



Section 1  
Physical-Chemical properties

**Test Guideline No. 124**  
Volume Specific Surface Area of  
Manufactured Nanomaterials

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**OECD Guidelines for the  
Testing of Chemicals**



*OECD GUIDELINE FOR THE TESTING OF CHEMICALS*

**Determination of the Volume Specific Surface Area of Manufactured  
Nanomaterials**

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## 1. INTRODUCTION

1. This Test Guideline (TG) describes a procedure to determine the Volume Specific Surface Area (VSSA) of powdered solid manufactured nanomaterials (MNs). This physical-chemical property may influence the behaviour and biological effects of manufactured nanomaterials (1)(2)(3)(4)(5), and thus can be requested for the safety testing of manufactured nanomaterials (6)(7)(8)(9)(10)(11). Data on VSSA or (mass) specific surface area (SSA) may provide information on the characteristic structure of the nanomaterial and can: a) help identify potential hazards or hazard modifications associated to similar structures, b) help to estimate nanomaterial fate in the environment and c) help to identify modification of exposure site-specific hazards related to the physico-chemical properties (4). Moreover, in some cases, VSSA or SSA data can be used to relate dose to observed fate, behaviour and effects of a specific nanomaterial, as the surface area may be the toxicologically relevant dose metric (5).
2. The VSSA (in  $\text{m}^2/\text{cm}^3$ ) of a material is calculated by multiplying its mass specific surface area (in  $\text{m}^2/\text{g}$ ) with its skeletal density (in  $\text{g}/\text{cm}^3$ ). A good overview on uses and limitations of VSSA can be found in (12).
3. Accordingly, this Test Guideline describes how to determine the external and internal (mass) specific surface area (SSA) of powdered solid MNs using the Brunauer, Emmett and Teller (BET) method (12)(13)(14)(15)(16).
4. This TG also provides instructions on how to determine the skeletal density ( $\rho$ ) of the MN by gas pycnometry. This is the ratio between sample mass and the volume of the sample including the volume of closed pores (if present) but excluding the volumes of open pores and the volumes of void spaces between particles within the bulk sample.
5. As these determinations can be carried out independently, this TG can also be used to determine separately (mass) specific surface area and/or skeletal density, if desired.
6. To determine the SSA of MNs, this TG uses the documentary standard ISO 9277:2022 "*Determination of the specific surface area of solids by gas adsorption – BET method*" (16) that is based on the ability of a material to physically adsorb gas molecules on its surface. The BET method is the most commonly used method for SSA determination, although other methods exist. Furthermore, the BET method is a fast and relatively inexpensive method that can measure the specific surface of a relative large, and hence more representative, sample volume (12).
7. Some instructions on how to address density and porosity by measuring the volume of material samples can be found in the OECD TG 109 "*Density of Liquids and Solids*" (17). However, as ISO 12154:2014 "*Determination of density by volumetric displacement – Skeleton density by gas pycnometry*" (18) allows rapid and efficient determination of the density (commonly referred to as 'skeletal density') of solid materials of regular or irregular particle shape, this documentary standard is used in this TG.
8. In the preparation of this TG, an interlaboratory comparison study (ILC) involving seven laboratories and seven materials (graphene, multiwalled carbon nanotubes, zinc oxide, two types of titanium dioxide, an organic pigment and fumed silica) was carried out (19) which allowed assessing the suitability of the overall method, identifying possible limitations, and obtaining general guidance. Overall, the examined variability<sup>1</sup>, both the within- and between-laboratory variability were within the ranges deemed acceptable despite the limited number of available datasets.

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<sup>1</sup> Evaluated by robust statistics according to the rules described in ISO 5725-5 (20).

9. This TG was developed to address specific information needs regarding manufactured nanomaterials; nevertheless, in principle, this does not preclude its applicability to other solid materials.

## 2. DEFINITIONS

10. Definitions of the terms used in this Test Guideline can be found in the Annex.

## 3. INITIAL CONSIDERATIONS AND LIMITATIONS

11. Most methods for determining the density and/or surface area of solids are the object of national and international standardizing bodies and are frequently specified by governmental agencies. In the following, an adapted very succinct description of the ISO 9277:2022 and 12154:2014 standards is provided, focused on their application to manufactured nanomaterials. The reader is referred to the mentioned ISO standards (16)(18) and reference (12) if additional general information is needed.

