Towards a method for detecting the potential genotoxicity of nanomaterials

Deliverable 4.1: Summary report on primary physicochemical properties of manufactured nanomaterials used in NANOGENOTOX

Key intrinsic physicochemical characteristics of NANOGENOTOX nanomaterials

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WP 4: Physicochemical Characterization of Manufactured Nanomaterials (MNs) and Exposure Media (EMs)

Deliverable 4.1: Summary report on primary physico-chemical properties of manufactured nanomaterials used in NANOGENOTOX

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Key intrinsic physicochemical characteristics of NANOGENOTOX nanomaterials

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National Research Centre for the Working Environment

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1 Introduction

This report summarizes the key physico-chemical data on the titanium dioxide ($TiO_{2)}$, Synthetic Amorphous Silica (SAS), and Multiwalled Carbon Nanotubes (MWCNT) Manufactured Nanomaterials (MN), which were analyzed as part of the Work Package 4 activities in the NANOGENOTOX Joint Actions Project.

The purpose of the report is to facilitate a quick overview of the main results obtained on the individual MN. For the reporting, different partner contributions from especially transmission electron microscopy (TEM) and X-ray diffraction (XRD) analyses have been averaged to simplify reporting and acknowledging that no "best method" or "best data set" has been defined as part of the project. Especially, for the elemental composition of the test materials, it should be noted that the NANOGENOTOX did not have high focus on this area. For TiO_2 and SAS, semi-quantitative data were obtained on pellets by Energy Dispersive Spectroscopy (EDS). The data reported for the MWCNT, were analyzed by Inductively Couple Plasma Mass Spectrometry (ICP-MS), using an extraction protocol, which was observed to generally have the highest yield. However, indications from thermogravimetric (TGA) data suggest that the extraction procedures were insufficient for full quantification. The reader is referred to the six topical reports listed hereafter for an in-depth presentation and discussion of the data:

- D4.1: Summary report on primary physiochemical properties of manufactured nanomaterials used in NANOGENOTOX

- D4.2: Transmission electron characterization of NANOGENOTOX nanomaterials

- D4.3: Crystallite size, mineralogical and chemical purity of NANOGENOTOX nanomaterials

- D4.4: Determination of specific surface area of NANOGENOTOX nanomaterials

- D4.5: Surface charge, hydrodynamic size and size distributions by zetametry, dynamic light scattering (DLS), and small-angle X-ray scattering (SAXS) in optimized aqueous suspensions for titania and silicon dioxide

- D4.6: Dustiness of NANOGENOTOX nanomaterials using the NRCWE small rotating drum and the INRS Vortex shaker

- D4.7: Hydrochemical reactivity, solubility, and biodurability of NANOGENOTOX nanomaterials.

The full references to the reports are found in the reference list at the end of the report.

Note that the results in the current report are considered the final data from the project.

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2 Key physical and chemical data on the test materials

In the following sub-chapters the key physical and chemical data obtained on the NANOGENOTOX test materials are summarized in material data sheets for each test material. The sheets contain the following data :

1) Representative transmission electron microscopy (TEM) image.
2) Information on the main phase from TEM and/or X-ray diffraction (XRD), and/or Raman spectroscopy.
3) The average area Equivalent Spherical Diameter (ECD) or in case of carbon nanotubes, the tubular diameter as determined by TEM.
4) The average maximum particle dimension/geodesic length of the MWCNT as determined by TEM.
5) The average spherical size calculated from Small Angle X-ray Scattering (SAXS)
6) The average Zeta aggregate size determined by Dynamic Light Scattering (DLS) of well-dispersed samples.
7) The BET (Brunauer-Emmet-Teller) specific surface area (SSA) determined by notrogen adsorption
8) The SSA determined from Small Angle X-ray Scattering (SAXS)
9) The major elements in the sample determined semi-quantitatively by either Energy Dispersive Spectrometry (EDS) or Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
10) Minor elements in the sample determined semi-quantitatively by either Energy Dispersive Spectrometry (EDS) or Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
11) The amount of organic coating as determined by Thermograviometric Analysis (TGA), considering only the mass-loss from 100 to 1000°C or for CNT the total percentage of un-combusted residual after TGA.
12) The Iso-Electrical Point (IEP) determined in a 0.036 mol/L constant ionic strength solution based on HNO_3, NaOH, and $NaNO_3$ mixtures for determination at fixed pH values.
13) The DLS average Zeta-size of 2.56 mg/ml sample in 0.05% w/v BSA water used for NM batch dispersion
14) The result after 24-hour dissolution of 0.32 mg/ml in 0.05% w/v BSA water at 37°C and 5% CO2 atmosphere.
15) The effect on pH during 24-hour dissolution in 0.05% w/v BSA water at 37°C and 5% CO2 atmosphere.
16) The effect on oxygen concentration ($dO2 = O_{2,sample} - O_{2,reference}$) during 24-hour dissolution in 0.05% w/v BSA water at 37°C and 5% CO2 atmosphere.
17) The inhalable dustiness determined using a down-scaled EN15051 rotating drum dustiness tester
18) The respirable dustiness determined using a down-scaled EN15051 rotating drum dustiness tester
19) The respirable dustiness determined using a Vortex shaker dustiness tester

