INTERNATIONAL STANDARD

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2013-03-01

Cleanrooms and associated controlled environments —

Part 10: **Classification of surface cleanliness by chemical concentration**

Salles propres et environnements maîtrisés apparentés —

Partie 10: Classification de la propreté chimique des surfaces

Reference number
ISO 14644-10:2013(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14644-10 was prepared by Technical Committee ISO/TC 209, *Cleanrooms and associated controlled environments*.

ISO 14644 consists of the following parts, under the general title *Cleanrooms and associated controlled environments*:

— *Part 1: Classification of air cleanliness by particle concentration*

— *Part 2: Specifications for testing and monitoring to prove continued compliance with* ISO 14644-1

— *Part 3: Test methods*

— *Part 4: Design, construction and start-up*

— *Part 5: Operations*

— *Part 6: Vocabulary*

— *Part 7: Separative devices (clean air hoods, glove boxes, isolators, mini-environments)*

— *Part 8: Classification of air cleanliness by chemical concentration (ACC)*

— *Part 9: Classification of surface cleanliness by particle concentration*

— *Part 10: Classification of surface cleanliness by chemical concentration*

The following part is under preparation:

— *Part 12: Classification of air cleanliness by nanoscale particle concentration*

Cleaning of surfaces to achieve defined levels of cleanliness in terms of particle and chemical classifications will form the subject of a future Part 13.

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Cleanrooms and associated controlled environments —

Part 10:
Classification of surface cleanliness by chemical concentration

1 Scope

This part of ISO 14644 defines the classification system for cleanliness of surfaces in cleanrooms with regard to the presence of chemical compounds or elements (including molecules, ions, atoms and particles). This part of ISO 14644 is applicable to all solid surfaces in cleanrooms and associated controlled environments such as walls, ceilings, floors, working environment, tools, equipment and devices.

NOTE 1 For the purpose of this part of ISO 14644, consideration is only given to the chemical characteristics of a particle. The physical properties of the particle are not considered and this part of ISO 14644 does not cover the interaction between the contamination and the surface.

NOTE 2 This part of ISO 14644 does not include the contamination generation process and any time-dependent influences (deposition, sedimentation, ageing, etc.) or process-dependent activities such as transportation and handling. Neither does it include guidance on statistical quality control techniques to ensure compliance.

2 Normative references

The following referenced documents are recommended for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14644-1:—[1)], *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness by particle concentration*

ISO 14644-6, *Cleanrooms and associated controlled environments — Part 6: Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14644-6 and the following apply.

3.1
air cleanliness by chemical concentration
ACC
level, expressed as an ISO Class *N*, which represents the maximum allowable concentration of a given chemical species or group of chemical species, expressed in grams per cubic metre (g/m^3)

Note 1 to entry: This definition does not include macromolecules of biological origin, which are judged to be particles.

3.2
contaminant category
common name for a group of compounds with a specific and similar deleterious effect when deposited on the surface of interest

1) To be published. (Revision of ISO 14644-1:1999.)

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3.3
chemical contamination
chemical (non-particulate) substances that can have a deleterious effect on the product, process or equipment

3.4
solid surface
boundary between the solid phase and a second phase

3.5
surface
boundary between two phases

Note 1 to entry: One of the phases is normally a solid phase and the other a gas, a liquid or another solid.

3.6
surface cleanliness by chemical concentration
SCC
condition of a surface with respect to its chemical concentration

3.7
surface cleanliness by chemical concentration class
N_{SCC}
common logarithm (to the base of 10) of the chemical concentration on a surface in grams per square metre (g/m^2)

4 Classification

4.1 Principles for establishing classification of clean surfaces in cleanrooms and controlled environments

Classification shall be specified by use of a classification descriptor. This descriptor is designated "ISO-SCC" and specifies the maximum total chemical concentration permitted on a surface for an individual chemical substance or group of substances. The classification of SCC is based upon the concentration of chemicals on a surface as calculated using Formula (1) (given in 4.2) and expressed in g/m^2. For calculation of the class, all other units shall be converted to g/m^2. In specific cases where low concentrations need to be specified, the maximum allowable concentration of chemicals on a surface may be expressed in atoms per square centimetre, ISO-SCC$_{_atomic}$, using Formula (2) in 4.4.

4.2 Classification for surface cleanliness by chemical concentration

The SCC class shall be designated by a classification number, N_{SCC}, where N_{SCC} is the common logarithm index of concentration C_{SCC}, expressed in g/m^2. The SCC class statement shall always be connected with a chemical substance or group of substances to which it is related. Intermediate concentrations may be specified, with 0,1 being the smallest permitted increment of N_{SCC}. C_{SCC} is determined from Formula (1), in terms of N_{SCC}:

$$C_{SCC} = 10^{N_{SCC}} \tag{1}$$

Therefore, $N_{SCC} = \log_{10} C_{SCC}$.

C_{SCC}, the maximum allowable concentration of the specified chemical substance or group of substances, is expressed in g/m^2. The measured chemical concentration on a surface shall not exceed the maximum allowable concentration of SCC, C_{SCC} to satisfy the predetermined SCC that is agreed between the customer and supplier.

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In all cases, N_{SCC} class numbers shall include the negative sign.

NOTE 1 An SCC class number is only valid in connection with a descriptor (see 4.3).

NOTE 2 For converting from gravimetric concentration (g/m^2) to numeric concentration (number of atoms, molecules or ions per unit area), see 4.4.

Table 1 and Figure 1 further illustrate the ISO-SCC classification as a function of chemical concentration on a surface.

Note also the parameters listed in Annex B that influence classification.

Table 1 — ISO-SCC classes

ISO-SCC class	Concentration (g/m^2)	Concentration ($\mu g/cm^2$)	Concentration (ng/cm^2)
0	10^{0}	10^{6}	10^{9}
−1	10^{-1}	10^{5}	10^{8}
−2	10^{-2}	10^{4}	10^{7}
−3	10^{-3}	10^{3}	10^{6}
−4	10^{-4}	10^{2}	10^{5}
−5	10^{-5}	10^{1}	10^{4}
−6	10^{-6}	10^{0}	10^{3}
−7	10^{-7}	10^{-1}	10^{2}
−8	10^{-8}	10^{-2}	10^{1}
−9	10^{-9}	10^{-3}	10^{0}
−10	10^{-10}	10^{-4}	10^{-1}
−11	10^{-11}	10^{-5}	10^{-2}
−12	10^{-12}	10^{-6}	10^{-3}

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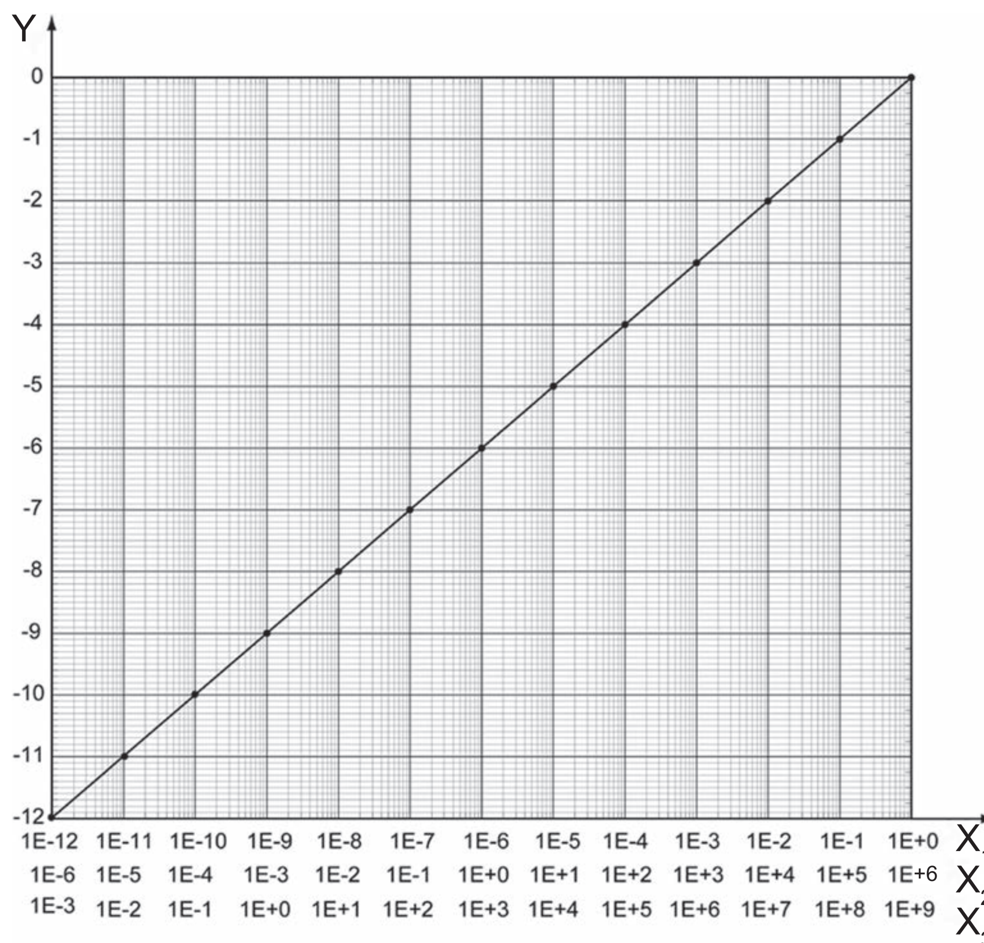

Key

X_1 surface mass concentration (g/m^2)

X_2 surface mass concentration ($\mu g/cm^2$)

X_3 surface mass concentration (ng/cm^2)

Y ISO-SCC class

Figure 1 — ISO-SCC classes as a function of concentration

4.3 ISO-SCC designation

An SCC class number is only valid in connection with a descriptor that includes the chemical substance or group of substances for which this class number is valid. The ISO-SCC descriptor is expressed in the format ISO-SCC Class *N* (*X*), where *X* is a chemical substance or group of chemical substances.