12. The ILC included inorganic materials (four metal oxides, two carbon based materials) and one organic pigment (see paragraph 8 and (19)). The results showed that the degassing step, or any other pre-treatment of the materials, is critical. This is particularly important for thermolabile nanomaterials and other challenging materials such as organic materials. The appropriate conditions have to be empirically verified for the testing, for instance using thermogravimetric analysis or spectroscopic methods, and under changing pressure and temperature conditions (19)(5). These conditions may vary depending on the material to be measured. Essentially, it has to be ensured that the material is not decomposed or transformed or its structure modified in the degassing conditions.

13. There remain some uncertainties as to whether the TG is applicable and valid for some more complex types of nanomaterials that were not represented in the ILC, such as coated nanoparticles, composite nanoparticles, microporous nanomaterials and nanomaterials with very high SSA (larger than  $600 \text{ m}^2\text{g}^{-1}$ ), or underrepresented (e.g. organic nanomaterials). Such materials may pose challenges when using ISO 12154 and calculating VSSA. High surface area materials can produce values of skeletal density that are significantly smaller than the true value (18), thus possibly introducing a systematic bias in the determination of VSSA. The skeletal density of coated or composite particles cannot be measured reliably if their precise composition and structure is unknown. If the coating has a very different density than the core and if it is thick, this will influence the VSSA, because VSSA is calculated from SSA by multiplying with the skeletal density. Special care should also be taken to verify the appropriate testing conditions when dealing with these classes of nanomaterials (19).

14. The BET method can be used to determine the external surface area of non-porous particles. It is applicable to nanomaterials that do not absorb the gas used and which exist as disperse powders that consist of particles that are either nonporous, mesoporous with pore diameters between 2 nm to 50 nm, or macroporous. The technique is not suitable for substances, which are microporous solids, although a specific annex is included in ISO 9277:2022, which contains a specific strategy for measurement of surface area for these substances as well. On the other hand, the BET gas adsorption model is inappropriate at small pores sizes (smaller than ca. 2 nm) and a correction may be needed, depending on the nanomaterial (adsorbent) and the gas (adsorptive) used. For larger pore sizes, the BET method may detect both internal pores area and external surface area. Therefore, special

attention must be paid how to address this, as well as to differentiate between internal pores and external surface area (2)(5).

15. The density determined by gas pycnometry is generally the skeleton or skeletal density of the powder material to be measured, which equals the true solid-state density only for particles without closed or inaccessible pores. If inaccessible pores or voids are present then the skeletal density will be lower than the true density. For very fine powders, particularly where the particles being assessed are made up of separate nanocrystals, the presence of a significant volume fraction of micro- or mesopores is considered unlikely (12), (18).

## 4. PRINCIPLE OF THE TEST

### 4.1. Specific Surface Area determination

16. The amount<sup>2</sup> of gas needed to physically adsorb to form a monolayer of gas atoms or molecules on the external surface of the solid is used to calculate the sample's surface area; this value divided by the sample mass gives the specific surface area (SSA).

17. The BET method determines the amount of adsorbate or adsorptive gas required to completely cover the external and the accessible internal pore surfaces of a solid with a monolayer of adsorbate (12)(13)(14)(15)(16). Any gas (e.g. nitrogen, argon, and krypton) may be used, provided that its molecules can be physically adsorbed by weak bonds (van der Waals forces) on the surface of the solid and can be desorbed by a decrease in pressure at the same temperature. Nitrogen is the most common gas used for this method. However, the N<sub>2</sub> molecule, due to its quadrupole moment, can interact with the solid surface (e.g. with highly polar or hydroxylated surfaces) changing the elongated N<sub>2</sub> molecule orientation, thus changing the cross-sectional area (Equation 2) and producing additional uncertainty on the surface area determination (15). Argon at 87 K – a monoatomic gas which is thus less sensitive to the surface chemistry – has become a more reliable alternative adsorptive, particularly in cases where such kinds of interactions can be presumed<sup>3</sup>. For nanomaterials with a very small SSA (SSA < 1 m<sup>2</sup>/g) krypton (18) is recommended as adsorbate, instead.

18. As the adsorption/desorption process is temperature dependent, the measurement of the gas adsorption on the sample surface is performed at equilibrium at a fixed constant temperature<sup>4</sup>.

19. The adsorption isotherm (Figure 1) is the graphical representation of the amount of adsorbed gas ( $n_a$ ), preferably expressed in moles per gram, versus the relative pressures of the gas.

