NanoDefine – figures and tables final report

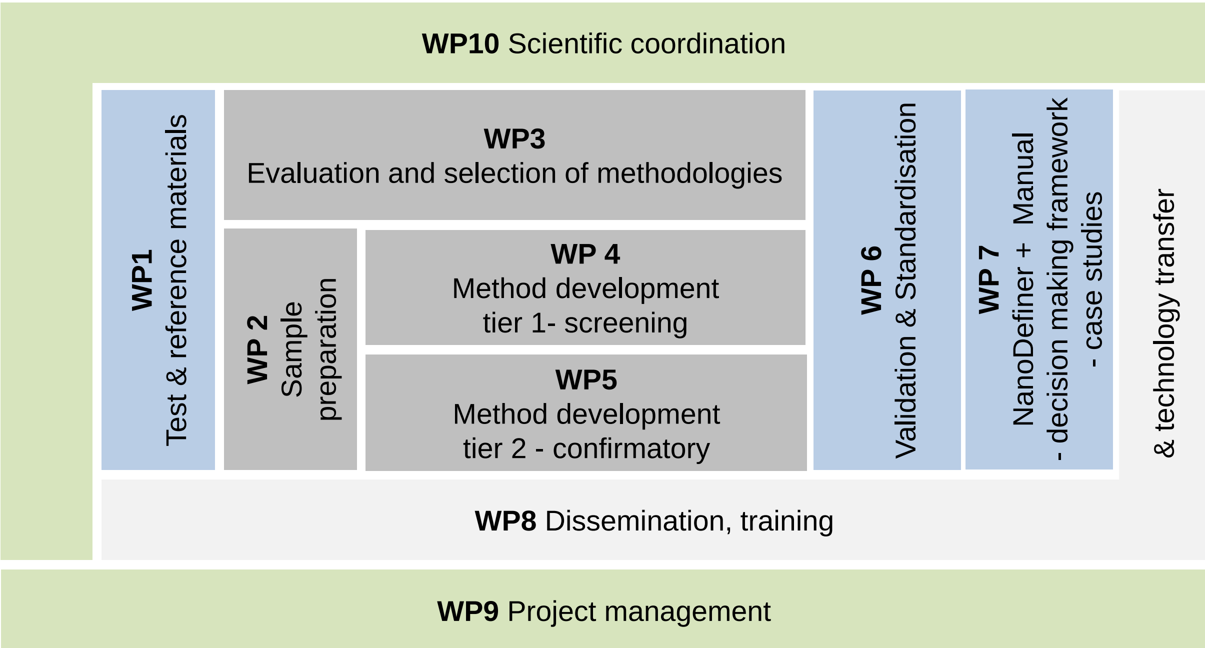

Figure 1: Structure of the NanoDefine work plan

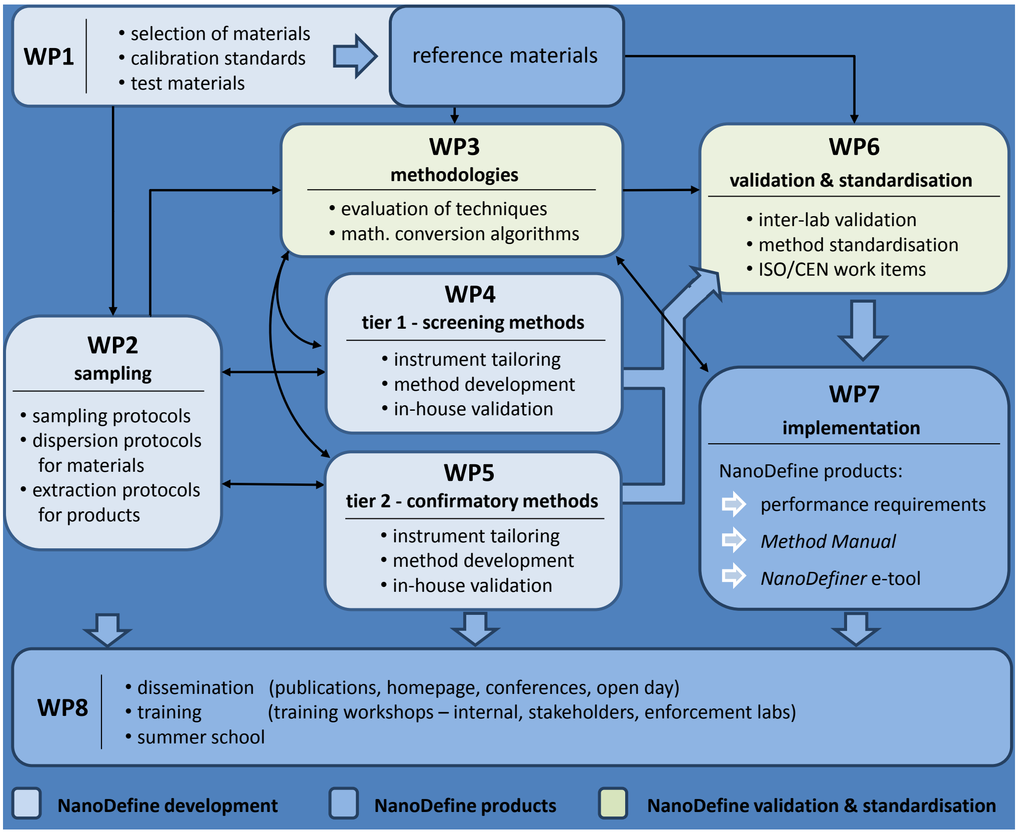

Figure 2: Overview on the coherence and interactions of the different WP, tasks and subtask

Table 1: Selection of representative test materials

able 1: Selection of representative test materials **Material ID**	**material**	**Subst.(S) / product (P)**	**pre-selected**	**nano (n) / coarse (c)**	**link to NANoREG WP2**	**Origin: maufactured (m); natural (n); incidental (i)**	**chem. composition**	**shape**	**presence of different sized particles**	**trade form**
IRMM-382	MWCNT	S	x	n	x	m	carbon	fiber	nano	powder
IRMM-380	Organic pigment	S	x	n		m	organic	particle	nano+(non-nano)	powder
IRMM-386	Organic pigment	S		c		m	organic	particle	(nano)+non-nano	powder
IRMM-384	$CaCO_3$ (fine grade)	S	x	c		m	inorganic	rod / cigar	(nano)+non-nano	powder
IRMM-383	Nano Steel	S	x	n		m	metal	platelets	nano+(non-nano)	powder
IRMM-387	$BaSO_4$ (ultrafine grade)	S		n	x	m	inorganic	particle	nano+(non-nano)	powder
