



Materials of construction: Flooring material

Hygienic requirements

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Cleanliness requirements in Hygienic Industries are constantly increasing!

■ Food industry

- ➔ Ceasing to use conservatives
- ➔ Ensuring product purity

■ pharmaceuticals:

- ➔ sterility, cross contamination,...
- ➔ Ensuring substance purity



User requirements of construction materials:

no generating and no emission of

- any substances or
- cleanliness critical contamination, that might be bad for the manufactured products



Outline: GMP - Good manufacturing Practice

- Quality system for **hygienic production**
(currently only Pharma, possibly in near future for Food, Cosmetics, Medtec)
- GMP guidelines outline all aspects of production and testing that can impact the **quality of a product**
- Many countries have **legislated** GMP guidelines in national law!

EU-GMP Annex 1...

- ...describes the production of **sterile medicinal products**
- ...defines the **manufacturing clean areas in zones**
- ...describes the **requirements on the used material**

Outline: GMP - cleanliness classes

Cleanliness classes according to EU-GMP Annex 1: **Airborne Particles**

Cleanliness Class	Maximum permissible particle count per m ³ in a resting state		Maximum permissible particle count per m ³ in an operating state	
	> 0.5 µm	> 5 µm	> 0.5 µm	> 5 µm
A	3,520	20	3,520	20
B	3,520	29	352,000	2,900
C	352,000	2,900	3,520,000	29,000
D	3,520,000	29,000	Not fixed	Not fixed



Outline: GMP - cleanliness classes

Cleanliness classes according to EU-GMP Annex 1: **Biocontamination**

Cleanliness Class	Recommended limiting values for microbiological contamination		
	Air sample [CBU/m ³]	Sedimentation plates (Ø 90 mm) [CFU/4 hours]	Contact plates (Ø 55 mm) [CFU/plate]
A	<1	<1	<1
B	10	5	5
C	100	50	25
D	200	100	50



Outline: GMP - requirements

Extract from EU-GMP-guideline Annex 1:

» ... in clean areas, all surfaces should be smooth, imperious and unbroken in order to **minimize the shredding or accumulation of particles** or microorganisms and to **permit repeated application of cleaning agents and disinfectants** where used ... «

» ... The manufacture of sterile products is subject to special requirements in order to minimize **risks of microbiological contamination**, and of **particulate or pyrogen contamination**. «

Particle

Biol. Resistance and
Microbizzidity

Cleaning and
Chem. Resistance

Relevant for safe hygienic production:

- Particle generation
- Chemical Resistance
- Bioresistance
- Cleanability
- Microbicidity

Hygienic-relevant construction materials? Examples:

- Coatings, eg. for walls
- **Flooring systems**
- Sealants and adhesives
- All surfaces with incidental product contact
- Doors, roofing

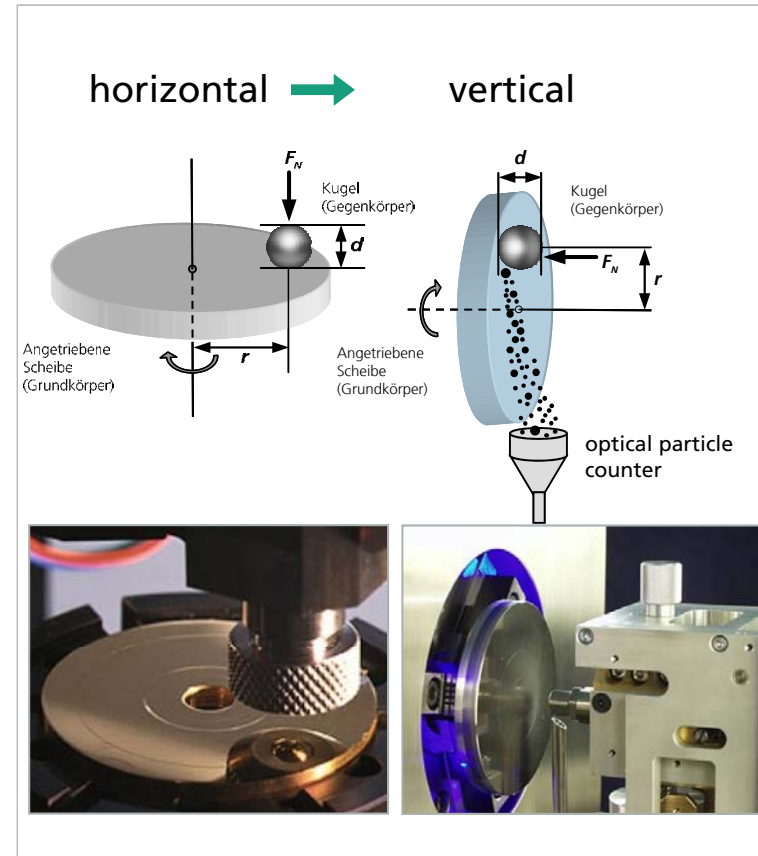
Particle generation

- Friction
 - Sliding (shoes etc.)
 - Rolling (trolleys etc.)
- Reduction of generated particles:
 - surface structure / roughness
 - contact pressure between materials
 - velocity / acceleration between materials
 - material-combination



Particle generation

- Innovative methodology & assessment
 - Measurement of emitted particles with particle counter
 - Roll-on-disc or ball-on-disc
- Result from these new conceptions:
 - Worldwide first explicit classification of materials



