Ministry of Environment and Food of Denmark
Environmental Protection Agency

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XMAX

The overall objective

The report proposes

suggested

conclude

It is concluded that

It is found

The report

delivers

mobility

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Det konkluderes

□ □

Rapporten finder

Rapporten leverer

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1) *Nanomaterial' means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm-100 nm.*

Nanotechnologies - Guidance on physico-chemical characterization of engineered nanoscale materials for toxicologic assessment

Nanomaterials -- Quantification of nano-object release from powders by generation of aerosols

after

Guideline for the validation of physico-chemical analytical methods

Nanotechnologies - Methodology for the classification and categorization of nanomaterials

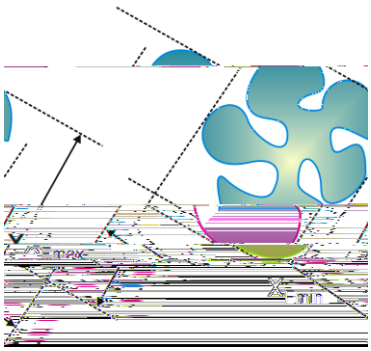
Comparative assessment of nanomaterial definitions and safety evaluation considerations

Commission Recommendation of 18 October 2011 on the definition of nanomaterial (2011/696/EU)

Requirements on measurements for the implementation of the European Commission definition of the term “nanomaterial”

Towards a review of the EC Recommendation for a definition of the term "nanomaterial"

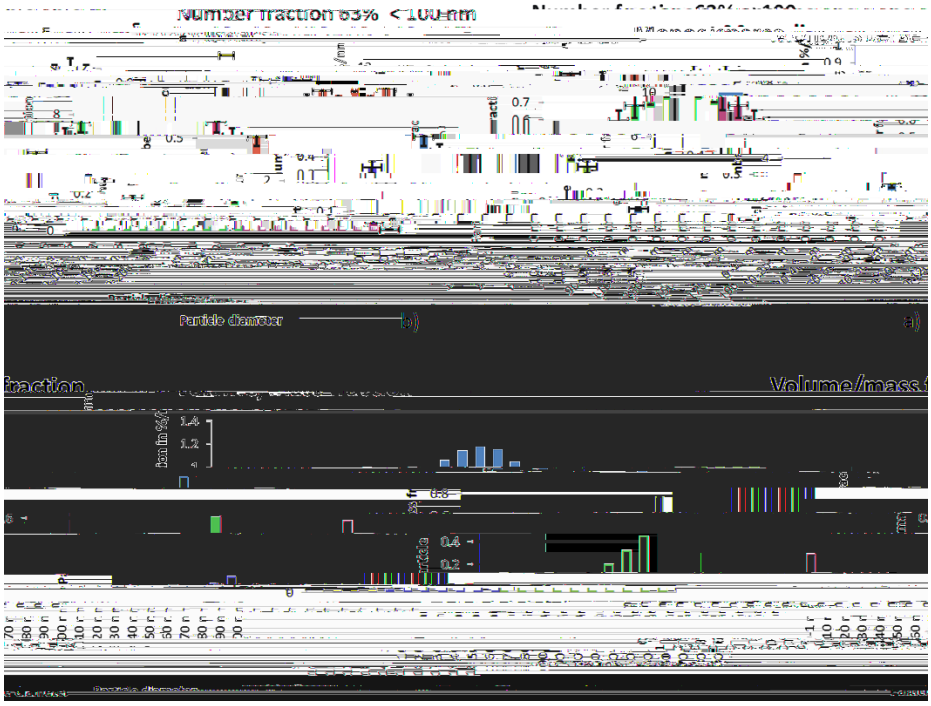
x_{Fmin}



Nanotechnologies - Vocabulary

Nanotechnologies – Terminology and definitions for nano-objects – Nanoparticles, nanofibre and nanoplates

Nanotechnologies -- Vocabulary -- Part 2: Nano-objects



counting methods

methods

q_k

k

q_k

q_k

concentrations

c *c* *c*

nanoparticle fractions f

Representation of results of particle size analysis — Part 1: Graphical representation

