Measurlabs

Test report ID 18956 - v1

Customer	DeeAnn Veeder Energy Strategy Associates, Inc. New York 203, Spencertown, NY 12165, USA
Assignment	Measurlabs provided **Nanoparticle testing and powder characterization according to REACH** as requested by the customer.
Sample(s)	Sampling was performed by the customer.

Measurlabs Sample name	Customer Sample name	Sample matrix	Lot/Batch/Reference number	Requested measurements
ID 18956-1	Nan-O-Sil ASD™ F040	Silicon dioxide/Synthetic amorphous Silica, Pyrogenic (fumed) amorphous Silica)	Lot #1658	Nanoparticle testing and powder characterization according to REACH

Samples received	13/03/2025 (dd/mm/yyyy)
Results	The results presented on the next page(s) relate to the tested sample(s) only.

On Wednesday, 16 April 2025, issued by

Kamal Raj R Mundoli

Kamal Raj R Mundoli
Project Manager

+358 50 386 4239
kamal.mundoli@measurlabs.com

Measurlabs

*Teollisuuskatu 33
00510 Helsinki
Finland*

This report is also available on our customer portal:
https://measurlabs.com/portal/

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Page 1(25)

Test results - Volume specific surface area (VSSA)

Methods Nitrogen adsorption sorptometry was used along with the application of the Brunauer, Emmett and Teller (BET) model to measure the specific surface area; Helium Pycnometry was used to measure the effective density. VSSA was calculated using the following formula:

$$VSSA \ = \ \rho \cdot S_{specific}$$

where ρ is the density of the material and $S_{specific}$ is the specific surface area of the material.

Nitrogen adsorption sorptometry

Measurements of the specific surface area using the BET method are carried out in accordance with ISO 9277:2010. The lab has Micromeritics Gemini VII specific surface analyser, a Micromeritics Smart VacPrep degasser and a Cryotec CRIOS - 55 freeze dryer. The analyses carried out in the context of this study were carried out with these different equipment. The sample was degassed at 50°C under vacuum for 2 hours

5.0 grade nitrogen was used for adsorption. Detailed technical presentation is given in Appendix B.

Helium Pycnometry

Pycnometry measurements are based on the ISO 12154:2014 standard. 10 purges and 5 measurements were made on Micromeritics- AccuPyc 1340 device. Detailed technical presentation is given in Appendix B.

The measurements were performed by an ISO IEC 17025 accredited external service provider.

Additional information **Table 1.** The reference material used for the performance test and validation of the results of Nitrogen adsorption sorptometry

Sample Reference	Analysis date	Specific Surface (m²/g)	Theoretical Specific Surface (m²/g)
MRC 20.3 m²/g	24/03/2025	20.45	20.3 ± 0.75

Table 2. The reference material used for the performance test and validation of the Helium Pycnometry results

Sample Reference	Analysis date	Volume (cm³)	Theoretical volume (cm³)
Tungsten beads	24/03/2025	6.3724	6.372350 ± 0.005

If (VSSA) < 6 m²/cm³: Raw material classified as non-nano. End of the study.
If (VSSA) > 6 m²/cm³: Need to perform SEM observation and potentially number based SEM size distribution to check the presence of nanomaterials.

Appendices **Appendix A** provides a reminder of regulatory materials relevant for this analysis.
Appendix B provides technical description of nitrogen adsorption sorptometry and Helium pycnometry part of VSSA calculations.
Appendix C presents Isotherm plots related to the VSSA analysis.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 2(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Results

Table 3. Specific surface area by nitrogen adsorption

Sample name	Test number	Weight of the sample (g)	Specific surface area, $S_{specific}$ (m²/g)
ID 18956 - 1	Test #1	0.4401	23.6231 ± 0.1268 *
	Test #2	0.5528	23.5661 ± 0.1224*

*uncertainty determined by the instrument

Table 4. Specific surface area, density, and VSSA of the sample.

Sample name	Mean specific surface area (m²/g)	Skeletal density (g/cm³)	VSSA (m²/cm³)
ID 18956 - 1	23.5946	2.2715	**53.60**

Conclusions of VSSA

According to the combined measurements of nitrogen adsorption sorptometry and helium pycnometry, the VSSA estimated is 53.6 m²/cm³, i.e. larger than 6 m²/cm³.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 3(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Test results - SEM-EDX + PSD

Methods   Around 5 µg of the powder is placed in two glass vials. 10 ml of isopropanol, filtered on a 0.45 µm filter, are added to the vial. It is placed in an ultrasonic bath for 10 minutes. It must be noted that it is not possible to know the amount of energy provided by the ultrasonic bath as the position of the vial and the number of samples in the bath might have an impact on it.

Silicon wafers are cleaned with an acidic solution (9:1 mix of ultrapure water and HNO_3) and isopropanol. 2.5 µl of the solution are deposited on it. The sample is dispersed on the wafer using spin coating (1000 rpm / 1 minute).

The sample was not coated as it prevents elemental analysis by EDX, and our SEM can perform at low vacuum, allowing the observation of materials with poor or no conductivity. A SEM-FEG with controlled pressure is used to obtain SEM images. Additional sample preparation details are given below.

Deposition method	Spin Coating
Dilution Medium	Isopropanol
Sample concentration	Approximately $5x10^{-4}$ g/l
pH of the solution	pH = 6
Ultrasonication	Yes
Ultrasonic bath power	240 W
Ultrasonic bath frequency	40 Hz
Coating	No

The measurements were performed by an ISO IEC 17025 accredited external service provider.

Appendices   **Appendix A** provides a reminder of regulatory materials relevant for this analysis.
Appendix D provides technical description of SEM EDX and methodology according to 2022 EC recommendation.
Appendix E provides additional SEM images showing EDX results and particle counting.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland
Page 4(25)
https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Results

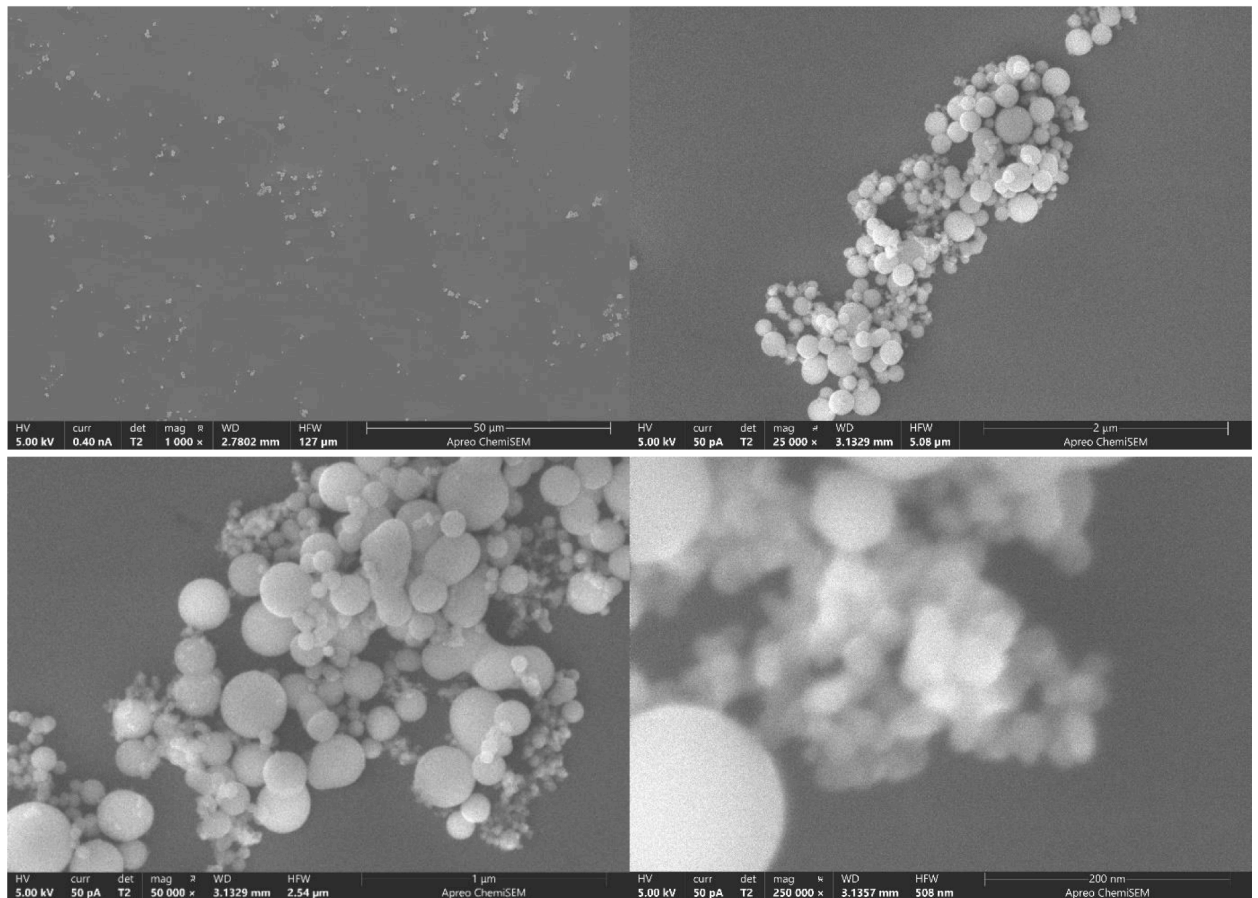**Morphological observations of particles by SEM**

Figure 1. SEM Observation of sample ID 18956-1. Magnification (M) = 1000 (top left); M = 25000 (top right); M = 50000 (bottom left) and M = 250000 (bottom right)
.