EXAMPLE 1 With an N-Methyl-2-pyrrolidone (NMP) sample, the measured value of chemical contamination on a surface was 9,8E-7 g/m^2. This is within the class limit of 1E-6 g/m^2 for Class −6. The designation would be: “ISO-SCC Class −6 (NMP).”

EXAMPLE 2 With an organic compound sample, the measured value was 6E-5 g/m^2 of total organic compounds (TOC). This is within the class limit of 1E-4 g/m^2 for Class −4. The designation would be: “ISO-SCC Class −4 (TOC).”

4.4 Converter for substances into surface atomic concentration

Very low concentrations are usually measured in surface number concentrations in the units of number of molecules, atoms or ions per surface area [$1/m^2$]. For classification purposes, these should be

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converted into surface mass concentrations in the unit mass per surface area [g/m^2]. This conversion is made using Formula (2):

$$C_{SCC} = \frac{M(C_{SCC_number})}{N_a} \quad (2)$$

where

C_{SCC_number}	is the surface number concentration = number of molecules, atoms or ions per surface area [1/m^2];
C_{SCC}	is the surface mass concentration [g/m^2];
N_a	is Avogadro's number [6,02 × 10^{23}/mol];
M	is the molar mass of atomic, molecular or ionic species [g/mol].

For information purposes, Figure A.4 in Annex A illustrates the relationship between chemical concentration on a surface (expressed in g/m^2) and the atomic concentration on a surface (expressed in atoms/m^2) for typical substances.

5 Measuring and monitoring the cleanliness of surfaces for chemical contamination and demonstration of compliance

5.1 Criteria for good cleanliness assessment

Figure D.2 in Annex D illustrates how to measure different types of contamination, showing differing sampling and measuring methods.

Tests performed to demonstrate compliance shall be conducted in a classified environment where the airborne chemical contaminant and the airborne particle contaminant levels do not negatively influence the classification. Suitable measurement methods and calibrated instruments shall be used for all tests. The environment, measurement methods and instruments shall be agreed upon between customer and supplier.

Additional test essentials are discussed in Annex C, while Annex D details measurement methods for demonstrating compliance.

The list of typical measurement methods is not exhaustive. Alternative methods that produce results with comparable accuracy may be specified by agreement between customer and supplier.

Measurement by different methods, even when those methods are correctly applied, may produce different results of equal validity.

Repeated measurements are recommended as part of the statistical approach.

Specific problems such as concentration spikes may occur when measuring high levels of cleanliness. Special quality control techniques will then be required, as explained in Figure D.4 in Annex D.

Precautions should be taken to reduce electrostatic charge around the test zone, as electrostatic charge enhances chemical deposition onto surfaces. If the surface is neither conductive nor grounded or charge-neutralized, electrostatic charges might occur. Therefore, test results may vary.

For typical methods of measurement for testing surface cleanliness by chemical concentration, refer to Annex D.

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5.2 Documentation and reporting

5.2.1 Principle

Compliance with surface cleanliness by chemical concentration (SCC) class requirements, as specified by the customer, is verified by performing measurements and by providing documentation of the results and conditions of measurement. Details for demonstrating compliance shall be agreed upon between customer and supplier in advance.

5.2.2 Testing

Tests performed to demonstrate compliance shall be conducted using suitable measurement methods together with calibrated instruments whenever possible.

Measurement methods for demonstrating compliance are described in Annex D. The list of typical methods described is not exhaustive. The testing environment shall be agreed between customer and supplier. Alternative methods of comparable accuracy also may be specified by agreement between customer and supplier.

Measurement by different methods, even when correctly applied, may produce different results of equal validity.

Repeated measurements are recommended.

The testing environment should be agreed between customer and supplier.

5.2.3 Test report

The results from testing each surface shall be recorded and submitted as a comprehensive report, together with a statement of compliance or non-compliance of the specified SCC class. The test report shall include as a minimum the following:

a) name and address of the testing organization;

b) name of the person performing the test;

c) measurement environment;

d) date, time and duration of sampling;

e) time of measurement;

f) number and year of publication of this part of ISO 14644, i.e. ISO 14644-10:2013

g) clear identification of the location of the surface measured and specific designations for coordinates of the surface, if applicable;

h) surface cleanliness by chemical concentration class with designation expressed as SCC class *N;*

i) acceptance criteria for the clean surface if agreed between customer and supplier;

j) specified measurement method(s), equipment resolution and detection limits;

k) details of the test procedure used, with any available data describing deviations from the test procedure (if agreed);

l) identification of the instrument(s) used and current calibration certificate(s);

m) number of measurements performed;

n) test results, including chemical concentration(s) data for given substances, for all measurements performed;

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o) surface condition, i.e. after final cleaning, before or after packaging, with agreement on type and quality of packaging required.

An example of how this test report may be constructed can be found in Annex E. Other variations of the test report which are agreeable to both the customer and supplier may be used.

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Annex A
(informative)

Conversion between different unit expressions of surface concentration for chemical substances

A.1 Principle

In addition to the unit of surface mass concentration of g/m^2, there exist several different units to express surface number concentration of an organic compound or a group of organics such as molecules/m^2, based on a number of organic molecules, and atoms C/m^2, based on a number of atoms of carbon composing organic compound(s) under consideration.

A.2 Examples

For information purposes, Tables A.1 to A.3 illustrate how the different units of surface number concentrations (molecules/m^2 or atoms C/m^2) can be converted into surface mass concentrations in terms of carbon (g C/m^2) or whole compound (g/m^2) respectively using the examples of heptane, hexadecane and di (2-ethylhexyl) phthalate.

Table A.1 — Illustration of the relationship between unit of surface concentration [g/m^2] and surface number concentration [molecules/m^2, atoms C/m^2] for heptane (C_7H_{16}), CAS No. 142-82-5

	Symbol	Unit	M = 100,2, N_c = 7			
			Example 1	Example 2	Example 3	Example 4
Surface number molecular concentration	$C_{molecule}$	[molecules/m^2]	1,00E+19	1,42E+18	7,16E+16	6,01E+16
Surface number concentration in terms of carbon	C_{carbon_number}	[atoms C/m^2]	7,00E+19	1,00E+19	5,00E+17	4,19E+17
Surface mass concentration in terms of carbon	C_{carbon_mass}	[g C/m^2]	1,39E-3	1,98E-4	1,00E-4	8,39E-6
Surface mass concentration	C_{SCC}	[g/m^2]	1,66E-3	2,36E-4	1,19E-4	1,00E-6

Table A.2 — Illustration of the relationship between unit of surface concentration [g/m^2] and surface number concentration [molecules/m^2, atoms C/m^2] for hexadecane ($C_{17}H_{34}$), CAS No. 544-76-3

	Symbol	Unit	M = 226,4, N_c = 17			
			Example 1	Example 2	Example 3	Example 4
Surface number molecular concentration	$C_{molecule}$	[molecules/m^2]	1,00E+19	6,20E+18	3,12E+16	2,60E+16
Surface number concentration in terms of carbon	C_{carbon_atom}	[atoms C/m^2]	1,59E+20	1,00E+19	5,00E+17	4,20E+17
Surface mass concentration in terms of carbon	C_{carbon_mass}	[g C/m^2]	3,19E-3	2,00E-4	1,00E-4	8,49E-6
Surface mass concentration	C_{SCC}	[g/m^2]	3,77E-3	2,35E-4	1,17E-4	1,00E-4

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Table A.3 — Illustration of the relationship between unit of surface concentration [g/m^2] and surface number concentration [molecules/m^2, atoms C/m^2] for di (2-ethylhexyl) phthalate ($C_{24}H_{38}O_4$), CAS No. 117-817-7

