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Nanotechnologies — Silica nanomaterials — Specification of characteristics and measurement methods for silica with ordered nanopore array (SONA)

Spécification des caractéristiques et méthodes de mesure de la silice à réseau de nanopores ordonnés (SONA)

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Foreword

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This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies.*

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Introduction

Silica with ordered nanopore array (SONA) is expected to act as novel catalysts and adsorbents because of the presence of their uniform nanopores. In addition to SONA, recently developed synthetic strategies have created a huge number of compositional and morphological variations. Therefore, SONA is expected to be applied in various fields such as electronics, optics and materials. They also have potential uses as electrodes for fuel cells and hydrogen-storage materials, all of which are owing to the presence of periodic nanopore and the physical properties of inorganic frameworks.

SONA, as described in the previous reports[1]-[5], has an amorphous structure like silica-gel and exhibits a honeycomb (hexagonal), 3D (cubic) and wormhole (gyroid) pore structure (see Annex A) with ordered cylindrical channels from 2 nm to 50 nm in diameter. The pores are constructed with thin silica walls which are connected to form the regular pore arrangements. The delicate structures of silica walls and their connected structures are influenced by their preparation, aging and storage conditions. The global SONA market is anticipated to witness significant growth on account of a wide range of existing and potential applications of the product in electronics, biomedical, drug delivery and optical fields. A market survey shows extensive use of SONA in the chemical industry as a catalyst support for synthesis of various chemicals[6].

SONA have a variety of industrial applications as catalysts, adsorbents, molecular sieve, where their properties and use cases highly depend on their production processes that affect their nanopore arrangements. They do not have long-range SiO_2 ordering confirmed by powdered X-ray diffraction, showing XRD peaks in low angle region (see Annex A). Having the ability to characterize these materials helps developers adapt to new research frontiers, such as bulky organometallic or inorganic complexes, biosensors from embedded enzymes on nanostructured silica,[7]-[8] to application in energy-efficient desiccation. Standardization of SONA can unify different types of SONA test reports in industry. This allows users to compare or select most suitable and qualified SONA for their applications.

Nanotechnologies — Silica nanomaterials — Specification of characteristics and measurement methods for silica with ordered nanopore array (SONA)

1 Scope

This document specifies the characteristics of samples of silica with ordered nanopore array (SONA) to be measured in powder form and the industrially available measurement methods used to determine said characteristics. This document provides a sound base for the research, development and commercialization of SONA for various applications.

This document excludes silica-gel, fumed silica and chemically modified SONA.

NOTE The pore size of SONA ranges usually from one nanometre to several tens of nanometres.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
area equivalent diameter
diameter of a circle having the same area as the projected image of the particle

Note 1 to entry: It is also known as the Heywood diameter or as the equivalent circular diameter.

[SOURCE: ISO 13322-1:2014, 3.1.1]

3.2
Feret diameter
distance between two parallel tangents on opposite sides of the image of a particle

[SOURCE: ISO 13322-1:2014, 3.1.5]

3.3
nanopore
cavity with at least one dimension in the *nanoscale* (3.4), which can contain a gas or liquid

Note 1 to entry: The shape and content of the cavity can vary. The concept of nanopore overlaps with micropore (i.e. pore with width of about 2 nm or less), mesopore (i.e. pore with width between approximately 2 nm and 50 nm), and macropore (i.e. pore with width greater than about 50 nm).

Note 2 to entry: When nanopores are appropriately interconnected, they can allow for transport through the material (i.e. permeability).

[SOURCE: SOURCE; ISO 80004-1:2023, 3.4.3, modified — Notes 1 and 2 to entry have been added.]

3.4
nanoscale
size range from approximately 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from a larger size will typically, but not exclusively, be exhibited in this size range. For such properties, the size limits are considered approximate.

Note 2 to entry: The lower limit in this definition (approximately 1 nm) is introduced to avoid single and small groups of atoms from being designated as nano-objects or elements of nanostructures, which can be implied by the absence of a lower limit.

[SOURCE: ISO 80004-1:2023, 3.1.1, modified — Notes 1 and 2 to entry have been added.]

3.5
ordered nanopore array
nanopores (3.3) formed and arranged in a pattern according to the specified rule

Note 1 to entry: The ordered nanopore array can be fully or partially regular depending on the *silica with ordered nanopore array* (3.7) sample.

Note 2 to entry: See Annex A for the types of pore array.

3.6
particle size
dimension that is representative of the size of an individual particle

Note 1 to entry: The particle size is usually expressed as Feret diameter or area equivalent diameter.

