# Should laser diffraction become the new standard for soil particle size analysis ?

Webinar: Determination of the clay content in soils | 25 June 2024


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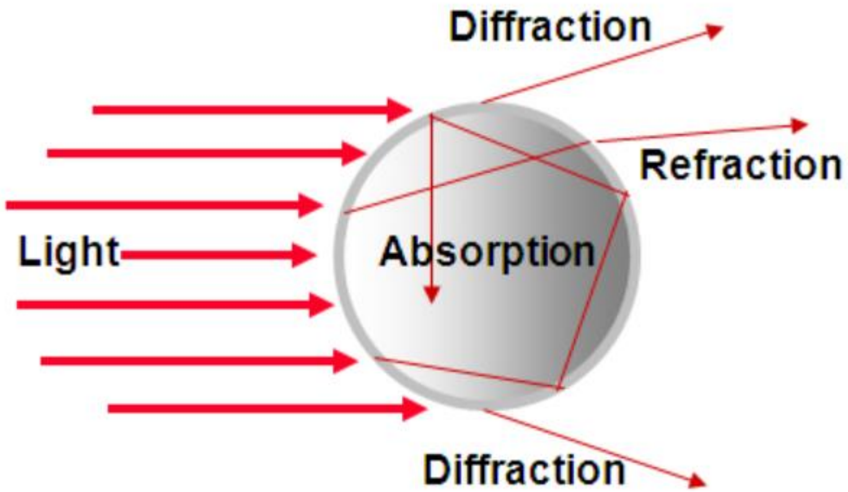
GLOBAL SOIL PARTNERSHIP

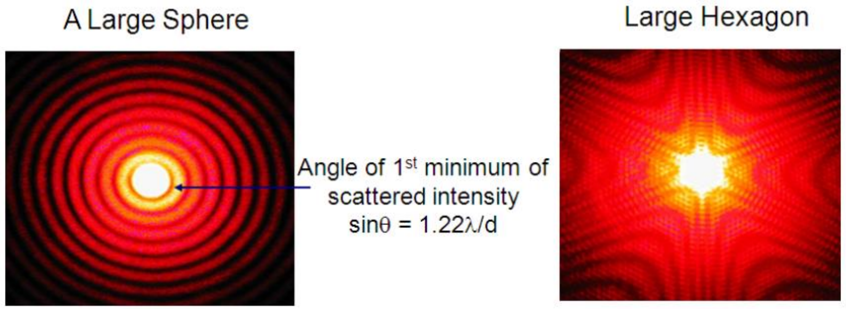
# Outline

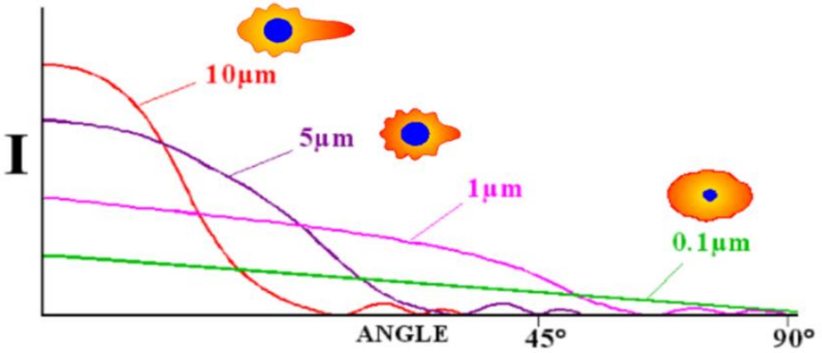
- Laser diffraction (LD) technique
- Procedure and accuracy of LD based clay determination
- Relevant International standards
- Any literature on LD of soil samples ?
- Comparison LD with pipette/sieve method
- Transfer functions and equivalent clay fraction limits
- Take-home messages

# Laser diffraction (LD) technique

VIS

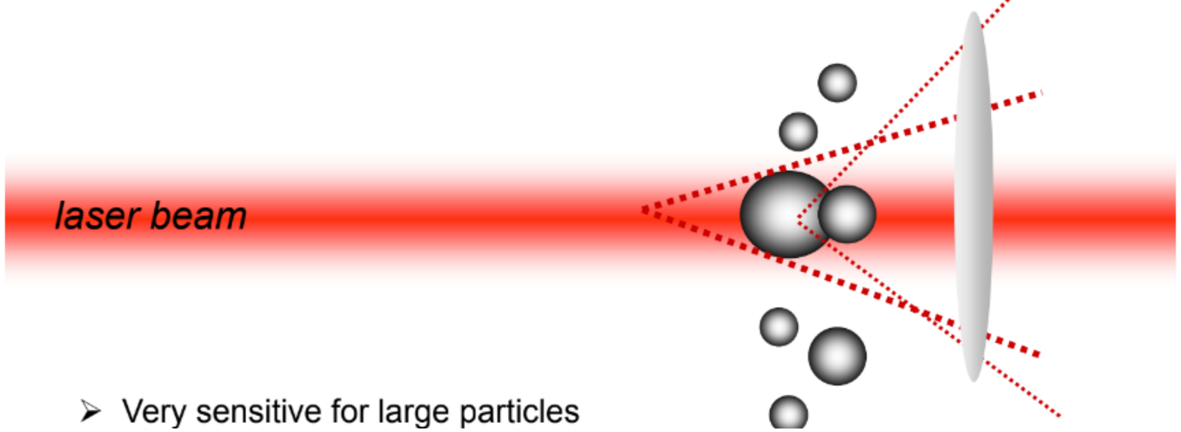



## Laser Diffraction

- ✓ Small particles scatter light at *wide* angles, with low intensity
- ✓ Large particles scatter light at *narrow* angles, with high intensity




- ➢ Very sensitive for large particles
  - ✓ When particles get too small.. (<1/10 wavelength)
  - ✓ No difference between angles, no difference in size

Source: Sysmex 2012

- the **scattered intensity** is a function of **scattering angle, particle shape, and particle size**
- sum of **spherical particles matches the measured scattering pattern**