IRMM-381	$BaSO_4$ (fine grade)	S		c		m	inorganic	particle	non-nano	powder
IRMM-385	Kaolin	S		c	x	n	inorganic	multiscale platelets	(nano)+non-nano	powder
BAM-11	Zeolite powder	S		c		m	inorganic	particle	(nano)+non-nano	powder
IRMM-389	Basic methacrylate copolymer particles (BMA)	S		c		m	organic	particle	non-nano	powder
IRMM-388	coated TiO_2	S		c		m	inorganic/ composite	core-shell	non-nano	powder
BAM-12a/b	Food with SiO_2, (NanoLyse-material+ commercial dry pancake mix)	P	x	n		m	inorganic	particle	nano+(non-nano)	suspension /powder
BAM-13a	TiO_2 +surface treatment with aluminum compound formulated in sunscreen (w/ Fe_2O_3, micro-TiO_2)	P	x	n		m	inorganic/ composite	particle	nano + (non-nano)	cream
BAM-13b	TiO2 +surface treatment with aluminum compound formulated in simplified sunscreen	P	x	n		m	inorganic/ composite	particle	nano	cream
BAM-14 BAM-14-1 BAM-14-2	Fe_2O_3 in HDPE (+ raw HDPE + raw Fe_2O_3 powder)	P		n		m	inorganic	particle	nano	granulate
BAM-15	Al_2O_3 in toothpaste	P		c		m	inorganic	particle	(nano)+non-nano	paste

