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# Determination of the clay content of soils

25 June 2024

Standard methodology for  
the determination of the  
clay content of soils  
(Brussels, Flemish and Walloon Region  
and Luxemburg)

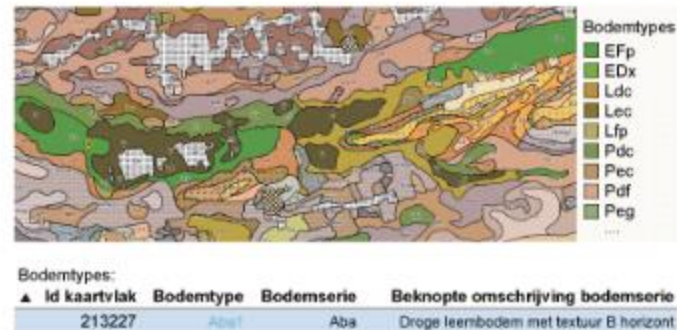
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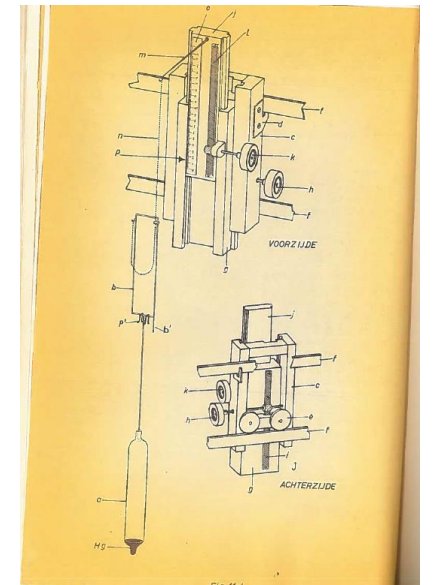
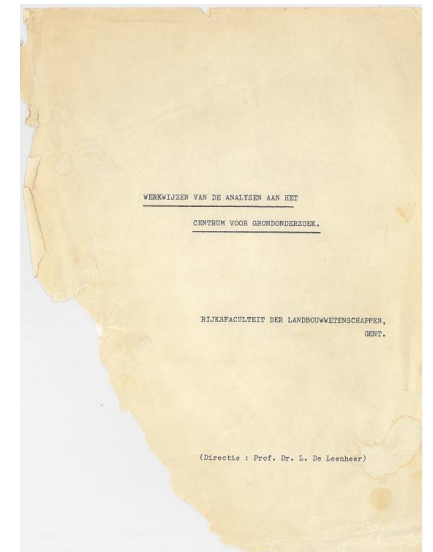
# Background

- The Belgian soil map was drawn up based on the results of intensive soil mapping during the 1950s to the 1970s. The soil profile was studied down to a depth of 1.25 m.
- In that time a lot of attention was paid to the optimization and development of the analytical method.

Bodemkaart (1:20.000) van België

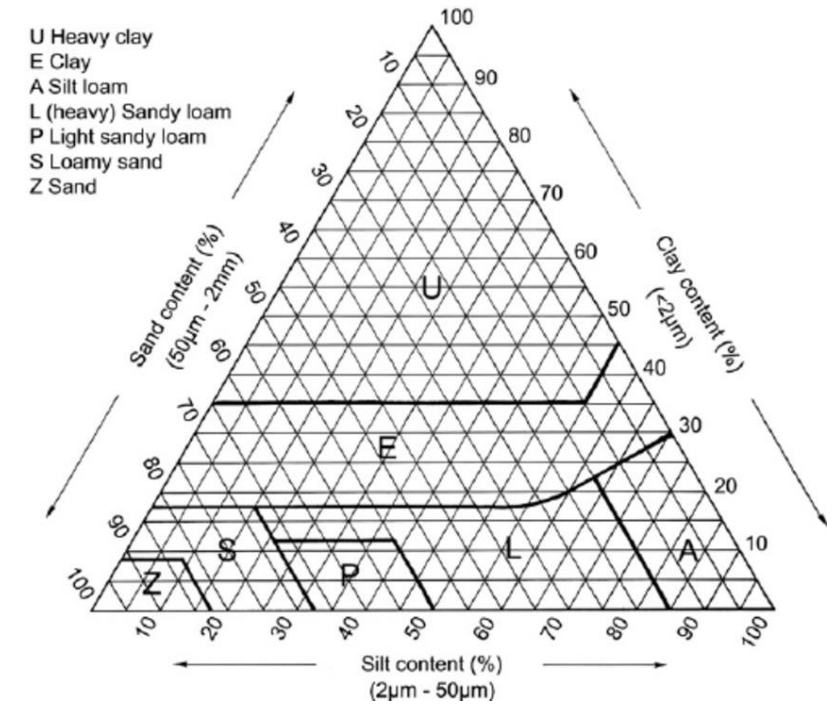


Source: [Bodemkaarten | DOV \(vlaanderen.be\)](https://www.dov.vlaanderen.be/bodemkaarten)