SEM observations reveal the presence of particles with a spherical shape category. Those particles are forming agglomerates of various sizes, but do not appear to be strongly bonded to each other. A wide range of particle size is observed, with particles ranging from a few nanometres to almost one micron. The particles are selected on a random manner on the wafer. Due to the wide range of particle size, it is not possible to acquire images showing clearly both ends of the dimension range. As such, the operator needs to take images at various magnifications. The selection of images for the particle size distribution is therefore not totally random and a human selection bias is introduced. This bias is, as of today, not avoidable. Even if an independent count over the two size ranges (few tens nanometres and few hundred nanometres) seems the most relevant, as the material analysed is not considered to be a mixture, a single distribution must be carried out. The operator tries to select images allowing to get a particle size distribution that stays true to the proportion of smalls, mediums et larges particles. It should be noted that, according to observations, the nanometric size range is greater in number than the large population (a few hundred nanometres).

These properties are described based on a series of representative and selected electron micrographs, recorded at several magnifications and covering the entire SEM magnification range.

The particles are mainly composed of Oxygen (O) and Silicon (Si).

EDX spectra and elemental composition is presented in Appendix **Figure E1.**

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 5(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Number based size distribution of particles by SEM

A counting of 322 particles was carried out using an image analyser. The counted particles were chosen randomly on the wafer. Only particles with a discriminable physical contour were counted.

The size of the primary particles takes precedence over agglomerates or aggregates, since the latter will be considered nanomaterials if the particles of which they are composed are nanometric. The distribution is presented using four parameters shown below. A selection of SEM images, with and without the count performed, is presented in the Appendix Figure E2.
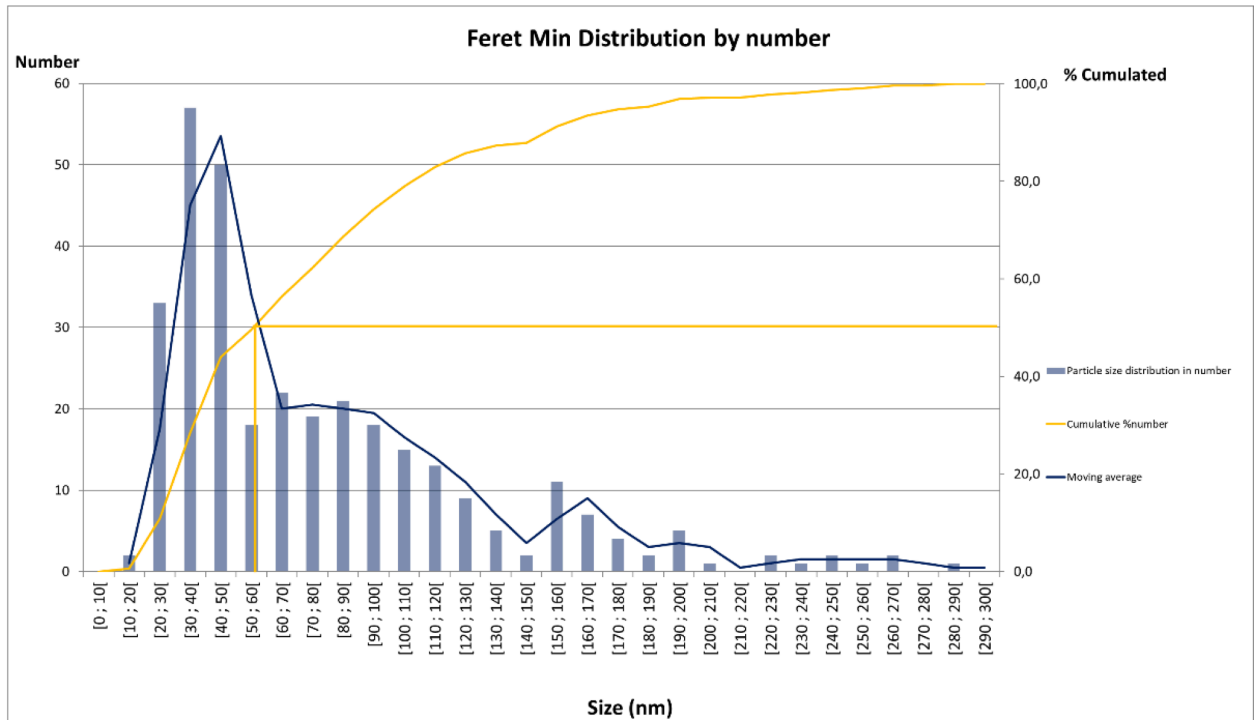
The minimum of Feret diameter:

Figure 2. SEM particle size distribution of the sample **ID 18956 - 1** by minimum Feret Diameter.