3.7
pore size
pore width, i.e. diameter of cylindrical pore or distance between opposite walls of slit

[SOURCE: ISO 15901-2:2022, 3.17]

3.8
pore size distribution
fraction by numbers or by volume of each classified *pore size* (3.7) which exists in a material

Note 1 to entry: The pore size distribution is usually expressed by the full width at half maximum of the distribution main peak.

[SOURCE: ISO 3252:2023, 3.3.47, modified — "percentage" has been replaced with "fraction" in the definition and Note 1 to entry has been added.]

3.9
silica with ordered nanopore array
SONA
amorphous silica containing internal structures in the form of *ordered nanopore array* (3.5)

4 Abbreviated terms

AAS	atomic absorption spectrometry
AFM	atomic force microscopy
BET	Brunauer–Emmett–Teller
BJH	Barrett-Joyner-Halenda
EDX	energy dispersive X-ray spectrometry

EPMA	electron probe micro analyser
ICP	inductively coupled plasma
SEM	scanning electron microscopy
TEM	transmission electron microscopy
TGA	thermal gravimetric analysis
XRD	X-ray diffraction
XRF	X-ray fluorescence spectrometry

See ISO/TS 80004-6 for the definitions of the abbreviated terms listed in this clause.

5 Characteristics and measurement methods

5.1 General

The characteristics to be measured or identified of a SONA sample and the applicable measurement methods are listed in Tables 1 to 2. The essential characteristics listed in Table 1 shall be measured by using the listed measurement methods. The optional characteristics listed in Table 2 should be measured by using the listed measurement methods.

Table 1 — Essential characteristics of SONA and measurement methods

Characteristics	Measurement methods	Relevant standards
Chemical composition content	ICP, AAS, XRF, SEM/EDX or EPMA	—
Pore size	Gas adsorption method	ISO 15901-2
Pore size distribution	Gas adsorption method	ISO 15901-2
Specific pore volume	Gas adsorption method	ISO 15901-2
Specific surface area	Gas adsorption method	ISO 18757
Type of ordered nanopore array	XRD	—
Moisture content	Oven drying and weighing	ISO 638-1

Table 2 — Optional characteristics of SONA and measurement methods

Characteristics		Measurement methods	Relevant standards
Hydrate content		TGA	—
Stability	against water	Gas adsorption method	—
	against humidity		
Morphology		SEM, TEM or AFM	—
Particle size		SEM, TEM or AFM	—

5.2 Descriptions of characteristics and measurement methods

5.2.1 General

The characteristics as well as the measurement methods listed in Tables 1 and 2 are described in 5.2.2 to 5.2.12.

Unless otherwise specified, measurements shall be performed under normal laboratory conditions in terms of temperature and humidity.

5.2.2 Chemical composition content

The chemical composition content refers to the ratio of the mass of chemical compounds to that of the total mass of a dried SONA sample. Impurities in a SONA sample can affect the stability and performance of SONA. The chemical composition contents shall be measured and the results expressed as a mass fraction percentage for individual compounds.

The content of SiO_2 and the minor components (e.g. Al_2O_3, FexOy) shall be measured. The chemical composition content shall be measured by ICP, AAS, XRF, SEM/EDX or EPMA for a SONA sample in powder form.

5.2.3 Pore size

When using SONA for heat pump, drug carrier for various adsorbents, catalyst carriers, separating agents, pharmaceuticals, etc., pore size and size distribution of SONA affects adsorption by capillary condensation.

Pore size refers to the inner diameter of pore of the SONA. The pore size shall be measured.

The measurement method shall be gas adsorption method. Pore size is calculated by Barrett-Joyner-Halenda (BJH) formula that is developed from Kelvin capillary condensation theory. Measurement procedures can be found in ISO 15901-2.

The average pore size shall be the median diameter by the volume base. The results of average pore size shall be expressed in nm.

5.2.4 Pore size distribution

When using SONA for heat pump, drug carrier for various adsorbents, catalyst carriers, separating agents, pharmaceuticals, etc., the performance of SONA is affected by the structural irregularity of pore size array, which is indicated by the pore size distribution.

The measurement method shall be gas adsorption method. The size distribution shall be displayed in the histogram. The full width at half maximum (FWHM) is measured for the main peak in the histogram chart. The pore size distribution of a SONA sample refers to the FWHM of the main peak. The smaller FWHM the better uniformity. Measurement procedures can be found in ISO 15901-2. See Annex B for information on pore size distribution.

5.2.5 Specific pore volume

When using SONA for heat pump, drug carrier for various adsorbents, catalyst carriers, separating agents, pharmaceuticals, etc., the specific pore volume affects how much of the host can be included.