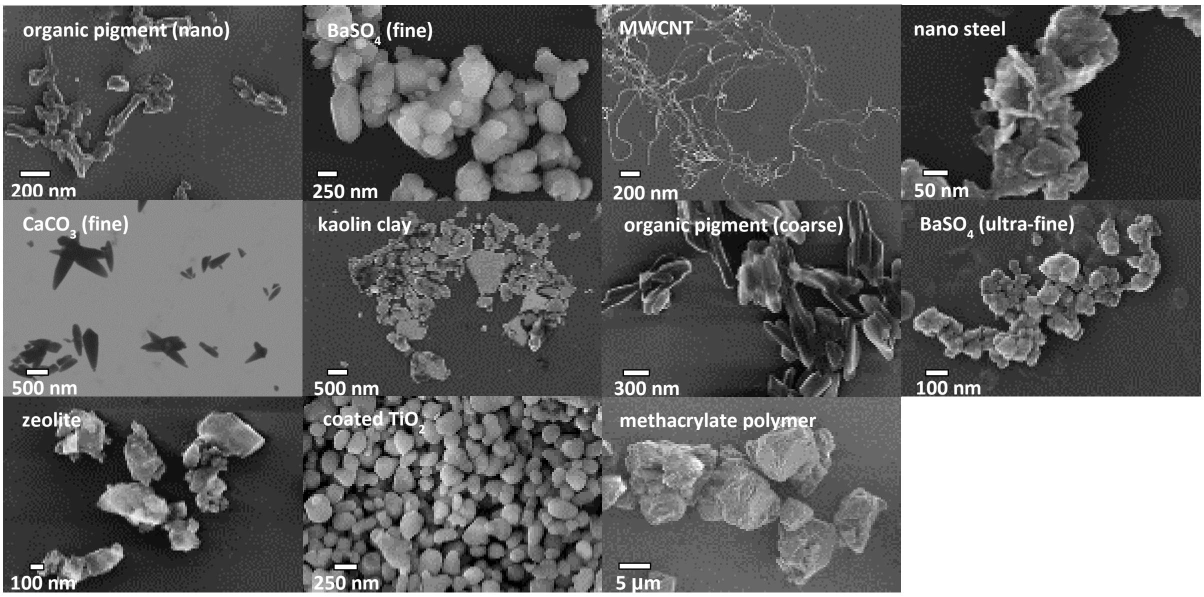

Figure 3: SEM images of the selected substances

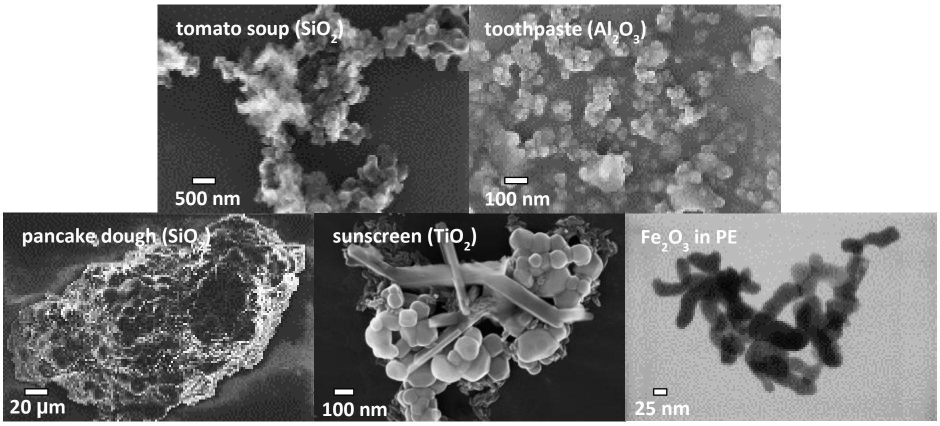

Figure 4: SEM images of the selected consumer products

Table 2: Results of the evaluation of all considered MTs and recommendations of MTs for further specific development or direct validation and standardisation.

