Measuring means comparing!

(An attempt to screen the actual available methods and instruments)

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I. Dimension

For thousands of years men sized their surrounding by comparison to parts of their body or their environment. Until now this has been brought to perfection in a big variety of measuring units.

Today the specification of measuring units is undergoing a further precision by changing the related base to exactly determined physical effects.

With respect to particle size, only a dynamic three dimensional image analysis covering a statistically relevant number of particles could give a reliable result, but this is not yet available. If we reduce particle size that in reality would need a three dimensional description plus shape information, to only a length or a diameter this will generally be based on the meter.

The meter is the length of the path travelled by light in vacuum during a time interval of $^{1}/_{299\,792\,458}$ of a second.
[17th General Conference on Weights and Measures. (1983). Resolution 1. International Bureau of Weights and Measures]

II. Measurement is comparison

Physical quantity = measure * measurement unit
G = {G} * [G]

Example:
Body length: l = 1.74 m: The unit for length [m] is 1.74 times contained in the body length.
Car inertia (weight): m = 885 kg: The unit for inertia [kg] is 885 times contained in the car inertia.

Basic parameters of SI

Parameter	***Symbol***	***Measurement-unit***	***Abbreviation***
Length	l	Meter	m
Time	t	Second	s
Inertia	m	Kilogram	kg
Current	I	Ampere	A
Temperature	T	Kelvin	K
Luminous intensity	I	Candela	cd
Amount of substance	n	Mole	mol

Prefixes

Prefix	***Symbol***	***Power***	***Prefix***	***Symbol***	***Power***
Tera	T	10^{12}	Centi	c	10^{-2}
Giga	G	10^{9}	Milli	m	10^{-3}
Mega	M	10^{6}	Micro	µ	10^{-6}
Kilo	k	10^{3}	Nano	n	10^{-9}
Hecto	h	10^{2}	Pico	p	10^{-12}
Deca	da	10	Femto	f	10^{-15}
Deci	d	10^{-1}	Atto	a	10^{-18}

III. Sizing methods for particle sizes below 1 millimeter

A. Human resources (fingertips, teeth/tongue, eye/lid)

Due to sensitivity it is possible to determine particle sizes between fingertips down to about 100µm, by teeth/tongue down to about 50µm and by eye/lid down to 5µm.

PROs: Instantly available at no cost.

CONs: Accuracy is based on experience and training.

B. Sieves

Dry, wet and jet sieving will measure down to about minimum 10µm.

PROs: Cheap and easy to use.

CONs: In case of non-spherical particles the result will be depending on the sieving duration.

C. Oversized grain control

A scraper generates a thin layer with decreasing thickness and the first particle lager than the actual gap width causes a trace on the layer. Minimum down to 1µm

PROs: Cheap and easy to use.

CONs: Mainly indicating the maximum particle size of oversized particles and its frequency.

D. Sedimentation / centrifugation / fractionation

The method has limitations on both ends and it is used for suspensions only. Very coarse particles will settle instantly with high speed and tend to drag down smaller particles with them in their vortex trail. This is why there is an upper limit at a few hundred µm. The lower limit is due to temperature and dispersion stability at about 0.1 µm. Flow fractionation respectively hydrodynamic fractionation as well as centrifugation may be looked at as a special case of "sedimentation".

PROs: Well established in geological analysis.

CONs: Because of very slow settling of the fines very time consuming.

Flake shaped particles are "parachuting" and by that tend to pretend smaller sizes.

E. Image analysis / microscopy

Sharp pictures of particles can only be taken within a given depth of sharpness defined by the optical setup. The higher the amplification (the smaller the particles) gets, the smaller the depth of sharpness is getting and raises limits to dynamic analysis. Dynamic image analysis with respect to diameter results ends at about 1 µm while static image analysis with restricted statistics can do down to 0.5 µm.

PROs: Very accurate size and shape information.

It is applicable for dry and wet dispersion.

CONs: Reasonable numbers of particles can only be measured using dynamic image analysis.

Static Image analysis forces particles into a preferential direction, only the largest area will be seen.

F. Light blockage / time of flight / time of sight

Depending on the detector devices particles passing through a detection area raise a signal for a certain time that due to the particle velocity represents a certain particle size. Passing different than one by one or out of focus causes errors and correction based on signal shape detection is doubtable. The measuring range is depending on the wavelength of the light source. The instruments work down to a some tenth of a µm. (minimum about 25 nm)

PROs: In case of focused single passing only, every particle is measured very exactly.