The maximum of Feret diameter:
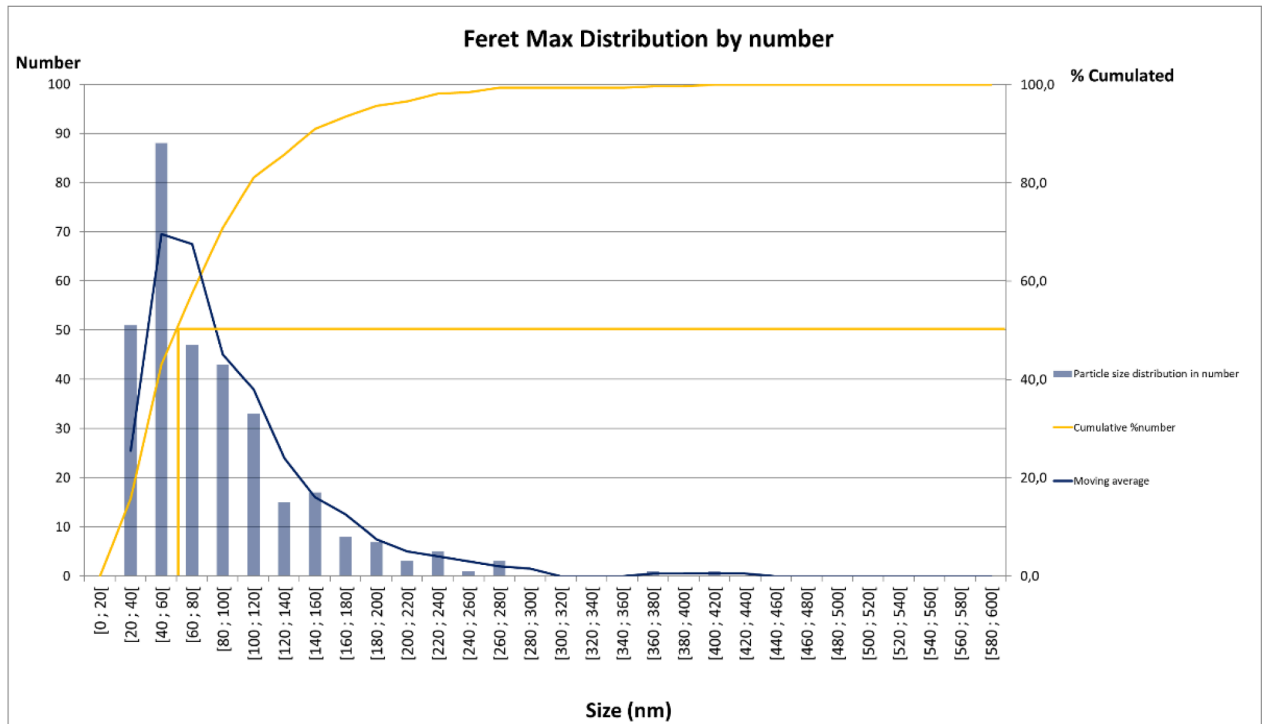

Figure 3. SEM particle size distribution of the sample **ID 18956 - 1** by maximum Feret Diameter .
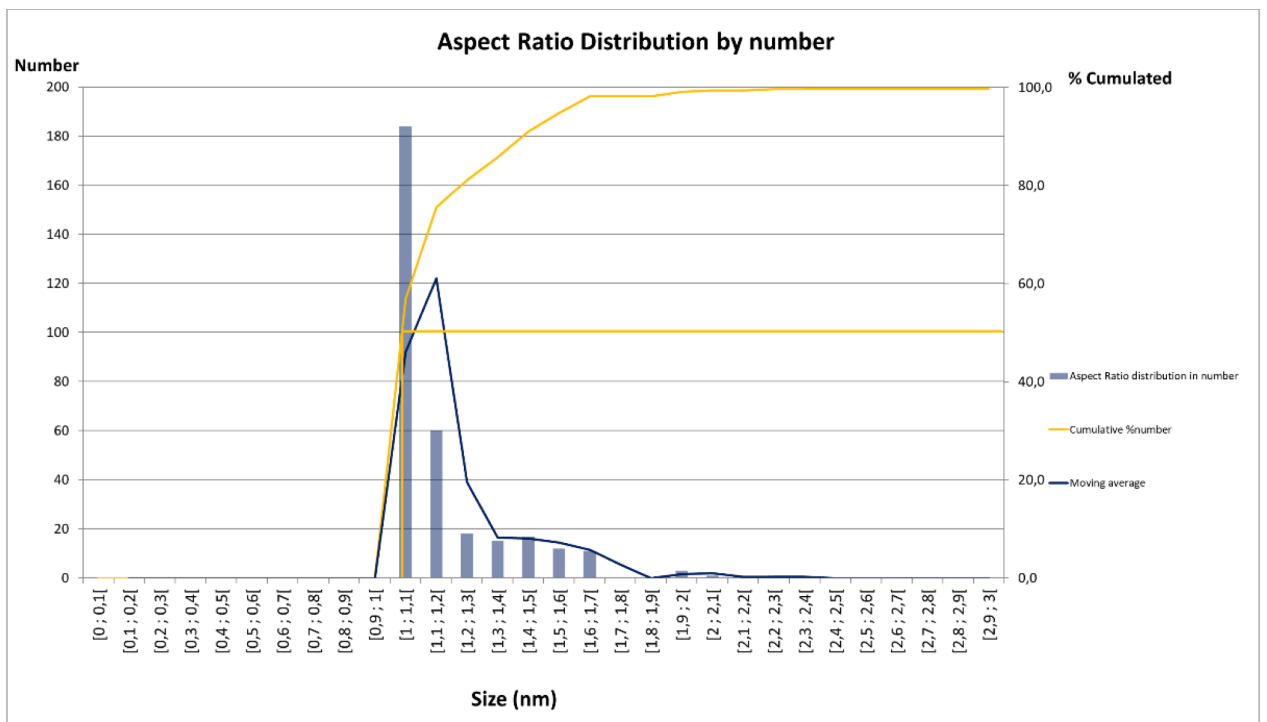
The Aspect Ratio:

Figure 4. SEM particle size distribution of the sample **ID 18956 - 1** by aspect ratio.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 7(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128
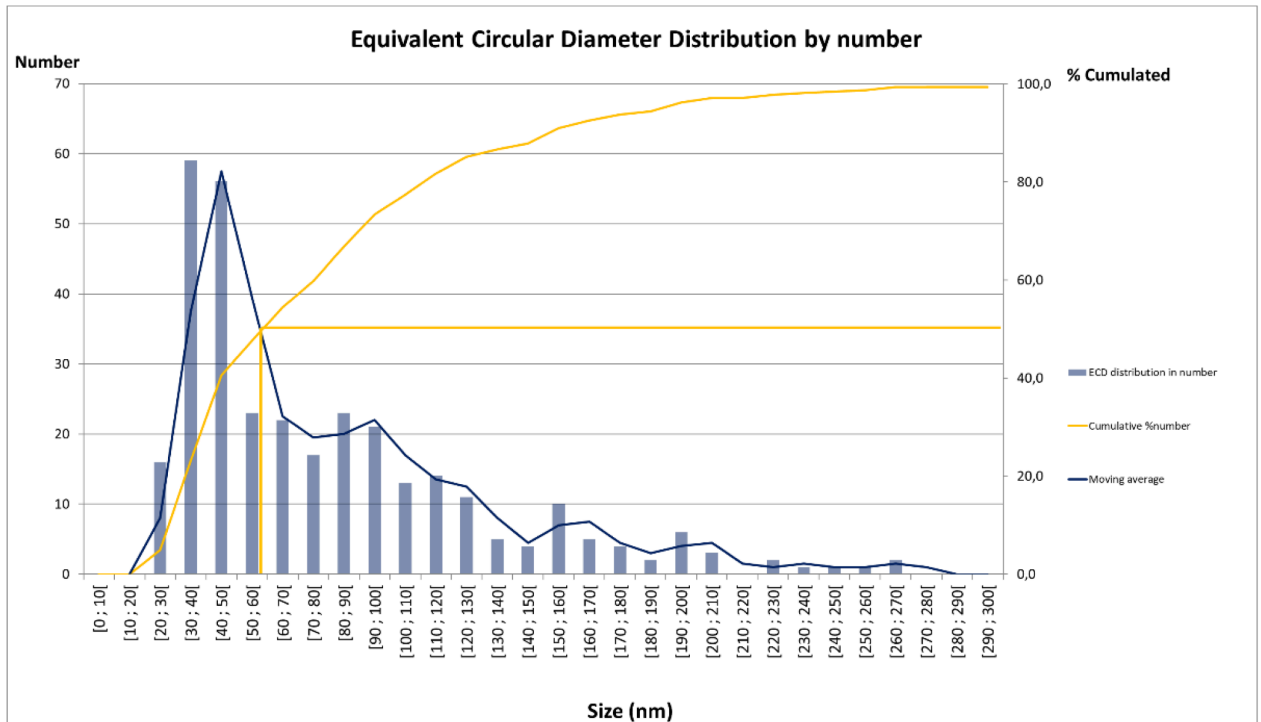
The Equivalent Circular Diameter (ECD):

Figure 5. SEM particle size distribution of the sample **ID 18956 - 1** by equivalent circular diameter.

Table 1. Summarized results from particle size distribution from sample **ID 18956 - 1**.

Minimal Feret diameter (nm)									
D10	D25	**D50**	D75	D90	**Mean**	Confidence Interval 95 %	**Fraction < 100 nm (%)**	Min	Max
29	37	**60**	102	155	**77**	5,73	**74**	17	287
Maximal Feret diameter (nm)									
D10	D25	D50	D75	D90	Mean	Confidence Interval 95 %	Min	Max	
37	46	66	107	159	86	6,04	27	401	
Aspect Ratio									
D10	D25	D50	D75	D90	Mean	Confidence Interval 95 %	Fraction AR < 3 (%)	Min	Max
1,01	1,02	1,07	1,20	1,49	1,17	0,03	99,69	1,00	3,12
Equivalent Circular Diameter (nm)									
D10	D25	D50	D75	D90	Mean	Confidence Interval 95 %	Min	Max	
34	41	63	105	157	81	5,87	24	333	

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland
Page 8(25)
https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

The minimal Feret diameter is the parameter used to determine if a particle is a nanoparticle or not. As such, this is the parameter that is discussed in the following point.

The particle size distribution of the sample is monomodal with a peak centred at about 40 nm, as well as a large shoulder at about 90 nm and a trail from 150 nm. The particle size ranges from 17 nm to 287 nm.

Conclusions of SEM-EDX PSD

The results of the particle size distribution (cumulative %) show that **more than 50% of the particles in the numerical size distribution are smaller than 100 nm**.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 9(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Test results - X-ray diffraction

Methods   Rigaku MINIFLEX II diffractometer equipped with a copper source (wavelength Kα 1.54 Å) is used for the analysis.

The measurements were performed by an ISO IEC 17025 accredited external service provider.

Appendices   **Appendix F** provides technical description of X-ray diffraction.