20. The amount of atoms or molecules forming the monolayer ( $n_m$ ) can be derived from the adsorption isotherm using the theory developed by BET (13) (see Equation 1) which is

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<sup>2</sup> Obtained by volumetric, gravimetric, calorimetric or spectroscopic measurement or by the carrier gas method using continuous or discontinuous operation (18).

<sup>3</sup> Typical molecular cross-sectional areas of the gases most commonly used are available in (15) (16). N<sub>2</sub> 0.162, Ar 0.142 (87.4 K), Kr 0.202.

<sup>4</sup> The technique usually involves measurement while keeping the chamber(s) immersed in liquid nitrogen or argon or in a cryostat (or cryocooler), after allowing enough time to reach temperature equilibrium. Moreover, most apparatus automatically provide for the evaporation of the nitrogen or argon, generally by moving the cryogenic bath, so the chamber is always below the surface of the liquid.

limited to the range where the relation between the amount of gas adsorbed  $n_a$  and relative pressure  $p/p_0$  is linear (generally between  $p/p_0 = 0.05$  and  $p/p_0 = 0.3$ ).

$$\frac{p/p_0}{n_a \left(1 - \frac{p}{p_0}\right)} = \frac{1}{n_m C} + \frac{C-1}{n_m C} \frac{p}{p_0} \tag{Equation 1}$$

Where

$p$  = Pressure of the adsorptive in equilibrium with the adsorbate (Pa)

$p_0$  = Saturation vapour pressure of the adsorptive (Pa)

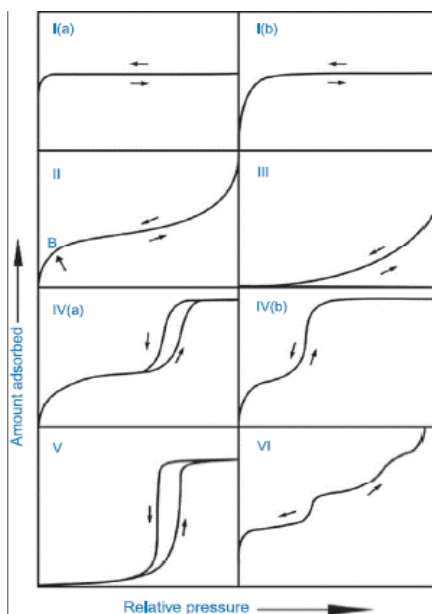
$n_a$  = Specific amount adsorbed (mol g<sup>-1</sup>)

$n_m$  = Specific monolayer capacity of adsorbate (mol g<sup>-1</sup>)

$C$  = BET parameter<sup>5</sup>

21. The BET method is applicable only to adsorption isotherms of type II (disperse, non-porous or macroporous solids) and types IVa and IVb (mesoporous solids) (15). However, in the case of Type IVb isotherms, caution is required since pore condensation may occur at quite low relative pressures  $p/p_0$  (15). Specific details concerning the other types of sorption isotherms are available in reference (15).

Figure 1. Adsorption isotherms classification according to IUPAC (15) (the typical BET method applicability range is between  $p/p_0$  0.05 to 0.3 and for type II and IV isotherms only) © IUPAC/De Gruyter 2015. Point B – the beginning of the middle almost linear section of the isotherm – usually corresponds to the completion of monolayer coverage (15).



22. A linear regression of  $(p/p_0) / [n_a(1 - p/p_0)]$  versus relative pressure,  $p/p_0$ , allows to calculate  $n_m$ , and the specific surface area per mass of the sample (SSA) expressed in m<sup>2</sup>/g is calculated as follows:

<sup>5</sup> The BET parameter  $C$  relates exponentially to the heat of adsorption of a first layer and the heat of condensation of the vapour.

$$SSA = n_m a_m L \quad (\text{Equation 2})$$

Where:

- $SSA$  = Specific surface area ( $\text{m}^2 \text{g}^{-1}$ )  
 $n_m$  = Specific monolayer capacity of adsorbate ( $\text{mol g}^{-1}$ )  
 $a_m$  = Molecular cross-sectional area of the adsorbate ( $\text{nm}^2$ )  
 $L$  = Avogadro constant ( $= 6.022 \times 10^{23} \text{mol}^{-1}$ )

## 4.2. Density Determination by Gas Pycnometry

23. Following ISO 12154:2014 (18), the skeletal density of solid material samples of regular or irregular shape, is determined volumetrically by means of a gas displacement pycnometer.

