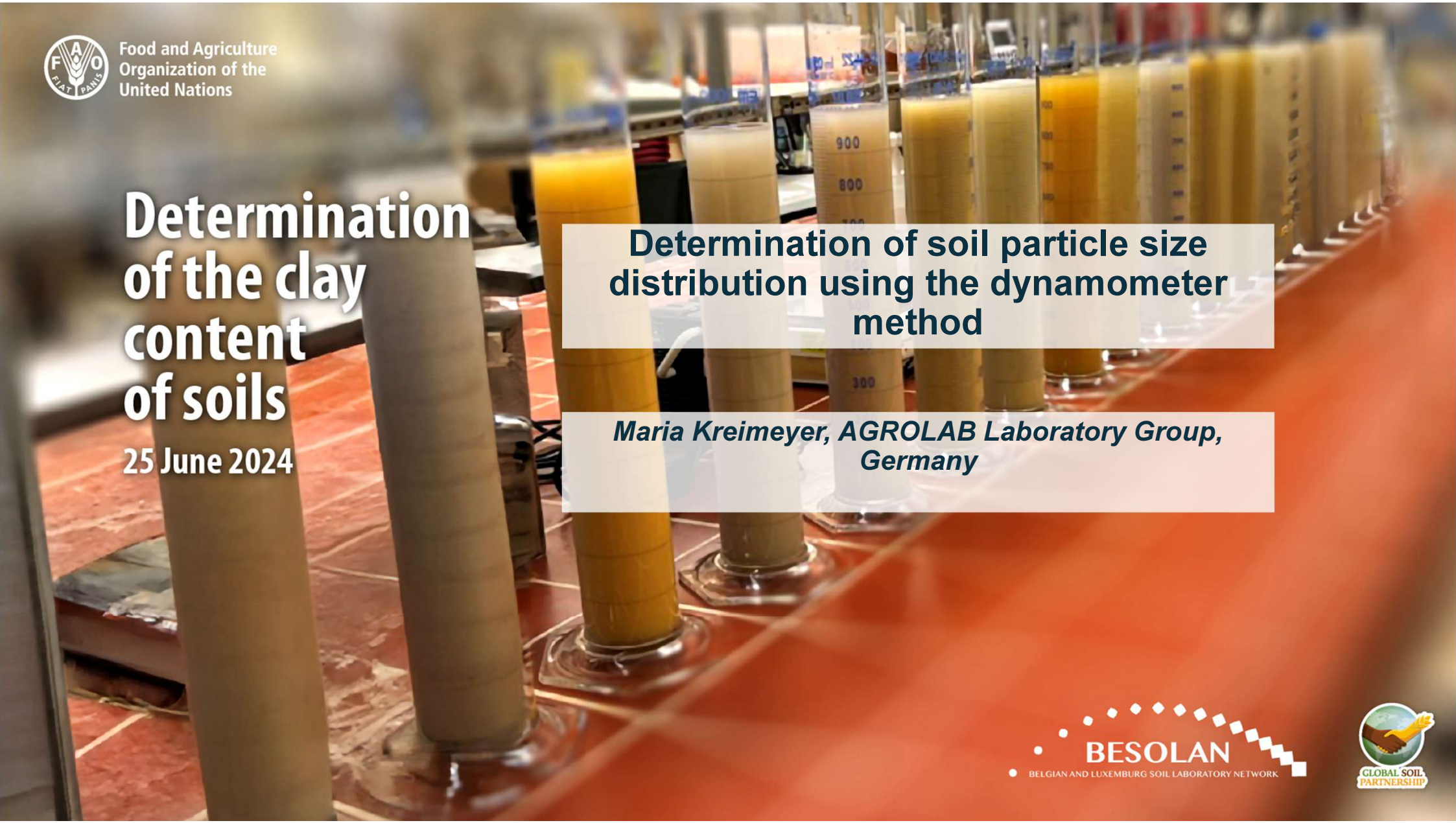
Food and Agriculture Organization of the United Nations
Determination of the clay content of soils
25 June 2024
Determination of soil particle size distribution using the dynamometer method
Maria Kreimeyer, AGROLAB Laboratory Group, Germany
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# Introduction & Motivation