CONs: One by one measurement takes very long and makes great demands to dispersion stability. Statistical relevance is very weak because of low total numbers. Multi passing and non-focused correction by signal shape analysis is doubtable.

G. Influencing the electrical field in an orifice

Depending on the width of an orifice, particles passing through raise a signal corresponding to the particle volume and surface loading. Passing different from one by one may block the orifice. Correction for multiple passing based on signal shape detection is doubtable. The measuring range is highly dependent on the orifice size and has a rather small dynamic range because comparatively small particles result in only minimal signals. Further for such small particles there is no sufficient homogeneity of the electrical field in a rather wide orifice and passing close to the wall will differ from passing through the middle. Modern instruments work down to about 50 nm.

PROs: In case of single passing only, every particle is measured very exactly.

CONs: One by one measurement takes very long and makes great demands to dispersion stability. Statistical relevance is very weak because of low total numbers. Multi passing correction by signal shape analysis is doubtable. Particles need to be dispersed in a conductive liquid.

H. Diffraction pattern evaluation (static light scattering)

This method is known best as laser diffraction (LD). The basics are very straight forward. Every particle diffracts light under an angle that is due to its size. Detecting the angle, under which diffracted light occurs, means measuring the corresponding particle size. The more angles are separately detected, the higher the accuracy of the result might be, if all detector elements are located on a single detector. The use of multiple detector devices needs to overcome offsets between those and decreases accuracy while increasing steady alignment control necessity. The measuring range of traditional LD applying Fraunhofer-theory ends at about 0.5 µm. Extended LD applying MIE-theory claims to measure down to 10 nm although there is no way to determine correct MIE-parameters for e.g. material mixtures.

PROs: Very fast measurements especially with dry dispersion.
It is applicable for dry and wet dispersion with flexible dispersing units.

CONs: Numerous detector units lower the possible accuracy due to offsets and possible misalignment.
Using a convergent beam setup increases focal errors.
Extending the measuring range into the MIE-area raises parameter problems.

I. Ultrasonic extinction

Depending on the size particles interact with a field of plain ultrasonic waves. Only particles larger than the wavelength cause extinction, smaller ones are just entrained. The impact is related to the elasticity of the particles. For very flexible ones e.g. droplets resonance as a special effect may show up. Scanning a sequence of wavelengths enables size distribution analysis.

PROs: The measurement can be applied for highest concentration down to less than 100 nm.

CONs: As the elasticity (based on a variety of parameters) of the particles is determining the impact, this method is mainly used in-line in processes calibrated to the elasticity. Benefit for lab use is limited as for every sample lots of parameters or calibration is needed.

J. Scattered light evaluation (dynamic light scattering)

When the particle size gets much smaller than the wavelength of the illuminating light source, only scattered light will still be emitted. The relation of the scattered light to the particle size is a non-linear increase of the intensity of the scattered light with increasing particle size. It cannot be used for size determination directly. But as particles in a suspension are subject to Brownian motion the scattered light intensity fluctuation in a focus area corresponds to the travelling speed of the particles initiated by the Brownian motion and this is directly related to particle size. Detection of the travelling speed measures the size.

PROs: The most reliable measurement in the nano-area (definition 1nm to 100 nm).

CONs: Very narrow measuring areas bastardize the Brownian-motion due to non-elastic pulsing at the walls. Short measuring times as well as selection of useful detection-time-slices or movement-traces lead to non-representative selected results. Simple evaluation leads to mono size forced results. Sophisticated methods need to be checked for mathematical artifact presentation by correlation control.

K. Cantilever scanning topography

All types of scanning electron microscopy use cantilever tips to gain a picture of the topography of a material surface. This topographical structure reveals information about particles on/in the surface and their size. They can measure down into the picometer area.

PROs: These instruments have the highest capacity to show details.

CONs: Their field of view is very limited and the number of particles measured cannot properly characterize a material with respect to statistical relevance.
Further most of these measurements need to be performed under ultrahigh vacuum.