24. This method determines the volume of a weighted dry outgassed solid sample by placing it in a chamber of known volume, which is connected via a valve to a second chamber of known volume, and it is based on the displacement of a volume of inert gas by the solid space under isothermal conditions. Before starting the measurement, all remaining air in both chambers is removed by flushing the whole system with an inert gas (usually He) and closing the valves afterwards (12). Two different configurations of the chambers are possible (18) (see paragraph 44), and in both cases, the volume of the sample is determined from the variation in pressure in the chambers, that are previously pressurized with inert gas, when opening the connecting valve. Once the volume of the sample and the mass of the sample have been determined, the skeletal density can be readily calculated.

25. The skeletal volume  $V_S$  is calculated from the initial pressure  $p_i$  and equilibrium pressure  $p_e$  and from the values of the calibrated volumes  $V_{cell}$  of the sample chamber and  $V_{ref}$  of the reference chamber according to ISO 12154:2014 (18)], using the following formula:

$$V_S = V_{cell} - \left( \frac{p_e}{p_i - p_e} \right) * V_{ref} \quad (\text{Equation 3})$$

Where:

- $V_S$  = skeletal volume ( $\text{cm}^3$ )  
 $V_{cell}$  = sample chamber volume ( $\text{cm}^3$ )  
 $p_e$  = equilibrium pressure (Pa)  
 $p_i$  = initial pressure (Pa)  
 $V_{ref}$  = reference chamber volume ( $\text{cm}^3$ )

26. The sample mass ( $m_S$ ) is determined by weighing and entered into the instrument software that automatically calculates the skeletal density ( $\rho_S$ ) according to Equation 4:

$$\rho_S = \frac{m_S}{V_S} \quad (\text{Equation 4})$$

27. The value of the skeletal density obtained will usually be equal or close to the density commonly reported for most materials (21). Particle porosity, while increasing the surface area and decreasing overall particle density, should not affect the skeletal density unless there is a significant volume fraction of closed pores (or voids) not accessible to the test gas. This, as discussed in (12) and (18), may be unlikely for most nanomaterial types.



## 5. REFERENCE MATERIALS

### 5.1. Specific Surface Area Determination

28. To ensure the reliability of the measurement results obtained, the performance of the apparatus used for the recording of the physisorption isotherms should be monitored periodically using a fit-for-purpose powder reference material with certified value for specific surface area (by the BET method) that can be obtained from providers listed in ISO 9277:2022. Alternatively, commercially available suitable reference materials for BET can be searched for in the COMAR database (<https://rrr.bam.de/RRR/Navigation/EN/Reference-Materials/COMAR/comar.html>). Certified reference materials are primarily intended to detect potential experimental significant biases and evaluate method accuracy. The reference materials used in the ILC (19), and shown in Table 1, may also be suitable for this provided they are still available.

**Table 1. Reference materials used in the interlaboratory comparison study (19)**

Source	Number	Material	SSA (m <sup>2</sup> /g)	Uncertainty (U) (m <sup>2</sup> /g)
KRISS	301-03-001	TiO <sub>2</sub>	57.00	1.32
NIST	SRM 2696	SiO <sub>2</sub>	22.92	0.36
BAM	P106	TiO <sub>2</sub>	96.60	1.70
KRISS	301-03-002	ZnO	13.51	0.25

Note: KRISS: Korea Research Institute of Standards and Science; NIST: National Institute for Standards and Technology (USA); BAM: Bundesanstalt für Materialforschung und Prüfung (Germany).

Note: Although NIST SRM 1898 and KRISS 301-03-001 represent the same TiO<sub>2</sub> product, the reference materials are prepared from different commercial lots and have significantly different specific surface areas. Skeletal Density Determination by Gas Pycnometry

29. Before starting sample analysis, the pycnometer has to be calibrated under the required measurement conditions and with the frequency established in the instrument user's manual. Stainless steel spheres of certified and traceable volume are generally used as calibration standards. The volume depends on the sample holder set used.

30. In addition to the materials used in the ILC or found in e.g. the COMAR database, the set of representative test materials available from the JRC Nanomaterials Repository (22), although they are not reference materials, can be useful in some cases for routine checking and developing of the actual standard operating procedure. Users must ensure the homogeneity and stability of samples of these materials before using them for routine quality control.