Abbreviations and symbols

NA Not available
ND Not Detected
- Negative
(-) Slightly negative
+ Positive
(+) Slightly positive

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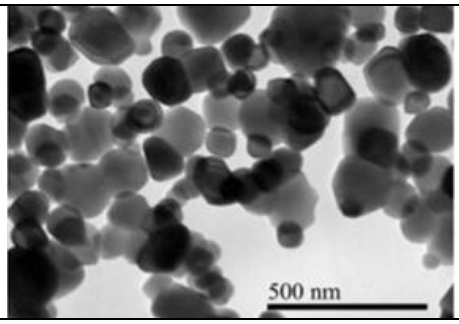
2.1 Material Data Sheet for NM-100

Representative TEM image	
Phase	Anatase (TiO_2)
Minor Impurity phases	ND
TEM primary particle size ECD [nm] (n=2[€])	110±57
TEM length of primary particle [nm]	NA
XRD size [nm] (average of 6 methods[£])	56.7 - > 100
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	9
SAXS SSA [m^2] (n=1)	NA
Major elements	Ti, O
Minor elements/coatings (EDS) Si [wt%] Al [wt%] Fe [wt%]	Fe, Si, Al 0.28 0.09 0.49
Organic coating [wt%]	ND
Organic compounds	NA
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 215±2 Ti trace (-) +
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 NA NA
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 1500

[€] data from n laboratories;[£] Six different calculations based on data from three from 3 laboratories

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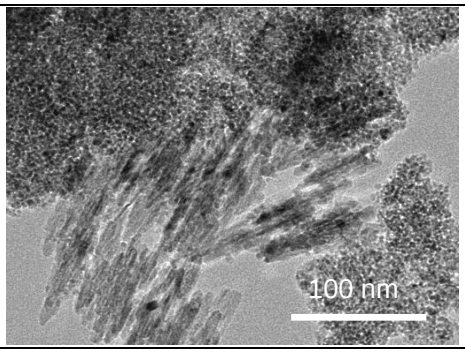
2.2 Material Data Sheet for NM-101

Representative TEM image	
Phase	Anatase (TiO_2)
Minor Impurity phases	ND
TEM primary particle size ECD [nm] (n=2)	5.5±0.7
TEM length of primary particle [nm]	NA
XRD size [nm] (average of 6 methods)	6.9±5.8
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	316
SAXS SSA [m^2] (n=1)	170±9
Major elements	Ti, O
Minor elements/coatings (EDS) Si (wt%) Al (wt%) Fe (wt%)	Si, Fe 0.09 0.29 -
Organic coating (wt%]	8
Organic compounds	silanes? hexadecanoic and oxyadecanoic acids
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 483±20 Al-coating unstable (-) +
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 728±10 24±9
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 5600

[€] data from n laboratories;[£] Six different calculations based on data from three from 3 laboratories

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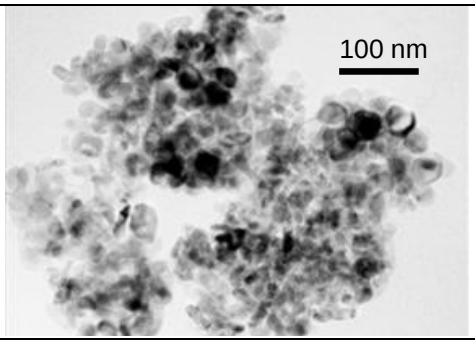
2.3 Material Data Sheet for NM-102