Particle generation and GMP

Regulatory				Limiting values of each air cleanliness class for differing particle sizes and reference volumes (acc. to ISO 14644-1)											
DIN EN ISO 14644-1	EU-GMP "at rest"	EU-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	
				per m³	per cbf	per m³	per cbf	per m³	per cbf	per m³	per cbf	per m³	per cbf	per m³	per cbf
1				10	0,3	2	0,1								
2				100	3	24	1	10	0,3	4	0,1				
3			1	1.000	30	237	7	102	3	35	1	8	0,2		
				1.240	35	265	8	106	3	35	1				
4			10	10.000	300	2.370	67	1.020	29	352	9,9	83	2		
				12.000	340	2.650	75	1.060	29	353	10				
5	A B	A	100	100.000	2.833	23.700	671	10.200	289	3.520	100	832	24	29	0,8
										3.520	100			20	0,6
										3.520	100			29	0,8
						26.500	750	10.600	300	3.530	100				
6			1.000	1.000.000	28.329	237.000	671	102.000	2.890	35.200	997	8.320	235	293	8
										35.300	1.000			247	7
7	C	B	10.000							352.000	9.972	83.200	2.357	2.930	83
										352.000	9.972			2.900	82
										352.000	9.972			2.900	82
										353.000	10.000			2.470	70
8	D	C	100.000							3.520.000	99.716	832.000	23.569	29.300	830
										3.520.000	99.716			29.000	821
										3.520.000	99.716			29.000	821
										3.530.000	100.000			24.700	700
9										35.200.000	997.167	8.320.000	235.694	293.000	8.300

Particle generation - CSM-Label



Chemical resistance

■ Hygienic requirements: »...all surfaces...*should permit repeated application of cleaning agents and disinfectants* where used ... «

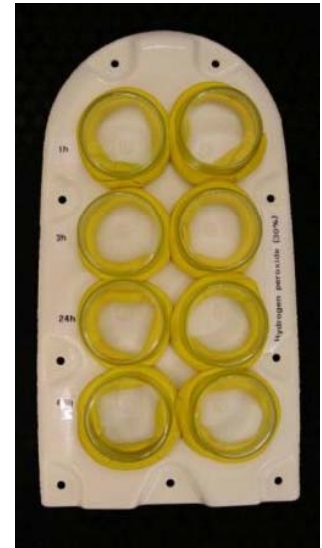
■ Standardized CSM worst-case-testing with following chemicals:

- | | |
|---------------------------------|----------------------------|
| • Formalin (37%) | • Hydrogen peroxide (30%) |
| • Sodium hydroxid solution (5%) | • Ammoniac (25%) |
| • Hydrochloric acid (5%) | • Isopropanol (100%) |
| • Peracetic acid (15%) | • Phosphoric acid (30%) |
| • Sulfuric acid (5%) | • Sodium hypochloride (5%) |

Test procedure according to

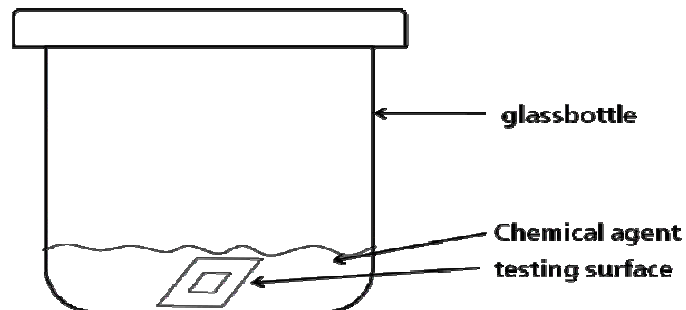
■ **ISO 2812-1: submers method**

■ ISO 2812-4: droplet method



Chemical resistance

ISO 2812-1: submers method



Chemical resistance

Assessment according to ISO 4628-1 to -6:

Tabelle 1 — Kennwerte zum Bewerten der Menge von Schäden

Kennwert	Menge der Schäden
0	keine, d. h. keine erkennbaren Schäden
1	sehr wenige, d. h. kleine, gerade noch signifikante Anzahl von Schäden
2	wenige, d. h. kleine, aber signifikante Anzahl von Schäden
3	mäßig viele Schäden
4	Schäden in beträchtlicher Anzahl
5	sehr viele Schäden

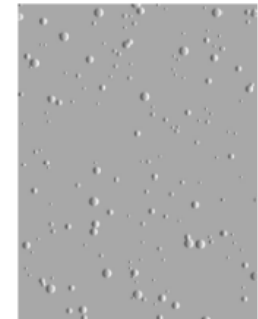
Tabelle 2 — Kennwerte zum Bewerten der Größe von Schäden

Kennwert	Größe der Schäden ^a
0	nicht sichtbar bei 10facher Vergrößerung
1	nur sichtbar bei bis zu 10facher Vergrößerung
2	gerade sichtbar mit bloßem Auge (auf Normalsichtigkeit korrigiertes Sehvermögen)
3	deutlich sichtbar mit bloßem Auge (auf Normalsichtigkeit korrigiertes Sehvermögen) (bis zu 0,5 mm)
4	Bereich 0,5 mm bis 5 mm
5	größer als 5 mm

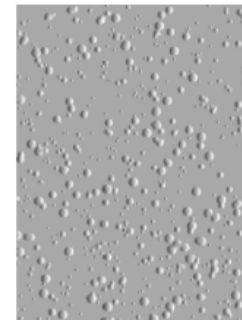
^a Falls in den Folgeteilen von ISO 4628 nicht anders festgelegt.