- The pipette method (ISO 11277) , accepted as the official reference method, is accurate but time-consuming and laborious
- Long processing times lead to delays and high costs
- **Aim:** Develop a method that maintains accuracy while reducing processing time and costs
- Methods considered: hydrometer measurement, laser diffraction and aerometer measurement

# History

- **Before 2014:** Grain size distribution in sediments from the tailings dam ”Żelazny Most” (the largest post-flotation waste reservoir in Europe)
- **2014:** Development of algorithm and the dynamometer method (Wroclaw University, Arcanum)
- **2015:** First device (Prototype)
- **2015 – 2016 :** Test and scientific evaluation


# History

- **2017:** Presentation of the method by Wrocław University and Arcanum to AGROLAB
- **2017 - 2018:** Development of automatic system for many samples
- **2018 - 2021:** Method optimizations and validation (AGROLAB/Wroclaw University)
- **From 2021 :** Routine measurements (> 30.000 samples)

# Innovative Cooperation between Science and Industry


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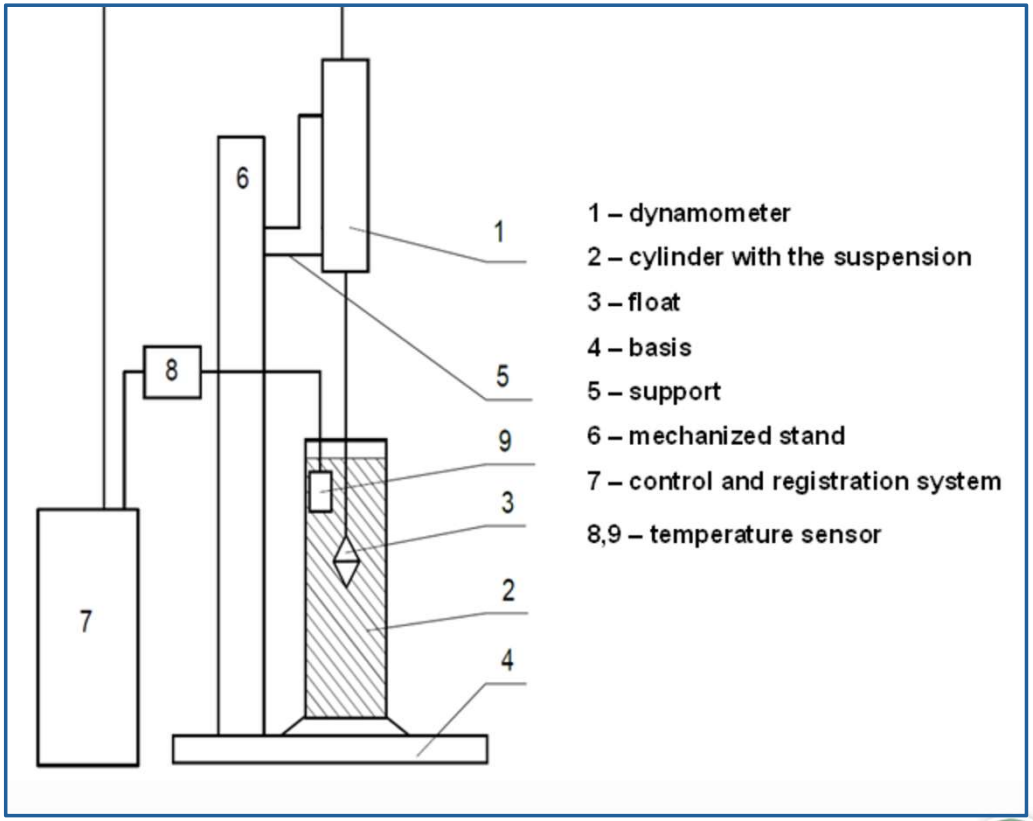
# Principle of the dynamometer method