Representative TEM image	
Phase	Anatase (TiO_2)
Minor Impurity phases	ND
TEM primary particle size ECD [nm] (n=3)	21.7±0.6
TEM length of primary particle [nm]	NA
XRD size [nm] (average of 6 methods)	22.5±18.3
SAXS aggregate size [nm]	560
DLS Zeta aggregate size [nm]	423±59
BET SSA [m^2] (n=1)	78
SAXS SSA [m^2] (n=1)	66±3
Major elements	Ti, O
Minor elements/coatings (EDS)	Si, Fe, Al
Si [wt%]	0.08
Al [wt%]	0.05
Fe [wt%]	0.07
Organic coating [wt%]	ND
Organic compounds	NA
IEP [pH]	6
Zeta-size and reactivity in 0.05% BSA water	
DLS Zeta-size BSA medium [nm]	545±14
24-hour dissolution	Ti trace
pH reactivity	(-)
dO_2 Reactivity	negligible
Rotating Drum Dustiness	
Inhalable dustiness index [mg/kg] (n=3)	268±39
Respirable dustiness index [mg/kg] (n=3)	15±2
Vortex shaker dustiness	
Respirable dustiness index [mg/kg] (n=1)	9200

€ data from n laboratories; £ Six different calculations based on data from three from 3 laboratories

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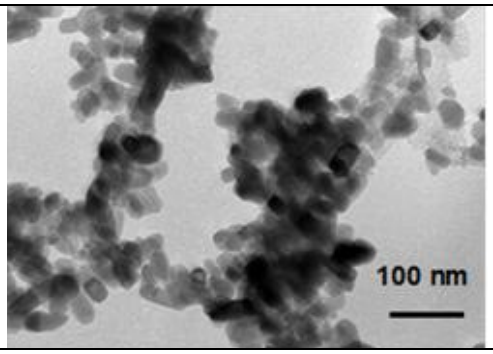
2.4 Material Data Sheet for NM-103

Representative TEM image	
Phase	Rutile (TiO_2)
Minor Impurity phases	ND
TEM primary particle size ECD [nm] (n=3)	24.7±2.3
TEM length of primary particle [nm]	NA
XRD size [nm] (average of 6 methods)	22.3±21.5
SAXS aggregate size [nm]	140
DLS Zeta aggregate size [nm]	113±3
BET SSA [m^2] (n=1)	51
SAXS SSA [m^2] (n=1)	51±2
Major elements	Ti, O
Minor elements/coatings (EDS) Si [wt%] Al [wt%] Fe [wt%]	Al, Si, Fe 0.68 3.43 0.06
Organic coating [wt%]	2
Organic compounds	dimethoxydimethyl-silane, silanes
IEP [pH]	8.2
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 194±2 Si and Al-coating unstable (-) negligible
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 9185±234 323±166
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 19000

€ data from n laboratories;£ Six different calculations based on data from three from 3 laboratories

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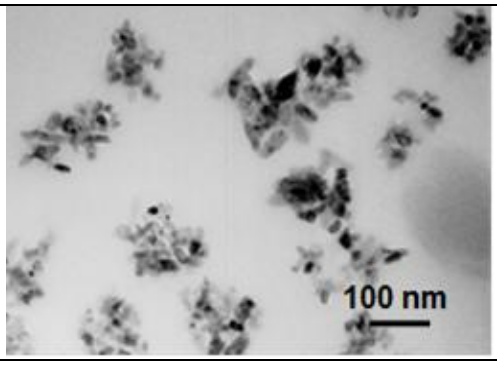
2.5 Material Data Sheet for NM-104