IV. Available instruments (known to SICO)

Type	Subtype	Manufacturer	Instrument	Webpage
B sieves		Fritsch Retsch Haver&Boecker ITECA SOCADEI SAS	Analysette3 / 18 AS200 / 300 / 400 Part'Sizer®	www.fritsch.de www.retsch.com www.retsch.com www.haver-partikelanalyse.com www.iteca.fr
C Oversized grain control	Grindometer	Eirichsen Zehntner Elcometer OMEC	Modell 232 ZGR2020-2024 Elcometer Grind Gauge	www.eirichsen.de www.zehntner.com www.elcometer.com www.beatop.com
D sedimentation	x-ray/light controlled	Miromeritics Topas	SediGraph 5120 USS791 / USS790	www.micromeritics.de www.topas-gmbh.de
	centrifuge	CPS Instruments Brookhaven Bettersize Instr.	CPS DC24000 UHR BI-DCP / BI-XDC BT1500	www.cpsinstruments.eu www.brookhaveninstruments.com www.bettersize.com
	fractination	ActiPix	TDA2000	www.paraytec.com
E image analysis	static	Malvern ParticleMetrix PSS Nicomp	Morphologi G3 Anatec SIA Alpaga 500 Nano	www.malvern.com www.particle-metrix.de www.pssnicomp.com
	dynamic	Sympatec Retsch Technology Malvern AnaTec MettlerToledo Haver&Boecker Bettersize Instr. OMEC	Qicpic Camsizer FPIA3000 Partan PVM Haver CPA BT1800 PIP 8.1 PIP 9.1	www.sympatec.com www.retsch-technology.de www.malvern.com www.ana-tec.com www.mt.com/lasentec www.haver-partikelanalyse.com www.bettersize.com www.beatop.com
F light blockage		Topas MettlerToledo Sequip Niwa PMT / PMS PSS Nicomp TSI Parsum	FAS362 / LAP322 FBRM MTS-PAT sensors Galai-CIS-100 LASAIR® II 110 Accusizer Aerotrac IPP70	www.topas-gmbh.de www.mt.com/lasentec www.sequip.de www.niwa.co.nz www.pmt-ag.com / www.pmeasuring.com www.pssnicomp.com www.tsiinc.de www.parsum.de
G e-field change		BeckmanCoulter Micromeritics Izon OMEC	Multisizer Elzone II qNano RC2100 RC3000	www.beckmancoulter.com www.micromeritics.de www.izon.com www.beatop.com
H static light scattering (diffraction)	convergent beam LD	Horiba Malvern Fritsch Cilas BeckmanCoulter Micromeritics ParticleMetrix Bettersize Instr. Jinan Winner OMEC	LA950 / LA300 Mastersizer Analysette22 990 / 1090 / 1190 LS13320 / LS200 Saturn DigiSizer Microtrac BT9300 BT2000-2003 Winner2000 - 3008 LS-*** Easysizer	www.retsch-technology.de www.malvern.com www.fritsch.de www.cilas.com www.beckmancoulter.com www.micromeritics.de www.particle-metrix.de www.bettersize.com Jinan Winner Particle instruments Stock Co. www.beatop.com
	parallel beam LD	Sympatec Malvern XoptiX Outotec	Helos Spraysizer XO01-XO03 L & P PSI300 PSI500	www.sympatec.com www.malvern.com www.xoptix.co.uk www.outotec.com

Type	Subtype	Manufacturer	Instrument	Webpage
I ultrasonic extinction		Sympatec Dispersion Technology Colloidal Dynamics	Opus DT1201 AcoustoSizer II	www.sympatec.com www.dispersion.com www.colloidal-dynamics.com
J dynamic light scattering	PCS	Horiba Malvern Fritsch/Cordouan BeckmanCoulter/ Otzuka Brookhaven ParticleMetrix PSS Nicomp Wyatt Technology Bettersize Instr. Jinan Winner OMEC	SZ-100 Zetasizer Nano Analysette12 / VASCO Delsa-Nano / ELSZ2 90 Plus BI-200SM Nanotrac Nicomp 380 DynaPro BT90 Winner801 Dylisizer 2	www.retsch-technology.de www.malvern.com www.fritsch.de / www.cordouan-tech.com www.beckmancoulter.com www.photal.co.jp/english www.brookhaveninstruments.com www.particle-metrix.de www.pssnicomp.com www.wyatt.eu www.bettersize.com Jinan Winner Particle instruments Stock Co. www.beatop.com
	PCCS	Sympatec	Nanophox	www.sympatec.com
	NTA	NanoSight	LM10 / LM20	www.nanosight.com
K electron microscopy	AFM	Atomic force mic.	see the list	list of AFM manufacturers
	STM	Scanning tunnel mic.		

V. Wrap up

This report is meant as a rather rough guide through the wide field of particle sizing only. To all types of particle sizing instruments you will find a lot of detailed information in the internet. It is a pity that the habit of manufacturers to publish detailed information on the technology of their instruments today has been replaced by blatant statements like “world best” etc. only. I don’t consider it as my life’s work to fill this gap, but I try at least to hand on my experience.