- Density of suspension determination based on apparent weight changes of a float submerged in it
- Measure weight of float with sensitive piezoelectric dynamometer to calculate soil fraction content
- Calculation of content of soil fractions with equivalent diameters in the range of 0.002 to 0.1 with the Stoke's equation
- Digital transmission of results enables automatic calculation of particle size composition

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# Determination with dynamometer method

Sample

pre-treatment

60 g (40g)

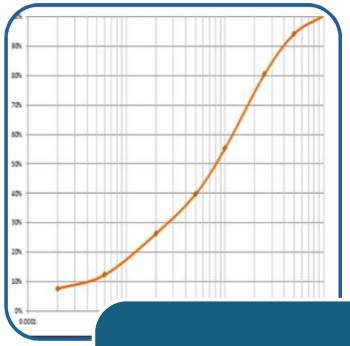
1 L $H_2O$

$N_4P_2O_7$ * 10 $H_2O$

shaking or mixing

Measurement:

depth, T, time, changes of the weight of the float

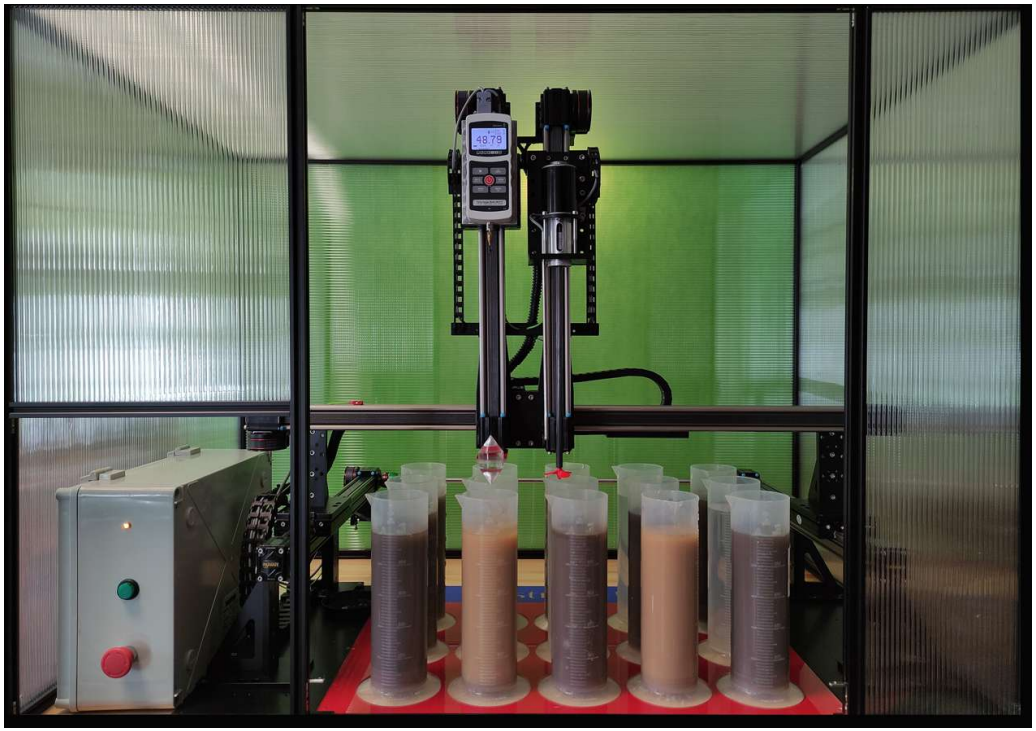


Algorithm:

results [%] of the multiple fractions

# Describtion of the Dynamometer Method

- Range 0,002 -0,1 mm
- With or without OM removement
- Free choice of sample pre treatment
- Short measurement time (ex: 14 samples, 3 fractions, 5:30 – 6:30 h)
- Measurement of many fractions in one run
- Fully automated measurement
- Flexible choice of fractions
- Graphical presentation of results possible
- A direct record of the results in digital form