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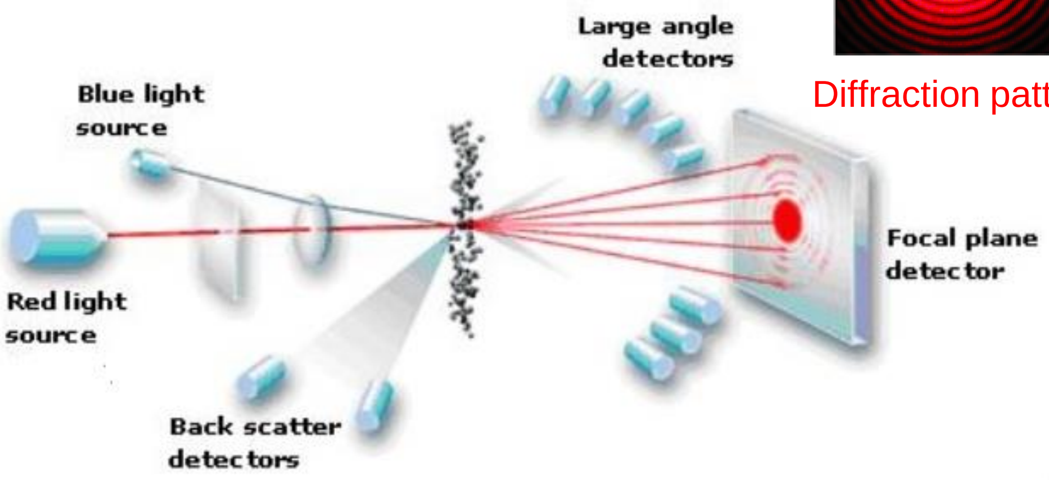
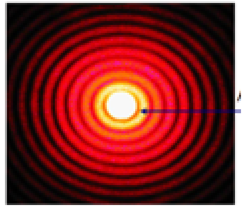
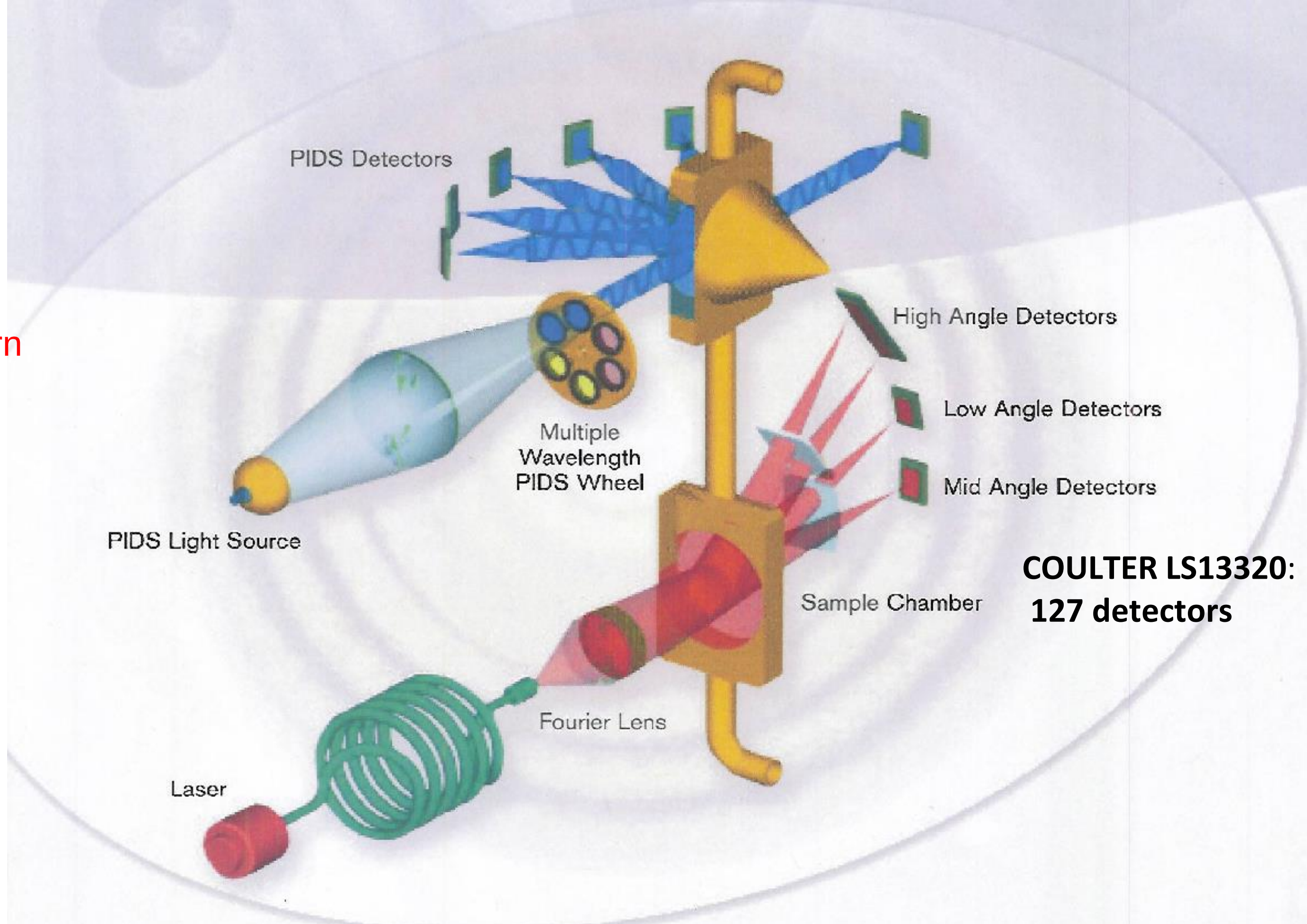
# Laser diffraction (LD) technique