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f

It is found

The fitness for purpose of analytical methods – A laboratory guide to method validation and related topics

Harmonized guidelines for single laboratory validation of methods of analysis

Commission decision 2002/657/EC

“Validation is the conformation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.”

recorded

both

suggests

matrix components

different chemical species

Nanoparticles in Food:

Analytical methods for detection and characterisation

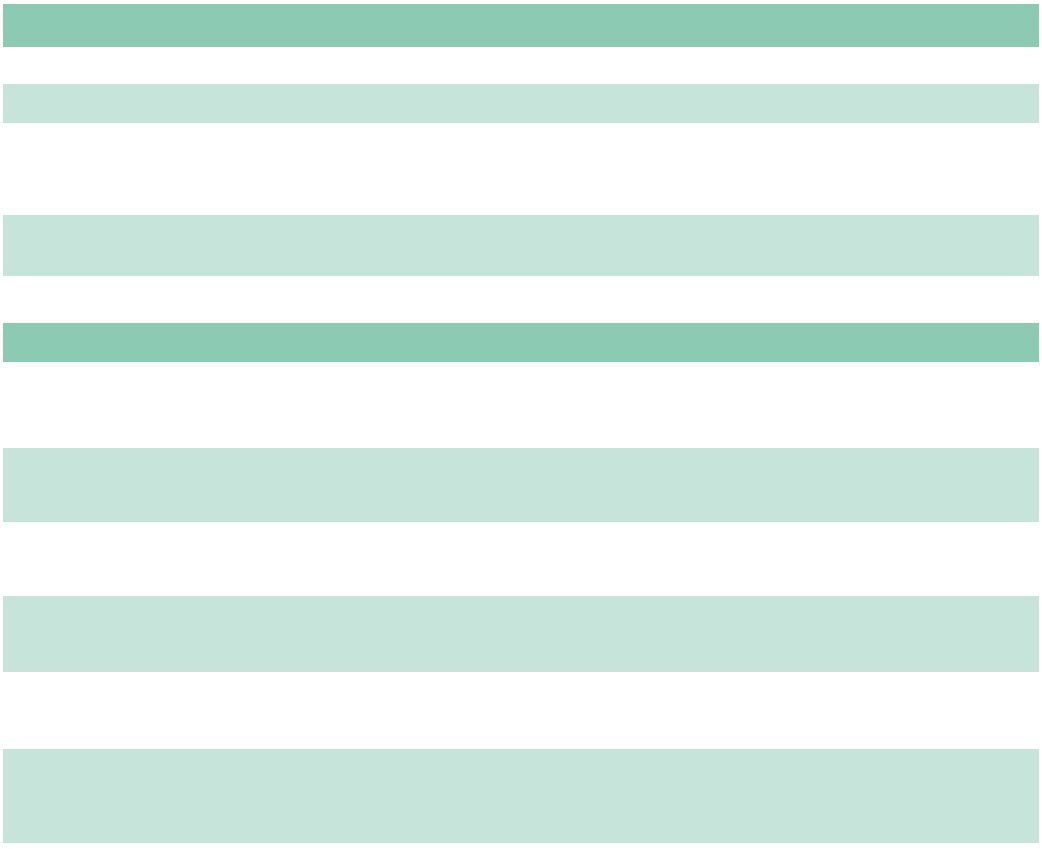
Validation of methods for the detection and quantification of engineered nanoparticles in food

IUPAC compendium of chemical terminology

International vocabulary of metrology --

Basic and general concepts and associated terms (VIM)

chemical equivalent particles



International vocabulary of metrology -- Basic and general concepts and associated terms (VIM)

particle size distribution

Q_k k k

k

International vocabulary of metrology -- Basic and general concepts and associated terms (VIM)

f

f ± U f

f x

X_{MAX} U f

X_{MAX} X_{MAX}

I_i k $b_{k-1} <$ $I_i \leq b_k$ counting efficiency $e(x)$

x $u(e(x))$

$e(x) < 1$ $u(e(x))$

$\boldsymbol{X_{MAX}}$

f x

X_{MAX}

X_{MAX}

x x x

x x x x

x

$\min u(x) \cong u(x_{min})$

$\max u(x) \cong u(x_{max})$

$\min u(x)$ $u(x), x_{min} < x < x_{max}$ $\max u(x)$

$u(x), x_{min} < x < x_{max}$ $u(x_{min})$ x_{min}

$u(x_{max})$ x_{max}

u x x x x

Nanomaterial number size distribution fraction, f

$u(f)$

$u(f)$

f

Diameter:

$u(x)$ x x x

$u(x)$

$u(x)$

Counting efficiency:

$e(x) < 1$ $u(e(x))$

Q_k

k

$u(Q_k)$

Q_k k

Q_k

$u(Q_k)/Q_k$

$u(Q_k)/Q_k$

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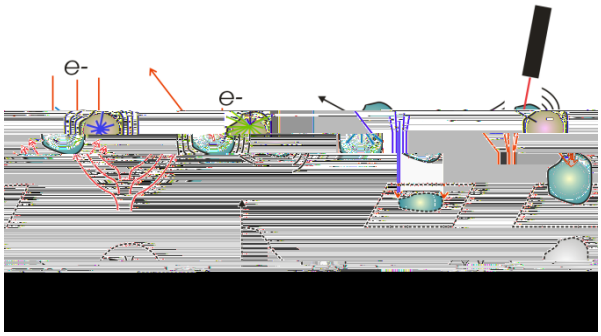
Chemical analysis, requirements to documentation of the method

Validation of methods for the detection and quantification of engineered nanoparticles in food



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Report of Investigation, Reference Material 8013, Gold Nanoparticles, Nominal 60 nm Diameter

Particle size analysis - Image analysis methods - Static image analysis methods

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Microbeam analysis -- Scanning electron microscopy -- Guidelines for calibrating image magnification

An electron microscopy based method for the detection and quantification of nanomaterial number concentration in environmentally relevant media

Microbeam analysis -- Analytical transmission electron microscopy -- Methods for calibrating image magnification by using reference materials having periodic structures

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"

Repeatability:

Zero measurement:

Background measurement:

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Calibration of equipment:

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Limitations:

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Additional discussion of DLS
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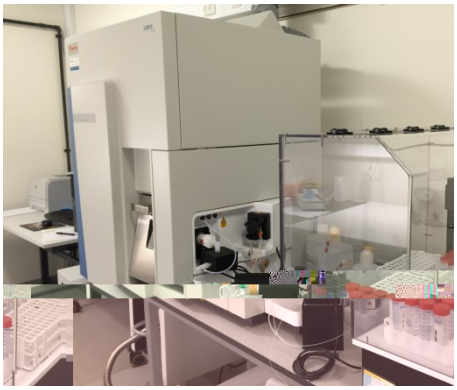
cannot

Photon correlation spectroscopy
Particle size analysis - Dynamic light scattering (DLS)

validate

Limitations:



Determination of trace elements in waters and wastes by inductively coupled plasma - mass spectrometry

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Zero measurement

Background measurement:

"ICP-MS: a promising tool in the detection and analysis of nanoparticles"