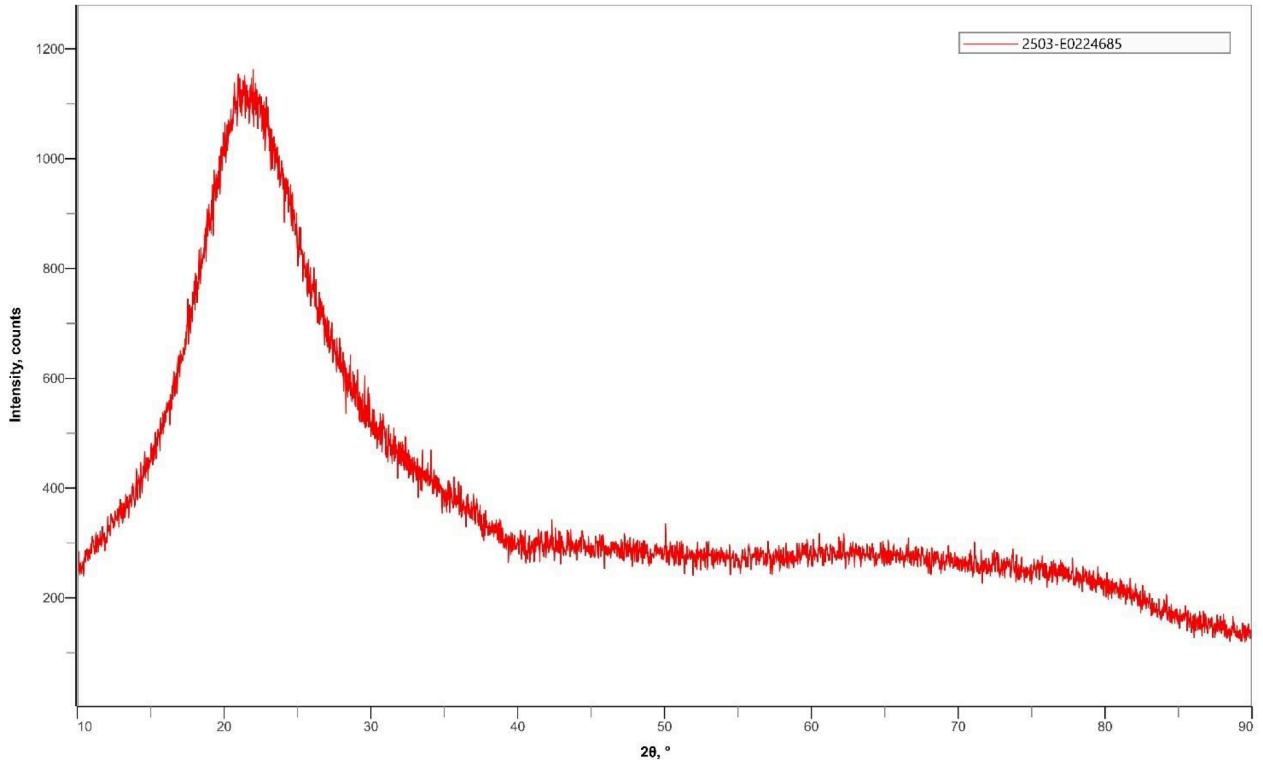**Results**   X-ray diffraction results from the sample are given in **Figure 1**.

Figure 1. Diffractogram obtained from the sample **ID 18956 - 1**

The diffractogram shows that the sample is composed of an amorphous phase, no crystalline phases are identified by our database for this sample.

Conclusions of XRD   The sample is completely amorphous.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 10(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Test results - ICP-AES analysis

Methods
A mass m = 0.1 g of sample is mineralized in a mixture of V = 10 mL of hydrochloric acid, V = 5 mL of nitric acid and V = 2 mL of hydrofluoric acid. After mineralization for 2 hours at 100 °C and cooling, the total volume is adjusted to 25 mL by ultrapure water.

Two independent sample preparations are performed. The solutions obtained in this way are analysed by ICP-AES according to the analytical conditions described below. It must be noted that mineralisation is total.

Details of the instrument and measurement settings are given below.

Instrument	Type	ICAP PRO
	Supplier	THERMO
Gaz	Nature	Argon
	Auxiliary gas flow	0.5 L/min
	Nebulising gas flow rate	0.5 L/min
	Plasma gas flow rate	12 L/min
Chamber	Type	Cyclonic Teflon
Measure		Axial and radial
Power	Radiofrequency power	1150 W

The measurements were performed by an ISO IEC 17025 accredited external service provider.

Appendices
Appendix G provides a technical description of ICP-AES.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland
Page 11(25)
https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Measurlabs

Results

Table 1. Results obtained for the samples, expressed in %w/w.

Element	Results in %w/w
Ag	<0.002
Al	<0.002
As	<0.002
B	<0.002
Ba	<0.002
Be	<0.002
Bi	<0.002
Ca	<0.002
Cd	<0.002
Co	<0.002
Cr	<0.002
Cu	<0.002
Fe	<0.002
K	<0.002
Li	<0.002
Mg	<0.002
Mn	<0.002
Mo	<0.002
Na	<0.002
Ni	<0.002
P	<0.002
Pb	<0.002
S	<0.002
Sb	<0.002
Se	<0.002
Si	>99.94
Sn	<0.002
Sr	<0.002
Ti	<0.002
Tl	<0.002
V	<0.002
Zn	<0.002

Conclusions   The ICP-AES analysis shows only the presence of Silicon (Si).
The purity of the sample is > 99.94%w/w.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 12(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Test results - CHNS by thermal/infrared conductibility

Methods   The sample is weighed with great precision into a tin capsule, which is then introduced into an oxidation/reduction reactor. A controlled supply of oxygen is performed and organic and inorganic substances are converted into gases (CO_2, N_2, H_2O, SO_2,...) and finally detected by detectors (thermal and infrared conductibility). The sample is analysed in its raw state, no preparation step is needed. Instrument details are given below. Details of the instrument and measurement settings are given below.

Equipment	ELEMENTAR UNICUBE	
Temperature	Combustion tube (right furnace)	1150°C
	Reduction tube (left furnace)	850°C
	Standby column absorption	40 °C
	Temp. Cooled col. Absorp.	90 °C

Results   **Table 1.** The results obtained for the samples, expressed in %w/w, are shown in the table.

Element	Results in %w/w
C	< 0.10
H	< 0.40
N	< 0.10
S	< 0.10

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland
Page 13(25)
https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Conclusion

The analyses implemented in this study has led to the following information:

1. **VSSA**

    The measured specific volume (VSSA) is estimated at 53.6 m²/cm3 and is therefore greater than 6 m²/cm3.

2. **SEM-EDX**

    The particle size distribution carried out by SEM on a sample of particles shows that more than 50% of the particles are smaller than 100 nm. In this study, this is the smallest size that was measured, as mentioned in the definition.

    Therefore, based on the EC recommendation of 10 June 2022 on the nanomaterials definition (2022/C 229/01), the sample ID 18956-1(Nan-O-Sil ASD™ F040, Lot #1658) seems to fulfil the criteria for the definition of Nanomaterials (item 1).

Sample Reference	Mean value	Value at 10% cumulative	Median value (50% cumulative, according to 2022/C 229/01)	% of nanoparticles in the sample of particles analysed
ID 18956 – 1	77 nm ± 6 nm	29 nm	60 nm **< 100 nm**	74

According to the EC recommendation of 2022 and the sampling used, this material is therefore to be considered as a "nanomaterial".

3. **XRD**

    The sample is completely amorphous.

4. **ICP-AES and CHNS**

    - The ICP-AES analysis shows only the presence of Silicon (Si).
    - The purity of the sample is > 99.94%w/w.
    - Infrared/Thermal conductibility analyses do not show the presence of C,H,N,S in the sample.

End of the test report

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 14(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Appendix A – Regulatory reminder on nanomaterials

- Definition of a nanomaterial according to EC recommendation of 06/10/2022– (CE) N°2022/C 229/01

  « 1. 'Nanomaterial' means a natural, incidental or manufactured material consisting of solid particles that are present, either on their own or as identifiable constituent particles in aggregates or agglomerates, and where 50 % or more of these particles in the number-based size distribution fulfill at least one of the following conditions:
  (a) one or more external dimensions of the particle are in the size range 1 nm to 100 nm;
  (b) the particle has an elongated shape, such as a rod, fibre or tube, where two external dimensions are smaller than 1 nm and the other dimension is larger than 100 nm;
  (c) the particle has a plate-like shape, where one external dimension is smaller than 1 nm and the other dimensions are larger than 100 nm.
  In the determination of the particle number-based size distribution, particles with at least two orthogonal external dimensions larger than 100 µm need not be considered. However, a material with a specific surface area by volume of < 6 m²/cm^3 shall not be considered a nanomaterial. »

- Important characteristic of a Nanomaterial according to EC recommendation of 06/10/2022– (CE) No 2022/C 229/01

  […]

  (7) The definition should be based on the relative fraction of particles in a defined range within the particle number-based distribution of the external dimension of the constituent particles of a material, irrespective to its potential inherent hazardous properties or risks to human health and the environment.

  […]

  (9) The term nanomaterial should address materials consisting of particles in solid state, present on their own or bound as constituent parts of aggregates or agglomerates. The term consist of' rather than 'contain' should be used to acknowledge that the particles are the principal component of the material. Other non-particulate components potentially present (e.g. additives necessary to preserve its stability or solvents that may be separated without affecting the particle size distribution) are part of the (nano-) material but should not be taken into account when assessing whether a material is a nanomaterial.

  (10) The definition should exclude non-solid (i.e. liquid and gaseous) particles. This should ensure that the highly dynamic nature of the external dimensions of non-solid particles, such as micelles for nanoscale droplets in emulsions or sprays, does not prevent the use of the external dimension as the defining qualifier in the definition.