# Background

- The soil triangle is used to classify soils based on their relative proportions of sand, silt, and clay whereby
  - Sand: fraction between 50  $\mu\text{m}$  and 2 mm
  - Silt: fraction between 2  $\mu\text{m}$  and 50  $\mu\text{m}$
  - Clay: fraction less than 2  $\mu\text{m}$
- Depending on the country, sometimes 63  $\mu\text{m}$  instead of 50  $\mu\text{m}$  is used as the limit to separate the silt fraction from the sand fraction
- Flanders (B): % of clay is also used for recalculation of the soil remediation limits for some heavy metals



# Introduction

- Determination of the clay content of the soil from the Belgian regions and Luxemburg is done according to the ISO 11277 method
- ISO 11277:2020 Soil quality – Determination of particle size distribution in mineral soil material – Method by sieving and sedimentation
- Different steps in the procedure are presented, highlighting the critical points and the variability within the procedure

# Principle of the method

- Soil sample, dried at 40°C and grinded to < 2 mm
- After removal of the organic fraction, the carbonates and soluble salts, the mineral fraction is left over.
- The mineral fractions is separated into different particle classes by **wet sieving and sedimentation**, followed by pipetting:
  - Addition of a peptizing agent to disperse the particles as much as possible.
  - Separation of sand fraction by wet sieving through a 50 µm sieve.
  - The suspension is transferred into a sedimentation column to separate the clay and silt fraction by sedimentation according to **Stokes' law** (the larger a particle is, the faster it moves in water under the action of gravity).
  - At constant temperature, pipetting is to be made twice at certain times and at a fixed depth (10 cm).The first pipetted volume contains the **clay and silt fraction**; the second only the **clay fraction**.

# General procedure

- Sample preparation
- Destruction of organic matter
- Removal of carbonates
- Removal of soluble salts
- Dispersion
- Wet sieving at 50  $\mu\text{m}$  (separation of sand fraction)
- Sedimentation (pipetting of the silt and clay fraction)
- Calculation of results for fraction  $< 2 \text{ mm}$

# Sample preparation

- Soil sample dried at 40°C
- Sieved < 2 mm, stamp gently



# Intake of the dried sample

- ISO 11277: 30 g for a sandy soil and 10 g for a clayey soil
- Intake varies between 10 – 20 g, laboratory depending
- For clayey soils: reduced intake of 7,5 g

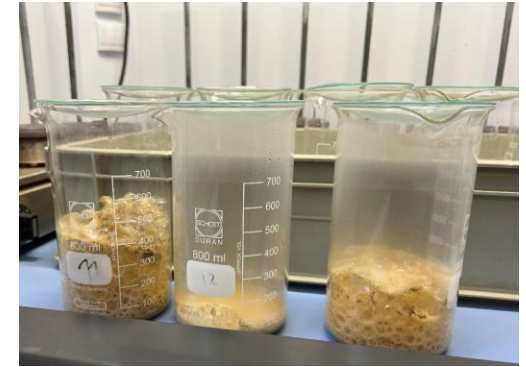




# Destruction of organic matter – method 1

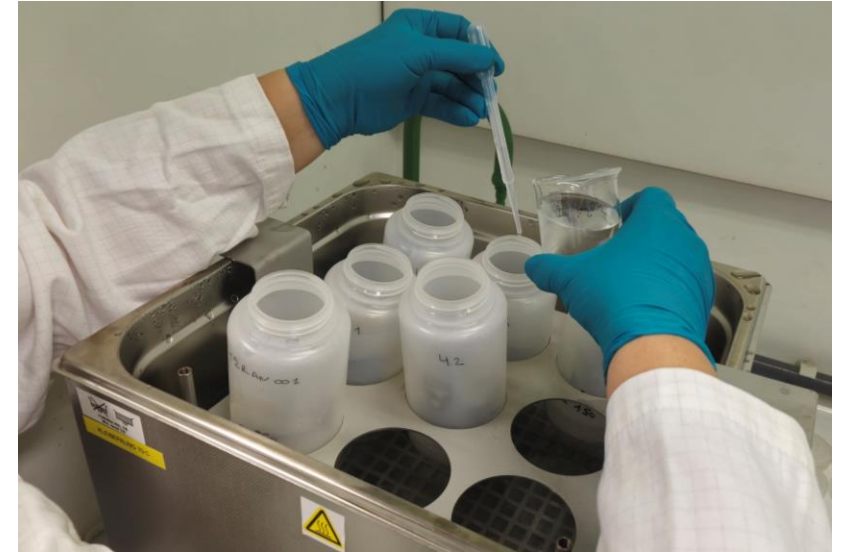


- Add 100 ml H<sub>2</sub>O<sub>2</sub> in 25 ml-steps to avoid the formation of too much foam!
- Cover the recipient with a watch glass
- Leave to settle until the next morning at room temperature
- Gradually increase the temperature to ±70°C in a laboratory hood.
- After 25-35 minutes, the temperature is set to the maximum position for approximately 15-20 minutes
- The volume of the suspension in the beakers should always be kept at around 150 ml by adding demineralized water!



