
Benzaldehyde	100-52-7	2,5E-08	1,2E-05	6,7E-12
Alkane		7,9E-08	4,0E-05	2,1E-11
n.i.		1,6E-08	7,8E-06	4,2E-12
Nonanal	124-19-6	2,9E-08	1,4E-05	7,8E-12
Decanal	112-31-2	1,5E-08	7,7E-06	4,2E-12
cis-Hexahydrophthalide?	6939-71-5	1,6E-07	8,0E-05	4,3E-11
Alkane		3,6E-08	1,8E-05	9,9E-12
Alkane		3,6E-08	1,8E-05	9,6E-12
Alkanone		5,6E-08	2,8E-05	1,5E-11
1-Oxaspiro(4,5)decan-2-one	699-61-6	1,5E-08	7,5E-06	4,1E-12
n.i.		1,5E-08	7,6E-06	4,1E-12
n.i.		1,6E-08	7,9E-06	4,3E-12
	Total:	**1,6E-06**	**7,8E-04**	**4,2E-10**

Figure 44 iQ Granit
Mass of outgassing components in relation to the surface area and mass of the partial sample at 90 °C and a normed outgassing duration of 1 s

Remarks about contaminant groups especially relevant to cleanrooms:
No outgassings of the following groups of components could be ascertained at 90 °:

- Amines
- Organophosphates
- Phthalates

In relation to the surface area, volume and mass of the test sample at a temperature of 90 °C and a normed outgassing duration of 1 s, the following values were obtained for critical contaminant groups:

SVOC:

		Concentration with regards to		
Substance	CAS-No.	Surface	Volume	Mass
SVOC (>C16)		[g/m2s]	[g/m3s]	[g/gs]
Benzophenone	119-61-9	3,8E-07	1,9E-04	1,0E-10
Heptadecane	629-78-7	9,9E-09	5,0E-06	2,7E-12
1-Decanol, 2-methyl-	18675-24-6	1,0E-08	5,0E-06	2,7E-12
Alkane		7,3E-09	3,6E-06	2,0E-12
Alkanole		1,3E-08	6,7E-06	3,7E-12
2,3-Dimethyldecane	17312-44-6	4,5E-08	2,2E-05	1,2E-11
Alkanol		1,2E-07	5,8E-05	3,1E-11
Dichloroacetic acid, nonyl ester	83004-99-3	1,0E-07	5,2E-05	2,8E-11
Trichloroacetic acid, tridecyl ester	74339-51-8	6,9E-08	3,4E-05	1,9E-11
Dichloroacetic acid, nonyl ester??	83004-99-3	4,5E-08	2,2E-05	1,2E-11
Alkane		6,3E-08	3,1E-05	1,7E-11
Alkanole		4,7E-08	2,4E-05	1,3E-11
Alkanole		2,1E-08	1,0E-05	5,7E-12
Alkanole		1,2E-08	5,8E-06	3,1E-12
	Total:	**9,4E-07**	**4,7E-04**	**2,5E-10**

Figure 45 iQ Granit
Sum of relevant critical contaminant groups at 90 °C and a normed outgassing duration of 1 s

Siloxanes:

		Concentration with regards to		
Substance	CAS-No.	Surface	Volume	Mass
Siloxanes		[g/m2s]	[g/m3s]	[g/gs]
Cyclotrisiloxane, hexamethyl-	541-05-9	4,1E-09	2,0E-06	1,1E-12
	Total:	**4,1E-09**	**2,0E-06**	**1,1E-12**

Figure 46 iQ Granit
Sum of relevant critical contaminant groups at 90 °C and a normed outgassing duration of 1 s

5.3.3.3 Material Classification

PARAMETER			SUBSTANCE GROUP TESTED	OUTGASSING MASS		CLASSIFICATION IN ACCORDANCE TO ISO 14644-8
SAMPLE AREA	TESTING TIME	TEMPERATURE		DETECTED	NORMALIZED	
A/m²	*t*/s	*T*/°C		*m*/g	g/m²s	ISO-AMC (or)
0.001	3600	23	TVOC	<1E-09	<2.8E-10	-9.6

Figure 47 iQ Granit
Classification results in accordance with ISO 14644-8 at the test temperatures

At a temperature of **23 °C** and an assumed sample surface area of 1 m^2, IQ Granit emitted a quantity of TVOC over a period of 1 s which did not exceed the stated limiting value for **Class -9.6** according to ISO 14644-8 in a reference volume of 1 m^3.

6 Comparison of Classifications of Airborne Particulate Contamination

The table below compares the limiting values for determining air cleanliness classes laid down in the international norm **ISO 14644-1** with the corresponding values given in the **EC-GMP Guideline Volume 4, Annex 1** and the American norm **US Federal Standard 209E** (since retracted). The comparison has been made for the particle size channels stated in ISO 14644-1.
The limiting values listed apply to the reference volumes of **1 m³** and **1 cft** (1 cubic foot = 0.0283 m³).

Nomenklatur				Maximal zulässige Partikelzahl gem. DIN EN ISO 14644-1 entsprechend verschiedener Partikelgrößen											
DIN EN ISO 14644-1	EG-GMP "at rest"	EG-GMP "in operation"	US Fed. Standard 209E*	0,1 µm		0,2 µm		0,3 µm		0,5 µm		1,0 µm		5,0 µm	
				pro m³	pro cbf	pro m³	pro cbf	pro m³	pro cbf	pro m³	pro cbf	pro m³	pro cbf	pro m³	pro cbf
1				10	0,3	2	0,1								
2				100	3	24	1	10	0,3	4	0,1				
3				1.000	30	237	7	102	3	35	1	8	0,2		
			1	1.240	35	265	8	106	3	35	1				
4				10.000	300	2.370	67	1.020	29	352	9,9	83	2		
			10	12.000	340	2.650	75	1.060	29	353	10				
5				100.000	2.833	23.700	671	10.200	289	3.520	100	832	24	29	0,8
	A	A								3.520	100			20	0,6
	B									3.520	100			29	0,8
			100			26.500	750	10.600	300	3.530	100				
6				1.000.000	28.329	237.000	6.710	102.000	2.890	35.200	997	8.320	235	293	8
			1.000							35.300	1.000			247	7
7										352.000	9.972	83.200	2.357	2.930	83
	C									352.000	9.972			2.900	82
		B								352.000	9.972			2.900	82
			10.000							353.000	10.000			2.470	70
8										3.520.000	99.716	832.000	23.569	29.300	830
	D									3.520.000	99.716			29.000	821
		C								3.520.000	99.716			29.000	821
			100.000							3.530.000	100.000			24.700	700
9										35.200.000	997.167	8.320.000	235.694	293.000	8.300

Figure 48 Overview of limiting values for airborne particles per m³ or cft for the norms ISO 14644-1, EC-GMP-Guideline Volume 4, Annex 1 and US Federal Standard 209E (since retracted)

Biotic airborne particle levels, for which limiting values are stipulated in the EG GMP Guideline Volume 4, Annex 1, were not investigated at the Fraunhofer IPA as part of these qualification tests. As each manufacturing environment possesses its own individual spectrum of microorganisms, it is impossible to carry out such tests in a reference laboratory. Rather, they can only be performed directly in the actual manufacturing environment concerned. The individual spectrum of microorganisms present is primarily influenced by production sequences, the prevailing environment and by the operating staff working in each specific manufacturing area.