Specific pore volume refers to the ratio of the total volume of pores to the mass of a dried SONA sample. The specific pore volume shall be measured by gas adsorption method.

The total specific pore volume of a SONA sample is obtained from the saturated adsorption amount of nitrogen by assuming that each pore has been filled with liquid nitrogen. The results of specific pore volume shall be expressed in cm^3/g.

The measurement procedures of specific pore volume by gas adsorption method are described in ISO 15901-2.

5.2.6 Specific surface area

When using SONA for drug carrier for various adsorbents, catalyst carriers, separating agents, pharmaceuticals, etc., it expresses the chemical properties and desired functions by controlling the physical properties of the surface such as the shape and size of the pores and the physical properties. The specific surface area of SONA is the most basic surface physical property and an important indicator of functional expression.

Specific surface area refers to the ratio of the total surface area to the mass of a dried SONA sample. The specific surface area shall be measured by gas adsorption method.

Specific surface area of a SONA sample shall be measured by gas adsorption method using BET analysis. The measurement procedures can be found in ISO 18757. The results shall be expressed in m^2/g.

The measurement procedures of specific surface area by gas adsorption method are described in ISO 9277.

5.2.7 Type of ordered nanopore array

SONA can have different types of ordered nanopore array such as hexagonal, cubic and wormhole. See Figure A.1.

Since various adsorption characteristics differ depending on the shape of the pores, it is necessary to identify the type of ordered nanopore array. Representative pattern showing clearly the form, structure and shape of pores in a SONA sample shall be taken by XRD.

Type of ordered nanopore array for a SONA sample shall be identified as hexagonal, cubic or others from XRD spectrum chart. See Annex A for the type of structure/shape of ordered pores.

5.2.8 Moisture content

SONA is likely to adsorb moisture due to the high porosity. The moisture content of a SONA product affects a lot the commercial value.

Moisture content refers to the ratio of the mass of the quantity of water in a SONA sample to the mass of the dried SONA sample. Moisture content shall be measured by the oven-drying method and be expressed as a percentage.

A SONA sample is heated up to 110 °C and kept for a given period in an oven. After cooling, the SONA sample is weighed. The heating is repeated until constant mass is reached so that the difference in measured masses between successive heatings becomes less than the specified value. Care should be taken to prevent moisture adsorption from the environment of the SONA sample under test in the measurement processes. ISO 638-1 is useful for the measurement procedures of moisture content.

NOTE Volatiles at 110 °C contained in a SONA sample, if any, can be included in the measured moisture content.

5.2.9 Hydrate content

Bound water, hydrogen-bonded hydroxyl groups and isolated hydroxy groups contained in SONA affects the catalytic performance and stability of the material. The amount of bound water, hydrogen-bonded hydroxy groups and isolated hydroxy groups can be estimated by dehydration of a SONA sample between 110 °C and 500 °C[18].

Hydrate content refers to one minus the ratio of the mass of a SONA sample at 500 °C where bound water, hydrogen-bonded hydroxy groups and isolated hydroxy groups have been removed to that of the SONA sample at 110 °C where adsorbed water has been removed.

The hydrate content should be measured by TGA. The measurement results should be expressed as a percent mass fraction.

NOTE Other volatile sources between 110 °C and 500 °C, if any, contained in a SONA sample are included in the TGA data.

5.2.10 Stability

5.2.10.1 General

Water and humidity have a significant impact on the stability of SONA in a wide range of applications. The stability of a SONA sample against water and humidity can be indicated by the ratio of the full widths at half maximum (FWHM) of the main peak in the pore size distribution before and after the stability test. The stability of a SONA sample against water and humidity should be measured by gas adsorption method and the results be expressed as a percentage.

5.2.10.2 Stability against water

The structural regularity of ordered nanopore array of a SONA sample can be deteriorated during the drying process in the environment after immersion of the sample into water[19].

The stability test condition should be immersion of a SONA sample into water for about 10 min at room temperature. Measurements of the pore size distribution are made in the environment condition before the immersion and after drying of the SONA sample.

5.2.10.3 Stability against humidity

The structural regularity of ordered nanopore array of a SONA sample can be deteriorated by humidity of the environment.

The stability test should be made in an environmental test chamber where a SONA sample is exposed to the relative humidity changing between more than 90 % and less than 10 % successively 10 times at the temperatures agreed between the buyers and sellers.