Astra 2.1

# Validation

Comparison with existing methods

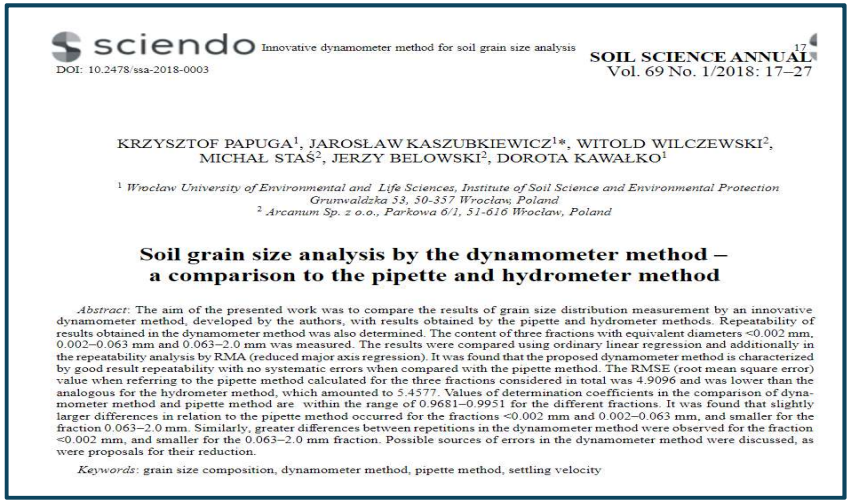
Proficiency testing,
Accreditation ISO/IEC 17025

Reproducibilty, MU, LOQ …

Scientific publications

# Validation

## Comparison with the pipette method



sciendo Innovative dynamometer method for soil grain size analysis

DOI: 10.2478/ssa-2018-0003

SOIL SCIENCE ANNUAL 17
Vol. 69 No. 1/2018: 17–27

KRZYSZTOF PAPUGA[1], JAROSŁAW KASZUBKIEWICZ[1]*, WITOLD WILCZEWSKI[2], MICHAŁ STAŚ[2], JERZY BELOWSKI[2], DOROTA KAWAŁKO[1]

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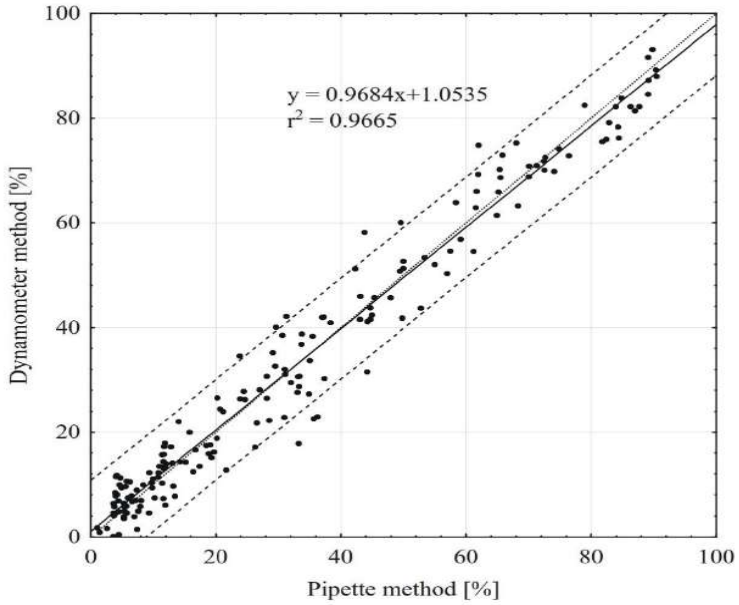
**Soil grain size analysis by the dynamometer method – a comparison to the pipette and hydrometer method**