Representative TEM image	
Phase	Rutile (TiO_2)
Minor Impurity phases	ND
TEM primary particle size ECD [nm] (n=3)	25.0±1.7
TEM length of primary particle [nm]	NA
XRD size [nm] (average of 6 methods)	22.9±21.1
SAXS aggregate size [nm]	160
DLS Zeta aggregate size [nm]	129±1
BET SSA [m^2] (n=1)	56
SAXS SSA [m^2] (n=1)	52±2
Major elements	Ti, O
Minor elements/coatings (EDS) Si [wt%] Al [wt%] Fe [wt%]	Al, Si 0.18 3.22 ND
Organic coating [wt%]	2
Organic compounds	tetramethyl-silicate?, glycerol, silanes? hexadecanoic and oxyadecanoic acids
IEP [pH]	8.2
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 234±34 Al-coating unstable (-) negligible
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 3911±235 38±7
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 6400

€ data from n laboratories; £ Six different calculations based on data from three from 3 laboratories

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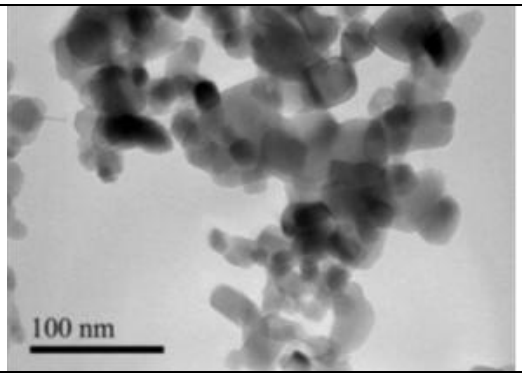
2.6 Material Data Sheet for NM-105

Representative TEM image	
Phase(s)	Anatase:Rutile (84:16) (TiO_2)
Minor Impurity phases	ND
TEM primary particle size ECD [nm] (n=3)	Anatase 24.3±3.5 / Rutile 15.0
TEM length of primary particle [nm]	NA
XRD size [nm] (average of 6 methods)	Anatase 23.3±19.3 / Rutile 56.9±69.3
SAXS aggregate size [nm]	130
DLS Zeta aggregate size [nm]	125±4
BET SSA [m^2] (n=1)	46
SAXS SSA [m^2] (n=1)	47±2
Major elements	Ti, O
Minor elements/coatings (EDS) Si [wt%] Al [wt%] Fe [wt%]	Si,Al 0.07 0.04 ND
Organic coating [wt%]	ND
Organic compounds	NA
IEP [pH]	6.6
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 155±1 < (-) negligible
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 1020±20 28±10
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 11000

€ data from n laboratories;£ Six different calculations based on data from three from 3 laboratories

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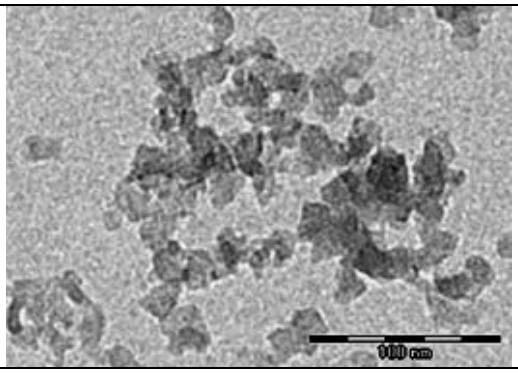
2.7 Material Data Sheet for NM-200

Representative TEM image	
Main Phase	Synthetic Amorphous Silica (SAS)
Minor Impurity phases	Na_2SO_4, AlO(OH), cristobalite (by XRD)
TEM primary particle size ECD [nm] (n=3)	18.3±4.5
TEM length of primary particle [nm]	NA
XRD size [nm]	not relevant
SAXS aggregate size [nm]	440
DLS Zeta aggregate size [nm]	208±12
BET SSA [m^2] (n=1)	189
SAXS SSA [m^2] (n=1)	123±5
Major elements	Si, O
Minor elements/coatings (EDS) Na [wt%] Ca [wt%] S [wt%] Al [wt%]	Na, S, Al 0.88 ND 0.46 0.87
Organic coating [wt%]	3
Organic compounds	ND
IEP [pH]	<2
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 185±5 SAS and Al-impurity partially soluble (-) negligible
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 6459±273 293±193
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 34000

