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Measurement Report (Draft Version)

Cleanroom suitability tests on materials used by NORD RESINE SPA

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Fraunhofer IPA

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1 Introduction and objectives

NORD RESINE SPA manufactures commercial floors which are especially durable, hygienic and easy to clean and care for.

The commercial floors are used for specific applications in a wide range of market segments such as healthcare where high standards of air quality, sterility and cleanliness must be fulfilled.

To secure the market position of NORD RESINE SPA, the company wishes to extend the application of its systems to clean manufacturing areas. To do this, it is crucial to gain information about the cleanroom suitability and cleanliness suitability of the floors.

2 Materials tested

TESTED MATERIALS
NAME (FULL LENGTH)
NORDPUR SW Manufacturing date: 11.04.2014 Batch number: A: 14031306; B: 14031113

Figure 1 Overview of materials tested

3 Overview of results

TESTED MATERIALS	PERFORMED TESTS				
NAME (FULL LENGTH)	PARTICLE EMISSION	VOC OUTGASSING	CHEMICAL RESISTANCE	BIOLOGICAL RESISTANCE	MICROBICIDITY
NORDPUR SW	x				
PA6 Nylon	x				

Figure 2 Overview of materials tested

PARTICLE EMISSION (CSM CLASSIFICATION ACCORDING TO ISO 14644-1)			
MATERIAL PAIRING			ISO CLASS
SPECIMEN	COUNTER SPECIMEN	LUBRICANT	
NORDPUR SW	PA6 Nylon	(none)	5

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.

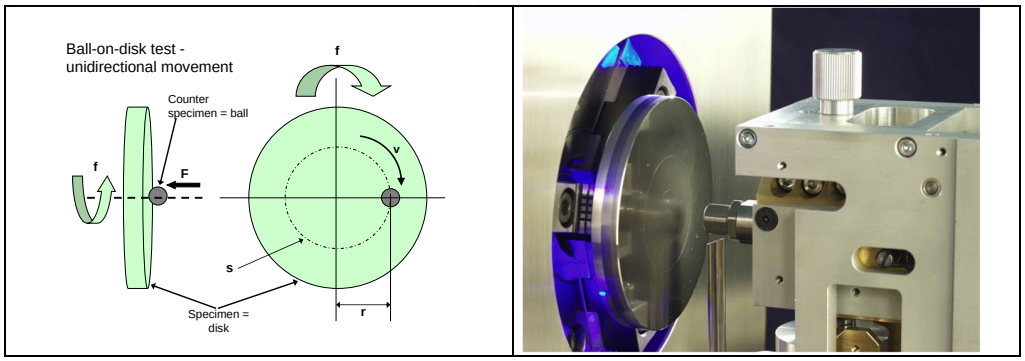

Figure 5 Tribological stress on material pairing – principle of **ball-on-disk test**

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.

The reel-on-disk test is based on the same principle as the ball-on-disk test with one difference: instead of a ball, a reel is used as counter specimen. According to the ball-on-disk test, the reel is pressed with a defined pressure onto the surface and the tribological generated particles are detected.

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
A 01	70	50	1	1500
A 02	90	50	3	1500
A 03	110	50	5	1500
A 04	130	100	6	1500
A 05	150	100	8	1500
A 06	170	100	10	1500
A 07	200	100	11	1500
A 08	220	100	13	1500
A 09	240	100	15	1500
A 10	260	150	16	1500
A 11	280	150	18	1500
A 12	300	150	20	1500

SET OF PARAMETERS	*s*/mm	*v*/mm/s	*F*/N	N
B 01	250	150	15	1500
B 02	250	150	45	1500
B 03	250	150	75	1500
B 04	250	150	90	1500
B 05	250	150	120	1500
B 06	250	150	150	1500
B 07	250	150	165	1500
B 08	250	150	195	1500
B 09	250	150	225	1500
B 10	250	150	240	1500
B 11	250	150	270	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 F	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, the following measuring device is used.

MODEL	COMPANY	PARTICLE SIZES DETECTED
LasAir II 110	PMT AG, Heimsheim	0.1 / 0.2 / 0.3 / 0.5 / 1.0 / 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.

Where possible, the **tribological test** is carried out using **one set of stress parameter**, taking into account the quantity of particles generated. To ensure reliability of the results, **10 repeated tests** are carried out for each set of stress parameter.

Before the 10 repeated tests, one test is performed to remove possible residues from the reel, this test is not incorporated into the assessment.

4.2 Material samples for particle emission tests

TESTED MATERIALS				LOAD
ID	SPECIMEN	COUNTER SPECIMEN	LUBRICANT	
IP NORD 01	NORDPUR SW	PA6 Nylon	(none)	Reel-on-disk-test

Figure 9 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 NORD 01, a floor covering founded on a 15 mm thick and a diameter of 140 mm disk is 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.