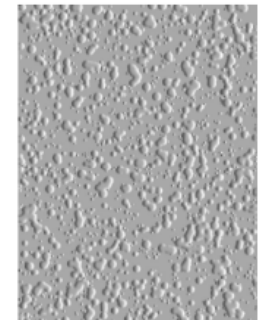
Menge 2 – 2(54)



Menge 3 – 3(54)



Menge 4 – 4(54)



Menge 5 – 5(54)

Chemical resistance

Assessment
according to ISO
4628-1 to -6:

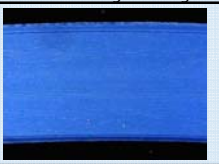
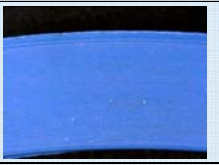
Chemical: Acetone

Tested material:

- Left:
Epoxy flooring
system

- Right:
Silicone sealant

Zeit	Mikroskopische Aufnahme 10 fache Vergrößerung	Wert
Blindwert		Ohne Belastung
1 h		No
3 h		No
6 h		No
24 h		No

Zeit	Mikroskopische Aufnahme 12 fache Vergrößerung	Wert
Blindwert		Ohne Belastung
1 h		Farbveränderung, Vergilbung I2
3 h		Farbveränderung, Vergilbung I2 – I3
6 h		Farbveränderung, Vergilbung I3, Rand gewellt N3 S2
24 h		Farbveränderung, Vergilbung I3, Rand gewellt N3 S2

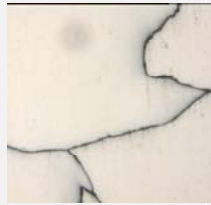
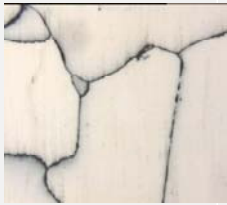
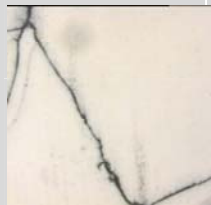
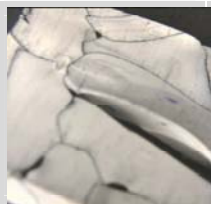
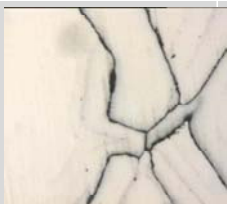
Chemical resistance

Assessment according to ISO 4628-1 to -6: Vinyl-Flooring System Forbo Colorex

Example:

left: Aceton

right: Isopropanol

Inkubationszeit	Bild (10x)	Wert	Bild (10x)	Wert
0 h		Ausgangsprüfung		Ausgangsprüfung
1 h		Schwellung, Deformation, Erweichung I3		NO
3 h		Schwellung, Deformation, Erweichung I4		NO
6 h		Schwellung, Deformation, Erweichung, Aufbrechen der Struktur I5		NO
24 h		Schwellung, Deformation, Erweichung, Aufbrechen der Struktur I5		NO

Chemical resistance

Assessment according to ISO 4628-1 to -6: Vinyl-Flooring System Forbo Colorex

Chemical resistance	CSM-Classification
0	excellent
1	very good
2	good
3	weak
4	very weak
5	none

Chemicals	Exposure 1 h	Exposure 3 h	Exposure 6 h	Exposure 24 h
Ethanol (100%)	excellent	excellent	excellent	excellent
Acetone (100%)	not resistant	not resistant	not resistant	not resistant
Benzine	excellent	excellent	excellent	excellent
Isopropanol (70%)	excellent	excellent	excellent	excellent
Ultrapure water	excellent	excellent	excellent	excellent
Formalin (37%)	excellent	excellent	excellent	excellent
Ammoniac (25%)	excellent	excellent	excellent	excellent
Hydrogen peroxide (30%)	excellent	excellent	excellent	excellent
Sulfuric acid (5%)	excellent	excellent	excellent	excellent
Hydrochloric acid (5%)	excellent	excellent	excellent	excellent
Peracetic acid (1%)	excellent	excellent	excellent	excellent
Sodium hydroxide (20%)	excellent	excellent	excellent	very good
Sodosil RA 08	excellent	excellent	excellent	excellent
Butyl acetate (100%)	week	week	none	none
Diethyl ether (100%)	week	week	week	week

Chemical resistance – Classification according to CSM

- Only if the 10 CSM-chemicals are tested after 24 hours incubation

Chemical/ Incubation duration	24 h
Isopropanol (70%)	0
Phosphoric Acid (30%)	0
Formalin (37%)	0
Ammoniac (25%)	0
Hydrogen peroxide (30%)	0
Sulfuric acid (5%)	0
Hydrochloric acid (5%)	0
Peracetic acid (1%)	0
Sodium hydroxide (20%)	1
Sodium hypochloride (5%)	0
Overall Result	0,1

**The average value of
all 10 chemicals
results in the
classification number
(rounded)**

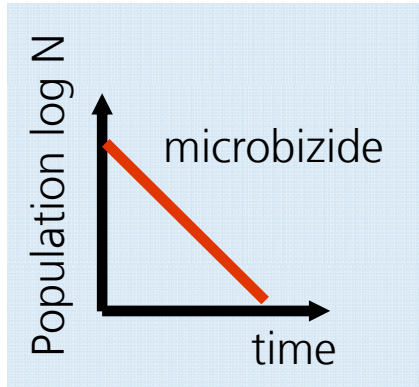
Chemical resistance	CSM- Classification
0	excellent
1	very good
2	good
3	weak
4	very weak
5	none

Chemical resistance – CSM label



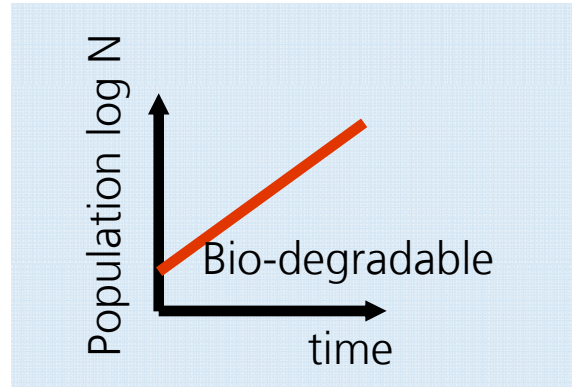
Bioresistance

Definition:



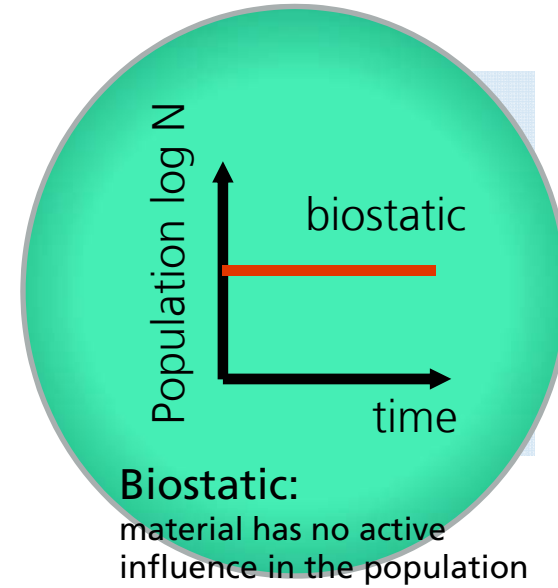
Microbicide:

Materials, which kill actively bacteria (z.B.: doping with silver ions, copper,..)



Bio-degradable:

material serves as nutrient for microorganisms (ex. Wood, some plastics, biopolymers as PHB or starch)



Biostatic:

material has no active influence in the population of microorganisms

Important:

Environmental conditions can kill microorganisms on surfaces easily, this is no material effect! Therefore, the test has to be done under idealistic conditions (humidity, temperature)

Bioresistance

INTERNATIONAL
STANDARD

ISO
846

Evaluation based on ISO 846

Second edition
1997-06-15

- Procedure A: Fungus
- *Procedure B: fungistatic activity*
- Procedure C: Bacteria
- *Procedure D: entrenching in earth*

**Plastics — Evaluation of the action
of microorganisms**

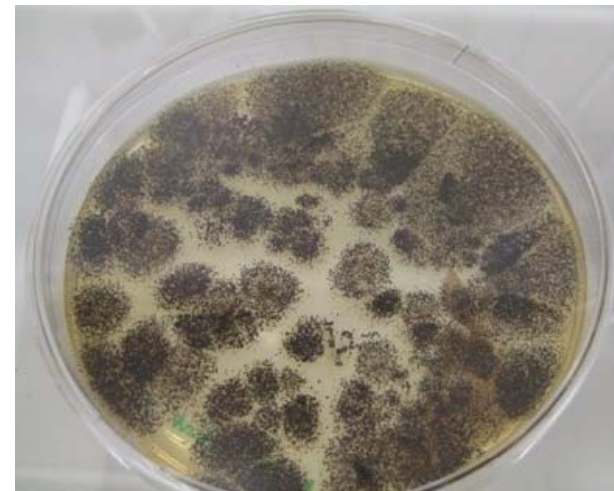


Bioresistance – Approach according to ISO 846

Used stock cultures:

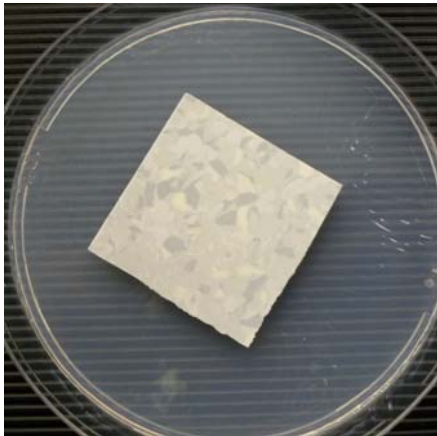
- *Aspergillus niger*
- *Penicillium funiculosum*
- *Peclomyces variotii*
- *Gliocladium virens*
- *Chaetominum globosum*

- *Pseudomonas aeruginosa*



Bioresistance – Approach according to ISO 846

- Incubation at 24 °C for 4 weeks
- Visual microscopic inspection of a caoutchouc flooring sample:



Bioresistance – Classification according to ISO 846

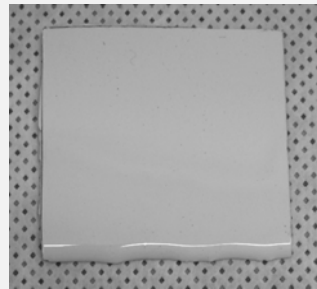
GROWTH-INTENSITY	ASSESSMENT
0	No growth visible under microscopic inspection
1	No growth visible by eyesight, but with microscopic inspection
2	Growth visible by eyesight; up to 25% of the sample surface is covered
3	Growth visible by eyesight; up to 50% of the sample surface is covered
4	Strong growth visible by eyesight; over 50% of the sample surface is covered
5	Strong growth visible by eyesight; whole sample surface is covered

Bioresistance – Classification according to CSM

REFERENCE NUMBER (OBTAINED AVERAGE)	CLASSIFICATION
0	Excellent
1	Very good
2	Good
3	Weak
4	Very weak
5	none

Bioresistance – Results: Epoxy-Flooring Sikafloor 269 CR

Fungus

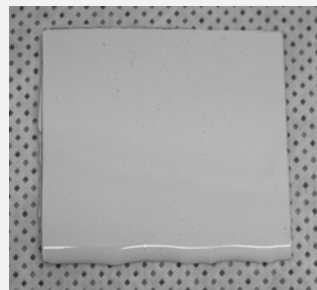


4) Sikafloor-269 CR



0; excellent

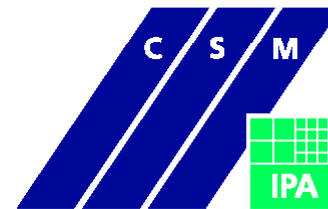
Bacteria



4) Sikafloor-269 CR



1; very good



Cleanroom[®]
Suitable
Materials

Sika AG
Report No. SI 1008-533

Sikafloor-269 CR
Particle (vs. PA6): GMP A
Biol. Resistance: very good

Flooring & Coating

Bioresistance – Results: Epoxy-Flooring Sika-ComfortFloor

Fungus

15) Sika ComfortFloor	0; excellent Comment: zone of inhibition. Fungistat be present