€ *data from n laboratories*

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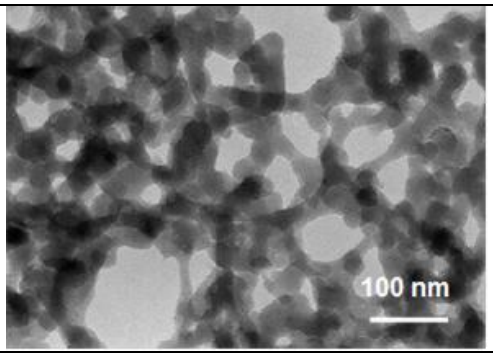
2.8 Material Data Sheet for NM-201

Representative TEM image	
Main Phase	Synthetic Amorphous Silica (SAS)
Minor Impurity phases	Na_2SO_4, AlO(OH) (by XRD)
TEM primary particle size ECD [nm] (n=3)	18.0±1.0
TEM length of primary particle [nm]	NA
XRD size [nm]	not relevant
SAXS aggregate size [nm]	180
DLS Zeta aggregate size [nm]	201±28
BET SSA [m^2] (n=1)	140
SAXS SSA [m^2] (n=1)	123±8
Major elements	Si, O
Minor elements/coatings (EDS) Na [wt%] Ca [wt%] Al [wt%] S [wt%]	Al, Na, S 0.44 ND 0.74 0.46
Organic coating [wt%]	3
Organic compounds	ND
IEP [pH]	<2
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 176±3 Al-impurity partially soluble (-) (+)
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 6034±199 218±24
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 6500

€ *data from n laboratories*

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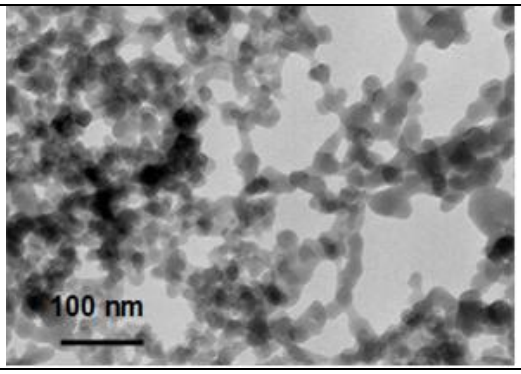
2.9 Material Data Sheet for NM-202

Representative TEM image	
Main Phase	Synthetic Amorphous Silica (SAS)
Minor Impurity phases	Na_2SO_4, AlO(OH) (by XRD)
TEM primary particle size ECD [nm] (n=3)	17.7±2.5
TEM length of primary particle [nm]	NA
XRD size [nm]	not relevant
SAXS aggregate size [nm]	100
DLS Zeta aggregate size [nm]	176±5
BET SSA [m^2] (n=1)	204
SAXS SSA [m^2] (n=1)	184±18
Major elements	Si, O
Minor elements/coatings (EDS) Na [wt%] Ca [wt%] Al [wt%] S [wt%]	Al, Ca ND 0.18 0.45 ND
Organic coating [wt%]	ND
Organic compounds	NA
IEP [pH]	2 to 4
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 134±4 SAS and Al-impurity partially soluble (-) +
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 4988±1866 91±11
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 17000

€ *data from n laboratories*

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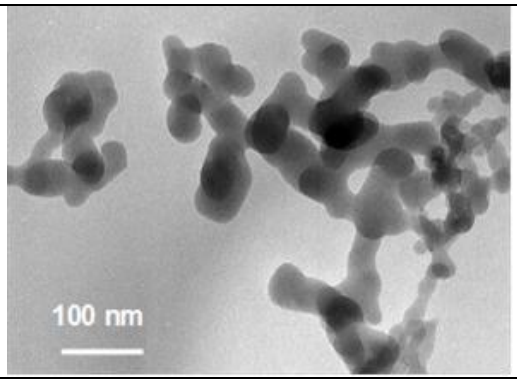
2.10 Material Data Sheet for NM-203

Representative TEM image	
Main Phase	Synthetic Amorphous Silica (SAS)
Minor Impurity phases	Na_2SO_4, AlO(OH) (by XRD)
TEM primary particle size ECD [nm] (n=3)	24.7±17.7
TEM length of primary particle [nm]	NA
XRD size [nm]	not relevant
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	175±7
BET SSA [m^2] (n=1)	204
SAXS SSA [m^2] (n=1)	167±13
Major elements	Si, O
Minor elements/coatings (EDS) Na [wt%] Ca [wt%] Al [wt%] S [wt%]	Al, S ND ND 0.43 0.04
Organic coating [wt%]	-
Organic compounds	ND
IEP [pH]	2 to 4
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 127±2 SAS and Al-impurity partially soluble (-) +
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 5800±1488 354±6
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 51000