		Size range: nm 1-10	nm 10-30	nm 30-100	µm 0.1–1	µm 1–10	µm >10	Direct counting CM	Convertibility to number weighted PSD / Quantitative accuracy	Access to the smallest dimension of each particle	Measurement of the material as it is	ISO standards available	Size Accuracy	Chemical selectivity	Access to primary particles?	Costs per sample	Analysis turnaround time	Widely used/ Availability	Hyphenation to other methods?	Potential for improvement in NanoDefine within 2014-2017?	Recommended to WP4	Recommended to WP5	Recommended to WP6
Counting	EM SEM	-	(+)	++	++	++	++	++		+	--	o	+	++[3]	+	-	-	++	-	+	-	++	+
Counting	EM TSEM	(+)	+	++	++	++	++	++		+	--	+	++	++3	+	-	-	+	-	+	-	++	++
Counting	EM TEM	++	++	++	++	++	+	++		+	--	+	++	++3	++	--	--	o	--	o	--	++	++
Counting	SFM	+	++	++	+	o	-	++		++	-	+	++	-	o	-	--	+	-	o	-		+
Counting	PTA	--	o	+	++	-	--	+		--	-	+	+	o	--	+	+	o	-	+	+		+
Counting	TRPS	--	--	o	+	+	--	++		--	-	++	++	-	--	+	+	-	-	-		-	-
Counting	sp ICP-MS	-	o	+	++	-	--	++		--	-	o	+	++	-	+	+	-	+	+		+	+
Fractionating	FFF	+	++	++	++	o	--	-	-	-	-	-	+	+[4]	-	o	o	-	++	+		++	+
Fractionating	AC	o	+	++	++	+	-	-	o	--	-	++	++	-	--	+	+	+	-	+	+	-	+
Fractionating	DMAS	+	++	++	++	--	--	+	+	-	-	++	+	-	-	+	+	o	+	o	+	-	o
Fractionating	SEC	o	+	+	+																		
Ensemble	DLS	+	++	++	++																		
Ensemble	SAXS	+	+	+	-																		
Ensemble	USSp	-	+	++	+																		
Ensemble	XRD	+	+	+	-																		
Ensemble	ALS	-	o	+	++																		
Ensemble	OS	+	+	+	o																		
Ensemble	FCS	--	--	--	--																		
Integral	BET	+	+	+	+																		

Recommended to WP4 (Screening methods)		Recommended to WP5 (Confirmatory methods)		Recommended to WP6 (Methods ready for direct validation or standardisation)	
Strongly recommended	recommended	**Strongly recommended**	recommended	**Strongly recommended**	recommended
PTA/DUM	TRPS	**EM**	SFM	**EM**	SFM
sp ICP-MS	FFF	**FFF**	sp ICP-MS[1]		PTA/DUM
AC	SAXS				sp ICP-MS
DMAS	USSp				FFF
DLS	ALS				AC
	BET[2]				

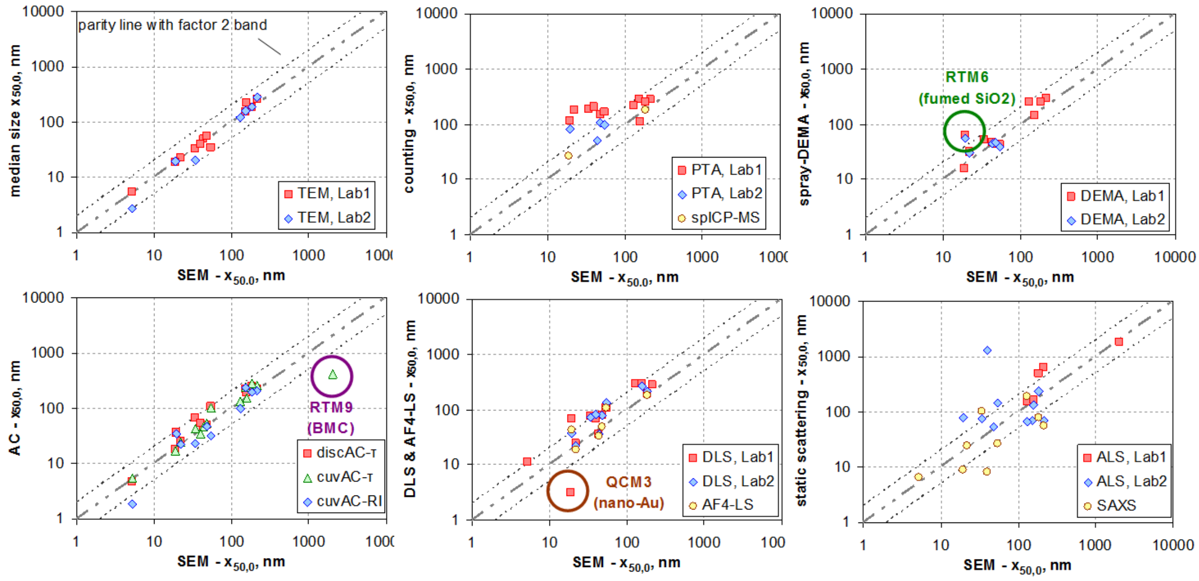

Figure 5: Parity plots of the number weighted medians $x_{50,0}$ as determined by the various MTs versus the SEM value; lines indicate parity and deviation from parity by a factor of 2.