Bacteria

15) Sika ComfortFloor	1; very good

**Cleanroom[®]
Suitable
Materials**

Sika AG
Report No. SI 1008-533

Sika-ComfortFloor
Particle (vs. PA6): GMP A
Biol. Resistance: very good

Flooring & Coating

Microbicidity

Based on the following standards

- JIS Z 2801:2000
(equals the ISO standard)
- ISO 22196:2007

JIS Z 2801 : 2000

Antimicrobial products—
Test for antimicrobial activity
and efficacy

**INTERNATIONAL
STANDARD**

**ISO
22196**

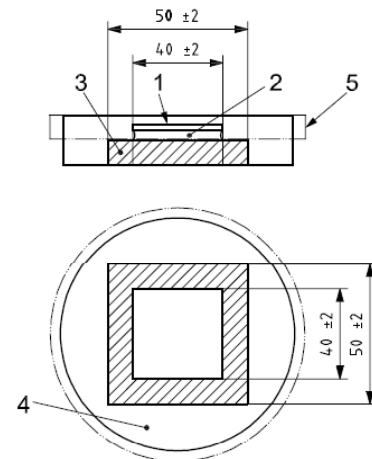
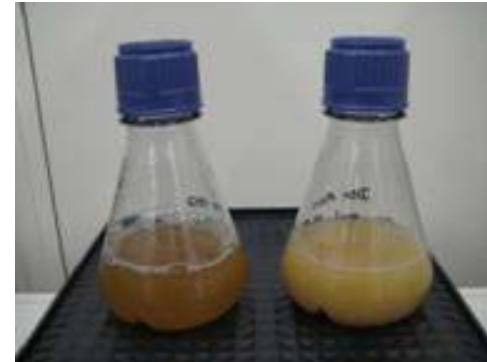
First edition
2007-10-15

**Plastics — Measurement of antibacterial
activity on plastics surfaces**

*Plastiques — Mesurage de l'action antibactérienne sur les surfaces en
plastique*

Microbicidity – Method description

- Test method according to ISO 22196
- Incubation of test sample with antibacterial property and blank sample of the same material but without antibacterial property
- Used organisms:
 - *Escherichia coli*
 - *Staphylococcus aureus*
- Covering with appropriate lid
- Incubation for 24 hours at 35 °C



Microbicidity – Classification

- Recovery of the bacteria from the test samples and determination of the CFU (colony forming units).
- Calculation of the R-Value using following equation:

$$R = \log(CFU_{untreated} / CFU_{treated})$$



Mikrobicidity R-value	CSM-classification
≥ 4	excellent
< 4	very good
< 3	good
< 2	weak
< 1	very weak
0	none

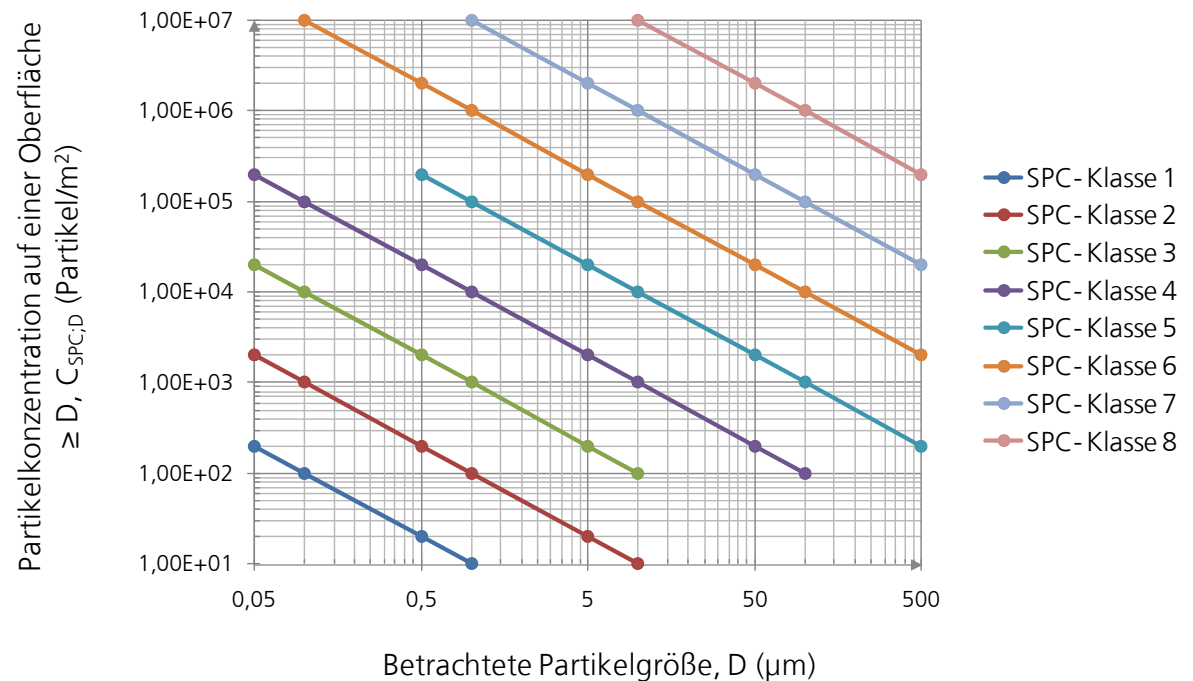
Microbicidity – CSM label



Cleanability

ISO/FDIS 14644-9: Surface cleanliness classes

Klassen der Partikelreinheit der Oberfläche (SPC)



Cleanability – Riboflavin-Test

- Minimal surface roughness of surfaces
➔ easy to clean
- but: Flooring with $\geq R9$ anti-slip resistance according to *Berufsgenossenschaftsrichtlinie (BGR) BGR 181*
- Measurement of anti-slip resistance according to DIN 51130!
- Qualitative Assessment of cleanability: Riboflavin-test according to VDMA