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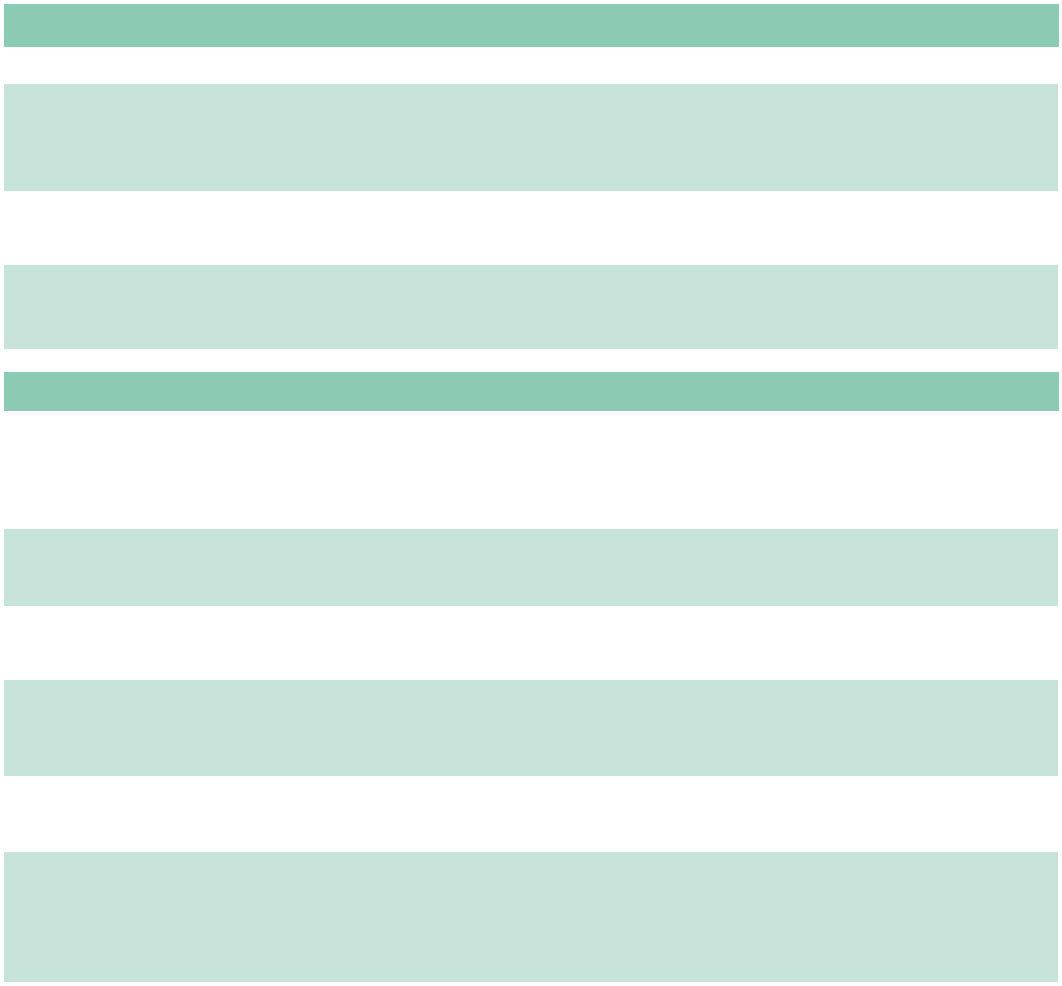
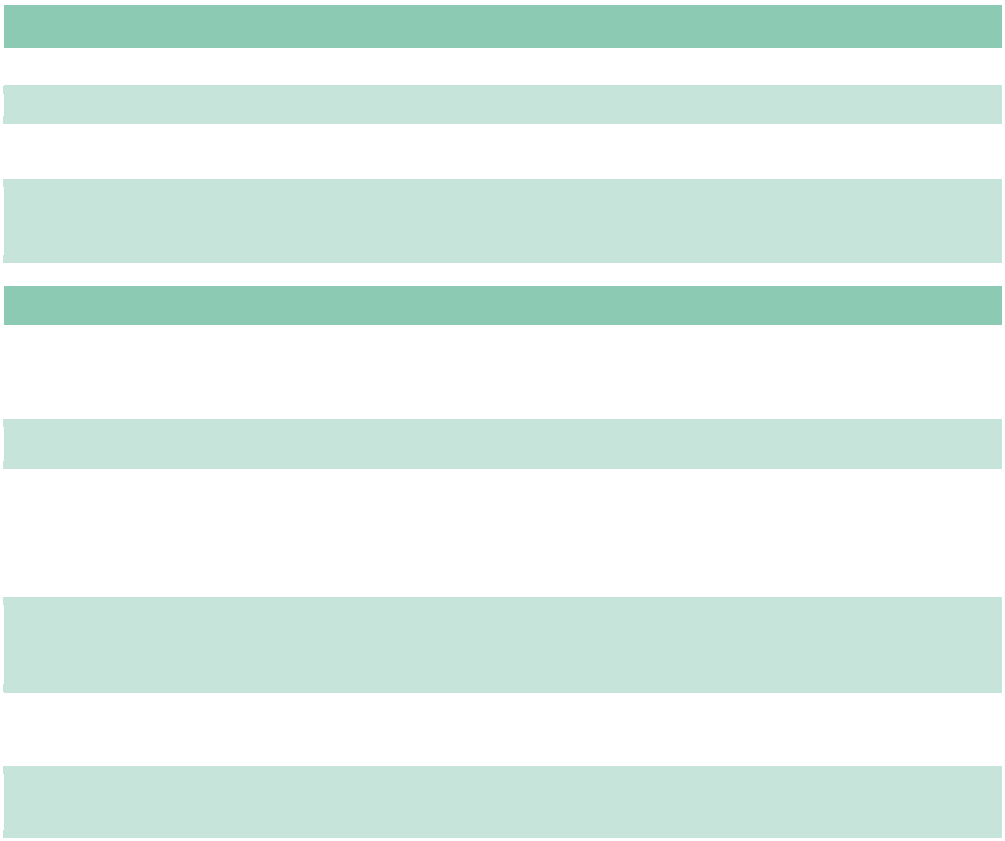
Zero measurement