	Symbol	Unit	M = 390,6, N_c = 24			
			Example 1	Example 2	Example 3	Example 4
Surface number molecular concentration	$C_{molecule}$	[molecules/m^2]	1,00E+19	4,20E+18	2,00E+16	1,50E+16
Surface number concentration in terms of carbon	C_{carbon_atom}	[atoms C/m^2]	2,39E+20	1,00E+19	4,89E+17	3,60E+17
Surface mass concentration in terms of carbon	C_{carbon_mass}	[g C/m^2]	4,89E-3	2,04E-4	1,00E-4	7,37E-6
Surface mass concentration	C_{SCC}	[g/m^2]	6,62E-3	2,76E-4	1,35E-4	1,00E-4

Each surface number concentration can be converted to the original surface concentration from Formulae (A.1) to (A.3):

$$C_{SCC}\left[\mathrm{g/m^2}\right] = C_{molecule}\left[\mathrm{molecules/m^2}\right] \times \frac{M[\mathrm{g/mol}]}{N_a[\mathrm{molecules/mol}]} \qquad \text{(A.1)}$$

$$C_{SCC}\left[\mathrm{g/m^2}\right] = \frac{C_{carbon_atom}\left[\mathrm{atoms\ C/m^2}\right]}{N_c[\mathrm{atoms\ C/molecule}]} \times \frac{M[\mathrm{g/mol}]}{N_a[\mathrm{molecules/mol}]} \qquad \text{(A.2)}$$

$$C_{SCC}\left[\mathrm{g/m^2}\right] = \frac{\left[\dfrac{C_{carbon_mass}[\mathrm{g\ C/m^2}]}{M_c[\mathrm{g/mol}]}\right]}{N_c\ [\mathrm{atoms\ C/molecule}]} \times M[\mathrm{g/mol}] \qquad \text{(A.3)}$$

where

N_c is the number of carbon composing organic compound(s) under consideration; and

M_c is the molar mass of carbon.

Surface number concentration with different types of unit expression based on Langmuir–Blodgett (LB) film for typical organics is also listed in Table A.4. The surface number concentration can be obtained from Formula (A.4):

$$C_{SCC_LB}\left[\mathrm{g/m^2}\right] = (M/N_a)^{1/3} d^{2/3} \qquad \text{(A.4)}$$

where

d is the density of organic compound(s) in g/m^3.

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Table A.4 — Monolayer concentration based on Langmuir–Blodgett film

	Heptane (C_7H_{16}), CAS No. 142-82-5	Hexadecane ($C_{17}H_{34}$), CAS No. 544-76-3	Di (2-ethylhexyl) phthalate ($C_{24}H_{38}O_4$), CAS No. 117-817-7
Surface number molecular concentration	2,55E+18	1,62E+18	1,34E+18
Surface number concentration in terms of carbon	1,79E+19	2,59E+19	3,21E+19
Surface mass concentration in terms of carbon	3,56E-4	5,18E-4	6,56E-4
Surface mass concentration	4,24E-4	6,10E-4	8,88E-4

For information purposes, Figures A.1 to A.4 illustrate how the different units of surface number concentrations (molecules/m^2 or atoms C/m^2) can be converted into surface mass concentrations in terms of carbon (g C/m^2) or whole compound (g/m^2), respectively.

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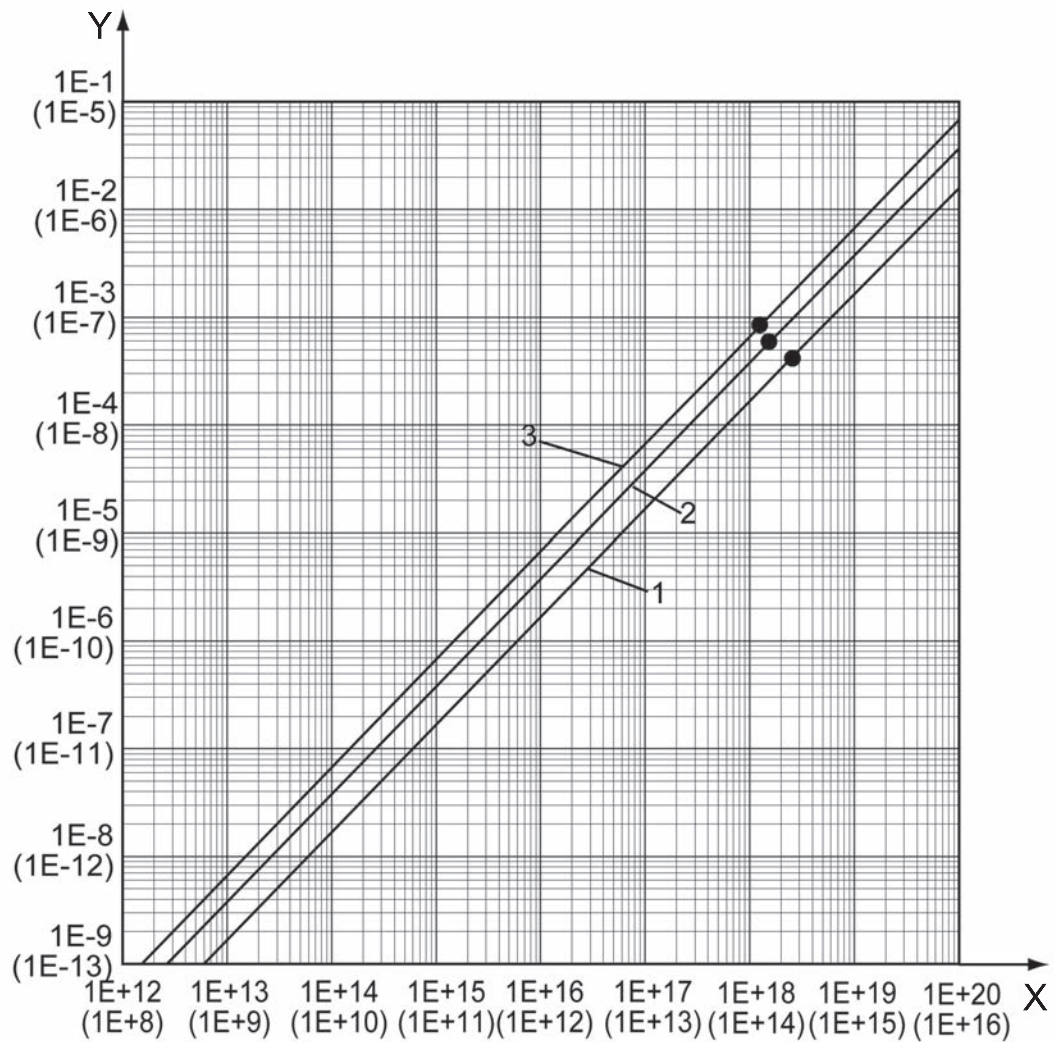

Key

X surface molecular concentration (molecules/m^2) [surface molecular concentration (molecules/cm^2)]

Y surface mass concentration (g/m^2) [surface mass concentration (g/cm^2)]

• surface concentration corresponding to monolayer based on LB model

1 heptane (C_7H_{16}), CAS No. 142-82-5

2 hexadecane ($C_{17}H_{34}$), CAS No. 544-76-3

3 di (2-ethylhexyl) phthalate ($C_{24}H_{38}O_4$), CAS No. 117-817-7

Figure A.1 — The relationship between the units of surface mass concentration (g/m^2) and surface molecular concentration (molecules/m^2) for typical organics