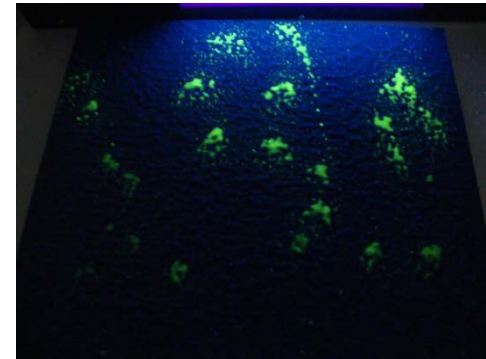
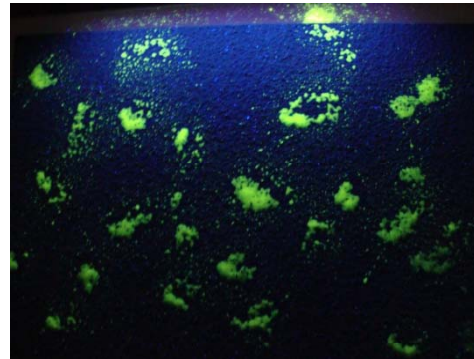
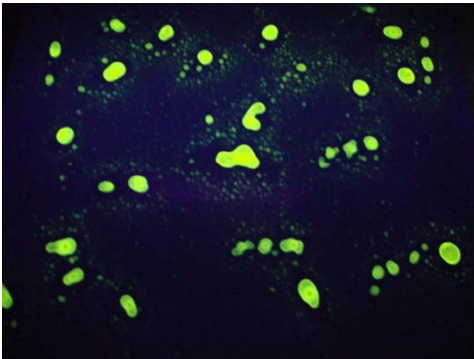
Cleanability - Riboflavin-test according to VDMA

Standard Epoxy-Flooring

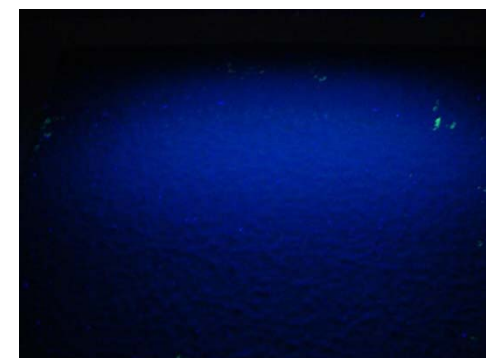
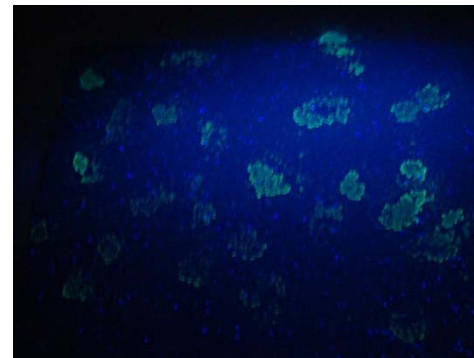
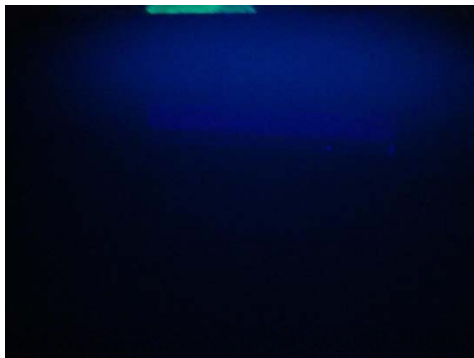
Sanded Epoxy-Flooring

Thixotropic surface

Prior
cleaning



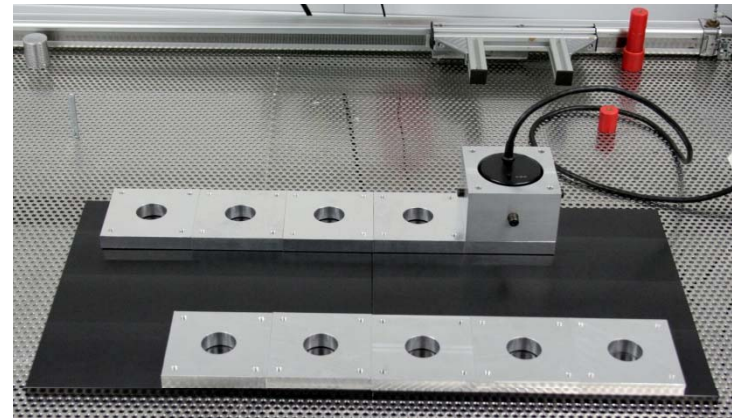
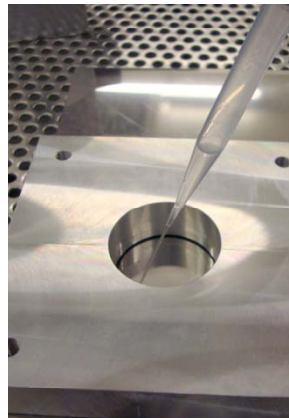
After
cleaning



Cleanability – Particle: Measurement method

Homogenous Contamination of the surface with particles

- Contamination: Test Dust (ATD testdust ISO 12103-1; silver particle (good for SEM-EDS, fluorescent particles for Laser Scanning Microscope LSM))
- Homogenous application of particles with liquid-suspension and evaporation of the Fluid (ex Ethanol, Isopropanol, Acetone,...):