5.2.11 Morphology

When SONA is used as a membrane, plate or column of chromatography, morphology of SONA particles has a significant impact on performance. Representative pictures showing clearly the form, structure and shape of SONA particles should be taken by SEM, TEM or AFM. The measurement method used should be indicated as SEM, TEM or AFM. The magnification scale should be shown on each picture. The number of pictures to be taken may be agreed between buyer and seller.

5.2.12 Particle size

The size of SONA particles are basic characteristics affecting the performance of SONA applications such as membrane, plate and column of chromatography.

Particle size refers to the outer diameter of the SONA primary particles. Feret diameter or area equivalent diameter of a SONA particle should be measured on a two-dimensional image taken by SEM, TEM or AFM. The SONA particle sample should be well dispersed before measurements. The median of the measured diameter data should be reported and expressed in µm. The number of particles to be measured may be agreed between buyer and seller.

6 Test report

The test report shall include:

a) sample identification:
   - sample name,
   - manufacturer's name, and
   - lot number,

b) storage conditions prior to testing;

c) name of characteristics measured or identified that are listed in Table 1;

d) measurement methods used for individual characteristics determination;

e) date of measurement and name of organization that made the measurements for individual characteristics;

f) quantitative and/or qualitative results of measurements for individual characteristics;

g) information on the uncertainty of measurement results;

h) additional information, if any, supporting the measurement results;

i) if there are deviations from this document, give the name of and detailed information on the measurement methods used and their justification;

j) any unusual features observed;

k) the International Standard used (i.e. ISO/TS 22298:2024).

When the characteristics listed in Table 2 are measured, the above items a) to k) can be applied.

Annex A
(informative)

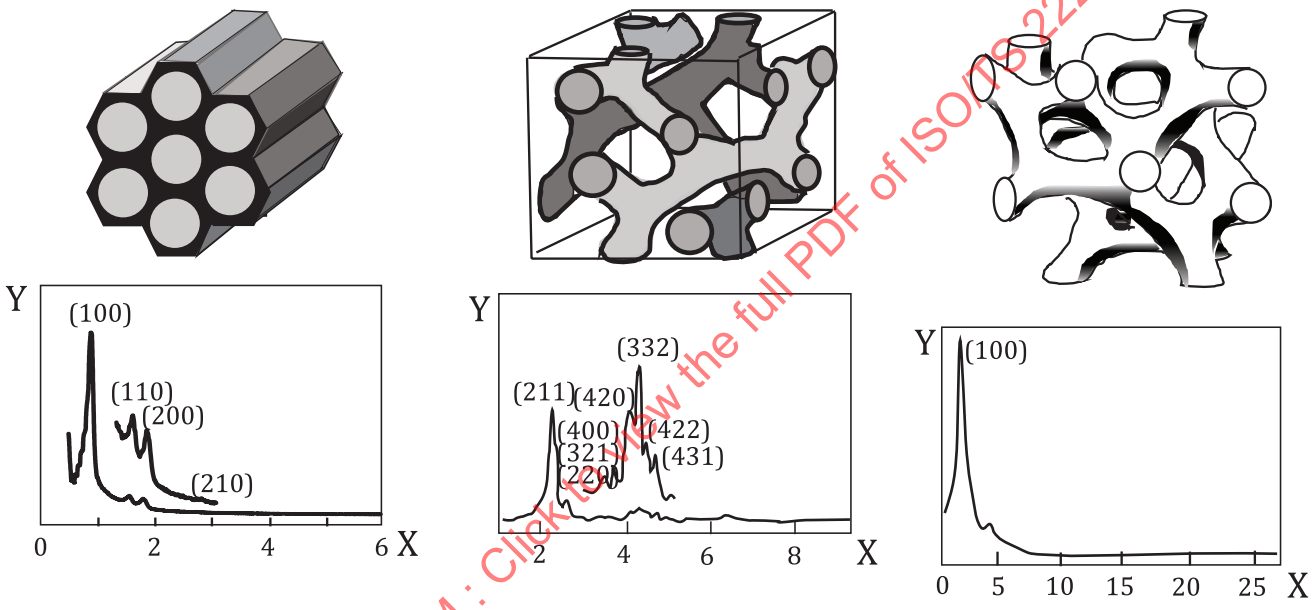
Type of ordered nanopore array

The SONA family of materials produced is classified according to the order of the pores as shown in Figure A.1:

a) hexagonal,

b) cubic, and

c) wormhole (gyroid).



a) Hexagonal **b) Cubic** **c) Wormhole (gyroid)**

Key

X 2θ (degree)

Y arbitrary units

Figure A.1 — X-ray diffraction patterns of different types of ordered nanopore array