  (11) The definition should not cover large solid products or components, even when they have an internal structure or a surface structure at the nanoscale(1), such as coatings, certain ceramic materials and complex nanocomponents, including nanoporous and nanocomposite materials.Some of these products or components may have been manufactured by using nanomaterials and may even still contain them.

  […]

  (14) The definition should cover both particles on their own and identifiable constituent particles(2) in agglomerates or aggregates. The review of the definition highlighted that the identification and measurement of constituent particles in aggregates can be very challenging. Thus,the qualifier 'identifiable' is bound by practical considerations pertaining to their identification.These considerations should be further elaborated in guidance.

  […]

  (18) Due to their much smaller number in all reasonably foreseeable and relevant situations,particles with at least two orthogonal external dimensions larger than 100 µm do not significantly influence the relative contribution of 1 nm to 100 nm particles in the total number of particles, and therefore do not affect to a significant extent the classification of materials. The definition should allow to restrict the determination of the particle number-based size distribution only to constituent particles with at least two orthogonal external dimensions below 100 µm, provided that the choice is documented by appropriate measurement results. Practical application of this option should be presented in guidance.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 15(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

(19) Experience has shown that use of specific surface area as a proxy indicator when identifying a nanomaterial can lead to interpretation and technical difficulties, for example, as a high specific surface area may be due to an internal nanostructure rather than indicate the presence of a large number of small constituent particles. Therefore, the review of the definition identified that the related option provided in point 5 of Recommendation 2011/696/EU was not appropriate and should be removed from being a qualifier in the definition of a nanomaterial.

(20) The NanoDefine project demonstrated, based on a large set of different industrial materials, that there were no inconsistencies in classification of non-nanomaterials, based on the median value determined from the particle number-based size distributions and on the volume specific surface area being less than 6 m² /cm3 (even if particle shape is unknown), respectively. Therefore, a material with a volume specific surface area less than 6 m² /cm3 should not be considered a nanomaterial. […]

- Definition of aggregates, agglomerates, and non-identifiable terms according to the "Guidance on the implementation of the Commission Recommendation 2022/C 229/01 on the definition of nanomaterial"

   - The term aggregate means a particle comprising of strongly bound or fused particles.
   - The term agglomerate refers to a collection of weakly bound particles or aggregates where the resulting external surface area is similar to the sum of the surface areas of the individual components.
   - When the constituent particles of aggregates are so strongly bound or fused together that their boundaries, or interfaces, cannot be discerned anymore, they are considered non identifiable. In this case, the relevant external dimensions are those of the aggregate.

The diagram below presents the different situations and how to delimit in these situations:
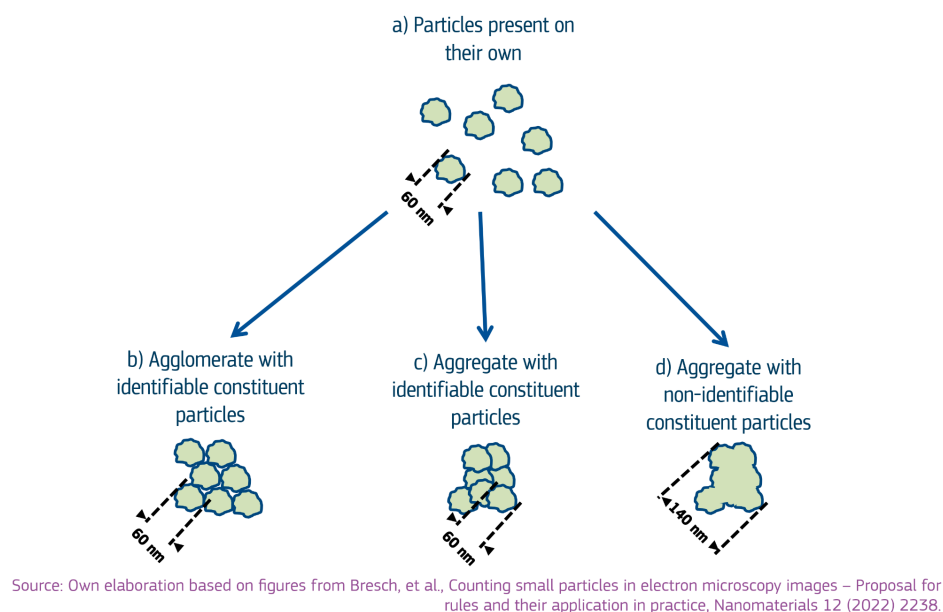

Source: Own elaboration based on figures from Bresch, et al., Counting small particles in electron microscopy images – Proposal for rules and their application in practice, Nanomaterials 12 (2022) 2238.

Figure A1. Presentation of the different ways to delimit particles when they are a) free, b) agglomerated and identifiable, c) aggregated and identifiable and d) aggregated and non-identifiable according to the Guidance on the implementation of the Commission Recommendation 2022/C 229/01 on the definition of nanomaterial (Figure 4 p.16)
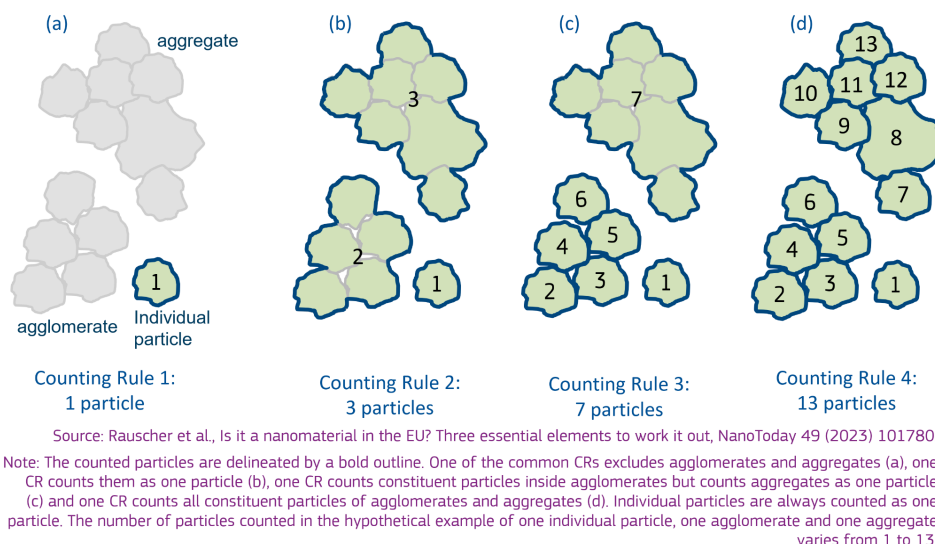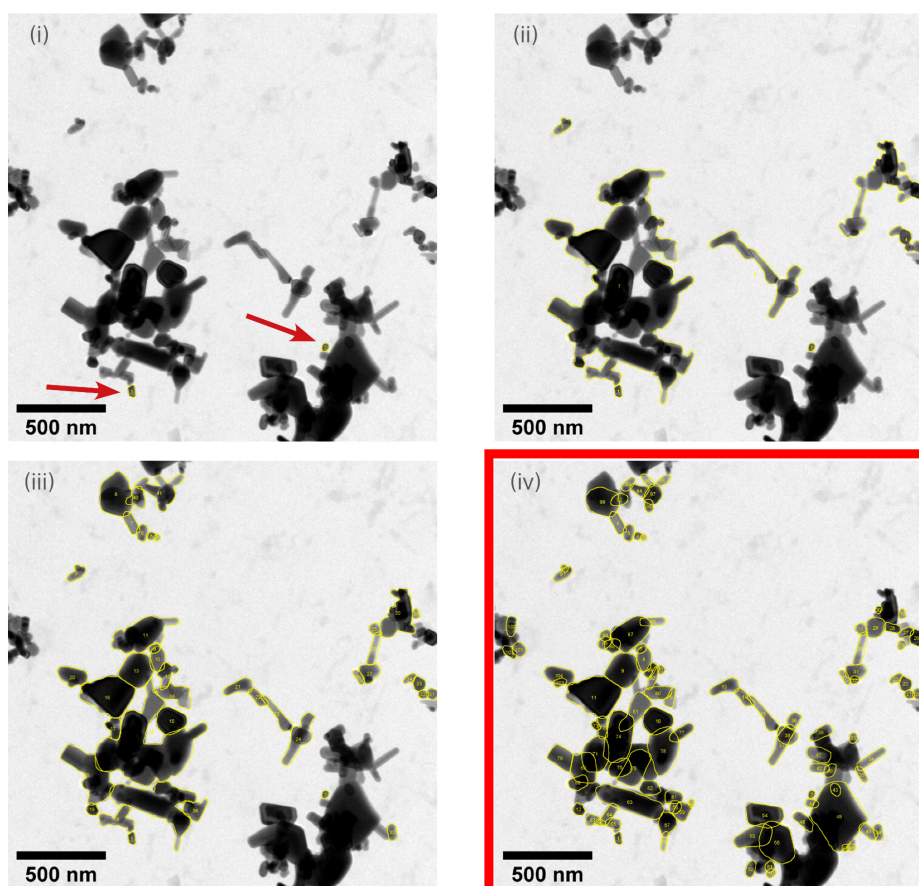
Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland
Page 16(25)
https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Figure A2. Presentation of the different counting rules according to the Guidance on the implementation of the Commission Recommendation 2022/C 229/01 on the definition of nanomaterial (Figure 5 p.16).