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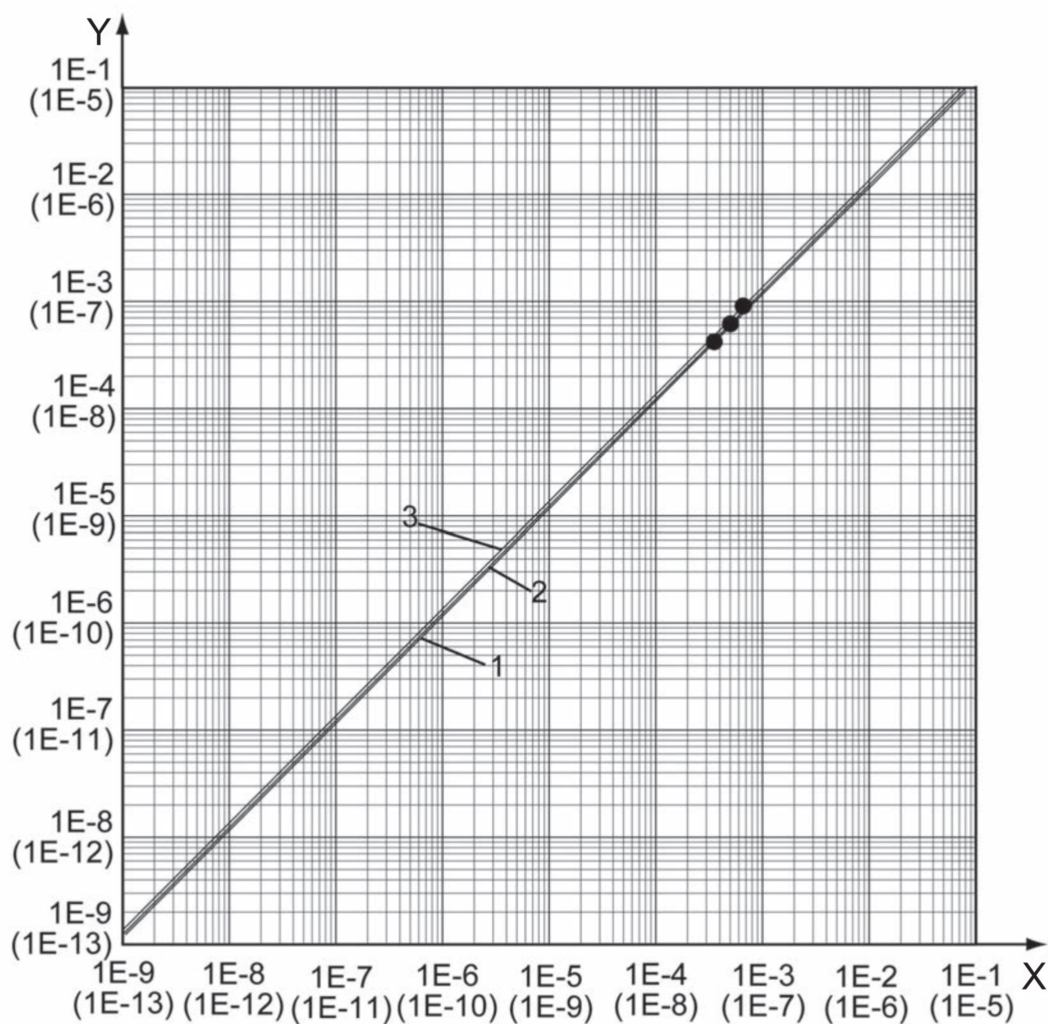

Key

X surface atomic concentration in terms of mass of carbon (grams C/m^2) [surface atomic concentration in terms of mass of carbon (grams C/cm^2)]

Y surface concentration (g/m^2) [surface concentration (g/cm^2)]

• surface concentration corresponding to monolayer based on LB model

1 heptane (C_7H_{16}), CAS No. 142-82-5

2 hexadecane ($C_{17}H_{34}$), CAS No. 544-76-3

3 di (2-ethylhexyl) phthalate ($C_{24}H_{38}O_4$), CAS No. 117-817-7

Figure A.2 — The relationship between the units of surface concentration (g/m^2) and surface atomic concentrations, in terms of mass of carbon (grams C/m^2) for typical organics

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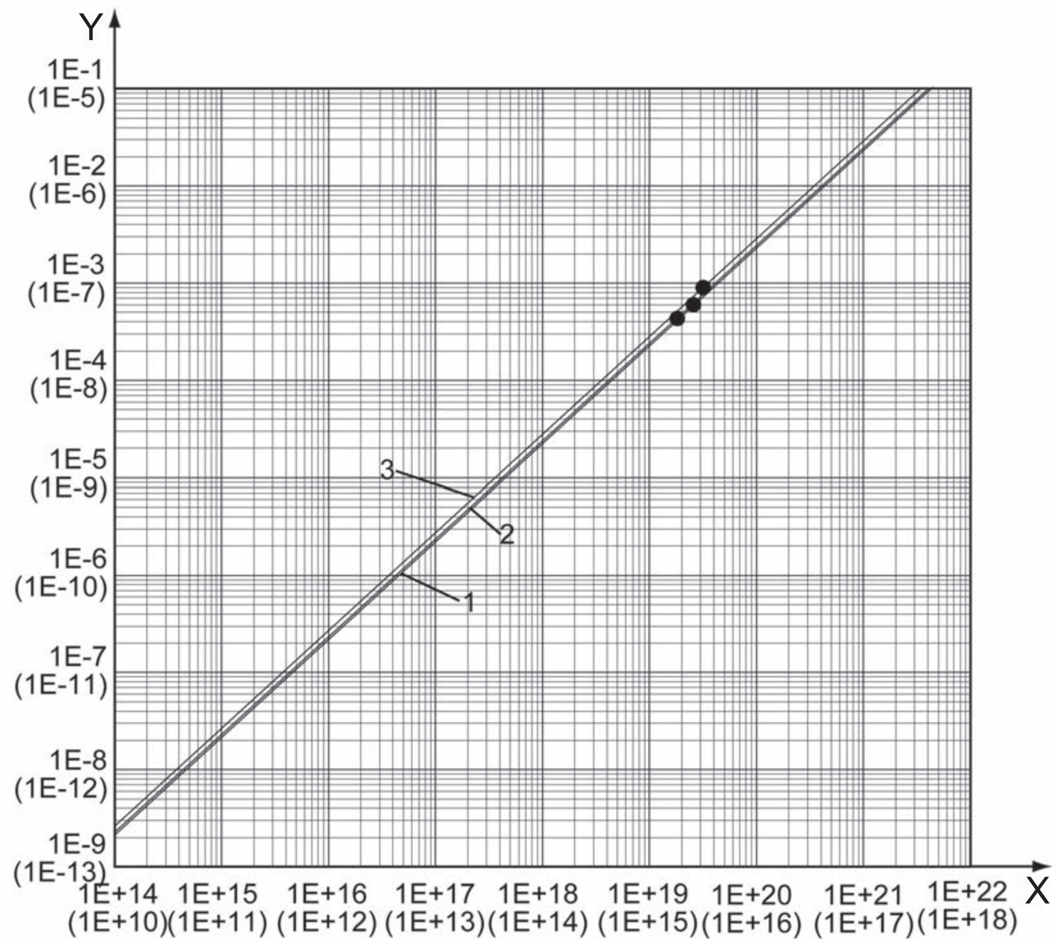

Key

X surface atomic concentration in terms of numbers of carbon (atoms C/m^2) [surface atomic concentration in terms of numbers of carbon (atoms C/cm^2)]

Y surface concentration (g/m^2) [surface concentration (g/cm^2)]

• surface concentration corresponding to monolayer based on LB model

1 heptane (C_7H_{16}), CAS No. 142-82-5

2 hexadecane ($C_{17}H_{34}$), CAS No. 544-76-3

3 di (2-ethylhexyl) phthalate ($C_{24}H_{38}O_4$), CAS No. 117-817-7

Figure A.3 — The relationship between the units of surface concentration (g/m^2) and surface atomic concentration, in terms of numbers of carbon (atoms C/m^2) for typical organics

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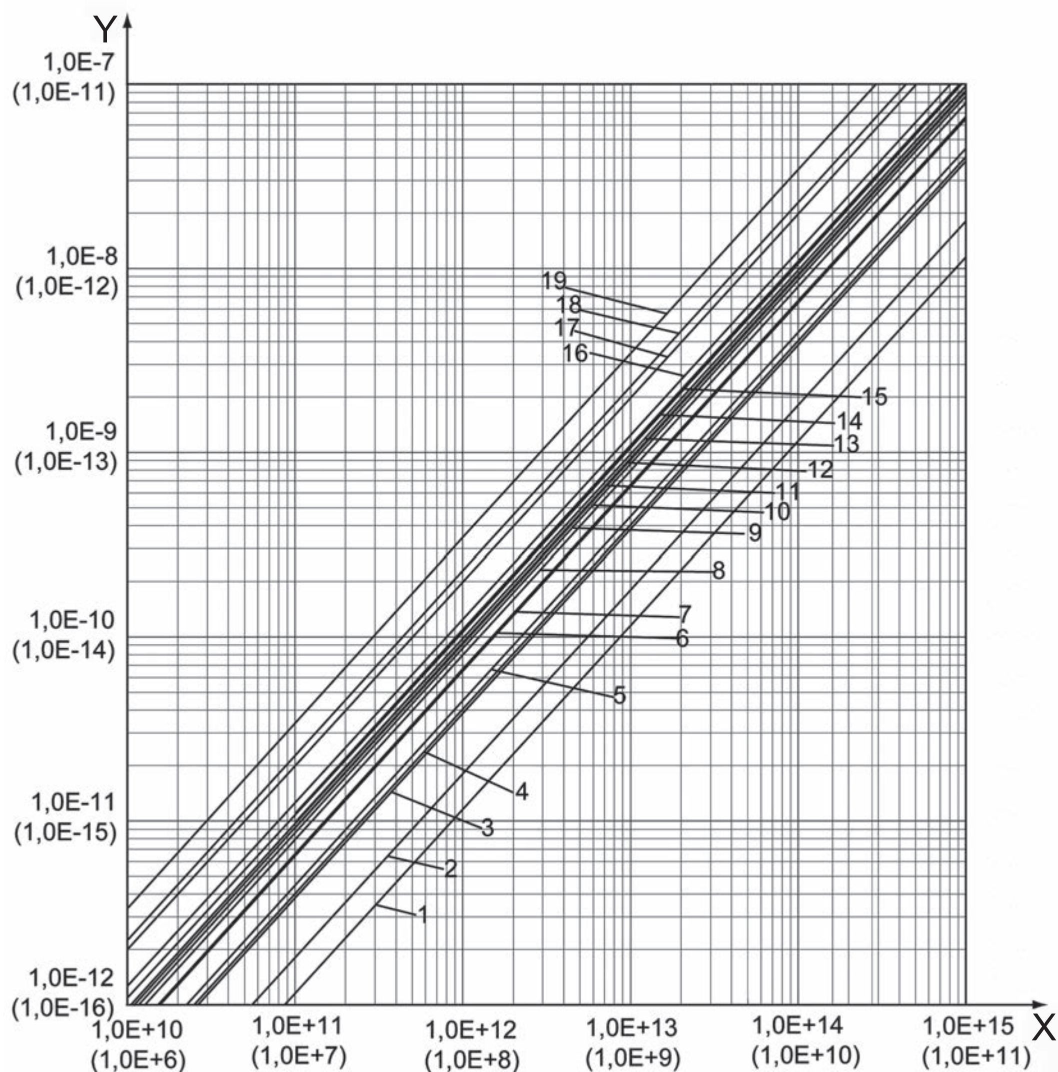