€ *data from n laboratories*

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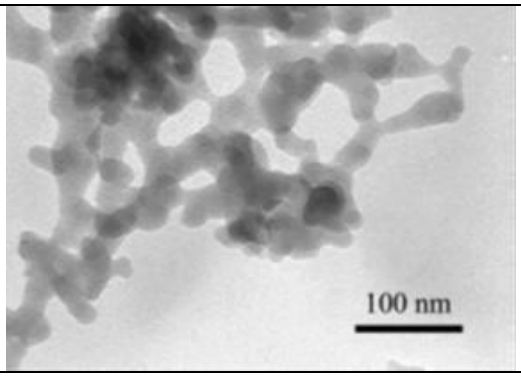
2.11 Material Data Sheet for NM-204

Representative TEM image	
Main Phase	Synthetic Amorphous Silica (SAS)
Minor Impurity phases	ND
TEM primary particle size ECD [nm] (n=2)	15.8±4.6
TEM length of primary particle [nm]	NA
XRD size [nm]	not relevant
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	137
SAXS SSA [m^2] (n=1)	132±23
Major elements	Si, O
Minor elements/coatings (EDS) Na [wt%] Ca [wt%] Al [wt%] S [wt%]	Al, S, Na 0.18 ND 0.48 0.21
Organic coating [wt%]	0.5
Organic compounds	silane, hexadecanoic and octadecanoic acids
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 174±7 SAS and Al-impurity partially soluble negligible (+)
Rotating Drum Dustiness Inhalable dustiness index [mg/kg] (n=3) Respirable dustiness index [mg/kg] (n=3)	 24969±601 1058±<1
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 14000

€ *data from n laboratories*

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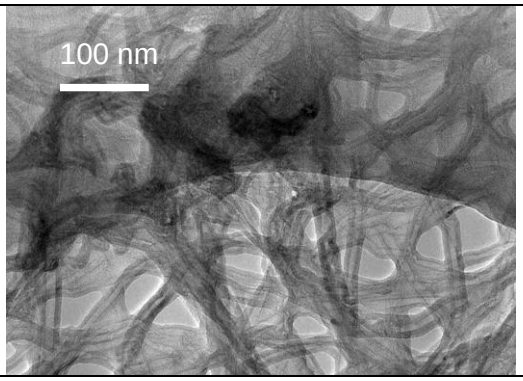
2.12 Material Data Sheet for NM-400

Representative TEM image	
Main Phase	Multi-walled Carbon Nanotube (MWCNT)
Minor Impurity phases	Al_2O_3 (XRD on TGA residual)
TEM size ECD [nm] (n=2)	13.6±3.7
TEM geodesic length [nm] (n=1)	846±446
XRD size [nm]	not relevant
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	254
SAXS SSA [m^2] (n=1)	189±8
Major elements	C
Minor elements/coatings (ICP-MS) Na [µg/g] Mg [µg/g] Al [µg/g] Mn [µg/g] Fe [µg/g] Ni [µg/g]	Al, Fe, Na, Ni 1345±151 ND 9951±331 ND 1988±26 4
TGA residual (oxidized) [wt%]	16.2
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 54.9±0.6 catalyst partially dissolved (-) negligible
Rotating Drum Dustiness Inhalable dustiness index Respirable dustiness index	 NA NA
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 <420 (below the detection limit)

€ *data from n laboratories*

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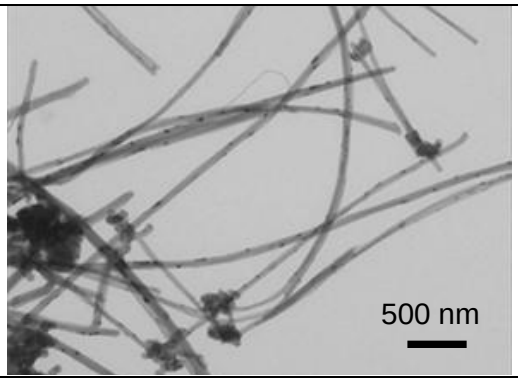
2.13 Material Data Sheet for NM-401