Diffraction pattern




**COULTER LS13320**: **127 detectors**

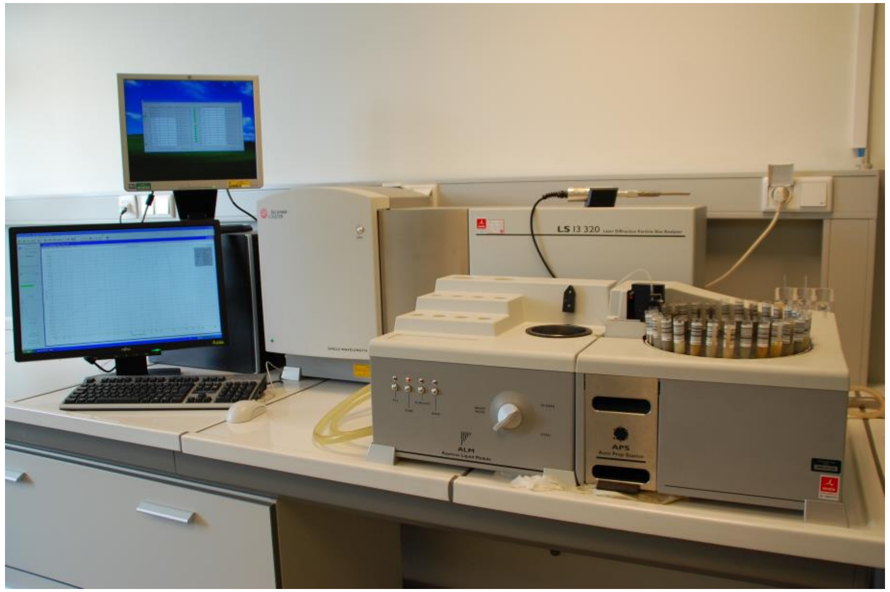
Assumption: **spherical particles**. Most particles in soils do not fulfill the assumption of sphericity
=> Leads to **broadening of the particle size distribution**, and usually **higher mean particle diameters**

**Diffraction theories:**

- **Fraunhofer model:** assuming complete diffraction (no absorption)
- **Mie model:** requires homogenous refractive index (RI) and absorption coefficient (AC) (different for each soil mineral)

# Our Laser diffraction equipment

COULTER LS 200



Beckman COULTER LS13320

Next ?
**New one in 2024**

Operational: **1998 - 2012 (2013)**
PSD range: **0.4 - 2000 µm**
Detectors: **126 / 92** size channels
**No autosampler**
Analysis time: **19-90 sec**
Cost: ~ **51 Keuro** (incl. VAT)

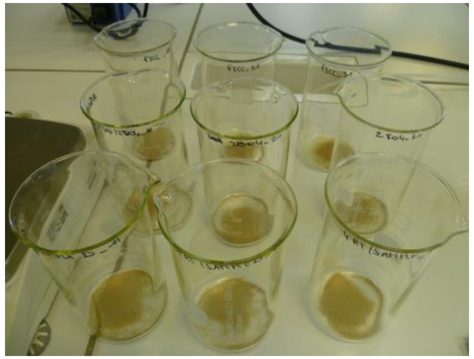
Operational: **2013 – 2024**
**Equipped with liquid module and autosampler 30 positions**
PSD range: **0.4 - 2000 µm**
Detectors: **127 / 92** size channels (without PID)
Forward scattering, no backscatter
Volume liquid module **1.25 l** / Automatic dillution system
Cost: ~ **72 Keuro** + **15 Keuro** autosampler (incl. VAT)

# Determination of clay and other particle sizes

**Pretreatment is laborious !!**

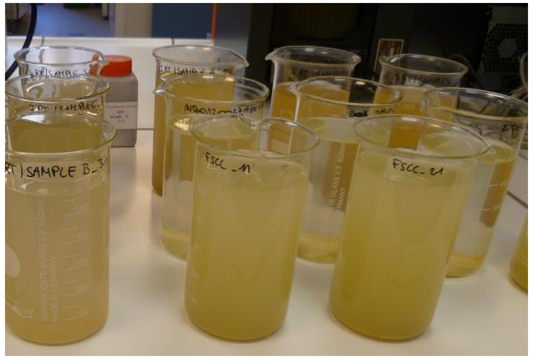
**Similar pretreatment as pipette.**

**INBO Procedure SAP-200B**



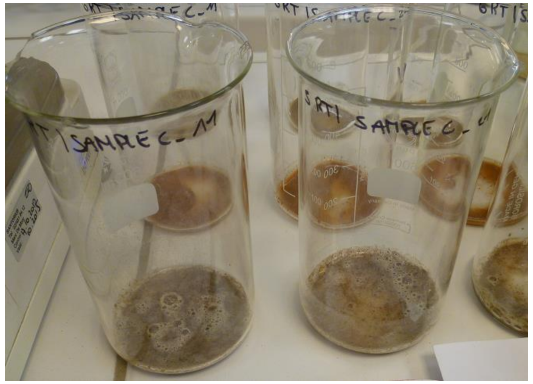
**Soil sample (< 2 mm)**
Dried at **40°C**
Sample mass: **1 - 5 g**
*Silt/clay => 1 g*
*Sand => 5g*
*OM => + 1g*