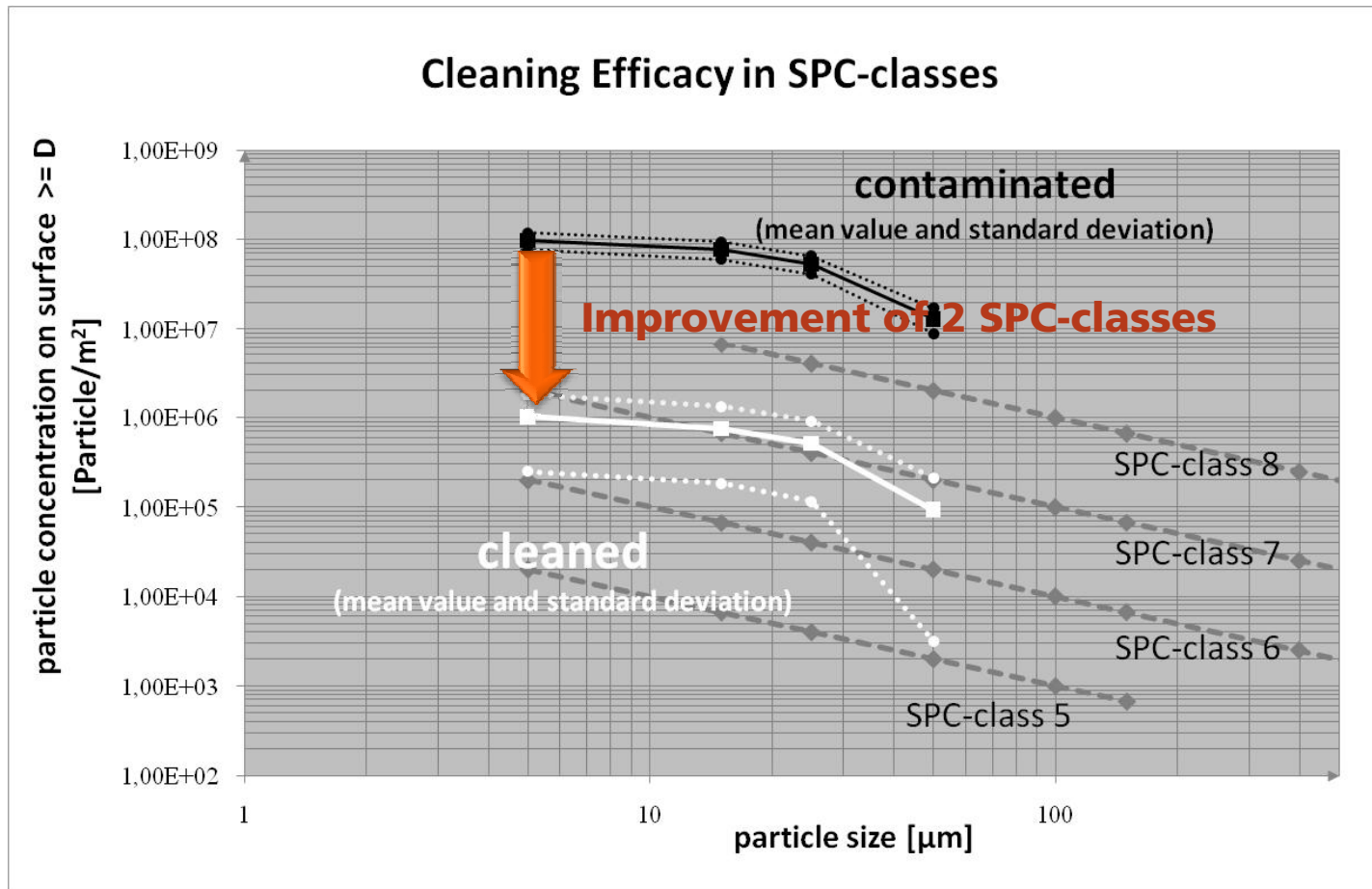
Cleanability – Particle: Measurement method

Cleaning of the surface with a cleaning simulator

- Weight $m = 1 \text{ kg}$; $v = 0,1 \text{ m/s}$, one stroke
- Surface particle measurements prior and after cleaning



Cleanability – Particle: Results



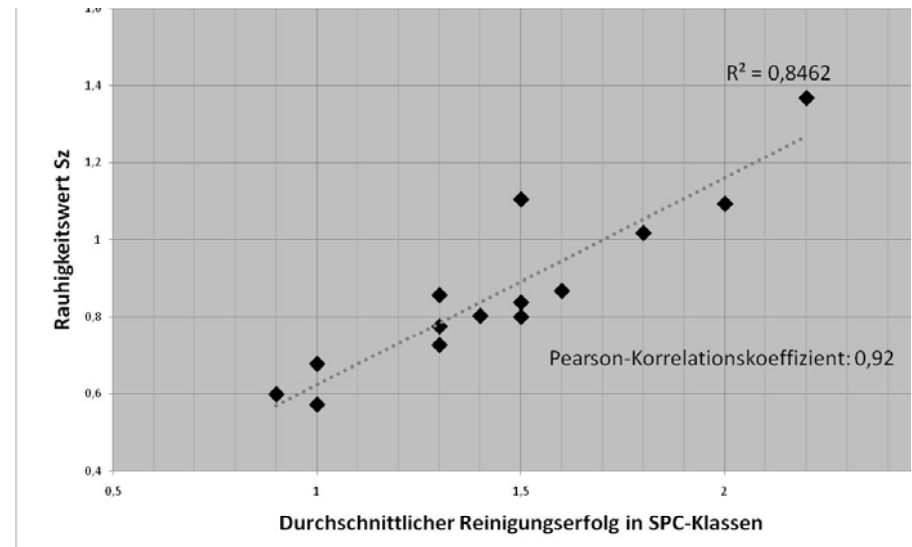
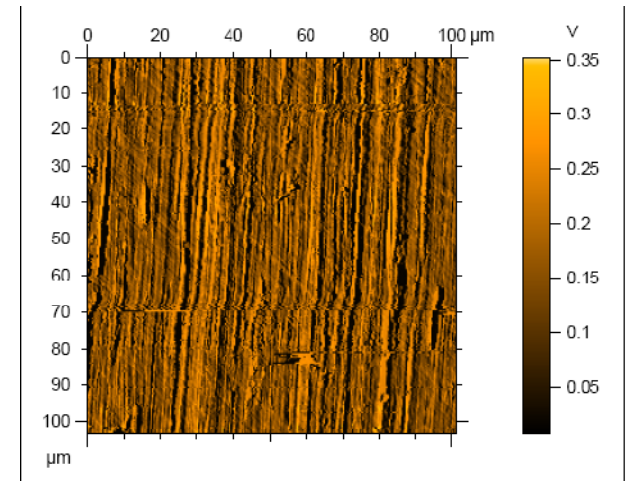
Cleanability – Particle: Results

- Cleaning efficacy does not correlate with Ra.