Representative TEM image	
Main Phase	Multi-walled Carbon Nanotube (MWCNT)
Minor Impurity phases	ND
TEM size ECD [nm] (n=2)	64.2±34.5
TEM geodesic length [nm] (n=1)	4048±2371
XRD size [nm]	not relevant
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	18
SAXS SSA [m^2] (n=1)	31±2
Major elements	C
Minor elements/coatings (ICP-MS) Na [µg/g] Mg [µg/g] Al [µg/g] Mn [µg/g] Fe [µg/g] Ni [µg/g]	Na, Fe, Al, Ni, Mg 581±32 0±0.3 59±4 ND 379±71 2
TGA residual (oxidized) [wt%]	18.1
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 710±17 catalyst partially dissolved (-) negligible
Rotating Drum Dustiness Inhalable dustiness index Respirable dustiness index	 NA NA
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 <1700 (below the detection limit)

€ *data from n laboratories*

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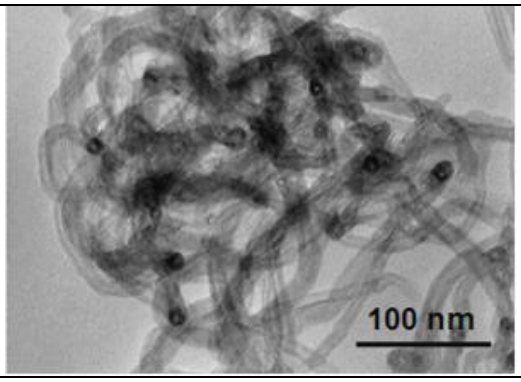
2.14 Material Data Sheet for NM-402

Representative TEM image	
Main Phase	Multi-walled Carbon Nanotube (MWCNT)
Minor Impurity phases	Fe_2O_3 (XRD on TGA residual)
TEM size ECD [nm] (n=2)	12.7±4.7
TEM geodesic length [nm] (n=1)	1372±836
XRD size [nm]	not relevant
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	226
SAXS SSA [m^2] (n=1)	130±4
Major elements	C
Minor elements/coatings (ICP-MS) Na [µg/g] Mg [µg/g] Al [µg/g] Mn [µg/g] Fe [µg/g] Ni [µg/g]	Fe, Al, Na, Ni, Mn 727±120 ND 12955±1530 9±1 16321±664 9±1
TGA residual (oxidized) [wt%]	10.6
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 240±58 catalyst partially dissolved (-) +
Rotating Drum Dustiness Inhalable dustiness index Respirable dustiness index	 NA NA
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 4200

€ *data from n laboratories*

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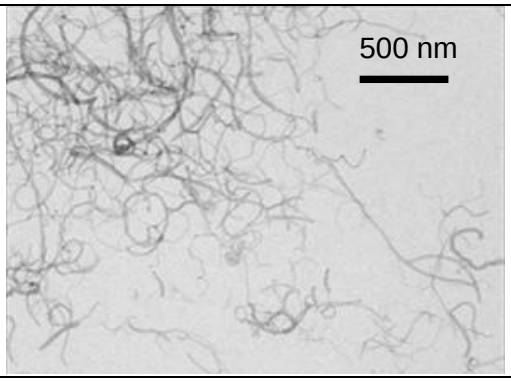
2.15 Material Data Sheet for NM-403

Representative TEM image	
Main Phase	Multi-walled Carbon Nanotube (MWCNT)
Minor Impurity phases	ND
TEM size ECD [nm] (n=1)	12.0±7.0
TEM geodesic length [nm] (n=1)	443±189
XRD size [nm]	not relevant
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	NA
SAXS SSA [m^2] (n=1)	189±11
Major elements	C
Minor elements/coatings (ICP-MS)	Mn,Mg,Al,Na,Ni,Fe
Na [µg/g]	893±443
Mg [µg/g]	2231±144
Al [µg/g]	2024±168
Mn [µg/g]	2706±182
Fe [µg/g]	7±4
Ni [µg/g]	58±4
TGA residual (oxidized) [wt%]	3.2
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water	
DLS Zeta-size BSA medium [nm]	354±46
24-hour dissolution	catalyst partially dissolved
pH reactivity	NA
dO_2 Reactivity	NA
Rotating Drum Dustiness	
Inhalable dustiness index	NA
Respirable dustiness index	NA
Vortex shaker dustiness	
Respirable dustiness index [mg/kg] (n=1)	4900