**Removal of organic matter 28,5% $H_2O_2$ @ 40°C until no reaction + Carbonate removal with 10% HCl**

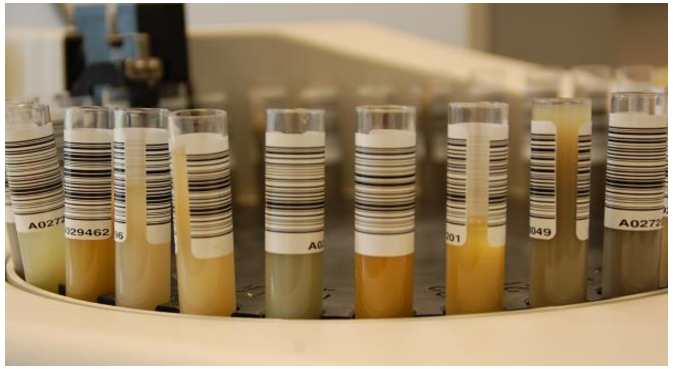


**Add demiwater + overnight**

**Removal of supernatant by jetpump**



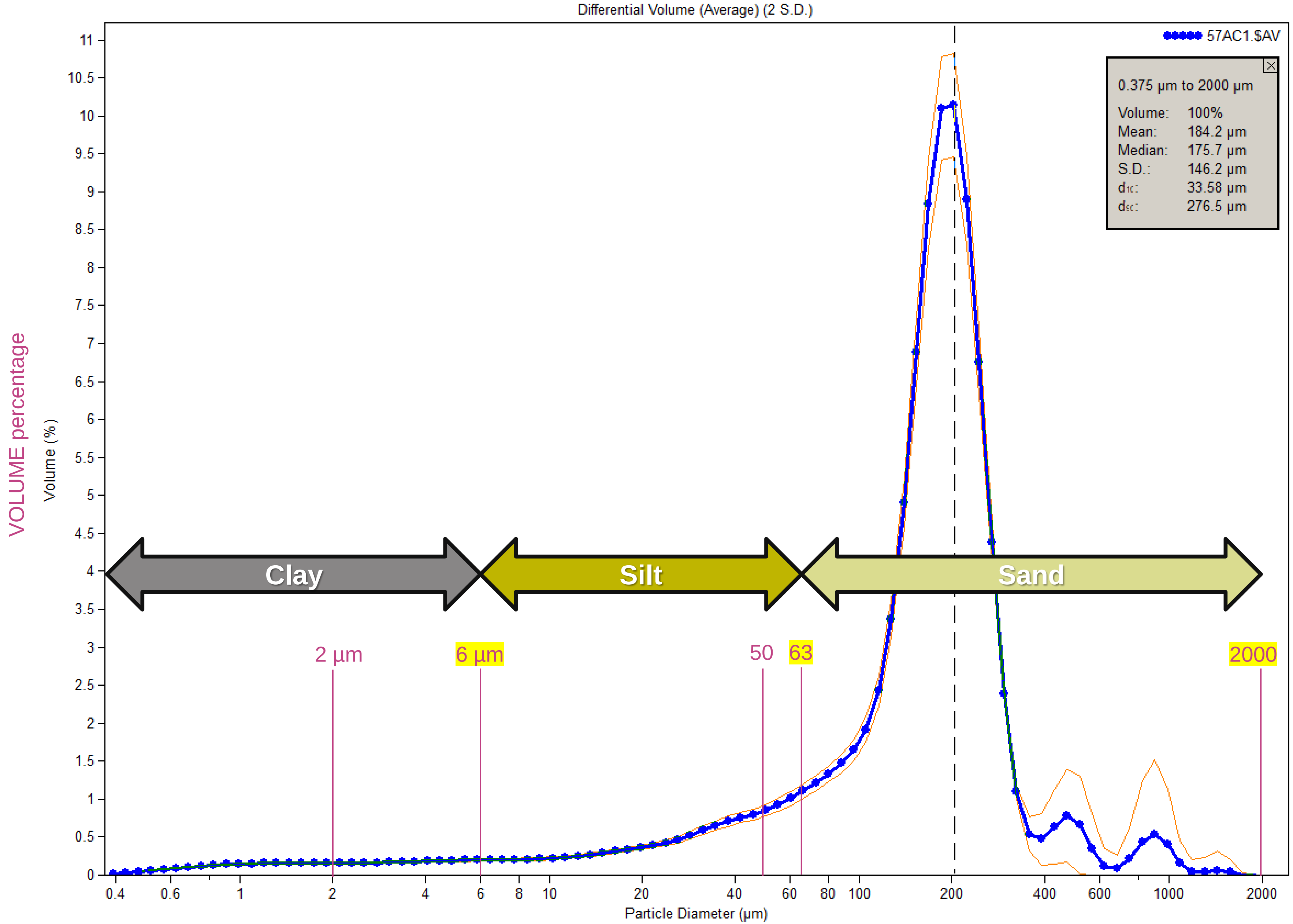
**Dispersion step with 6% $Na_4P_2O_7$*10H2O**



**Volume reduction to 10 ml for autosampler**

**Laserdiffractometry @ recommended obscuration (10-15%)**

# Typical graphical output: texture fingerprint




Detector intensity value

2 SD of 5 replicate measurements

Clay-Silt-Sand fractions
2 – 50 – 2000 µm standard limits
6 – 63 – 2000 µm equivalent limits

| Fraction | Pipette limits (µm) | Eq. LD limits (µm) | % |
|---|---|---|---|
| Clay | 0-2 | < 6 | 4.1 |
| Silt | 2-50 | 6-63 | 11.7 |
| Sand | 50-2000 | 63-2000 | 84.2 |

**Texture class:**
Belgian class: Z – Zand
USDA class: LS: loamy Sand

# Typical numerical output (CSV list : fingerprint)

| LabSampleID | FieldSampleID | lower_boundary | upper_boundary | fraction | sd |
|---|---|---|---|---|---|
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,38 | 0,41 | 0,016 | 0,001 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,41 | 0,45 | 0,031 | 0,001 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,45 | 0,5 | 0,052 | 0,002 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,5 | 0,54 | 0,07 | 0,003 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,54 | 0,6 | 0,087 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,6 | 0,66 | 0,102 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,66 | 0,72 | 0,114 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,72 | 0,79 | 0,123 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,79 | 0,87 | 0,13 | 0,003 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,87 | 0,95 | 0,134 | 0,002 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 0,95 | 1,05 | 0,135 | 0,001 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 1,05 | 1,15 | 0,134 | 0 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 1,15 | 1,26 | 0,131 | 0,002 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 1,26 | 1,38 | 0,127 | 0,003 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 1,38 | 1,52 | 0,123 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 1,52 | 1,67 | 0,12 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 1,67 | 1,83 | 0,118 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 1,83 | 2,01 | 0,117 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 2,01 | 2,21 | 0,118 | 0,003 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 2,21 | 2,42 | 0,121 | 0,004 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 2,42 | 2,66 | 0,127 | 0,006 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 2,66 | 2,92 | 0,135 | 0,01 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 2,92 | 3,21 | 0,146 | 0,014 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 3,21 | 3,52 | 0,157 | 0,02 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 3,52 | 3,86 | 0,17 | 0,026 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 3,86 | 4,24 | 0,183 | 0,031 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 4,24 | 4,66 | 0,195 | 0,037 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 4,66 | 5,11 | 0,206 | 0,042 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 5,11 | 5,61 | 0,214 | 0,046 |
| 23-001304 | CMON_P91e31cf_laag_0_10_MG_2022 | 5,61 | 6,16 | 0,22 | 0,048 |