# Destruction of organic matter – method 2

- Add 75 mL H<sub>2</sub>O<sub>2</sub> in small portions
- Additions spread over 3 hours
- Apply **ultrasonic power** to the bath for 1 min at (70°± 5) °C every time after addition of H<sub>2</sub>O<sub>2</sub>

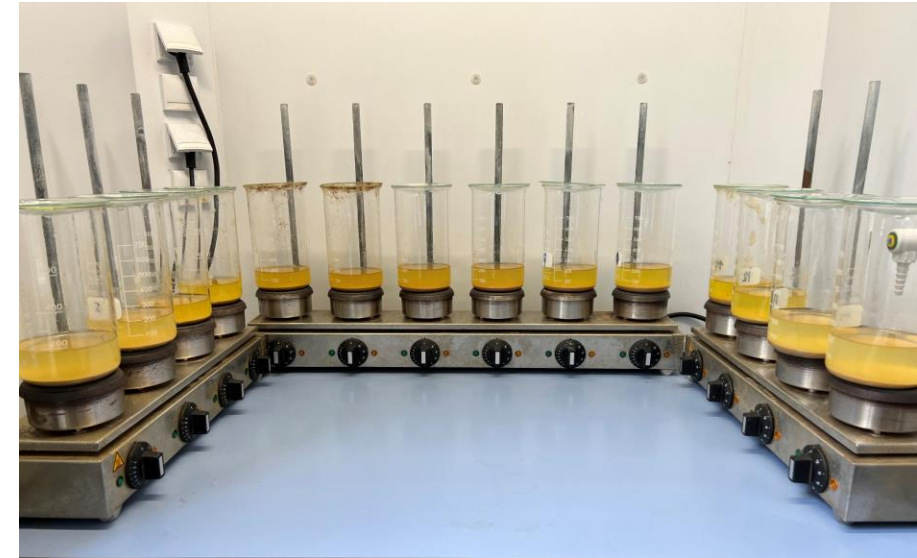


## *ISO 11277 Annex D – use of ultrasonic bath*

*Systems operating with an ultrasonic bath may be used, provided that comparability to the procedure described above without the use of ultrasonic power is proven (see Annex D). Comparability has to be proven for all soils to be analysed in relation to their Reference Soil Groups according to WRB (2015), as soils are from many different climate zones and parent materials which influences the comparability of particle size analyses depending on pre-treatment.*

# Removal of carbonates – method 1

- Add 10 ml of HCl 1M with a pipette to destroy carbonates
- Let it react for 1 hour at room temperature
- Gradually increase the temperature to  $\pm 70^{\circ}\text{C}$  in a laboratory hood to destroy the dolomites!
- Let the solution boil for at least 10 min
- Let the solution cool down



# Removal of carbonates – method 2

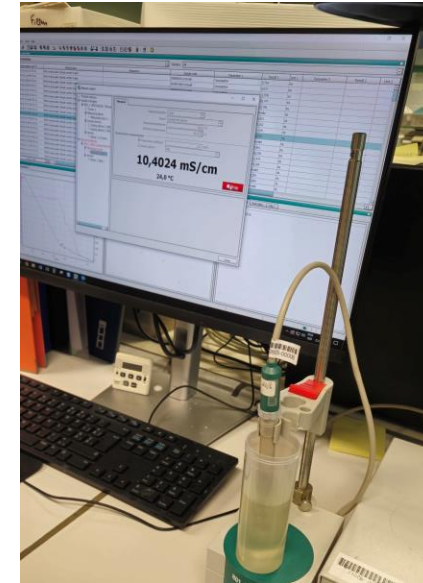
Add a few drops of HCl to check presence of carbonates

- Sparkling reaction → carbonates are present
  - Addition of 50 mL 1N HCl, **ultrasonic power** for 1 min, and 2 hrs in a heating bath ( $70^{\circ} \pm 5$ ) °C
  - After 30 min, the pH is measured with a pH strip
  - At pH < 3: the removal of the carbonate was successful
  - At pH > 3: carbonate is assumed to be present in significant amounts
  - Extra HCl is added, repeat steps
- No sparkling reaction → no carbonates
  - Addition of 45 mL water and 5 mL 1N HCl (pH < 3)



# Removal of soluble salts

- The soluble salts are leached and decanted several times after centrifugation.
- This operation is repeated until the electrical conductivity of the supernatant is  $< 40 \text{ mS/m}$



# Dispersion

- After removal of clear supernatant, 25 mL dispersion agent is added
- Dilute to 150 mL and shake thoroughly.
- Apply high **ultrasonic power** in the water bath for 10 min. at  $(70 \pm 5) ^\circ\text{C}$ .