## 6. VALIDITY OF THE METHOD - TEST CONDITIONS

31. The measurement results depend on careful sampling and sample preparation (see examples in ISO 8213 (23) or ISO 14488 (24)), the purity of the adsorptive gas, the control of temperature and pressure and other method parameters described respectively in ISO 9277:2022 (16) for specific surface area and ISO 12154:2014 (18) for skeletal density.

### 6.1. Specific Surface Area Determination

32. The appropriate sample size for analysis depends on the anticipated surface area of the material and on the instrumentation available. Instrument manufacturers often

recommend measuring an amount of test chemical with an estimated total surface area (e.g. 10 m<sup>2</sup>) in the sample holder. Nevertheless, for unknown samples, a preliminary test should be considered in order to get information on the approximate surface area (19).

33. At least five points of the BET isotherm in the relative pressure range,  $p/p_0$ , 0.05 to 0.3 should be recorded to ensure that at least four measuring points are available for determining the correlation curve. If the nanomaterial contains micropores, the linear range of the BET equation shifts to lower relative pressures, it is thus recommended to record the isotherm in the range of relative pressure  $p/p_0$  from 0.005 to 0.3.

34. A correlation coefficient of 0.999, or better, must be obtained in the determination of the BET linear fit range. If this is not the case, check the proper function of equipment, check the data points and, if necessary, change the measurement conditions (for example, increase the sample mass).

35. The BET plot should show a linear correlation within the usual relative pressure range 0.05 to 0.3 with an intercept that must be positive. If a negative intercept ordinate results, then the measurement has to be repeated since the BET equation does not apply. In any case, guidance using the software of a particular instrument manufacturer should be followed carefully (12) (19).<sup>6</sup>

## 6.2. Density determination by gas pycnometry

36. For density determination by gas pycnometry, an analysis gas of sufficient purity that is neither reacting with nor adsorbing onto or absorbing into the solid sample, and that behaves as ideally as possible, should be used. Helium (He, purity  $\geq 99.996$  % by volume) is, therefore recommended for most applications. Moreover, He is able to penetrate even the smallest pores or cracks of a material. However, He may be problematic for some organic materials like cellulose and some low density polymeric materials, due to its ability to permeate through thin walls of particles with closed pores (18).

37. For each sample analysis, a measurement cycle of at least five (sometimes more than 20) single consecutive volume measurements are carried out. The measurement cycle ends when the relative standard deviation (RSD) of at least five consecutive results of the volume determination falls below a predefined value (typically  $RSD < 0.01$  % of the volume of the sample holder) (19).

38. Consecutive volume measurements may show a consistent increase or decrease in surface values. Such a correlation indicates interaction between the sample and the gas or an incomplete removing of atmospheric gases during the degassing step. When interaction is observed, the gas used for the measurement should be substituted by another gas (19).

39. It is essential to ensure that, in each preparation conducted, exposure of the dried sample to atmospheric moisture is avoided (18), (19).

## 7. DESCRIPTION OF THE METHOD AND PROCEDURES

40. For the technical details of the measurement apparatus and procedures, both ISO 9277:2022 and ISO 12154:2014, respectively, may be consulted.

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<sup>6</sup> Once the linear correlation has been established for a particular nanomaterial and for large C parameter, it may be possible to use a simplified procedure requiring only the determination of a single point on the isotherm in the range of relative pressures between 0.2 and 0.3 (16).

41. The same specimens analysed for the determination of the SSA can be used for the density measurement, provided they are appropriately degassed again before the density measurement.

42. Some commercial instruments allow for the simultaneous determination of surface area and skeletal density and are equally acceptable under this Test Guideline. These instruments usually have software incorporated to allow for all the necessary calculations.

### 7.1. Surface Area Determination by the BET Method

43. Several commercial apparatuses for the determination of physisorption isotherms exist on the market, including degassing stations and high vacuum systems in several configurations<sup>7</sup> that enable to perform the determinations. The manometric method is generally considered the most suitable technique for undertaking physisorption measurements with nitrogen, argon and argon at cryogenic temperatures (16). The specific apparatus user's manual applies for further detailed operating instructions.