**30 subfractions**

**for clay**

**92 bins/ detectors**
**Size channels**

**Fraction in vol%**

**SD:** variation of 5 repetitions in each BIN

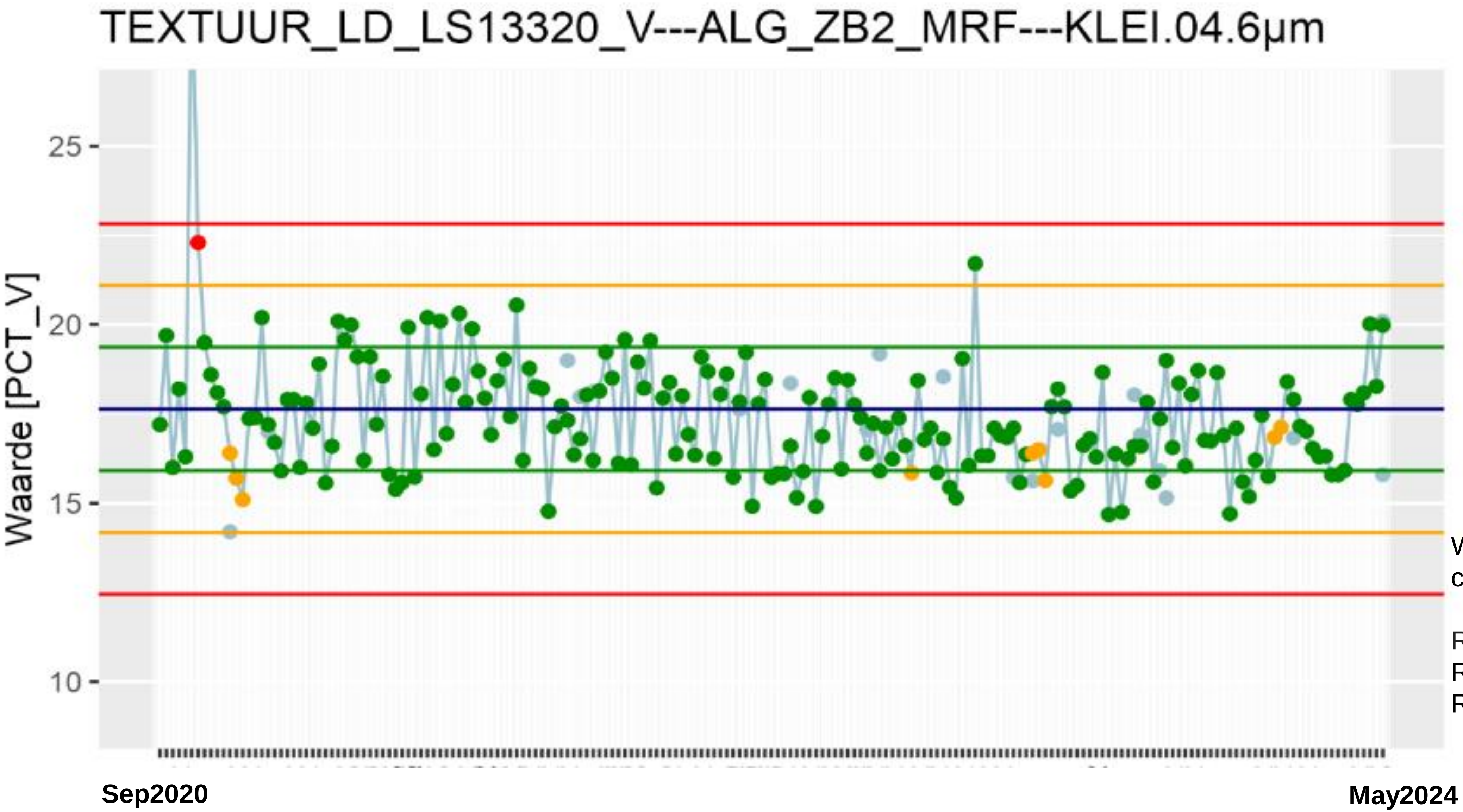
**RSD all bins:**
Mean: RSD: 23,4
Median: RSD: 4.7 %

For larger particles bin RSD's up to 250%

RSD clay fraction:
**Mean RSD: 7%**

**Excellent repeatability within subfraction bins**

# Control charts: within lab reproducibility




IRM: ZONBOD

Silt loam forest soil (Zoniën forest)

N: 193

Mean: 17.3 % Clay
SD: 1.5

RSD: 8.4%

We have 3 IRMs for control charts:

RF-ISE958: 11,2 %
RF-ZONBOD2: 17,3 %
RF-BAGBOD2: 37.8 %

# International Standardisation ?

## ISO 11464:2006

Soil quality — Pretreatment of samples for physico-chemical analysis

drying, crushing, sieving, dividing and milling

**Published** (Edition 2, 2006)

This standard was last reviewed and confirmed in 2022. Therefore this version remains current.

## ISO 11277:2020

Soil quality — Determination of particle size distribution in mineral soil material — Method by sieving and sedimentation

**Published** (Edition 3, 2020)

↳ This standard has 1 amendment.

First edition 1998-05-15

## ISO 13320:2020

Particle size analysis — Laser diffraction methods

**Published** (Edition 2, 2020)

First edition 2009-10-01

- Particles in many two-phase systems **including soil-water suspensions**
- Standard for particle sizes ranging from **0,1 µm to 3 mm**
- For **non-spherical particles** the resulting PSD **is different** from that obtained by methods based on other physical principles (e.g. **sedimentation, sieving**).