Table 3: u_{IP} obtained for the measurement of the median Feret min of the different materials by P1, P2, P3, TP1 and TP2.

u_{IP}	P1	P2	P3	TP1	TP2
ERM-FD100 18500x	1.8%	1.9%	1.7%	1.7%	2.0%
ERM-FD100 68000x	1.9%	2.3%	2.4%	Test 1: 31.7% Test 2: 1.9%	5.3%
ERM-FD304 18500x	1.3%	3.0%	1.4%	1.4%	1.4%
ERM-FD304 68000x	1.5%	2.2%	1.8%	6.1%	6.1%
Au nanorods	3.4%	7.4%	4.4%	Test 1: 29.5% Test 2: 23.8%	7.2%
NM-100	2.1%	2.6%	2.3%	3.9%	15.8%
NM-103	2.5%	3.1%	2.1%	6.3%	2.3%
NM-212	6.7%	9.0%	6.3%	4.4%	4.5%

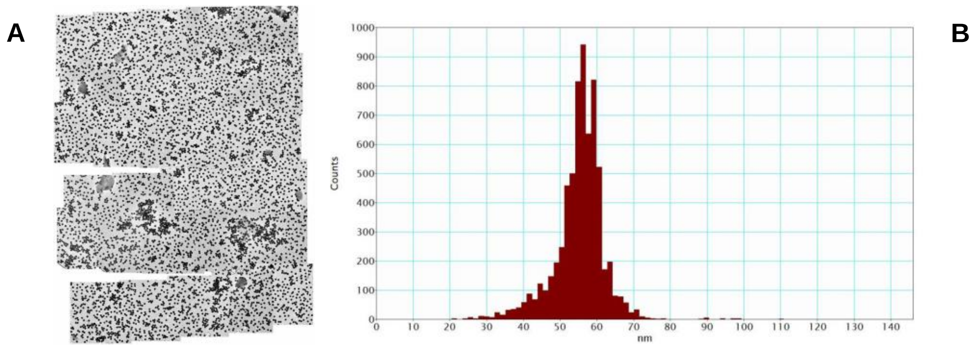

Figure 6: (A) A mosaic image (5x6) of Au NIST NPs (JEOL JEM-2800); (B) PSD of NIST Au NPs (X_{50} value is 56 nm and 6636 particles were analysed).

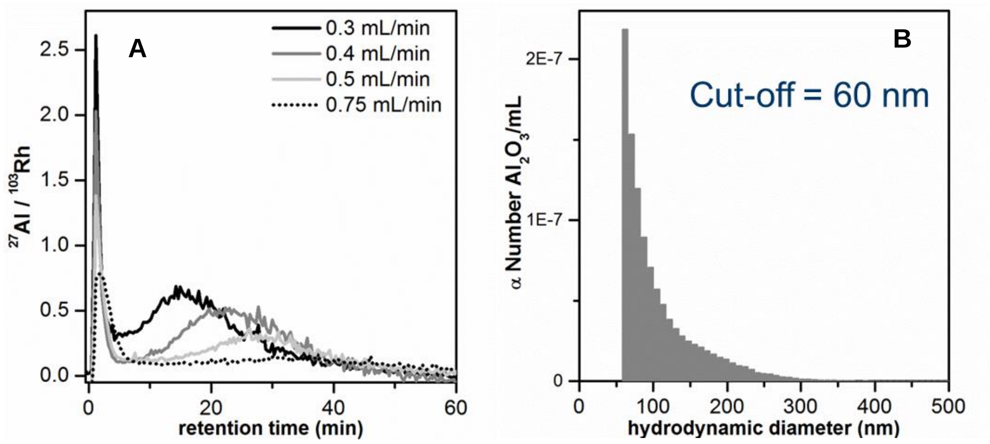

Figure 7: Fractogram of toothpaste with Al_2O_3 particles after AF4-ICP-MS (A) and conversion to number based particle size distribution (B).