Key

X surface atomic concentration in terms of numbers of atoms (atoms/m²) [surface atomic concentration in terms of numbers of atoms (atoms/cm²)]

Y surface concentration (g/m²) [surface concentration (g/cm²)]

1	Li (M = 6,9)	8	Ti (M = 47,8)	15	Zn (M = 65,4)
2	B (M = 10,8)	9	Cr (M = 52,0)	16	As (M = 74,9)
3	Na (M = 23,0)	10	Mn (M = 54,9)	17	Sn (M = 118,7)
4	Mg (M = 24,3)	11	Fe (M = 55,9)	18	Ba (M = 137,33)
5	Al (M = 27,0)	12	Co (M = 58,9)	19	Pb (M = 207,2)
6	K (M = 39,1)	13	Ni (M = 58,7)		
7	Ca (M = 40,1)	14	Cu (M = 63,4)		

Figure A.4 — The relationship between the units of surface mass concentration (g/m²) and surface atomic concentration in terms of numbers of atoms (atoms/m²) for typical substances

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Annex B
(informative)

Parameters influencing testing and interpretation of results

B.1 Parameters

Parameters influencing testing and interpretation of results include the following:

a) skill level of personnel doing sampling or making the test;

b) reliability, repeatability and capability level of the testing laboratory;

c) cleanliness level of sample vessels, tools and test equipment;

d) cleanliness level of packaging used for shipping the samples;

e) use of an agreed sampling, blank and error management procedure for collecting and measuring samples (see Figure D.3 in Annex D);

f) unauthorized opening of the sample;

g) opening the sample outside a controlled environment;

h) quality of chemical or water used to strip the chemical from its substrate;

i) loss of analyte during sampling, shipping or testing;

j) loss or change of analyte by desorption or thermal decomposition during test heating;

k) failure to continually recalibrate test equipment or to investigate unusual data excursions (test spikes);

l) errors in measuring or calculating the sample area.

B.2 Considerations

The list in B.1 is not comprehensive.

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Annex C
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Essential considerations for a good cleanliness assessment

C.1 Principle

The following considerations should be established, agreed and managed before starting and during the assessment:

a) surface wettability state, i.e. hydrophilic or hydrophobic;

b) substance or group of substances used in the subject material;

c) substance or group of substances of interest used in the subject process;

d) substance or group of substances used in the maintenance process;

e) substance or group of substances found in the outside and inside air;

f) existence of a general chemical/molecular or product-specific problem;

g) substance or group of substances intended to be measured;

h) nature, volume and velocity of the air during sampling;

i) odour detection during sampling (Precautions should be taken to prevent inhalation.);

j) temperature, relative humidity and pressure levels during sampling;

k) visualization test or an ACC measurement prior to the SCC measurement;

l) nanoparticle measurement prior to the SCC measurement;

m) cleaning substance or process used;

n) expected level of the measurement result;

o) type of measurement instrument needed;

p) point of interest representative of the entire sample;

q) contamination from the outside environment;

r) recontamination from the measurement process or instrument;

s) restriction of electrostatic discharge and electrical transients at the measurement site;

t) measurement results within the specification of the classification;

u) application of adequate quality control to the collection and measurement of data.

C.2 Considerations

Also see Annex D for detail on measurement methods.

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Annex D
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Methods for testing surface cleanliness by chemical concentration

D.1 Method selection

D.1.1 Principle

In order to obtain quantitative information regarding surface cleanliness, appropriate measurement methods should be selected. In some cases where quantitative information cannot be ascertained for a surface, it is possible at least to obtain a qualitative result. Qualitative results cannot be used for surface cleanliness by chemical concentration classification as defined in Clause 5.

D.1.2 Measurement matrix

Figure D.1 provides a matrix for selecting an appropriate method for measuring surface cleanliness by chemical concentration.

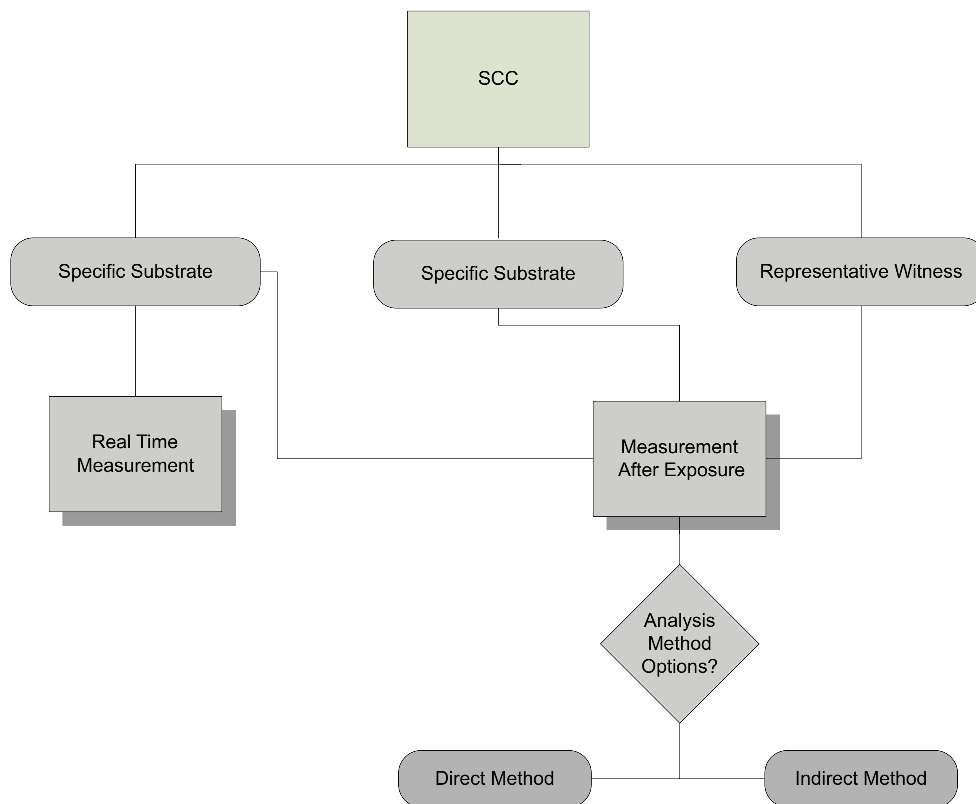

Figure D.1 — Analysis of surface cleanliness by chemical concentration: measurement matrix chart

D.1.3 General application range of principal measurement methods

Figure D.2 illustrates how to measure different types of contamination, indicating which measurement methods are appropriate for measuring particular substances.