The figure above illustrates the four commonly encountered counting rules for particles. Counting rule 4 is the one applied in recommendation 2022/C 229/01

Figure A3. Illustration on zinc oxide particles of the different counting rules according to the Guidance on the implementation of the Commission Recommendation 2022/C 229/01 on the definition of nanomaterial (Figure 6 p.17).

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 17(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

1. Interpretation of point (11) of the EC recommendation of 06/10/2022− (CE) No 2022/C 229/01

Point (11) refers to components with a surface nanostructure and nanoporous materials. This point defines such materials as non-nanometric. Nanoparticle-coated nacres or nanostructured silica beads are examples of such materials. Examples of these materials are presented below.
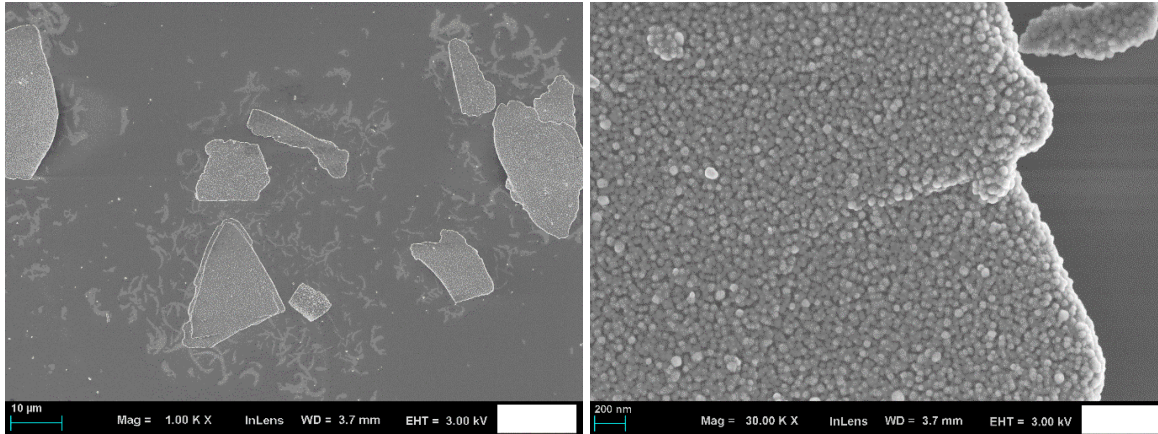

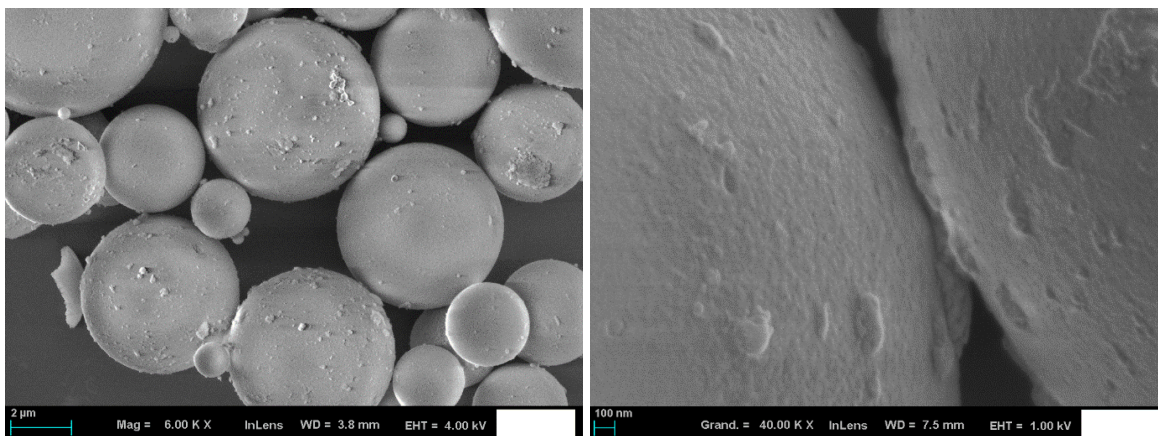**Figure A4**. Examples of nacres morphologies covered with Nanoparticles

Figure A5. Examples of morphologies of nanostructured beads.

2. Interpretation of point (14) of the EC recommendation of 06/10/2022− (CE) No 2022/C 229/01

In point (14) of the EC recommendation of 2022, the term "identifiable constituent particles" is mentioned. At the moment, the notion "identifiable" is submitted to interpretation, and is not defined by any precise criteria.

The pictures below show examples of particles morphology that would be considered to be identifiable by the lab, as well as examples of particles morphology that are not:
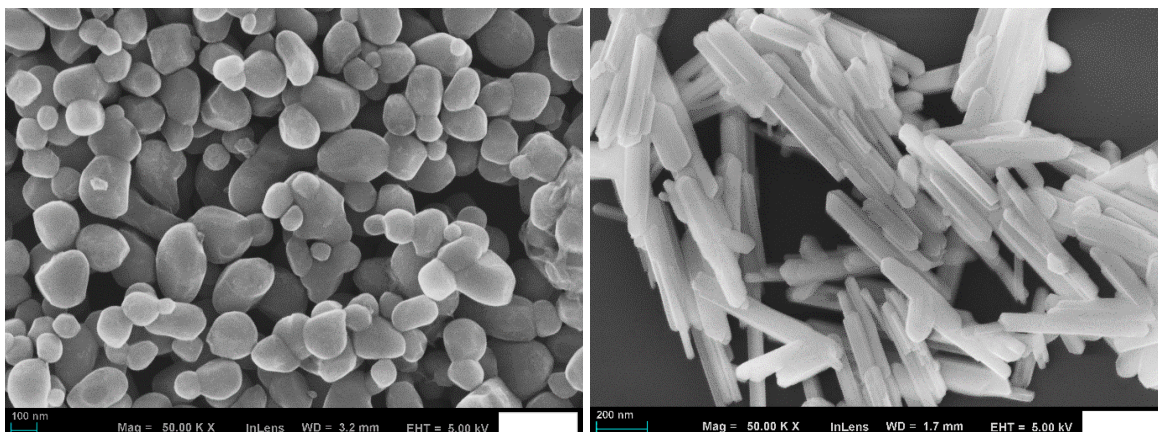

Figure A6. Examples of particle morphology that would be considered __identifiable__ by the lab.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 18(25)

https://measurlabs.com/
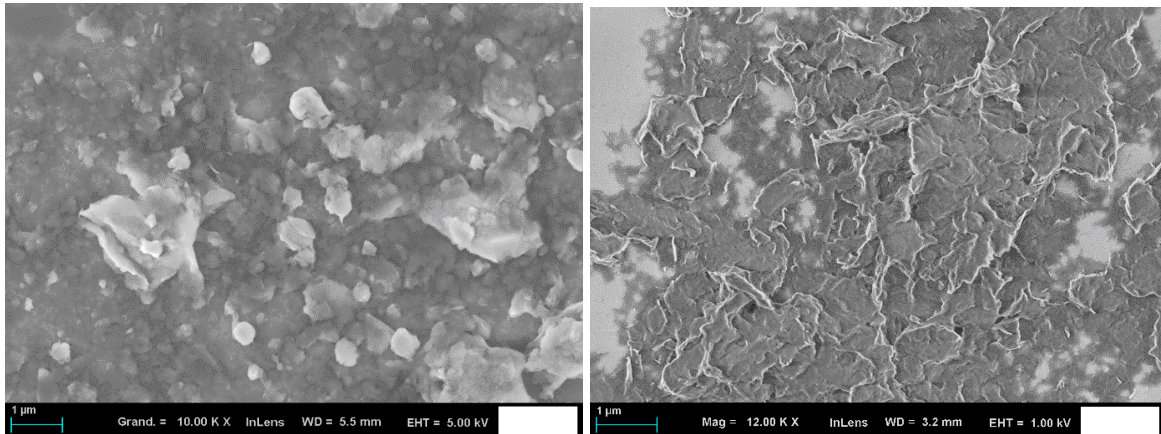info@measurlabs.com
+358 50 336 6128

Figure A7. Examples of particles morphology that would be considered **not-identifiable** by the lab

It should be noted that other morphologies exist and that the above examples do not constitute an exhaustive list of what is considered identifiable by the lab and what is not.