	Material	Cleaning Efficacy in SPC-classes	R _a along	R _a cross
PO01	Stainless steel 1.4301 brushed	2,0	0,2 µm	0,3 µm
PO02	Stainless steel 1.4301 Brushed and electro polished	1,5	0,2 µm	0,2 µm
PO04	Aluminium AlMg 4,5 Mn Milled and anodized	1,0	0,6 µm	0,5 µm
PO05		1,3	1,1 µm	0,9 µm
PO06		1,1	3,1 µm	1,0 µm
PO07	Aluminium AlMg 4,5 Mn Milled + nickel coated	1,6	0,5 µm	0,3 µm
PO08	Aluminium AlMg 4,5 Mn milled + Comp Cote H	2,2	1,2 µm	1,5 µm
PO09	Aluminium AlMg 4,5 Mn milled + Safe Coat 1327	0,9	3,5 µm	4,5 µm
PO10	Aluminium AlMg 4,5 Mn Milled and nickel coated	1,8	1,2 µm	0,7 µm
PO11	Aluminium AlMg 4,5 Mn Milled, sand blasted and Comp Cote H	1,5	1,4 µm	1,3 µm
PO12	Aluminium AlMg 4,5 Mn milled+ Comp Cote H	1,3	1,4 µm	1,1 µm
PO13	Aluminium AlMg 4,5 Mn milled + Safe Coat 1327	1,3	2,8 µm	2,5 µm

Cleanability – Particle: Results

- Cleaning efficacy does not correlate with Ra. (ISO 4287)
- But: it correlates very good with Sz. (ISO 25178-2)
- In scientific discussion, Sz is seen as a better value for the characterization of surface quality.
- Standardization is in progress
- results will be published in TechnoPharm 1/2013



CSM-Database www.ipa-csm.com

– Results from public domain - extract

Material	Verarbeitung	TVOC-Ausgasung: ISO-AMC _m -Klasse (or)	Partikel: ISO-Material-Klassifizierungszahl N	Mikrobiologische Resistenz: Klassifizierung
Gerflor WPT PURline P6334UF Silver Grey	Plattenmaterial		7	
Gerflor Technic EL 5	Plattenmaterial		5	
Gerflor GTI Prisma	Plattenmaterial		4	
Gerflor MIPOLAM ESPRIT 500	Plattenmaterial		4	
StoPox BB OS	Epoxy-System	-6,6	4	
StoPox WHG Deck 100	Epoxy-System	-6,6		
Caparol Disboxid 472	Epoxy-System		7	
noraplan mega ed, recipe 948	Plattenmaterial		3	
Forbo ColoRex EC Everest	Plattenmaterial		4	0
Sikafloor-169	Epoxy-System			2
Sikafloor-266 CR	Epoxy-System	-7,8	3	1
Sikafloor-266 ECF CR	Epoxy-System	-7,7	4	2

CSM-Database www.ipa-csm.com

– Results from public domain - extract

Sikafloor-269 CR	Epoxy-System	-9,6	5	1
Sikafloor-269 ECF CR	Epoxy-System	-9,6	4	2
Sikafloor-235 ESD	Epoxy-System	-6,8	5	3
Sikafloor-264	Epoxy-System	-6,5	4	3
Sikafloor-263 SL	Epoxy-System	-6,5	5	2
Sikagard-183 CR	Epoxy-System	-9,6	5	0
Sikafloor-304 W	Epoxy-System			1
Sikafloor-381	Epoxy-System	-9,6	1	2
Sikafloor-381 AS	Epoxy-System	-9,6	1	2
Sikafloor-390	Epoxy-System	-9,6	4	2
Sikafloor-390 AS	Epoxy-System	-9,6	4	2

CSM-Database www.ipa-csm.com

– Results from public domain - extract

Sika ComfortFloor	Systemboden		2	1
Sika ComfortFloor Decorative	Systemboden		1	1
Sika DecoFloor	Systemboden		4	1
Sika DecoFlake	Systemboden		4	2
Sika CompactFloor	Systemboden		5	2
Sikaflex AT Connection	Dichtungsstoff	-4,6		0
Sikaflex PRO 3 WF	Dichtungsstoff	-4,8		2
Sikasil-DW	Dichtungsstoff			2
Sikasil-Pool	Dichtungsstoff			0
Sikafloor-20 N PurCem	PU-System			1
Sikafloor-21 N PurCem	PU-System			1
Sikaflex Pro 3 (i-Cure)	Dichtungsstoff	-6,8		1
Sikaflex Wallcoat N	Epoxy-System	-9,6		0
Sikagard-206 W	Acryl-System			0
Sikagard-216 W	Acryl-System			0
Sikafloor-325	Epoxy-System	-7,1	4	0

Conclusions

- The Food-Industry needs hygienically safe flooring systems
- Following material testing are proposed:

- Particle emission
- Chem. Resistance
- Biol. Resistance
- Microbicidity (if applicable)
- Cleanability

-> The CSM test seal enables an easy material selection according to the addressed requirements for the flooring system

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