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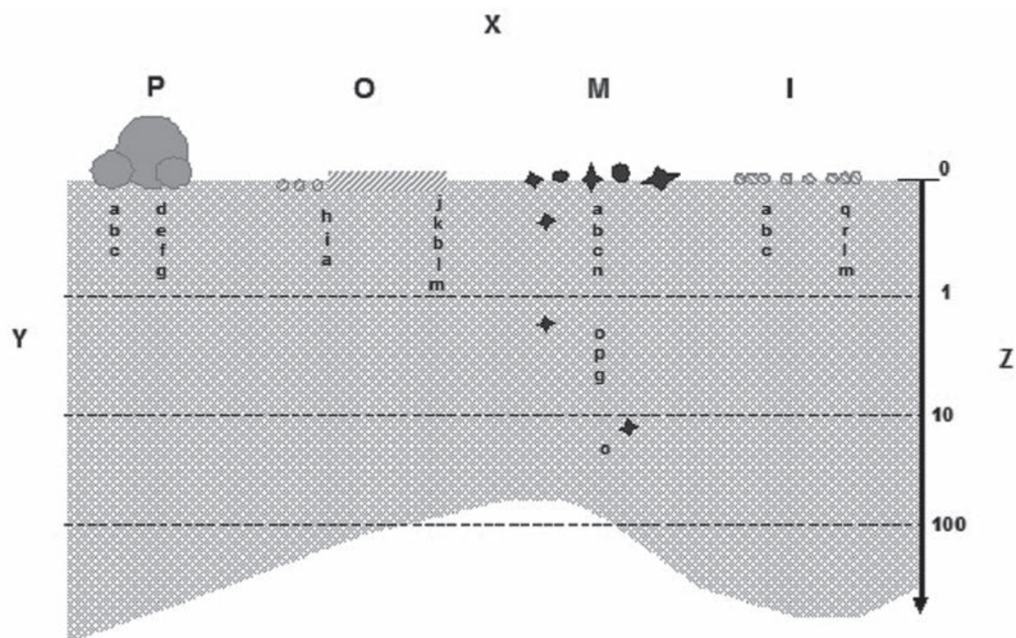

Key

X	surface chemical contamination	
Y	main measurement methods	
Z	detection depth (nm)	
P	particles	
O	organics	
M	metals	
I	ions, i.e. acids and bases	
a	TOF-SIMS	time of flight–secondary ion mass spectrometry
b	XPS	X-ray photo-electron spectroscopy
c	AES	Auger electron spectroscopy
d	SEM	scanning electron microscopy
e	SEM-EDX	SEM–energy dispersive X-ray spectroscopy
f	SEM-WDX	SEM–wavelength dispersive X-ray spectroscopy
g	TXRF	total reflection X-ray fluorescence spectroscopy
h	TD-GC/MS	thermal desorption–gas chromatography/mass spectrometry
i	TD-IMS/MS	TD–ion mobility spectroscopy/MS
j	FTIR	Fourier transform infrared spectroscopy
k	MIR-FTIR	multiple interlayer reflection–FTIR
l	QCM	quartz crystal microbalance
m	SAW	surface acoustic wave
n	VPD-ICP/MS	vapour phase decomposition–inductive coupled plasma/MS
o	SIMS	secondary ion mass spectrometry
p	VPD-TXRF	vapour phase decomposition–TXRF
q	SE-IC/MS	solvent extraction–ion chromatography/MS
r	SE-HPLC	SE–high-performance liquid chromatography

Figure D.2 — General application range of principal measurement methods

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D.2 Criteria for the measurement of surface cleanliness by chemical concentration

D.2.1 General

The classification of surface cleanliness by chemical concentration can be determined as soon as the chemical contamination has been measured.

As a measurable and quantitative criterion for the evaluation and classification of the surface cleanliness, the number and type of all adherent chemical substances should be determined.

D.2.2 Requirements of the measurement method

The measurement method is selected according to criteria determined by the surface being tested and its characteristics. Some of the most important requirements are summarized as follows:

a) information regarding chemical characteristics and resistance (e.g. material, concentration);

b) feasible measuring positions (direct, real-time measuring device such as an SAW instrument);

c) feasible measuring positions (indirect, analytical measuring device such as an SEM stub zone);

d) testing speed and effort involved (i.e. use of random sampling or series testing);

e) flexibility (i.e. whether the method can be rapidly implemented on various surfaces of different components);

f) little or no surface alteration caused by the measurement procedure (i.e. measurement surfaces are not altered as a result of being wetted with flushing fluids).

Due to requirements a) through f) in D.2.2, the measurement methods described in D.2.3 could be classified and be limited for each of the applications.

D.2.3 Direct or indirect measurement methods

D.2.3.1 Principle

Ideally, surface cleanliness by chemical concentration is best assessed when the test surface can be accessed by the chosen measurement device. In principle, the following methods can be utilized to measure the surface cleanliness:

a) direct methods;

b) indirect methods where a pre-treatment technique may also be introduced.

Direct methods that do not require sample pre-treatment should generally be given priority. Generally, these methods involve less measurement activity and are also associated with fewer errors, thus giving more reproducible results than indirect methods. However, depending on component and sampling feasibility for complex components, indirect methods are often the only possible alternative in order to determine the chemical cleanliness of a surface.

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D.2.3.2 Collection method

The chemical contamination under consideration may be presented in one of three groups:

1) total product — where the whole contaminated specimen is sent to test;

2) deposition sample — from proximity exposure onto witness discs or wafers;

3) extracted sample — collected from materials, work tops or equipment, using appropriate physical tools such as swabs, scalpels or adhesive tapes of predetermined contamination level or by solvent extraction.

D.2.3.3 Detachment techniques

“Total product”-type contamination may be collected and analysed as per Figure D.3, using either wet or dry techniques. Deposition samples may also follow the same procedure, wherein the contaminant is either displaced by surfactant in a water solution or dissolved by an organic solvent. The resulting analyte is then fed into the appropriate test system for indirect assessment. Alternatively if “dry” assessment is to be made, the material will be cut into appropriately sized pieces and loaded directly into the instrument.

The third type — extracted samples — requires the contaminant to be transferred to adhesive tapes or SEM stubs that can be directly loaded into the instrument. Low-tack adhesive is used for this work, which involves placing the tape or stub over the surface and making firm contact with the film. The adhesive or stub is then placed in a clean, sealed transport pouch for shipment to the laboratory. Non-leaching cleanroom gloves should be used when taking the sample. Depending on the extraction method used and the contamination form, either analysis can be carried out directly by placing the physical collection sample into the analytical instrument, or further preparation by chemical or thermal techniques will be required.

D.2.4 Packaging of test samples

D.2.4.1 Packaging of samples for chemical examination

Samples that are to be assessed for SCC outside the area of origin should be packaged as follows:

a) Preparation should take place inside the area of origin. The preparer should wear appropriate cleanroom clothing.

b) To handle samples, a technician should wear a new pair of washed nitrile or latex cleanroom gloves and use tweezers or vacuum pickup.

c) When a cleaning process has been used, the samples should be allowed to cool and dry before wrapping in clean aluminium foil.

d) Each sample should be foil-wrapped separately before being placed in a cleanroom bag.

e) Cleanroom-produced polyethylene bags with a fully detailed chemical test certificate should be used. The minimum thickness of the bags should be 80 µm to avoid tearing.

f) The bag should be sealed with specified adhesive tape and an identity label should be fixed to the outside, worded to prevent the bag from being opened outside a controlled environment.

g) A second cleanroom bag should then be placed over the first bag to eliminate particulate contamination.

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D.2.4.2 Removal from packaging

a) The exterior bag should be removed immediately before entering the controlled test environment.

b) The interior bag should not be removed before arrival in the controlled test environment.

c) Full cleanroom garments, including hood and face mask, should be worn when handling the interior pack.

d) A new pair of washed nitrile or latex cleanroom gloves should be worn when examining the samples.

D.2.5 Pre-treatment methods

In situations where a direct measurement technique is not practical, it may be necessary to condition the sample before measurement using an additional surface refinement technique. See also Figure D.3.

Examples of pre-treatment are:

a) Field-ion beam (FIB) microscopy — a preparation technique using an ion beam to create a cross section in the specimen which is then analysed using a high-resolution microscope such as a scanning electron microscope (SEM), a transmission electron microscope (TEM), or a scanning transmission electron microscope (STEM).

b) Thermal desorption (TD) spectroscopy — the contaminant is thermally evaporated into a gaseous substance, which is then analysed using controlled analytical methods. Derivatives of this are:

— TD-GC/MS, used on inorganic and metallic surfaces where the final analysis is carried out using gas chromatography with mass spectrometry;

— TD-QMS, using thermal desorption and measuring ion intensity with a quadrupole mass spectrometer;

— TD-API-MS, using a Thermally Desorbed Atomic Pressure Ionization Mass Spectrometer;

— wafer thermal desorption (WTD), used for semiconductor wafers.

c) Solvent extraction (SE) — a suitable solvent is used to dissolve the surface contaminant creating a test solution for use in one of the following wet microanalysis methods:

— solvent dissolution (SD), where both the contaminant and the substrate matrix are dissolved by the solvent for a few nanometres depth, as shown in D.2.8;

— vapour phase decomposition (VPD), where the contaminant is dissolved using vaporized hydrofluoric acid and tested as shown in D.2.8.