As stated in the Guidance, for some samples (such as industrial fine powder) "even the application of high-energy sonication will not be sufficient to achieve a complete disintegration of the secondary structures and the only solution to reliably assess such materials is by measuring the constituent particles within the agglomerates/aggregates using high-resolution imaging methods". This means that even if the particles show some certain degree of agglomeration, the result of the particle size distribution should be considered reliable.

Moreover, it is clearly stated that the "Counting rule 4 is the one applied in Recommendation 2022/C 229/01". As such, any particles observed by SEM in this study that is sufficiently visible must be considered and counted, just like it's shown in the zinc oxide example presented above.

Appendix B – VSSA technical description

Nitrogen adsorption sorptometry

Principle: The measurement of the specific surface area (most commonly given in m²/g) is carried out via the adsorption isotherm of the sample to be analyzed. The isotherm is determined by the physical adsorption of nitrogen molecules on the surface of the sample at a temperature of 77 Kelvin (liquid nitrogen temperature). To determine the specific surface area, the BET method (model established by Brunauer, Emmett and Teller) is applied to the adsorption isotherm in the range of law validity, i.e. between 0.05 and 0.30 P/P0. The BET transform is thus plotted and the specific surface area is deduced.

Hypothesis of the BET method: The main assumptions of the BET method are:

- The molecules in the last adsorbed layer are in equilibrium with the surrounding vapour,
- No lateral interaction between the adsorbed molecules,
- Only one type of adsorption site, energetically homogeneous surface,
- Probability of adsorption of the molecule is the same for all layers.

Acceptance parameter of the BET method: The two criteria considered are:

- Positive BET constant
- Correlation coefficient > 0.995.

Measurements of the specific surface area using the BET method are carried out in accordance with ISO 9277:2010.

Density by Helium pycnometry

Principle: Helium Gas Pycnometry is a technique based on the Perfect Gas relationship (see below). This relation enables to measure the exact volume of a sample, excluding inter and intra granular porosity. With the knowledge of the mass of the sample, the effective density (g/cm3) can be calculated.

Pycnometry measurements are based on the ISO 12154 :2014 standard.

Perfect Gas relationship:

$$P_1(V_{CELL} - V_{SAMP}) = n_c RT_a$$

Where ,
T_a = Ambient Temperature ,
n_c = number of moles of gas in V_{CELL},
R = Perfect Gas Constant (8,3144621 J.mol^{-1}.K^{-1})

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland
Page 20(25)
https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Appendix C – Isotherm plots from VSSA analysis
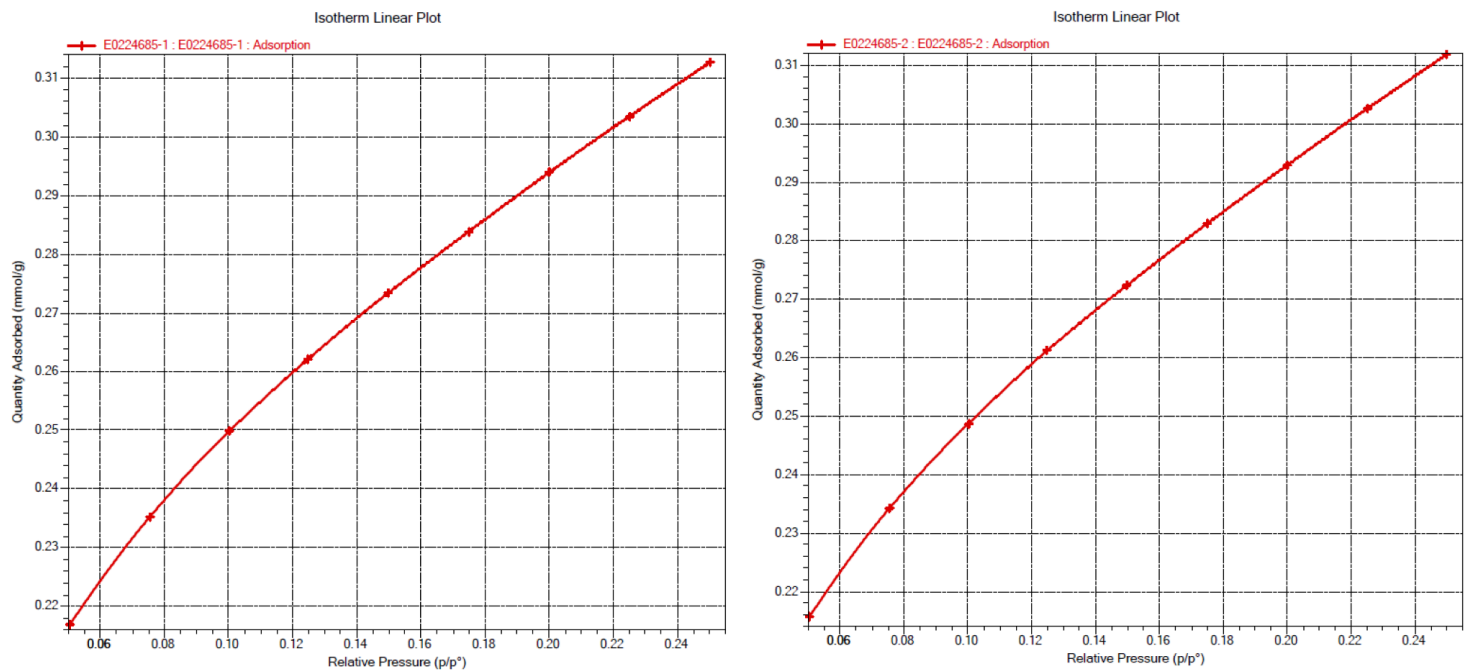
Figure C1. Isotherm plot acquired by Test #1 (left), and Test #2 (right) from sample **ID 18956 - 1**

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 21(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Appendix D - SEM EDX technical description & methodology

Principle: Scanning electron microscopy involves the emission of an electron beam onto the sample surface and then the analysis of the interactions between electrons and the material. The electrons are generated by a field effect gun. Compared to a beam emitted via a filament, the latter is thinner and its brightness is increased. The emission can thus be more targeted on the surface and the information obtained of better quality. Three types of "material/electron" interactions are exploited by this technique.

1. Secondary electrons (SE): Incident electrons collide and eject the outlying electrons of the atoms located on the surface to be analysed. The ejected electron is then called secondary. A specific detector allows to visualize the morphology of the sample. The images obtained via secondary electrons (SE mode) give access to the topographic contrast of the sample.

2. Backscattered electrons (BSD): They result from the interaction between incident electrons and those present in the nucleus of the atoms constituting the sample. Backscattered electrons are re-emitted at a different velocity from their initial one, depending on the size of the atom with which they interact. The image obtained (BSD mode) is called chemical composition contrast image.

3. X-RAYS (EDX mode): The bombardment of the material surface by the incident beam electrons also leads to the emission of X-rays, whose wavelength is specific to each atom. The analysis of these rays gives access to the chemical nature of the atoms constituting the sample, via an EDX probe. Spectra are thus obtained that indicate the elemental chemical compositions from a semi-quantitative point of view.

Methodology of the 2022 EC Recommendation on the definition of nanomaterials (2022/C 229/01)

Morphological observation by SEM

Observation of particle morphology is necessary in order to determine the organization of the material on a very small scale (composed of identifiable or unidentifiable particles). In the case of identifiable primary particles whose two orthogonal dimensions are not greater than 100 µm, then a particle size distribution by SEM is required to determine whether the material is classified as nanoscale or not.
If the particles are considered to be large, and have an internal or surface structure on the nanoscale, then the material is classified as non-nanoscale.

Particle size distribution by number on raw material by SEM

"Nanomaterial" means a natural, incidentally formed or manufactured material containing free particles, in aggregate or agglomerate form, of which at least 50% of the particles, in numerical size distribution, have one or more external dimensions between 1 nm and 100 nm.

- Dispersion of the sample on a slide with a spin coating step: detection of nanoparticles + primary particle size + determination of the D50 + establishment of a number distribution.

- SEM study then counting by image analyser.