*Abstract*: The aim of the presented work was to compare the results of grain size distribution measurement by an innovative dynamometer method, developed by the authors, with results obtained by the pipette and hydrometer methods. Repeatability of results obtained in the dynamometer method was also determined. The content of three fractions with equivalent diameters <0.002 mm, 0.002–0.063 mm and 0.063–2.0 mm was measured. The results were compared using ordinary linear regression and additionally in the repeatability analysis by RMA (reduced major axis regression). It was found that the proposed dynamometer method is characterized by good result repeatability with no systematic errors when compared with the pipette method. The RMSE (root mean square error) value when referring to the pipette method calculated for the three fractions considered in total was 4.9096 and was lower than the analogous for the hydrometer method, which amounted to 5.4577. Values of determination coefficients in the comparison of dynamometer method and pipette method are within the range of 0.9681–0.9951 for the different fractions. It was found that slightly larger differences in relation to the pipette method occurred for the fractions <0.002 mm and 0.002–0.063 mm, and smaller for the fraction 0.063–2.0 mm. Similarly, greater differences between repetitions in the dynamometer method were observed for the fraction <0.002 mm, and smaller for the 0.063–2.0 mm fraction. Possible sources of errors in the dynamometer method were discussed, as were proposals for their reduction.

*Keywords*: grain size composition, dynamometer method, pipette method, settling velocity




| Fraction mm | No. of samples | Regression equation | Correlation coefficient | Std. error of slope coefficient | Root mean square error | Std. error of regression | Critical value* |
|---|---|---|---|---|---|---|---|
| 0.063–2.0 | 59 | y=0.9625x+0.2739 | 0.9951 | 0.0127 | 3.7275 | 3.2931 | -0.2846 |
| 0.002–0.063 | 59 | y=0.9992x–0.5335 | 0.9681 | 0.0343 | 5.4107 | 5.4750 | 0.5340 |
| <0.002 | 59 | y=1.0217x–1.4148 | 0.9699 | 0.0340 | 5.3985 | 5.1183 | -1.3848 |
| All fractions | 177 | y=0.9684x+1.0535 | 0.9831 | 0.0136 | 4.9096 | 4.8634 | -1.0879 |

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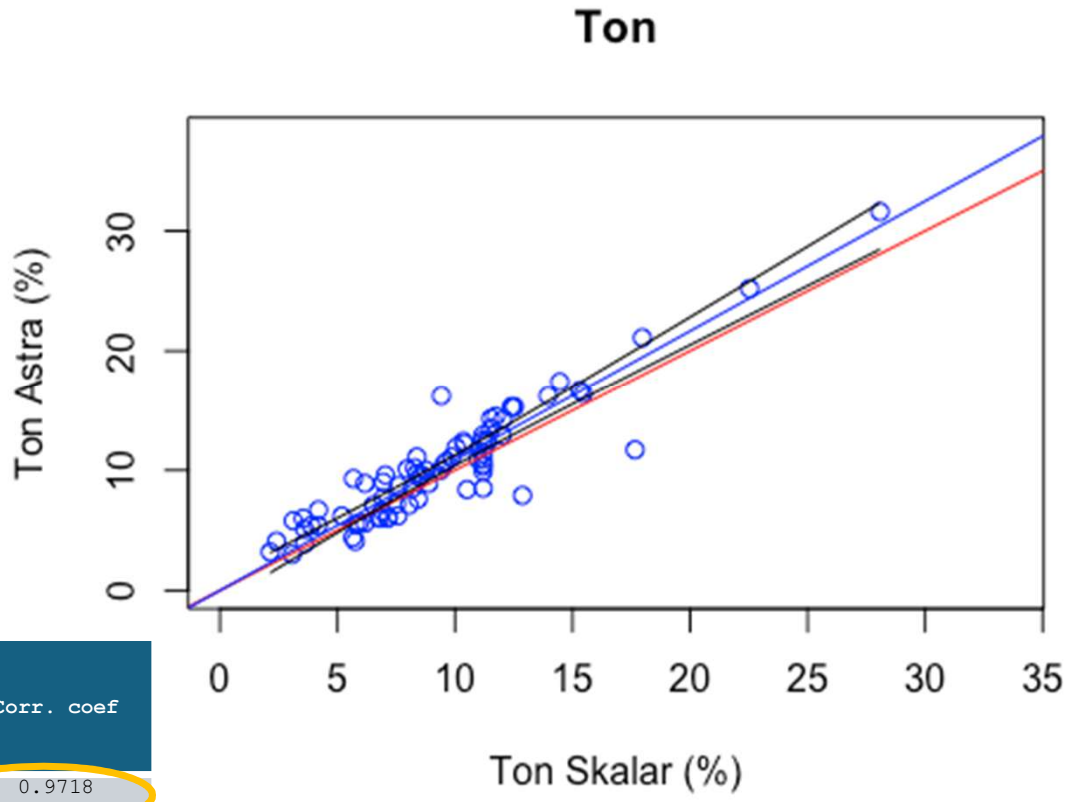
# Validation