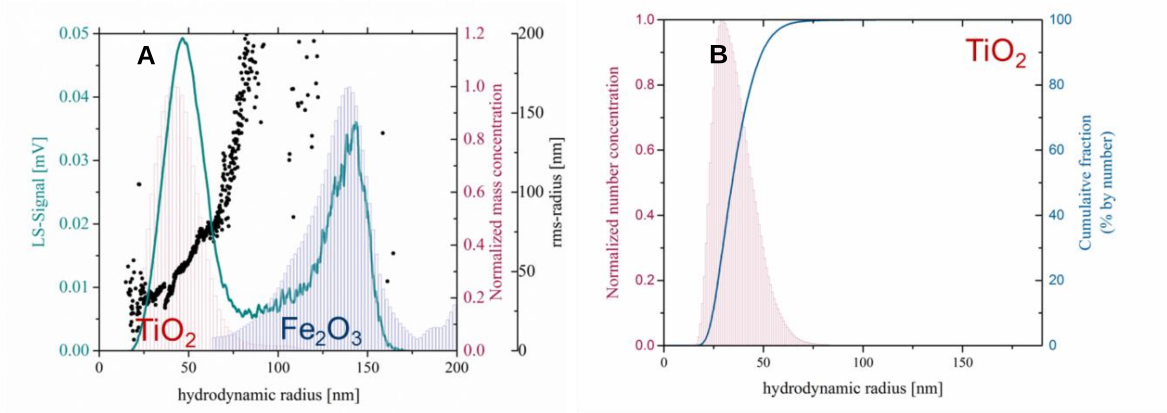

Figure 8: TiO_2 mass-based PSD (A) and number-based PSD (B) of BAM-13a (complex sunscreen) after dilution method. LS-signal (A) is referred to MALS data for a detector angle of 90°.

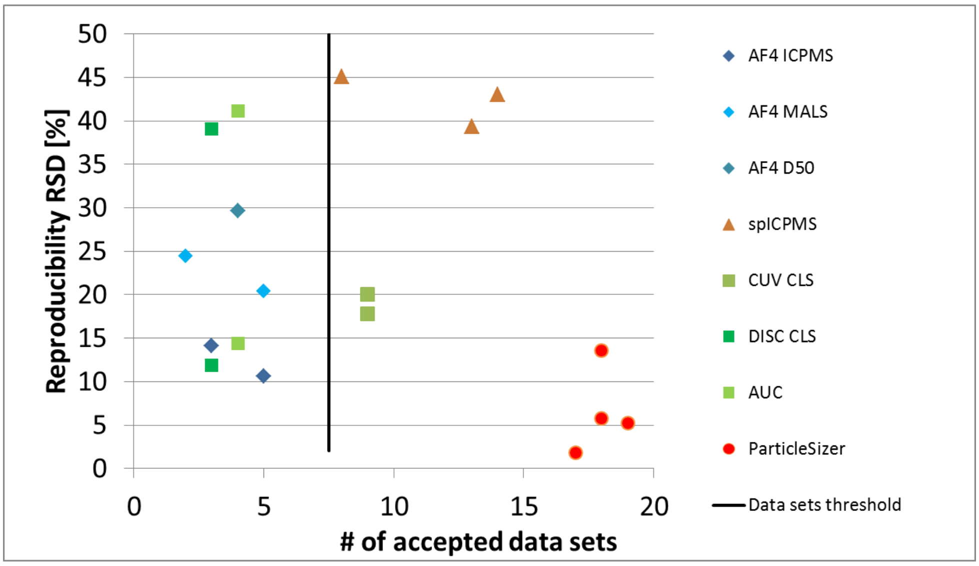

Figure 9: Selected relative reproducibility standard deviations vs. the number of accepted data sets in the inter-laboratory validation exercises. Only the most prominent parameters are shown (average particle sizes of number-based or mass-based distributions). The black bar represents the minimum number of valid data sets of eight. Identical symbols indicate where different test materials were available (CLS) or the particle distribution was bimodal (AF4).