D.2.6 Comparative test methods

The following tables are for guidance only and are not intended to be exhaustive or comprehensive. Table D.1 lists direct test methods and Table D.2 lists indirect methods, which may include pretreatment.

D.2.7 Direct measurements

Complex methods: X/Y (for example, GC/MS), where X is the main measuring method to be used and chromatographic separation (qualification) and determination (quantification) are the basic functions. The classification is based on the ‘X’ process. ‘Y’ describes the additional functions, e.g. sensitivity of mass spectrometry for each chromatogram peak to make up quantification. Additional information for ‘X’, such as a mass chromatogram or the mass spectra of each peak to ascertain qualification, may also be shown.

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Table D.1 — Direct measurement methods and their applications

Technique	Acronym	Principle	Information gained	Sensitivity	Resolution lateral depth		Quantitative analysis?	Typical applications
Auger electron spectroscopy	AES	Method in which an electron spectrometer is used to measure the energy distribution of Auger electrons emitted from a surface	Structure composition	0,1 at %	< 10 nm	20 nm	Semi-quantified with standard materials	Characterization of surface contaminants; multilayer structure
X-ray photoelectron spectroscopy (also known as electron spectroscopy for chemical analysis)	XPS (or ESCA)	Method in which an electron spectrometer is used to measure the energy distribution of photoelectrons and Auger electrons emitted from a surface irradiated by X-ray photons	Quantified elemental and oxidation state	0,1 at %	< 10nm	< 10 nm	Semi-quantified with standard materials	Surface analysis of organic and inorganic materials or residues; depth profiling for thin-film composition; oxide thickness measurements (SiO_2, Al_2O_3); determination of functional polymer groups
Secondary ion mass spectrometry	SIMS	Mass spectrometry is used to measure the mass-to-charge quotient and abundance of secondary ions emitted from sample after ion bombardment	Elemental, simple inorganic molecules	ppm-ppb	2 nm	30 nm	Quantified with standard materials	Trace contamination of surfaces, thin films, multilayer structures and interfaces; composition and impurity measurements of thin films
Time-of-flight secondary ion mass spectrometry	TOF-SIMS	Same as SIMS	Elemental and molecular	ppm-ppb	<5 nm	<0,2 µm	Semi-quantified with standard materials	Microanalysis of organic and inorganic materials; mass spectra direct from surfaces; ion surface imaging
Scanning electron microscopy	SEM	High-power microscope using a focused scanned electron beam to produce images	Structure and morphology	N/A	N/A	N/A	N/A	Characterization of micro-structural surface topography, grain size, oxides and contamination
Energy-dispersive X-ray spectroscopy	SEM EDX	Analytical capability that uses a solid-state cooled detector system to detect and measure X-ray pulses emitted during SEM interaction.	Elemental. May be combined with imaging tools (SEM, TEM, STEM).	0,1 %	≅ 1 µm	≅ 1 µm	yes	Elemental microanalysis
Total reflection X-ray fluorescence spectroscopy	TXRF	X-ray spectrometer measuring energy distribution of fluorescent. X-rays emitted from surface irradiated by primary X-rays under condition of total reflection.	Quantified elemental	10 ppb	1 nm	1 mm	Quantified with standard materials	Qualitative elemental screening of unknown samples, metallic surface contamination on semiconductor wafers, composition ratios of binary thin films

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Table D.1 *(continued)*

Technique	Acronym	Principle	Information gained	Sensitivity	Resolution lateral depth		Quantitative analysis?	Typical applications
Fourier transform infrared spectroscopy	FTIR	Measurement for infrared absorption spectra in which a spectrometer is used to measure the infrared energy	Chemical bonding and molecular structure	1E-19 carbon atoms/m^2	0,1–2,5 µm	10–100 µm	Quantified with standards	Identification of molecular structure organic compounds, films, particles, powders liquids; quantification of O and H in Si wafers; quantification of H in SiN wafers
Multiple internal reflection FTIR	MIR-FTIR	Optically modified version of FTIR; low-angle incident IR is multi-reflected between infrared transparent prism and substrate surface to enhance detection activity	As above	Less than 1E-18 carbon atoms/m^2	As above	As above	As above	Similar to above on surfaces
Quartz crystal microbalance	QCM	Device in which piezoelectric quartz crystals respond to the condensation of gasses by reducing operating Hz in a linear mass-to-Hz relationship	Molecular flux measurement	ng/cm^2-Hz	N/A	N/A	Vibration frequency is compared with reference materials of known concentration	Thin film deposits of gaseous contaminants onto quartz detector surface
Surface acoustic wave detector	SAW	Item as above	Molecular flux measurement	0,01 ng/cm^2-Hz	N/A	N/A	Acoustic wave is compared with reference materials of known concentration	Thin film deposits of gaseous contaminants onto a detector substrate surface
Atomic force microscope	AFM		Shape and molecule size					Visual imaging
Wavelength dispersive X-ray spectroscopy	SEM-WDX	Measuring X-ray wavelength of elements of atomic number of lithium during SEM interaction; measuring X-ray wavelength elements > atomic number of lithium during SEM interaction	Element distribution can be combined with imaging tools (SEM, TEM, STEM, etc.)					Visual imaging
NOTE Resolution and sensitivity are interdependent parameters. Resolution is related to the concentration on the sample. Sensitivity can be influenced by optimizing the sample area.								

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D.2.8 Indirect measurement methods (pre-treatment and measurement)

Table D.2 — Indirect measurement methods and their applications

Technique	Acronym	Principle	Information gained	Typical sensitivity	Typical quantitative analysis applications	
Thermal desorption–gas chromatography/mass spectrometry	TD-GC/MS	Contaminants on substrate surface are thermally desorbed and concentrated into a specific absorption column and analyte. Concentrates are then introduced to a gas chromatograph/mass spectrometer.	Quantified molecular	10 ng/m^2	Quantified with standards	Trace analysis of organic species; evaluation of contaminants on silicon wafers (wafer thermal desorption GC/MS)
Solvent dissolution–ion chromatography/mass spectrometry	SD-IC/MS	Contaminants on substrate surface are dissolved in an appropriate solvent, via a concentration stage if required. An optimum amount of solution is then injected into an ion chromatograph/mass spectrometer.	Quantified ionic	10 ng/m^2	Quantified with standards	Trace ionic contamination analysis; measurement of extractable ionic constituents of powders, encapsulation compounds and glasses, after sample preparation by reflux
Thermal desorption–ion mobility spectrometry	TD-IMS	Contaminants on substrate surface are thermally desorbed and are introduced to an ion mobility spectrometer. Contaminants are decomposed using β rays into ionized fragments, which are separated according to their ion mobility. Ions are then determined with quadrupole mass detector.		parts per billion (ppb) in gas phase 1 μg/m^2	Quantified with standards	Inorganic NH_3 and organic contaminants on substrate surface
Solvent dissolution–capillary electrophoresis-mass spectrometry	SD-CE/MS	The contaminants are dissolved by an appropriate solvent and are introduced into capillary electrophoresis (CE). The contaminants are separated from each other according to their conductive properties and evaluated by mass spectrometer.		200 ng/m^2		
Solvent dissolution–high performance liquid chromatography	SD-HPLC	The contaminants are dissolved with appropriate solvent and are introduced to high-performance liquid chromatograph to be separated from each other and evaluated		2000 ng/m^2	Quantified with standards	Measurement of extractable organic components

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Table D.2 *(continued)*

Technique	Acronym	Principle	Information gained	Typical sensitivity	Typical quantitative analysis applications	
Solvent dissolution–gas chromatography/mass spectrometry	SD-GC/MS	The contaminants on the substrate surface are dissolved with appropriate solvent. A portion of solvent is introduced to GC/MS.		500 ng/m^2 to 10,000 ng/m^2		
Vapour phase decomposition– inductively coupled plasma/mass spectrometry	VPD-ICP/MS	Vapour-phase hydrofluoric acid (HF) is condensed on the surface to dissolve the SiO_2 with the contaminants in it. The HF with contamination forms a droplet on the resultant hydrophobic surface. This is transferred by appropriate method to the measuring instrument ICP/MS.	Quantified elemental	1E-14 atoms/m^2		Ultra trace analysis of metallic contaminants on silicon wafers

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D.2.9 Sampling, analysis and associated quality control (QC) elements