Background measurement

Calibration of equipment:





Kortlægning af tekstiler med nanosølv på det danske marked
Anbefalinger om kvalitetskrav til identifikation og kvantifikation af Ag nanopartikler i tekstiler

Preparation of silver nanoparticle loaded cotton threads to facilitate measurement development for textile applications

Certification Report of CRM BAM-N001, Particle size parameters of nano silver

Nanotechnologies - Vocabulary

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Report on the BIPM workshop on metrology at the nanoscale

Diameter measurements of polystyrene particles with atomic force microscopy

Workplace atmospheres -- Characterization of ultrafine aerosols/nanoaerosols -- Determination of the size distribution and number concentration using differential electrical mobility analysing systems

Workplace exposure - Metrics to be used for the measurements of exposure to inhaled nanoparticles (nano-objects and nanostructured materials) such as mass concentration, number concentration and surface area concentration

Sample preparation -- Dispersing procedures for powders in liquids

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Towards a review of the EC Recommendation for a definition of the term "nanomaterial"

Comparative assessment of nanomaterial definitions and safety evaluation considerations

Sunscreens with titanium dioxide (TiO2) Nano-Particles: A Societal Experiment

The structure, composition, and dimensions of TiO2 and ZnO nanomaterials in commercial sunscreens

Suspensions of modified TiO2 nanoparticles with supreme UV filtering ability

Improved sample preparation and quality control for the characterization of titanium dioxide nanoparticles in sunscreens using flow field flow fractionation on-line with inductively coupled plasma mass spectrometry