# Separation of the sand fraction

- Wet sieving at 50  $\mu\text{m}$
- Transfer the dispersed suspension from the centrifuge bottle quantitatively onto the sieve, and wash the soil using a jet of water and gently rub with the fingertips
- Wash the sieve's content sufficiently with water until the jet of water is clear
- The total volume of the washings should not exceed 1000 ml



# Separation of sand fraction

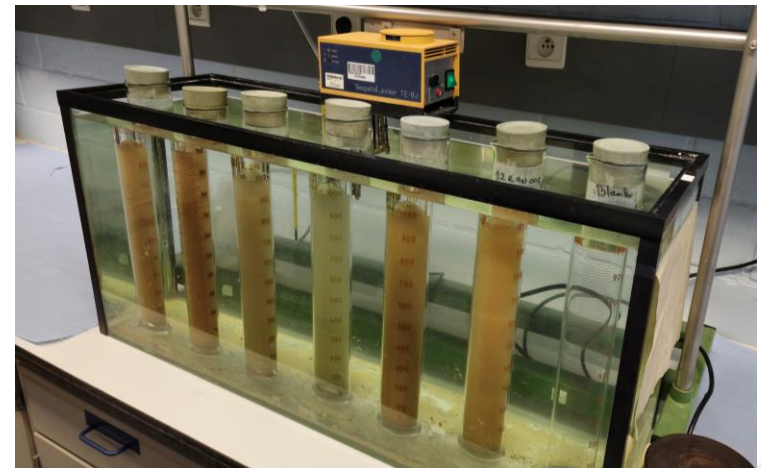
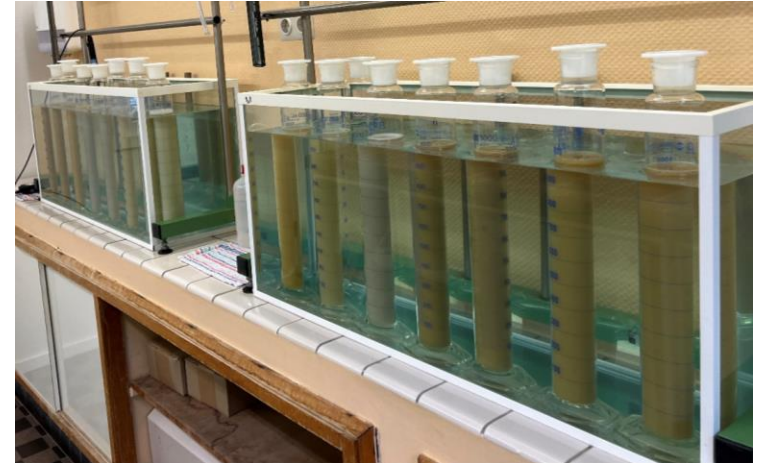
- The sand fraction is quantitatively transferred into a weighing vessel by a gentle spray from the wash-bottle.
- The weighing vessel is dried at 105 °C until a constant weight is reached.