Table 4: Overview of new results (foreground) with high potential for commercial exploitation

Exploitable product	**Main producer**	**Innovation and commercialization potential**
NTA Validation Protocol	Malvern	Describes a set protocol for in-house and inter-laboratory validation of NanoDefine samples. Offers a method for generating values for instrument validation. A technique for evaluating PTA technology.
NTA Validation SOP for sample preparation and measurement	Malvern	Describes a set protocol for sample preparation (including powder dispersion) and measurement setup for NanoDefine supplied samples. To be used in conjunction with NTA Validation protocol. A technique for evaluating PTA technology
HRMS for nanoparticle characterization	RAMEM	There is no commercial High-resolution DMA for particles in the range of 1-5 nm. High Resolution, detection of the smallest nanoparticles.
Electrospray deposition for EM	RAMEM	Sampling system to avoid aggregation and agglomeration of NP for EM. No other sampling system exists to avoid aggregation and homogeneous distribution of NP
Coupling FFF and ESI for aerosol detec-tors	RAMEM	Increase in the number and type of detectors for FFF. First development apart from FFF-ESI-ICP-MS
Concentration Calibration of NTA in-struments	Malvern	Current NTA instruments use fixed values across all instruments and capture settings in determining concentration measurements. This innovation aims to calibrate individual system at a range of capture and analysis settings thereby removing concerns of low reproducibility and user dependence. Additionally, accuracy of measurements will be improved. Increases accuracy and robustness of concentration measurement producing a unique, high throughput, number based concentration tool
spICP-MS data evaluation software	RIKILT	The Single Particle Calculation tool version 2 (SPCTv2) allows to generate spreadsheet-based analysis data of spICP-MS measurements. Data can be imported into the NanoDefiner e-Tool for an automatic nano/non-nano/borderline decision for a specific material.

		http://www.wur.nl/nl/show/Single-Particle-Calculation-tool.htm
Auto-EM toolbox for automated operation and image analysis	EAWAG	The ParticleSizer is an ImageJ plug-in for TEM image analysis. Measures automatically distributions of characteristic size and shape prop-erties of potential nanomaterials. Analysis results exportable in a text-based file format and imported into the NanoDefiner e-Tool for an automatic nano/non-nano/borderline de-cision on a specific material. https://imagej.net/ParticleSizer
The NanoDefiner e-tool	FHDO	a Standardised automated procedure for method selection and NM classification for the most economic implementation of the definition. https://labs.inf.fh-dortmund.de/NanoDefiner/ and http://www.nanodefine.eu/index.php/nanodefiner-e-tool
The NanoDefine Method Manual	JRC	Technical guidance on the use of available methodologies
New Standard operation procedures (SOPs) for dispersion control and various particle size measurement and imaging methods	WP1-7 partners	SOPs described in WP1-7 deliverable reports developed and validated by intra- and/or inter-laboratory comparison studies

Table 5: Number of females and male researchers/technicians as well as postgraduates, PhD students and postdocs involved in NanoDefine

Beneficiary name	Female	Male	Postgraduate	PhD students	Postdocs
RIKILT	5	6	0	0	0
NOMI	2	2	0	0	0
JRC	8	12	0	0	0
UNIVIE	2	4	0	1	4
DTU	4	2	0	0	1
BfR	2	2	0	0	0
EAWAG	0	3	0	0	2
CEA	1	1	0	0	0
TUD	0	3	1	1	2
CODA-CERVA	1	1	0	0	1
UoB	3	0	0	1	1
FHDO	1	5	0	2	0
BAM	6	3	1	1	1
DIN	3	2	0	0	0
BASF	0	4	0	0	0
CLARIANT	0	3	2	1	0
SOLVAY	0	2	0	0	0
MBN	0	6	3	2	0
L'OREAL	2	2	0	0	0
MALVERN	2	4	2	4	0
RAMEM	3	8	0	0	0
SUPERON	0	5	2	0	0
THERMO FISHER	1	4	1	0	0
EUROFINS	0	2	0	0	0
NIA	2	5	0	0	0
VdMi	2	0	0	0	0
Cosmetics Europe	0	1	0	0	0
LNE	1	2	0	0	0
TOTAL	**50**	**94**	**12**	**13**	**12**