### 7.2. Density Determination by Gas Pycnometry

44. In this case, two experimental configurations are possible, which are both equally valid<sup>8</sup>. The specific apparatus user's manual applies for further detailed operating instructions. The skeletal volume  $V_s$  is obtained either by measurement or by calculation depending on the type of pycnometer used.

## 8. DATA AND REPORTING

### 8.1. Data Analysis/ Evaluation of Test Results

#### 8.1.1. Surface Area Determination

45. The evaluation of the raw data of adsorption (and desorption) measurement takes place usually by means of software supplied with the measurement instrument, as described in the Principle of the Method section and in ISO 9277:2022.

#### 8.1.2. Density Determination by Gas Pycnometry

46. The evaluation of the raw data of pycnometry takes place usually by means of software supplied with the measurement instrument, as described in the Principle of the Method section and in ISO 1254:2014.

#### 8.1.3. Calculation of the Volume Specific Surface Area (VSSA)

47. The Volume Specific Surface Area is calculated using the formula

$$VSSA = SSA \times \rho_s \quad (\text{Equation 5})$$

<sup>7</sup> They may rely on volumetric, gravimetric, calorimetric or spectroscopic measurement or by the carrier gas method, using continuous or discontinuous operation.

<sup>8</sup> Essentially, both experimental configurations are very similar. They both consist of a have fixed size sample chamber and a reference chamber connected by tubes, a pressure-measuring sensor and three valves. The only difference is the position of the pressure-measuring sensor in relation to the valve that connects the sample chamber to the reference chamber.

where

$SSA$  = specific surface area ( $\text{m}^2 \cdot \text{g}^{-1}$ )

$\rho_s$  = density ( $\text{g} \cdot \text{cm}^{-3}$ )

## 9. TEST REPORT

48. The test report should include, but not be limited to, the following information:

- Reference to this Test Guideline;
- Any variation from the standard method should be described in detail.

### 9.1. Specific Surface Area

#### 9.1.1. Test sample and reference material:

- chemical identification, such as IUPAC or CAS name, CAS number, SMILES or InChI code, structural formula, purity, chemical identity of impurities as appropriate and practically feasible, (preliminary purification step, if any), certified value (in case of reference material), etc.;
- source, lot number, limit date for use, if available;
- method of sampling, sample division;
- stability of the test sample itself, if known; (e.g. by Thermogravimetric Analysis (TGA) or Differential Scanning Calorimetry (DSC) results)
- density (see also next section on density results);
- all information and remarks relevant for the interpretation of the results, especially with regards to impurities and physical state of the substance.

#### 9.1.2. Test conditions:

- pre-treatment and degassing conditions, e.g. degassing in a vacuum or in inert gas flow, temperature and duration of degassing, residual pressure, flushing gas;
- mass of degassed sample, measured with at least 0.1 mg precision in a calibrated balance;
- details of the sample and sample preparation;
- experimental procedure for adsorption isotherm determination. e.g. volumetric, gravimetric, static or continuous gas admission, single point-determination, calibration of dead volume or buoyancy;
- reference of the commercial apparatus and configuration used;
- chemical identity and impurities of any certified specific surface area of a BET reference material, if used for calibration or comparison, including the expanded uncertainty ( $U$ )

#### 9.1.3. Adsorptive:

- chemical nature, purity

**9.1.4. Results:**

- Adsorption isotherm ( $n_a$  plotted against relative pressure,  $p/p_0$ ), measurement temperature;
- pressure range of linearity and the correlation coefficient;
- SSA value obtained, together with its expanded measurement uncertainties,  $U$  (according to the ISO/IEC Guide 98-3 (25));
- results obtained with the reference material when used for calibration or performance verification.

**9.1.5. Discussion of results:**

- Evaluation parameters: multipoint or single-point determination, BET plot or range of linearity, monolayer amount  $n_m$ , BET parameter  $C$ , molecular cross-sectional area used;
- Specific surface area.