With AGROLAB samples

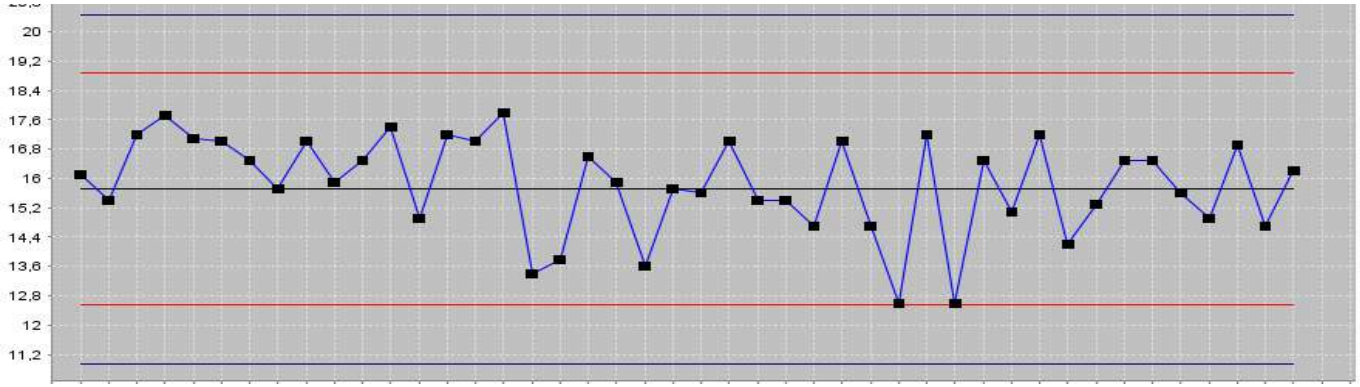
AGROLAB / Wrocław University (2021)