€ *data from n laboratories*

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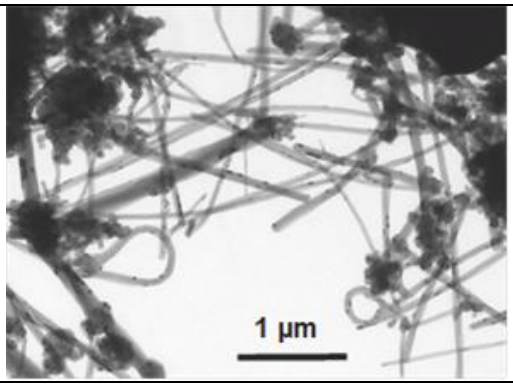
2.16 Material Data Sheet for NRCWE-006

Representative TEM image	
Main Phase	Multi-walled Carbon Nanotube (MWCNT)
Minor Impurity phases	Fe_2O_3 (XRD on TGA residual)
TEM size ECD [nm] (n=1)	74±28
TEM geodesic length [nm] (n=1)	5730±3674
XRD size [nm]	not relevant
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	22
SAXS SSA [m^2] (n=1)	29±2
Major elements	C
Minor elements/coatings (ICP-MS) Na [µg/g] Mg [µg/g] Al [µg/g] Mn [µg/g] Fe [µg/g] Ni [µg/g]	Na, Mn, Al, Ni, Mg 499±103 1±1 66±19 ND 355±2 1
TGA residual (oxidized) [wt%]	17.6
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 682±13 catalyst partially dissolved negligible negligible
Rotating Drum Dustiness Inhalable dustiness index Respirable dustiness index	 NA NA
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 NA

€ *data from n laboratories*

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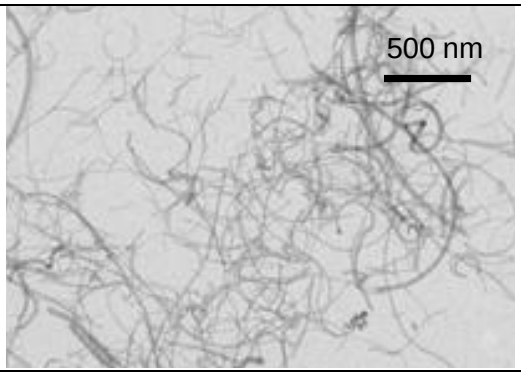
2.17 Material Data Sheet for NRCWE-007

Representative TEM image	
Main Phase	Multi-walled Carbon Nanotube (MWCNT)
Minor Impurity phases	$NiFe_2O_4$ (XRD on TGA residual)
TEM size ECD [nm] (n=1)	17±7
TEM geodesic length [nm] (n=1)	465±340
XRD size [nm]	not relevant
SAXS aggregate size [nm]	NA
DLS Zeta aggregate size [nm]	NA
BET SSA [m^2] (n=1)	75
SAXS SSA [m^2] (n=1)	117±3
Major elements	C
Minor elements/coatings (ICP-MS) Na [µg/g] Mg [µg/g] Al [µg/g] Mn [µg/g] Fe [µg/g] Ni [µg/g]	Ni, Fe, Na, Al, Mg, Mn 505±190 54±16 86±24 14±2 480±13 4843±289
TGA residual (oxidized) [wt%]	6.2
IEP [pH]	NA
Zeta-size and reactivity in 0.05% BSA water DLS Zeta-size BSA medium [nm] 24-hour dissolution pH reactivity dO_2 Reactivity	 223±12 catalyst partially dissolved negligible +
Rotating Drum Dustiness Inhalable dustiness index Respirable dustiness index	 NA NA
Vortex shaker dustiness Respirable dustiness index [mg/kg] (n=1)	 NA

€ *data from n laboratories*

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THIS REPORT IS BASED ON THE SIX FINAL TOPICAL DELIVERABLE 4 REPORTS

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