The report proposes *description*

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conclude

It is concluded

It is found

The report deliver

concluded

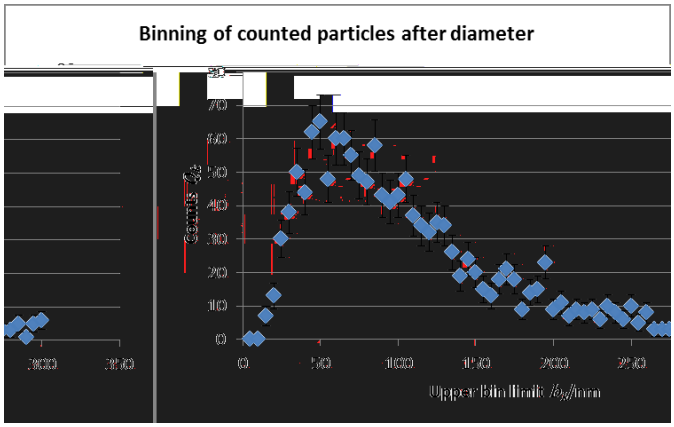
concluded

proposed

concluded

N

x_i i



I_i

x_i

$x_i = aI_i,$

a

$u(a)$

a

Q_K

I_I $B_{K-1} < I_I \leq B_K$ $B_0 =$ $B_{K-1} =$ $B_K - B_{K-1} =$

$u(a)$

I_i x_i

I_i

$u(I_i)$

N

$b_0 < b_1 < \ldots < b_K$ K

I_i k $b_{k-1} < I_i \leq b_k$

k q_k

Q_k $u(Q_k)$

k N

$Q_k = q_k + 1, \quad u(Q_k) = \sqrt{Q_k},$

N=1479 $K =$

$b_k - b_{k-1} =$ $b_0 =$

$b_{K-1} =$ K $b_K =$

$u(Q_k)$

counting efficiency

$e(x)$ x

$e(x)$

$$e(x) = A\left(1 - \exp\left(-\frac{x}{x_c}\right)\ln(2)\right),$$

A x_c

$e(x_c) =$ A x_c

$u(A)$ $u(x_c)$

$u(e(x))$ $e(x)$

$u(e(x))/e(x)$

Q_k e_k

k i

x_i I_i $1/e(x_i)$

Q_k^c $u(Q_k) =$

$\sqrt{Q_k}$

f x

$b_K =$

f

f

f

$$f = \frac{n_{1-100}}{n_{1-5000}}$$

n_{1-100} n_{1-5000}

f f

$u(f)$ M_{nano}

$f \geq 0.5$

p_{nano}

$$p_{\text{nano}} = \frac{M_{\text{nano}}}{M}$$

$p_{\text{nano}} \geq 0.95$
$p_{\text{nano}} \leq 0.05$

f

p_{nano}

p_{nano}

a A

x_c $u(I_i)/I_i$

$f =$

$u(f) =$ $p_{\text{nano}} =$

☐ q_k ☐ ☐ $b_{k-1} < I_i \leq b_k$
☐ ☐ $Q_k = q_k + 1$ ☐ ☐
e $u(Q_k) = \sqrt{Q_k}$

f

f

$Q_k, k = 1, \ldots, K$ $\mathrm{G}(Q_k, 1)$
Q_k Q_k Q_k

$I_i, i = 1, \ldots, N,$ $\mathrm{N}(I_i, u^2(I_i))$ I_i
$u(I_i)$
$s_r = u(I_i)/I_i$
a $\mathrm{N}(a, u^2(a))$ a
$u^2(a)$
A $\mathrm{N}(A, u^2(A))$ A
$u^2(A)$
x_c $\mathrm{N}(x_c, u^2(x_c))$ x_c
$u(x_c)$

f
I_i
$b_{k-1} < I_i \leq b_k$
$u(I_i)$ I_i
I_i

$n_{1-100} = n_{1-5000} = 0$ k Q_k

I $b_{k-1} < I \leq b_k$
I_i $\mathrm{N}(I, \sigma^2)$
I $\sigma = s_r I$ $s_r = u(I_i)/I_i$
$x_i = aI_i$
$1\ \mathrm{nm} \leq x_i \leq 100\ \mathrm{nm}$ n_{1-100} $1/e(x_i)$
$1\ \mathrm{nm} \leq x_i \leq 5000\ \mathrm{nm}$ n_{1-5000} $1/e(x_i)$

f

$$f = \frac{n_{1-100}}{n_{1-5000}}$$

f

k Q_k' $\mathrm{G}(Q_k, 1)$

a' $\mathrm{N}(a, u^2(a))$ a
$u(a)$
A' $\mathrm{N}\big(A, u^2(A)\big)$
A $u(A)$
x_c'
$\mathrm{N}(\, x_c, u^2(x_c))$
x_c
$u(x_c)$

$Q_k', k = 1, \ldots, K$ a' A' x_c'

f

M
f
f
$u(f)$
M_{nano}
$f \geq 0.5$

Calibration and validation:

Detection limit, measurement interval and measurement uncertainty:

Particle size analysis -- Dynamic light scattering (DLS)

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Preparation for measurements:

Results:

Limitations of the method:

Additional possibilities when using the method:

The objective of this project was to establish a set of validation parameters which can be used to document the performance of measurement methods to detect and quantify nanoparticles. The core of the report has been to specify, interpret and clarify this set of validation parameters so they meet the requirements that are relevant for nanomaterials in the regulatory context.

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