**No specific ISO standard for Soil quality !**

# Before year 2000 → 20 years later

**Little progress**

1997

Sedimentology (1997) 44, 523–535

## Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction

MARTIN KONERT and JEF VANDENBERGHE
Faculty of Earth Sciences, Vrije Universiteit, De Boelelaan 1085, 1081 HV Amsterdam, The Netherlands

2022

Contents lists available at ScienceDirect
Soil & Tillage Research
journal homepage: www.elsevier.com/locate/still
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## An investigation in laser diffraction soil particle size distribution analysis to obtain compatible results with sieve and pipette method

David Nimblad Svensson*, Ingmar Messing, Jennie Barron

1986

14 Part. Charact. 4 (1987) 14–19

## Laser Diffraction Spectrometry: Fraunhofer Diffraction Versus Mie Scattering

Gerben B. J. de Boer*, Cornelis de Weerd*, Dirk Thoenes*, Hendrik W. J. Goossens**

(Received: 21 October 1986)

2017

European Journal of Soil Science
European Journal of Soil Science, 2017 doi: 10.1111/ejss.12456

## Pedotransfer functions for converting laser diffraction particle-size data to conventional values

A. Makó[a], G. Tóth[a,b], M. Weynants[c], K. Rajkai[a], T. Hermann[b] & B. Tóth[a,b]
[a]Hungarian Academy of Sciences ISSAC-CAR, Herman Ottó út 15, Budapest, H-1022, Hungary, [b]Department of Crop Production and Soil Science, Georgikon Faculty, University of Pannonia, Deák F. u. 16, Keszthely, H-8360, Hungary, and [c]European Commission, Joint Research Centre (JRC), Via Enrico Fermi 2749, 21027, Ispra, Italy

2023

frontiers | Frontiers in Forests and Global Change
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## Soil texture analysis by laser diffraction and sedimentation and sieving–method and instrument comparison with a focus on Nordic and Baltic forest soils

Ingeborg Callesen[1*], Marjo Palviainen[2], Kęstutis Armolaitis[3], Charlotte Rasmussen[4] and O. Janne Kjønaas[5]

ELSEVIER CATENA
Catena 32 (1998) 193–208

1998

## Grain-size analysis by laser diffractometry: comparison with the sieve-pipette method

L. Beuselinck[a,*], G. Govers[a,b], J. Poesen[a,b], G. Degraer[a], L. Froyen[c]
[a] Laboratory for Experimental Geomorphology, K.U.Leuven, Redingenstraat 16, 3000 Leuven, Belgium
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[c] Department of Metallurgy and Materials Engineering, K.U.Leuven, de Croylaan 2, 3001 Heverlee, Belgium

Received 10 March 1997; revised 6 April 1998; accepted 6 April 1998

**Is the soil science community too conservative ?**

PLOS | ONE

2017

RESEARCH ARTICLE

## Adequacy of laser diffraction for soil particle size analysis

Peter Fisher*, Colin Aumann, Kohleth Chia, Nick O'Halloran, Subhash Chandra
Agriculture Research & Development Division, Department of Economic Development, Jobs, Transport & Resources, Tatura, Victoria, Australia

2022

Contents lists available at ScienceDirect
Geoderma
journal homepage: www.elsevier.com/locate/geoderma
ELSEVIER

## Experimental evidence of laser diffraction accuracy for particle size analysis

Marco Bittelli[a,*], Sergio Pellegrini[b], Roberto Olmi[c], Maria Costanza Andrenelli[b], Gianluca Simonetti[d], Emilio Borrelli[a], Francesco Morari[d]

# Laser diffraction for soil particle size analysis

## Strengths

- Fast measurement (< 1min)
- Generate the entire particle size distribution (PSD)
- Order of magnitude more precise than conventional PSD methods
- Allows various sample pretreatment methods
- Can be automated (autosampler)
- Allows post-processing for factors such as diffraction models & optical parameters