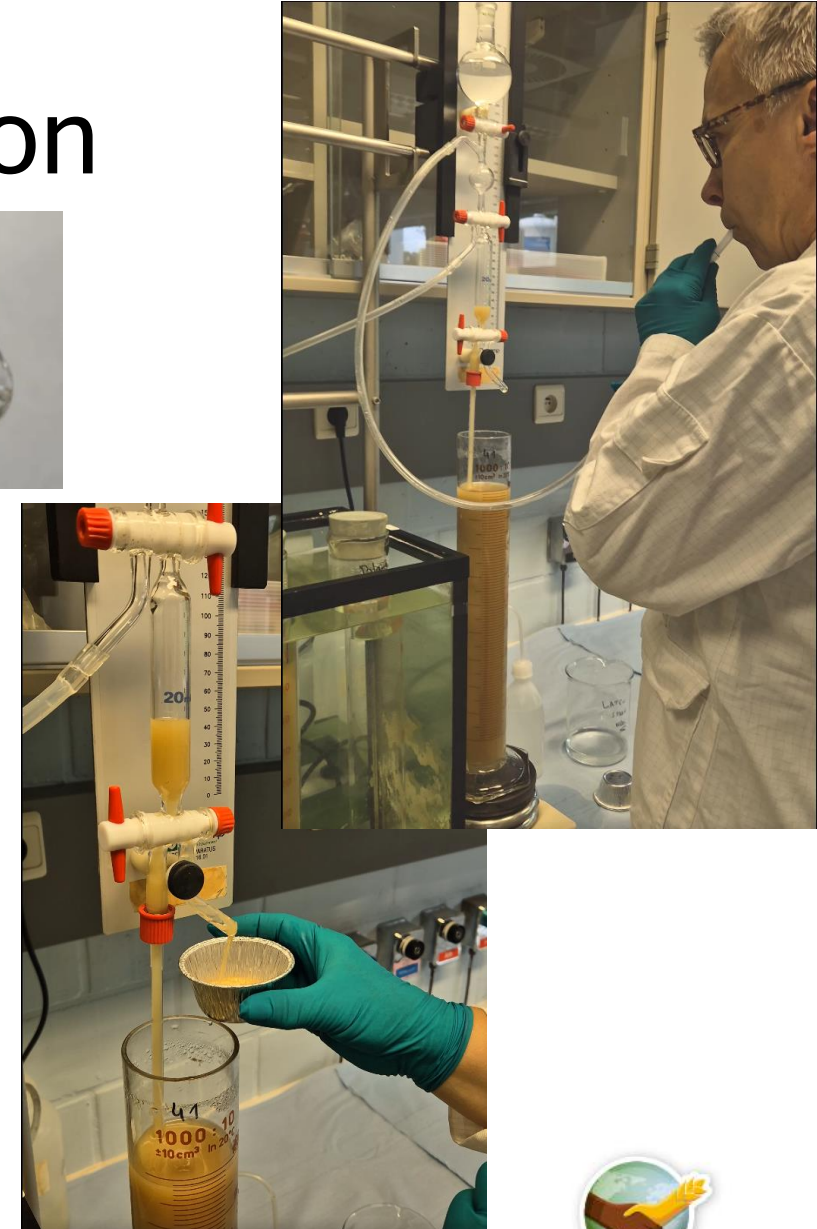
# Separating the silt and clay fraction

- Suspension is transferred to the sedimentation cylinder
- Bring the total volume of the sedimentation cylinder to exactly 1000 ml and insert a magnetic stirrer
- Place the sedimentation cylinder with a cap in the constant-temperature environment (e.g. water bath at 30 °C or room with climatization)



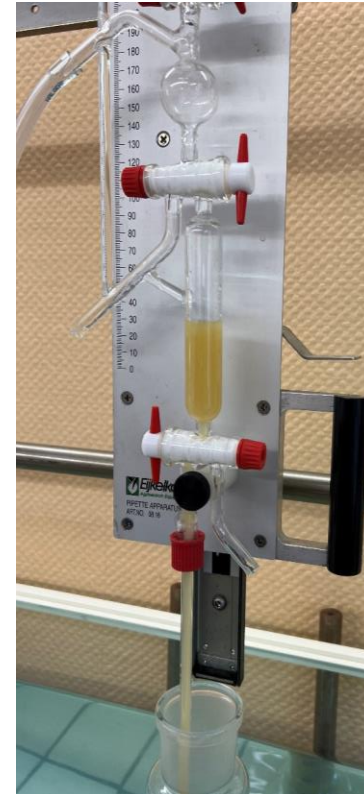
# Separating the silt and clay fraction

- First sampling: clay + silt (+ dispersion agent)
- Agitate the suspension vigorously (e.g. by stirrer)
- Pipette with sideways openings
- Pipette insertion depth is 10 cm
- 20 or 25 mL was aspirated using a Robinson-Köhn pipette (calibrated volume)
- Transfer the volume of the pipette in the weighing vessel
- The weighing vessel is dried at 105 °C until a constant weight is reached.

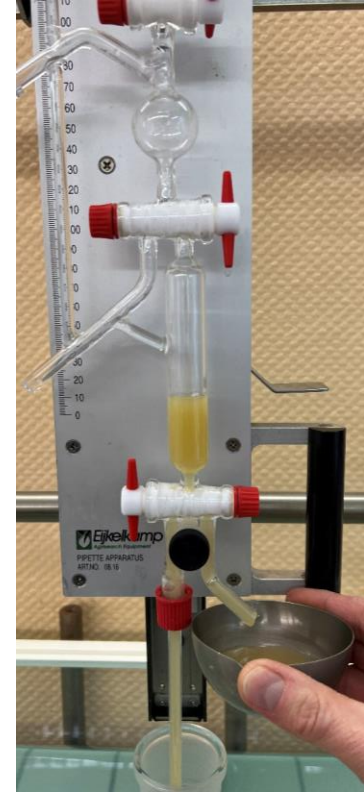


# Separating the silt and clay fraction

- Second sampling: clay (+ dispersion agent)
- Sampling after exactly 06.09 h (at 30°C), 30 seconds before the end of the sedimentation time, the pipette is placed into the suspension
- Pipette insertion depth of 10 cm
- 20 or 25 mL was aspirated using a Robinson-Köhn pipette
- The weighing vessel is dried at 105 °C until a constant weight is reached.