Photographs of the materials tested:

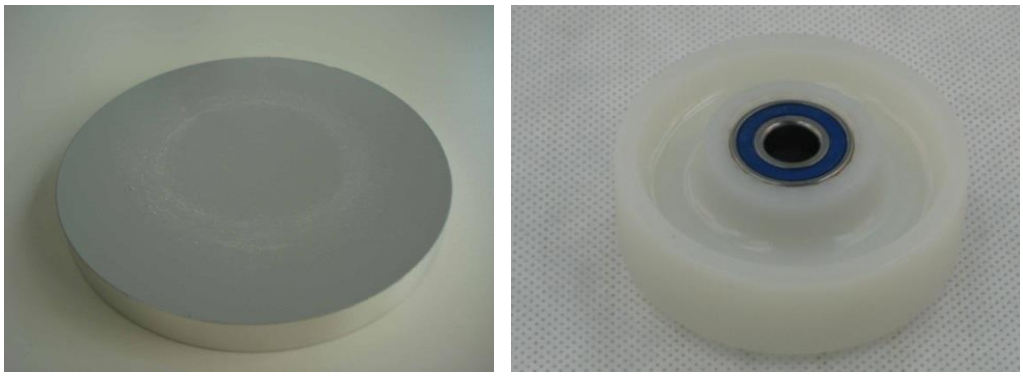

Figure 10 Materials tested – left: NORDPUR SW; right: PA6 Nylon

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 NORD 01: NORDPUR SW versus PA6 Nylon

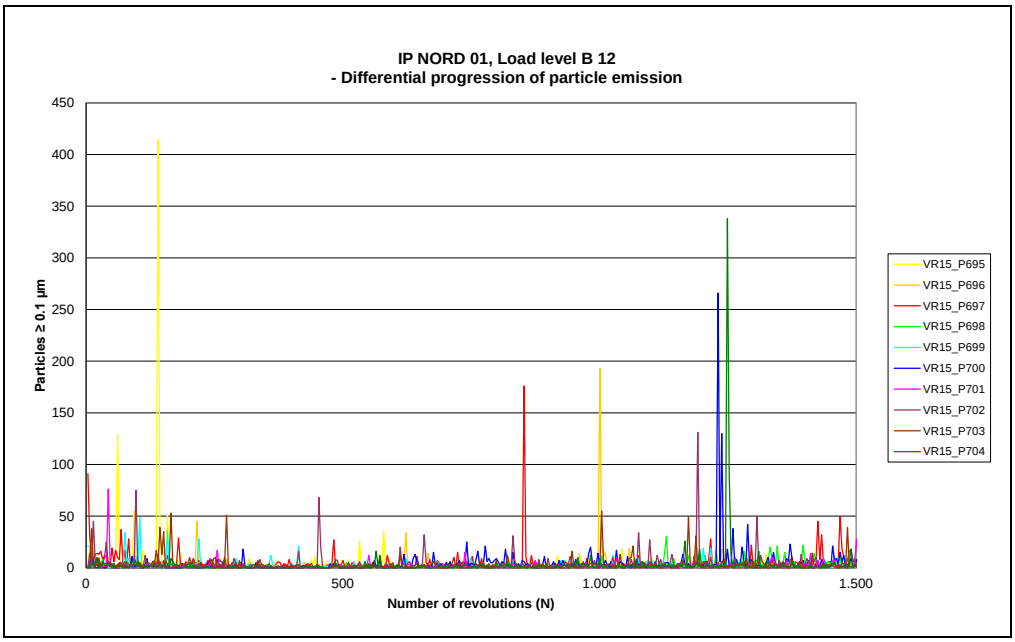

Figure 11 IP NORD 01 – progression of particle emission, particle size **0.1 µm**, set of stress parameters **B 12**

4.3.1.3 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 1.0 µm relates to particles with a diameter of 1.0 µm to 5.0 µm,
- Particles in the size channel 5.0 µm relates to particles with a diameter equal to or greater than 5.0 µm.

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.