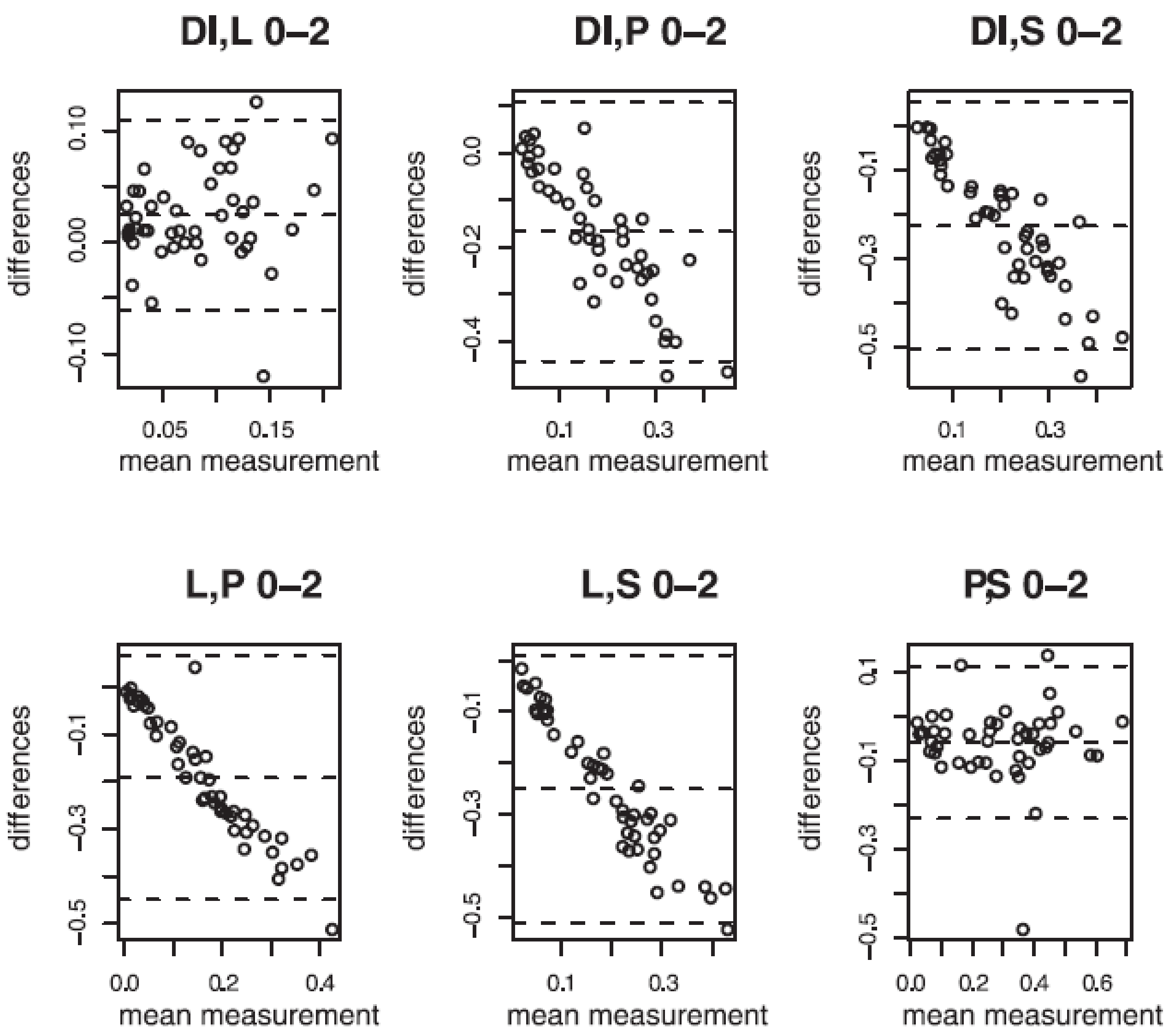
## Weaknesses

- Requires consistent & standardized sample preparation
- Amount of sample may be limited (< 1g) compared to > 10g sedimentation
- Sample introduction is critical for new LD equipment: taking a representative subsample from pretreated sample (best is complete sample in > 1 L liquid module)
- Controlling pump speed for circulating sample
- Correct way of dilution to lower the obscuration within recommended range
- Which scatter model to use for soils: Fraunhofer or Mie (which Refractive index)

# LD compared with pipette/sieve method

| Property | Conventional Pipette/Sieve | Laser diffraction |
|---|---|---|
| **Cost equipment** | Low | High |
| **Labour intensiveness**<br>Sample preparation<br>Analytical phase | <br>High<br>High | <br>High<br>Low |
| **Throughput time** | 6 samples/day (manual Pipette) | 60 samples/day |
| **Output particle sizes** | Discrete fractions | Almost continuous (PSD fingerprint) |
| **Standard measurement unit** | Mass% | Vol% |
| **Autosampling** | Possible, but difficult | Yes |
| **Operator error** | high | Low |
| **Measurement grain sizes** | 2 runs (sedimentation, sieving) | 1 run (uniform physical method) |
| **Definition of grainsize** | 2 definitions | one definition |
| **Accuracy**<br>Repeatability<br>Reproducibility | <br>Moderate<br>Low | <br>High<br>High |
| Analysis cost/sample (INBO) | 81 € (7€ without personnel costs) | 27 € (12€ without personnel costs) |

# LD compared with other methods




**Bland-Altman graphs**
comparing **0-2 µ Clay fraction** determined by:

**DI:** Digital Imaging
**L:** Laser diffraction
**P:** Pipette (sedimentation)
**S:** Sieving/Sedigraph

**Pipette is the standard method but is it the golden standard ?**

**Digital Imaging ≈ Laser Diffraction**

Bittelli et al. 2022 (Geoderma)

# Transfer functions based on regression



**Table 2**
Linear regression relationship between sieve and pipette method (SPM) and laser diffraction method (LDM).

| Class (μm) | Equation | $R^2$ | P-value | Equation with intercept locked to zero | $R^2$ | P-value |
|---|---|---|---|---|---|---|
| Clay[a] < 2 | LDM= 0.714SPM-2.163 | 0.917 | < 0.001 | LDM= 0.662SPM | 0.897 | < 0.001 |
| Fine silt[a] 2–6 | LDM= 1.222SPM+ 2.721 | 0.874 | < 0.001 | LDM= 1.400SPM | 0.882 | < 0.001 |
| Medium silt[a] 6–20 | LDM= 1.133SPM+ 3.180 | 0.934 | < 0.001 | LDM= 1.280SPM | 0.93 | < 0.001 |
| Coarse silt[a] 20–63 | LDM= 0.833SPM+ 1.786 | 0.888 | < 0.001 | LDM= 0.919SPM | 0.894 | < 0.001 |
| Silt[a] 2–63 | LDM= 1.083SPM+ 6.758 | 0.939 | < 0.001 | LDM= 1.217SPM | 0.936 | < 0.001 |
| Sand[b] 63–2000 | LDM= 0.976SPM+ 0.746 | 0.999 | < 0.001 | LDM= 0.987SPM | 0.998 | < 0.001 |

[a] pipette (SPM) and laser diffraction (LDM) analyses (LDM fraction limits based on nearest bin).
[b] sieving for both SPM and LDM analyses.

Svensson et al. 2022

Conversion from mass to volume percent is incorporated in transfer function.