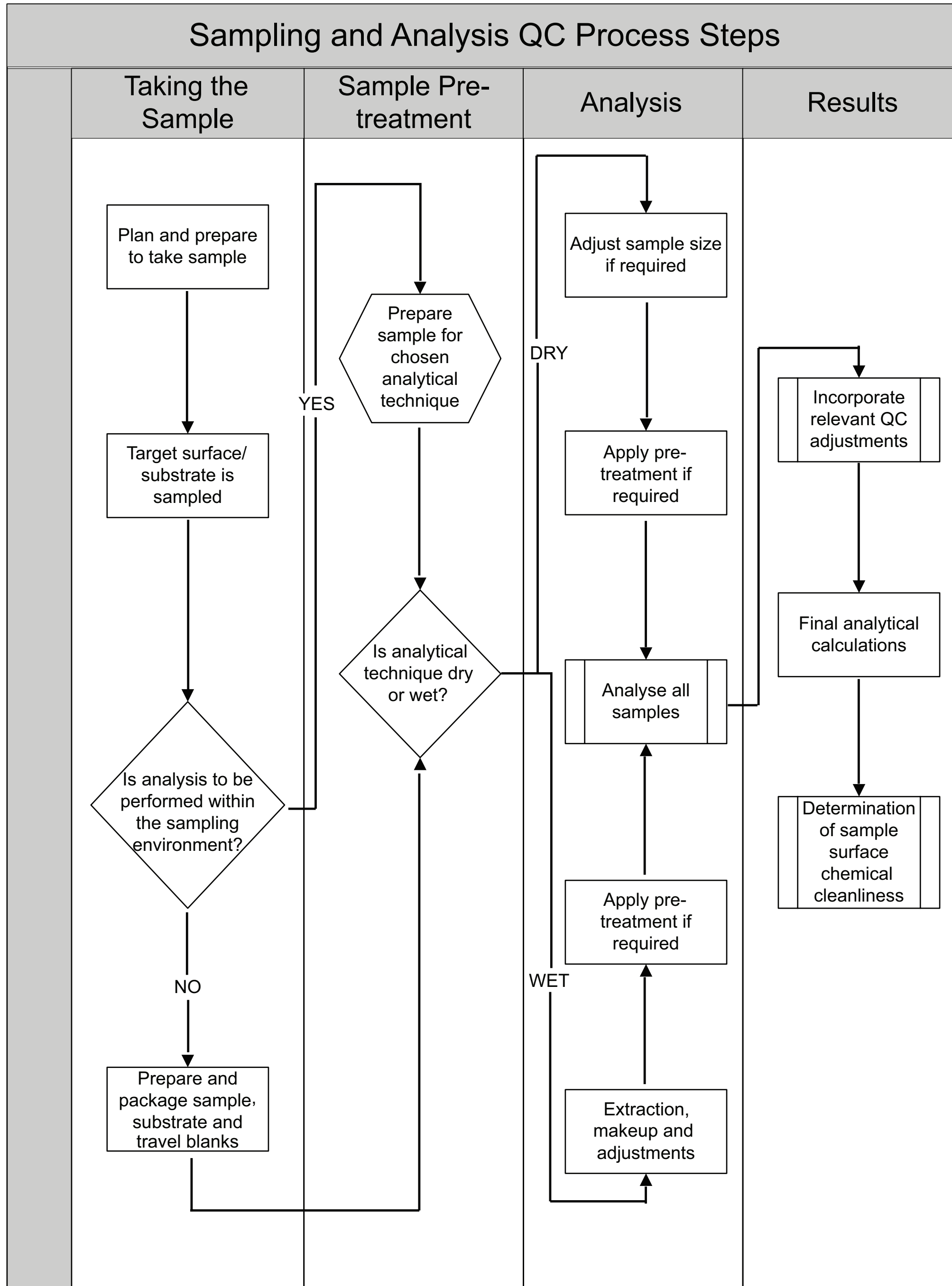

Figure D.3 — Overview flowchart of sampling, analysis and associated QC elements

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D.2.10 Analysis quality control

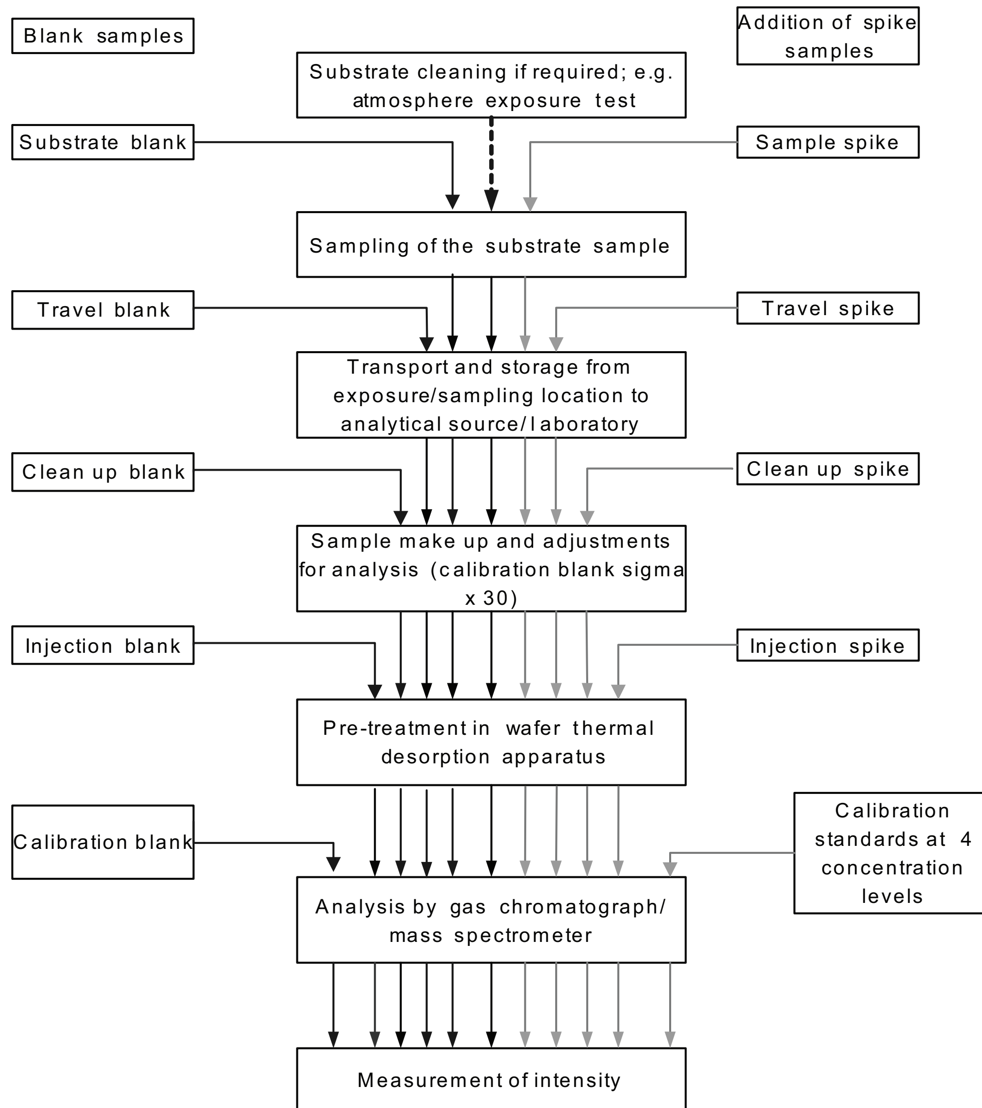

Figure D.4 — Analysis quality control flowchart using silicon wafer TD-GC/MS

In general, spike samples are introduced at critical sampling and analysis stages in order to monitor method performance or quantify the variance and bias of preparation and testing stages. The analysis method will describe spike levels at each stage. In this example of a silicon wafer, intensity and total ion chromatogram (TIC) of an injection spike analyte should be the same as that of the calibration standard, of the same amount added, within the required reliability range. For example, a 100-ng injection spike added to wafer thermal desorption is equivalent to a 100-ng calibration standard in intensity and TIC pattern (see Reference[14] in the Bibliography).

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Annex E
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Test record documentation

Table E.1 is an example of how a test report might be constructed. Other variations of the test report which are agreeable to both the customer and supplier may be used.

Table E.1 — Supporting documentation for testing surface cleanliness by chemical concentration

Formalities Title: Date: Tester: Customer:
Environmental conditions/ cleanroom conditions Temperature: Relative humidity: Locations used for the measurements:
References of standards and guidelines consulted:
Specimen Description of the operating facility and identification: Identification of the test object:
Test set-up (photo and/or sketch) Description of operating parameters: Description of measurement points and measurement methods:
Measurement devices Identification number of measuring device: Measurement range of measuring devices used; equipment resolution and detection limits: Reference of calibration certificate:
Performing the test Test and measurement procedure: Noticeable observations made during the measurement where applicable: Test duration and sampling time:
Reference to ACC or nanoparticle measurements if applicable:
Results and analysis Measurement values and/or their analysis: Visual inspection of the test surface before and after the measurement, where applicable:

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