If D50 < 100 nm: Raw material classified as nano. End of the study.
If D50 > 100 nm: Raw material classified as non-nano. End of the study.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 22(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Appendix E – SEM EDX additional results

SEM-EDX

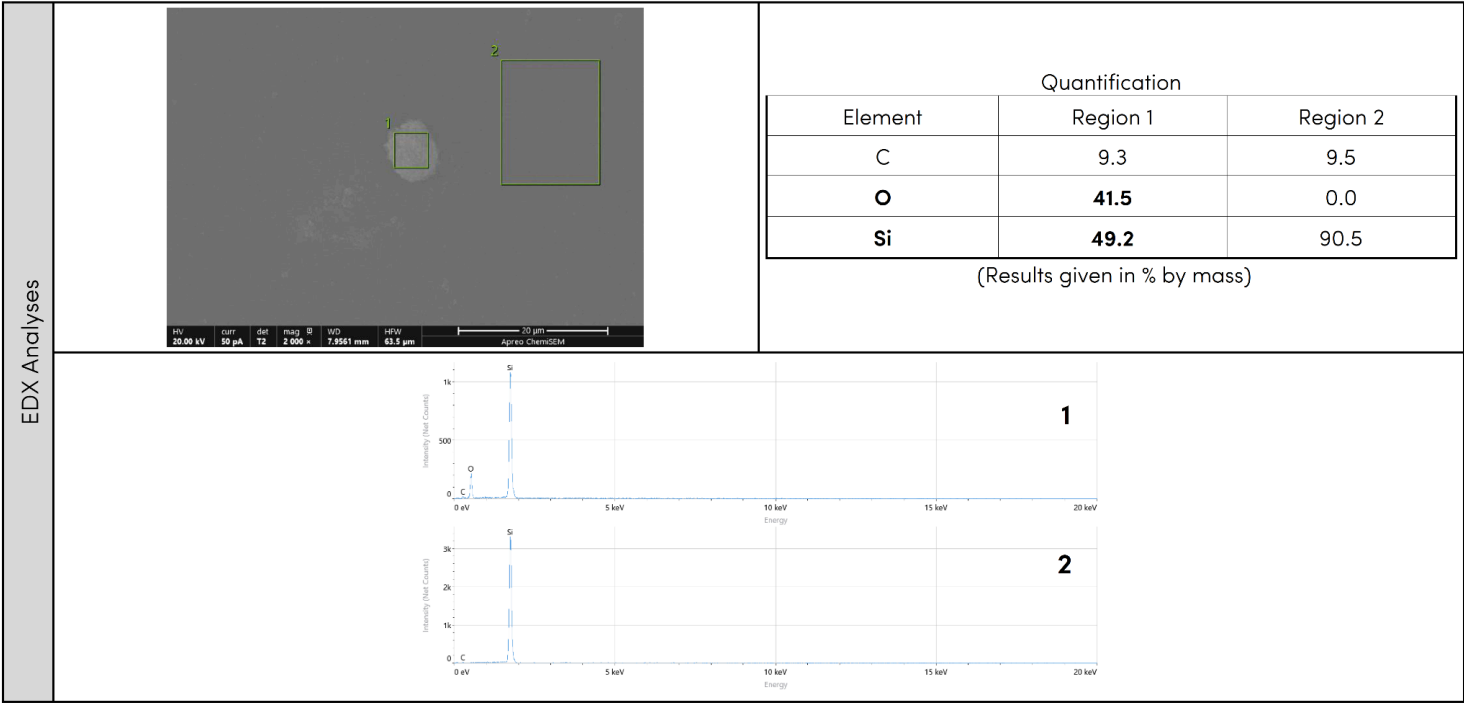| EDX Analyses		Quantification		
		Element	Region 1	Region 2
		C	9.3	9.5
		O	**41.5**	0.0
		Si	**49.2**	90.5

(Results given in % by mass)

Figure E1. SEM image (top left), EDX spectrum from sample (bottom left), from background (bottom right), and elemental composition (top right) from sample ID 18956 - 1
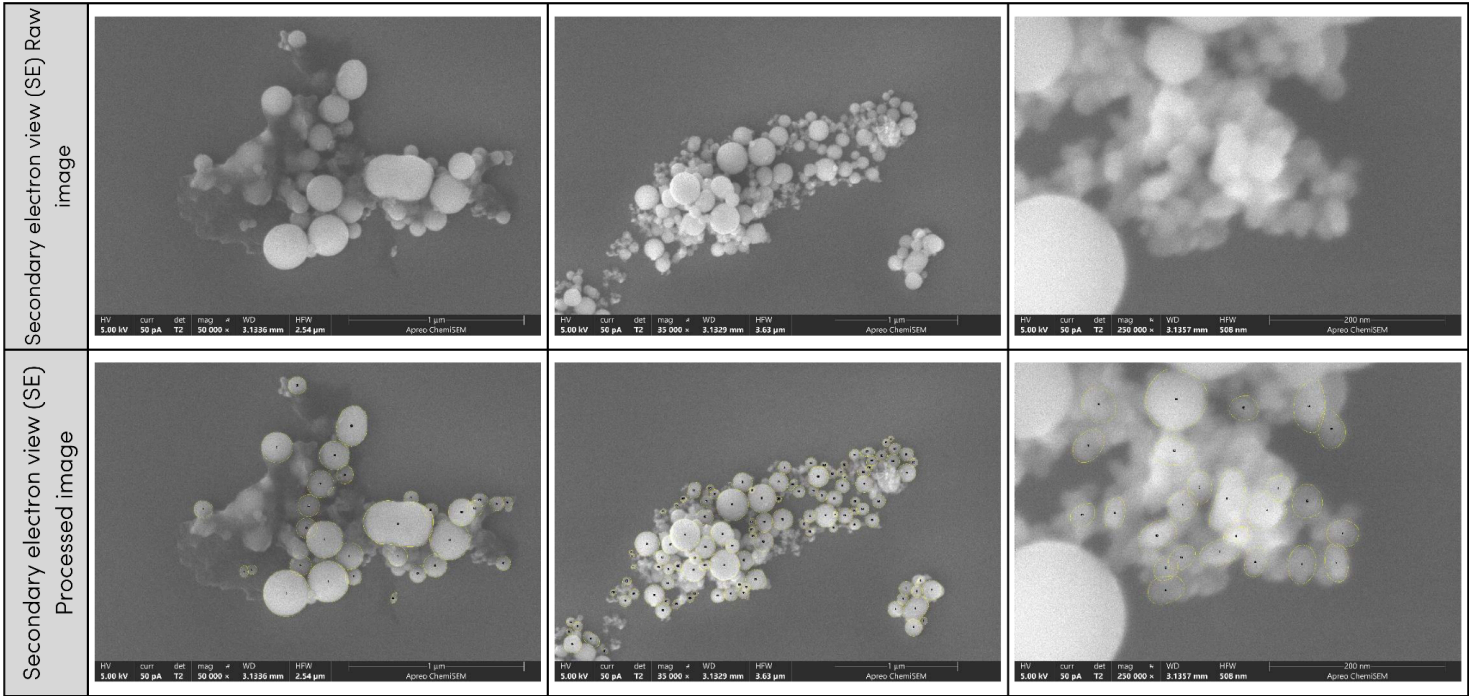
SEM-EDX particle counting

Figure E2. SEM images before and after image treatment showing boundaries of counted particles from sample ID 18956 - 1

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 23(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Appendix F – XRD method description

Principle: X-ray diffraction (XRD) is an analytical method that provides information on the arrangement of the constituent elements of the material. The wavelength of X-rays is similar in order of magnitude to the size of an atom, namely Angstrom (10^{-10} m). If the material is a crystal, X-rays are reflected in specific directions, determined both by the initial characteristics of the radiation and by the parameters of the crystal lattice itself. These returned rays are called diffracted, their analysis makes it possible to accurately identify the crystalline phase with which they interfered.

The crystallographic phases are identified both via the angle of the returned rays, which constitutes the diffraction peaks, and their intensities, which is a function of symmetries, absorption capacity and the position of the atoms that constitute the material.

Crystalline structure: A crystal lattice is the repetition in space of a basic volume called an elementary lattice. They are an assembly of atoms of different types. Rich in planes of symmetry, angles and vectors, these are all parameters that influence the response of X-rays when they diffract by scanning the material.

Diffractogram: It represents the intensity of the diffracted beam as a function of its deflection angle. It is the signature of the crystalline phase.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland
Page 24(25)
https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128

Appendix G – ICP–AES technical presentation

ICP-AES for 'Inductively Coupled Plasma', is an inductively coupled plasma analytical technique used to measure the content of an inorganic element in a sample. This technique is applicable to all types of elemental chemical elements.

ICP sample analysis consists of several steps. Firstly, the solid sample must be dissolved using a strong acid, a mixture of strong acids or microwaves. This is known as mineralisation. The preparation is then vaporized in argon plasma and placed at very high temperatures. These thermal excitations lead to ionization and separation of the elements, making it possible to characterize and detect each element, depending on the analysers used.

Each of the solutions prepared is introduced directly by a nebuliser into a very high temperature plasma (8000 K), which ionizes the atoms in the form of monovalent cations. The ions thus obtained are focused and analyzed by a detector that quantifies the intensity of the signal for each wavelength of the element according to a synthetic calibration range (this range is based on standards certified for the element in question and prepared under the same conditions as the samples).

A calibration is carried out at the start of each sequence, using an analytical standard.

Measurlabs
Teollisuuskatu 33
00510 Helsinki, Finland

Page 25(25)

https://measurlabs.com/
info@measurlabs.com
+358 50 336 6128