**9.1.6. Conclusion****9.2. Skeletal Density****9.2.1. Test substance:**

- chemical identification, such as IUPAC or CAS name, CAS number, SMILES or InChI code, structural formula, purity, chemical identity of impurities as appropriate and practically feasible, (preliminary purification step, if any), etc.;
- source, lot number, limit date for use, if available;
- purity, method of sampling, sample division;
- stability of the test chemical itself, if known;
- all information and remarks relevant for the interpretation of the results, especially with regards to impurities and physical state of the substance;

**9.2.2. Test conditions:**

- pre-treatment and degassing conditions, e.g. degassing in a vacuum or in inert gas flow, temperature and duration of degassing, residual pressure, flushing gas;
- mass of degassed sample, measured with at least 0.1 mg precision in a calibrated balance
- certified or local reference material(s) used for performance testing of the instrument and validation of results;
- details of the sample and sample preparation;
- the type of pycnometer and its manufacturer, configuration, chamber volumes, sample mass, sample volume, temperature;
- purity of the gas

**9.2.3. Results:**

49. the skeletal density of the sample, including its expanded measurement uncertainty,  $U$  (according to the ISO/IEC Guide 98-3 (25)).

50. results obtained with the reference substances when used for calibration or comparison;

**9.3. VSSA****9.3.1. Calculated VSSA value.**

- When reporting VSSA values, it should be specified whether reference tabulated density values have been used (e.g. from the CRC Handbook of Chemistry and Physics (21), or skeletal density values obtained by Helium Pycnometry or another measurement. If measured skeletal density values are used, the test report should include the details on the measurements as indicated above.

## 10. LITERATURE

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## Annex A. Definitions

51. For the purposes of this Test Guideline, the following definitions apply:

<b>Absorption:</b>	diffusion of particles of gas or liquid into liquid or solid materials.
<b>Adsorbate:</b>	adsorbed gas.
<b>Adsorbed amount:</b>	quantity of gas adsorbed at a given pressure and temperature. Normally expressed in moles.
<b>Adsorbent:</b>	solid material on which adsorption occurs.
<b>Adsorption:</b>	enrichment of the adsorptive gas at the external and accessible internal surfaces of a solid material.
<b>Adsorptive:</b>	gas or vapour to be adsorbed.
<b>Closed pore:</b>	pore totally enclosed by its walls and hence not interconnecting with other pores and not accessible to fluids.
<b>Density:</b>	ratio of the mass of a certain amount of a sample to the volume occupied by that mass (g/m <sup>3</sup> ).
<b>Dead volume:</b>	volume of the sample holder not occupied by the sample. This volume is also known as ‘free space’, ‘headspace’, or ‘dead space’.”
<b>Equilibrium adsorption pressure:</b>	pressure of the adsorptive gas in equilibrium with the adsorbate.
<b>Isotherm:</b>	relationship between the amount of gas adsorbed and the equilibrium pressure of the gas, at constant temperature.
<b>Macropore:</b>	pore with a width greater than approximately 50 nm.
<b>Mesopore:</b>	pore with a width between approximately 2 nm and 50 nm.
<b>Micropore:</b>	pore with a width of approximately 2 nm or less.
<b>Molecular cross-sectional area:</b>	molecular area of the adsorbate, i.e. the area occupied by an adsorbate molecule in the complete monolayer (nm <sup>2</sup> ).
<b>Monolayer amount:</b>	number of moles of adsorbate that form a monomolecular layer over the surface of the adsorbent.
<b>Open pore:</b>	pore not totally enclosed by its walls and (potentially) interconnecting with other pores and accessible to fluids

<b>Particle:</b>	minute piece of matter with defined physical boundaries.
<b>Physisorption:</b>	weak bonding of the adsorbate, reversible by small changes in pressure or temperature.
<b>Relative pressure (<math>p/p_0</math>):</b>	ratio of the equilibrium adsorption pressure ( $p$ ) to the saturation vapour pressure ( $p_0$ ) at analysis temperature.
<b>Saturation vapour pressure:</b>	vapour pressure of the bulk liquefied gas at the temperature of adsorption.
<b>Skeleton density:</b>	ratio between sample mass and the volume of the sample including the volume of closed pores (if present) but excluding the volumes of open pores and the volumes of void spaces between particles within the bulk sample.
<b>Specific Surface Area (SSA):</b>	absolute surface area of the sample divided by sample mass ( $\text{m}^2/\text{g}$ ). Sometimes may be denoted as mass-specific surface area or specific surface area by mass
<b>Surface area:</b>	extent of available area as determined by a given method under stated conditions. For the purposes of this Test Guideline, the area includes the external surface of a solid plus the internal surface of its accessible, macro-, meso- and micropores.
<b>Volume specific surface area (VSSA):</b>	amount of surface area per unit volume of adsorbent (effectively mass SSA multiplied by the sample density). Sometimes denoted as surface-area-to-volume ratio or surface-to-volume ratio.