Wait the settle time for the clay and loam fraction at a depth of 10 cm and absorb the solution



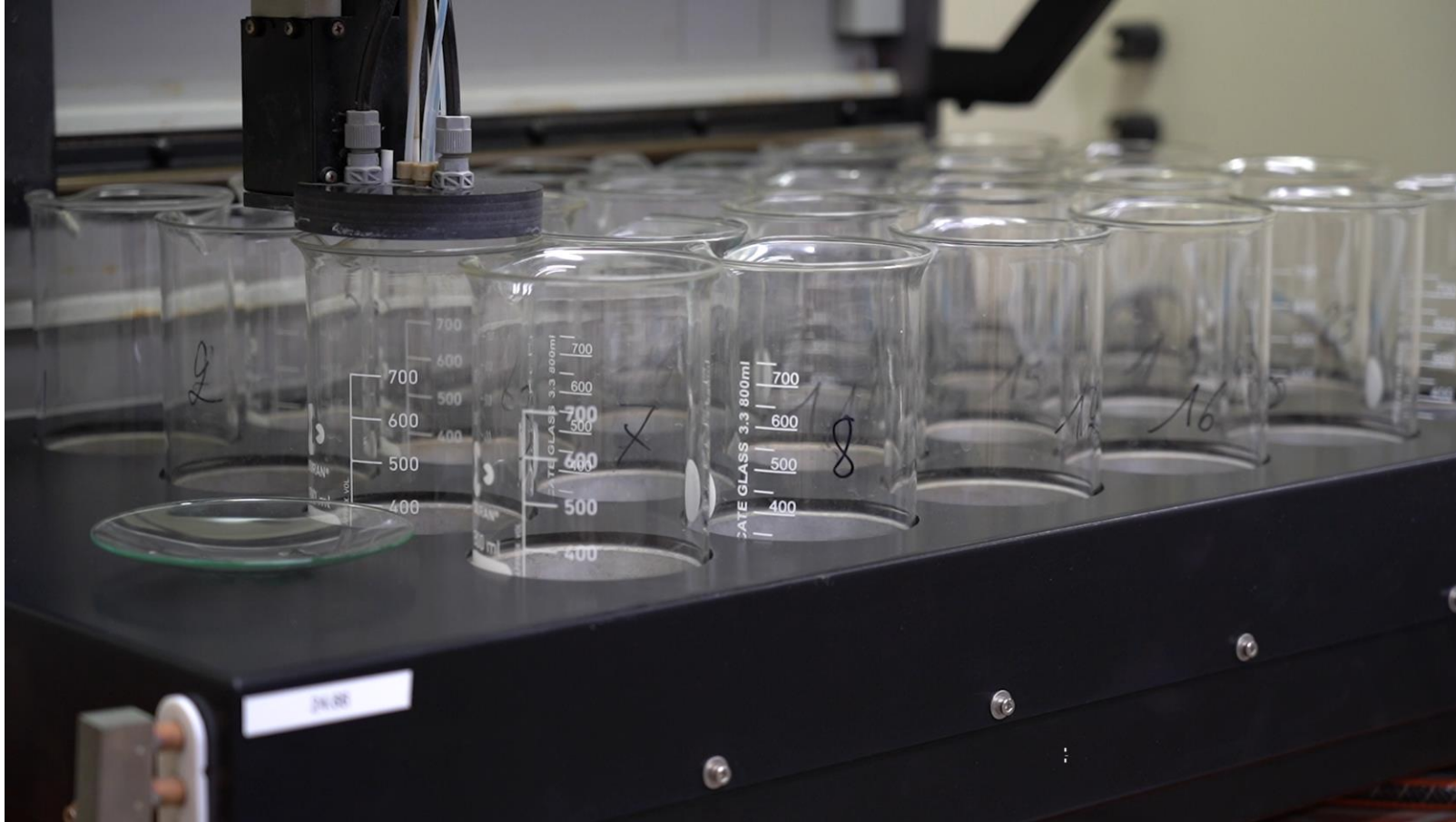
Transfer the volume (20 or 25 ml) of the pipette in a drying cup

# Calculations

- % fraction of clay is determined on the mineral fraction (after removal of the organic fraction, the carbonates and soluble salts)
- % clay ( $< 2 \mu\text{m}$ ) =  $(m_{\text{clay}} / m_{\text{total}}) \times 100\%$
- $m_{\text{total}} = m_{\text{sand}} + m_{\text{silt}} + m_{\text{clay}}$
- $m_{\text{total}} \neq m_{\text{initial}}$