# Equivalent clay fraction limits for textural classification

LD 0-8 µm

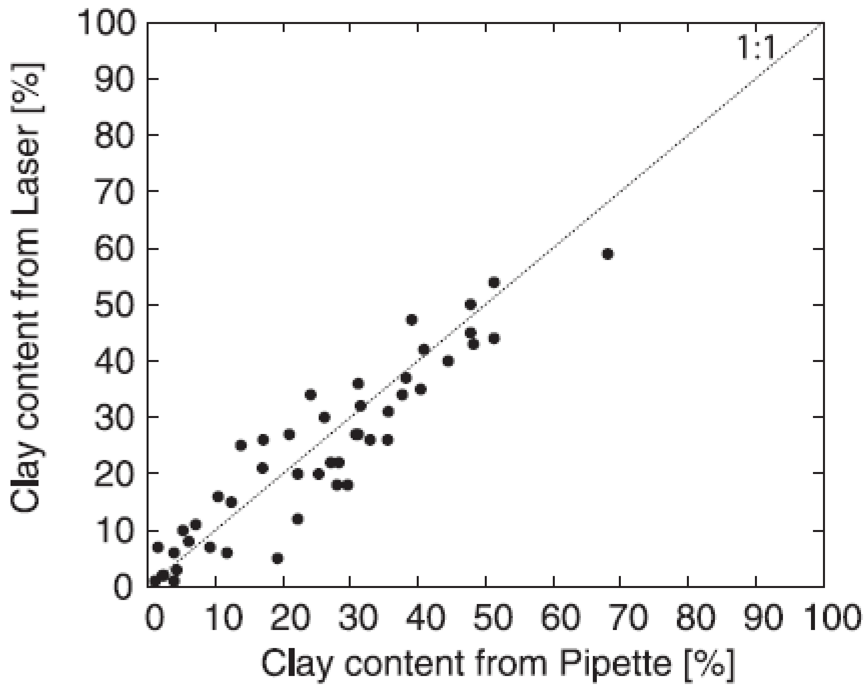



Fig. 10. Linear regression between clay percentage from P and from L, with clay size limits for L in the range 0–8 µm.

Bittelli et al. (2022)

**Using: Malvern Mastersizer 2000, Mie model**

LD 0-6 µm

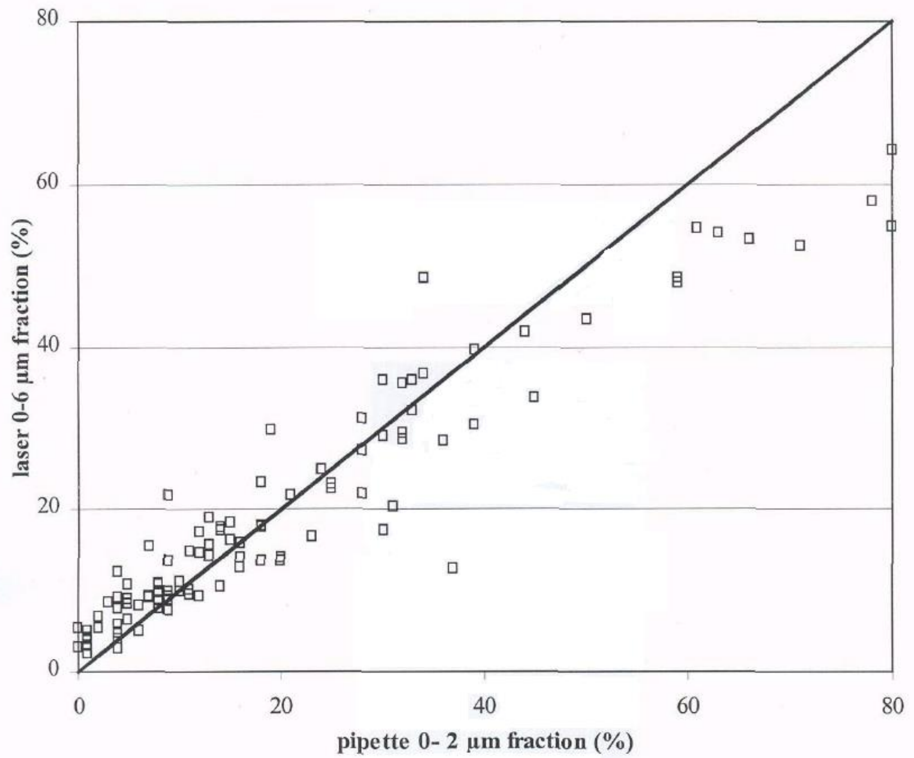



**Figure** 7 Scatterplot of the pipette 0-2 µm fraction and the laser 0-6 µm fraction. The 1:1 ratio line is also shown.

Vandecasteele & De Vos (2001)

**Using: Coulter LS13320, Fraunhofer model**

Other clay equivalent diameters found in literature: **4 µm** (Thomas et al., 2021), **4.6 µm** (Antoine et al. 2009), **5 µm** (Qiu et al., 2021), **7 µm** (Mako et al., 2019), **9 µm** (Fisher et al. 2017): so **requires lab and device specific determination**

# Take home messages

- Laser diffraction (LD) **looks differently to soil particle sizes** than conventional methods (Pipette, Sieving, Sedigraph) due to the underlying physical laws and assumptions

- LD provides a total **fingerprint of the soil particle size up of the fine earth (< 2 mm)**, allowing (re)calculation of various fractions. Therefore, we recommend to **store the complete fingerprint in soil databases,** also for **post-processing**.

- The **equivalent diameter** for the upper limit of the clay fraction (**2 µm**) shifts to a LD diameter between **6-9 µm,** depending on sample pre-treatment, device, diffraction model and working conditions

- Soil textural analysis by LD is in the end **cheaper, faster and more accurate** than the sedimentation-sieving combination method

- LD is well suited for **high-throughput** soil textural analysis, especially for (large) **soil surveys**

- **Increasing evidence,** based on digital imaging techniques, shows that **clay and silt fractions** measured by LD **better approximates the "real" particle sizes** than the conventional pipette/sedimentation method

- Hence, the conventional standard for soil particle size analysis should be changed from sedimentation to Laser Diffraction methodologies. There is a **need** for **a specific Soil Quality ISO** for Particle Size Distribution **of mineral soils by Laser Diffraction**, building further on the general ISO 13320 for laser diffractometry.

# Thank you for your attention

If you want to reach out: Bruno.devos@inbo.be

# Recent literature on LD analysis of soil PSD

## **advantages** and **disadvantages**

| Reference | Title |
|---|---|
| Konert & Vandenberghe 1997 | **Comparison** of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction |
| Fisher et al. 2017 | **Adequacy** of laser diffraction for soil particle size analysis |
| Makó et al. 2017 | **Pedotransfer functions for converting** laser diffraction particle-size data to conventional values |
| Bittelli et al. 2022 | **Experimental evidence** of laser diffraction accuracy for particle size analysis |
| Svensson et al. 2022 | An investigation in laser diffraction soil particle size distribution analysis to obtain **compatible results** with sieve and pipette method |
| Callesen et al. 2023 | Soil texture analysis by laser diffraction and sedimentation and sieving–method and instrument **comparison** with a focus on Nordic and Baltic forest soils |