4.3.1.4 IP NORD 01: NORDPUR SW versus PA6 Nylon

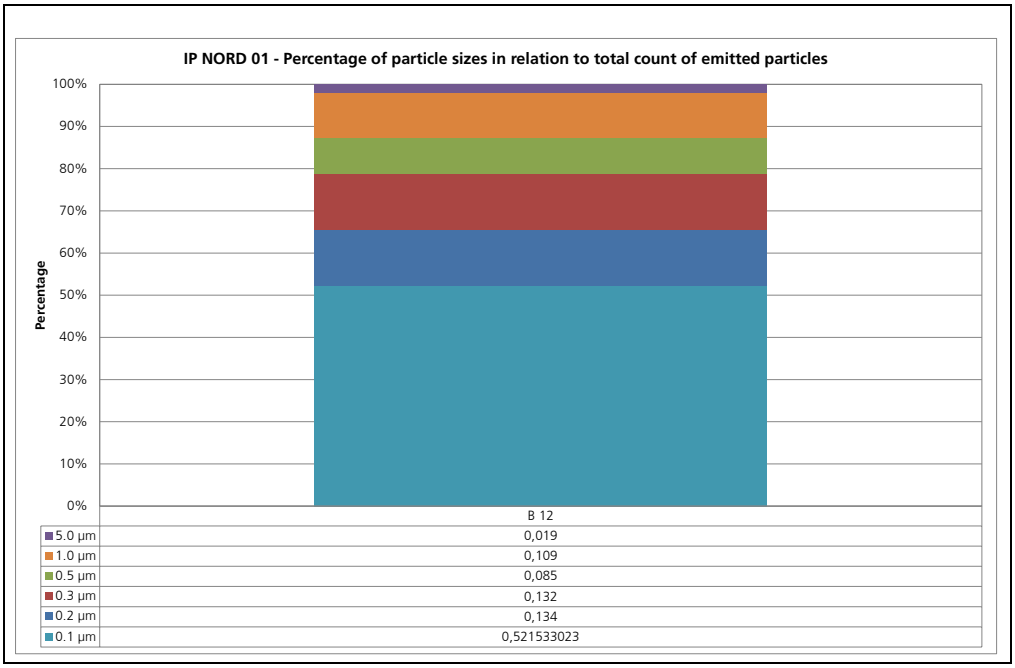

Figure 12 IP NORD 01– required minimum count of 100 particles was not recorded

4.3.2 Classification

4.3.2.1 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 greater than 5.0 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.

4.3.2.2 Overview of classification results

NORDPUR SW							
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	3.2	3.5	3.7	4.0	4.4	5.0
CLASSIFICATION RELEVANT TO DOCUMENTS							5

Figure 13 IP NORD 01: NORDPUR SW 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 **NORDPUR SW versus PA6 Nylon** lies within the permissible values of the corresponding Air Cleanliness Classes **ISO Class 5** in accordance with ISO 14644-1.

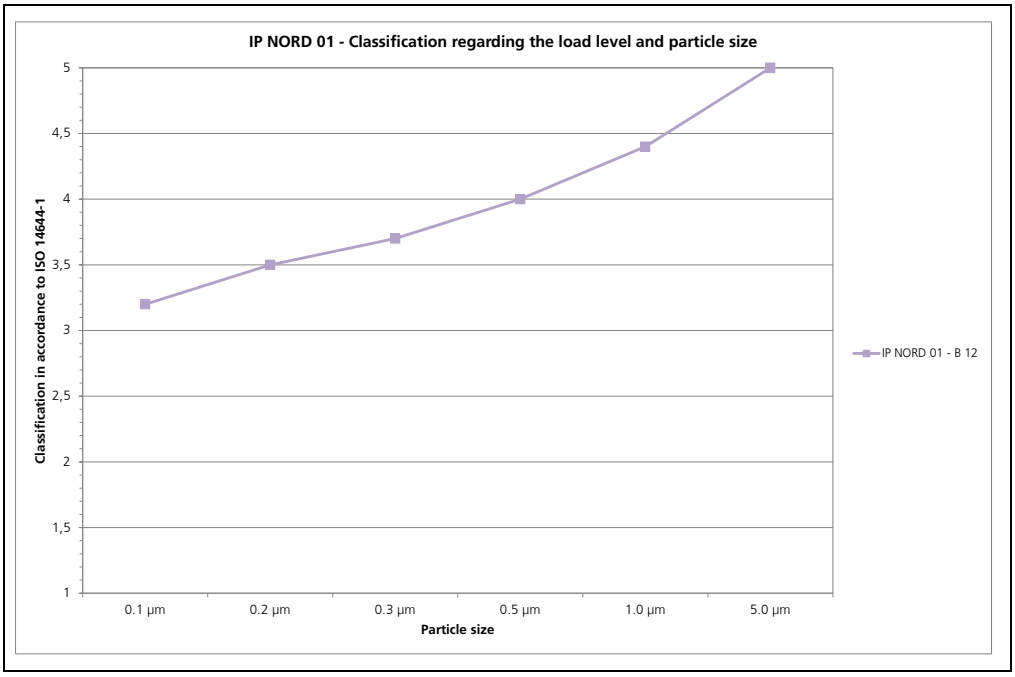

Figure 14 CSM IV 03: NORDPUR SW versus PA6 Nylon
Classification in accordance with ISO 14644-1 in dependence upon the particle size