# Automated procedure – sample prep



*Thanks to Guy D'Hond of Labo De Vlieger – Van Vooren for providing the movies*

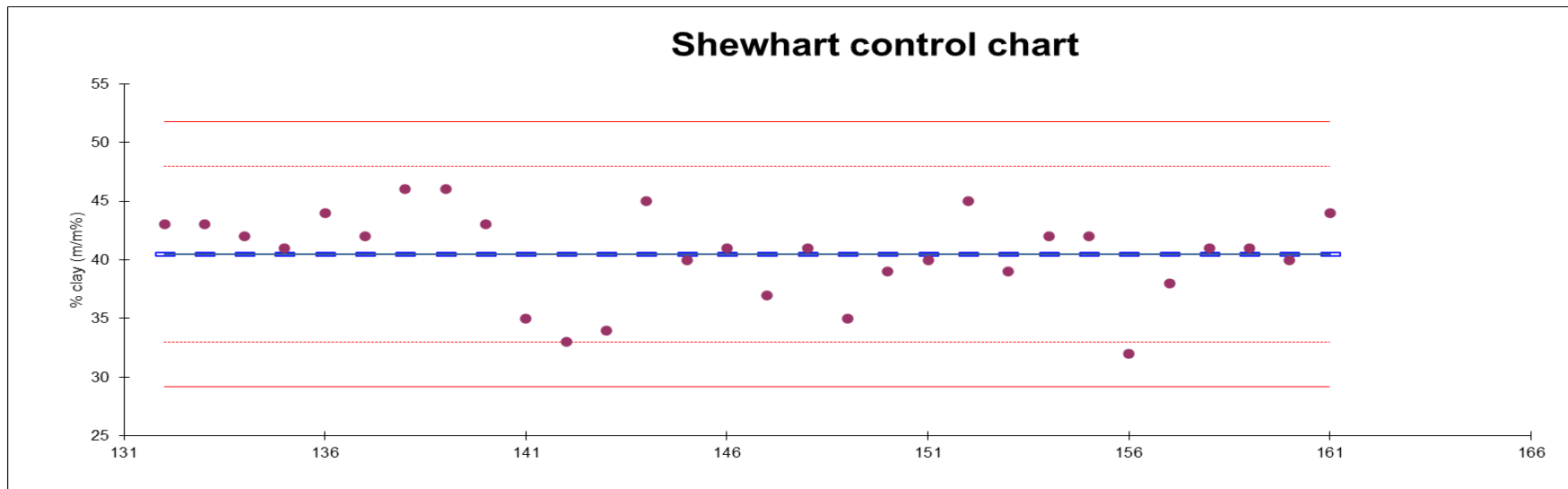
# Automated procedure - pipetting



*Thanks to Guy D'Hond of Labo De Vlieger – Van Vooren for providing the movies*

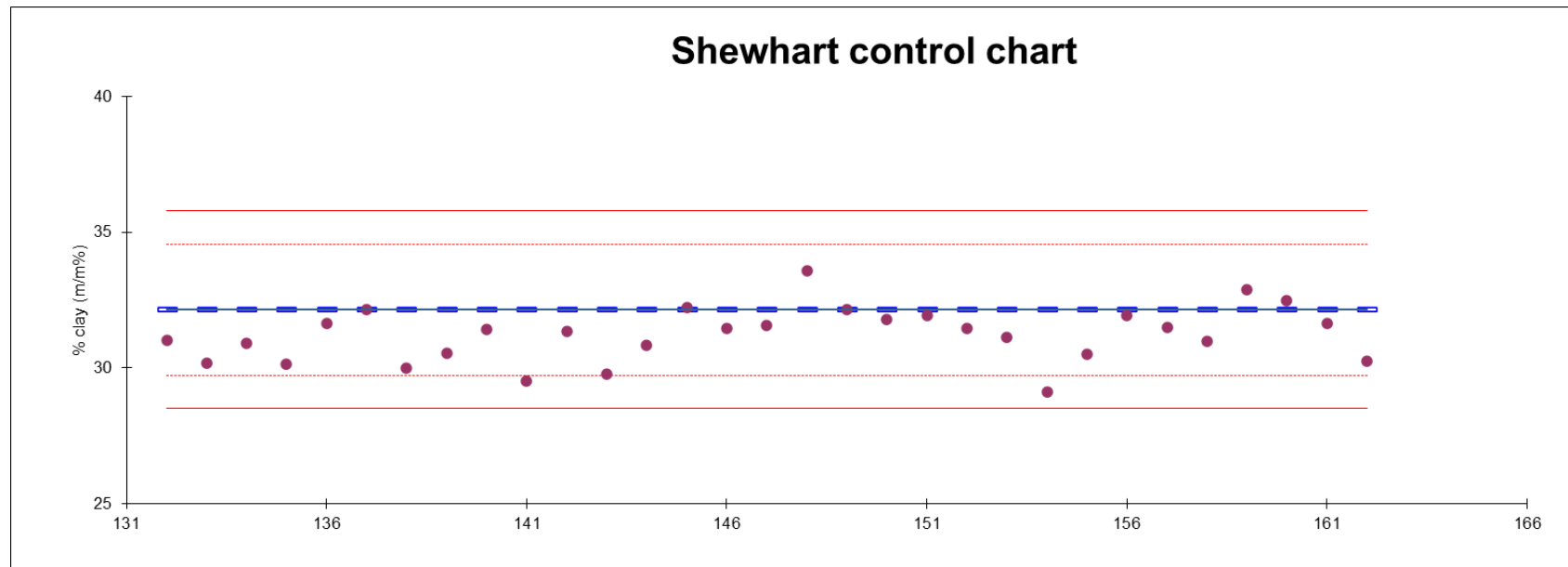
# Intra-laboratory reproducibility results