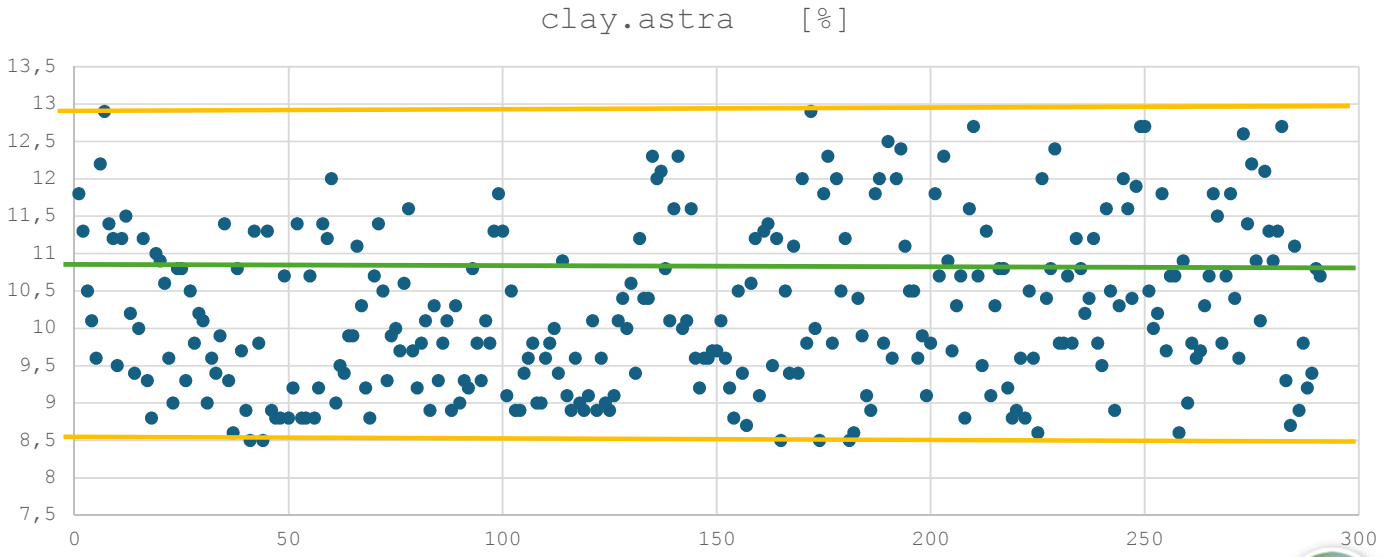



| Parameter | a-coefficient | Standard error | Number of samples | Corr. coef |
|---|---|---|---|---|
| < 0,002 | 1.08260 | 0.02102 | 77 | 0.9718 |
| 0,002 – 0,063 | 0.95607 | 0.02771 | 77 | 0.9393 |

# Validation

- Proficiency testing : BIPEA
- Measurement of PT samples
- Accreditation ISO/IEC 17025 (DakkS)
- Quality Control Charts

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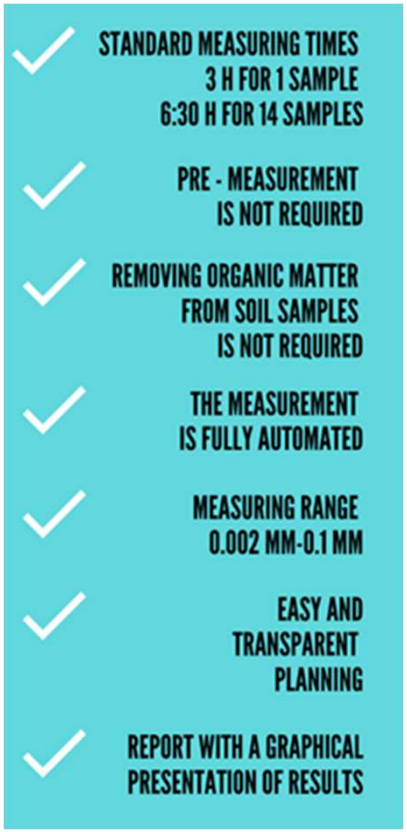
# Validation

- **Reproducibility 11 %**
- **Measurement uncertainty 24 %**
- **LOQ 0,5%**

| | clay.astra | clay.astra. |
|---|---|---|
| | tobi | qcb |
| average | 10,2 | 15,3 |
| std deviation | 1,1 | 1,5 |
| rsd% | 10,9 | 10,0 |
| n | 296,0 | 1274,0 |
| expanded uncertainty on mean | 0,13 | 0,1 |

# Main Advantages / Conclusion

- The consistency of the results with the reference method
- No systematic differences (in relation to the reference method)
- Very good validation data
- A direct record of the results in digital form
- The capacity to analyse multiple fractions with arbitrarily chosen ranges of diameters
- Simple and user friendly  automatic process
- Reduction of workload
- The reduction of the analysis time in relation to other sedimentation methods
- Cheaper than laser diffraction methods
- Great alternative for the pipette method



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Food and Agriculture Organization of the United Nations

# Thank you

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