- Typical Shewhart control chart for the full process of the clay determination according to ISO 11277 (manual pre-treatment steps)
- $n = 30$  – 9,3% RSD



# Intra-laboratory results

- Control chart obtained from clay determination using the automated pre-treatment steps – n = 30 – 3,8% RSD
- Weight of clay, related to the initial intake





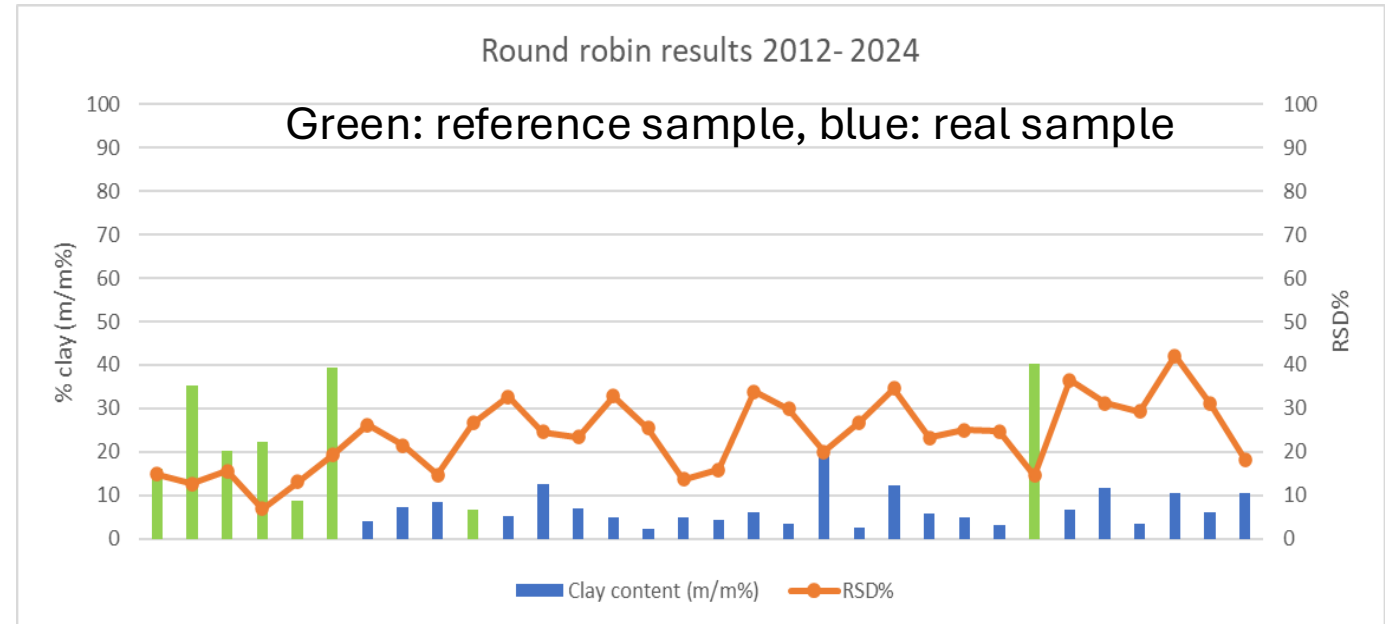
# Round robin results

Round robin trial organized on a yearly base in Flanders by VITO, commissioned by OVAM

Participation of 9 labs from B, NL, D

Method: according to ISO 11277, but with different pre-treatment steps

Combination of manual and automated methods



# Conclusion

- ISO 11277 is the reference method for the Belgian regions and Luxembourg
- Method is applicable for soil classification, but in Flanders (B) also applicable for adapting the soil remediation limit values
- For high-throughput analysis (sanitation), automation is encouraged and introduction of the use of ultrasonic bath to reduce the time for sample pre-treatment
- Inter-laboratory reproducibility ranges 10 – 35 %, differences in operational pre-treatment procedures
- Exchange of experiences about the method is of importance to reduce the variability
- Via mirror committees with the reference labs and the recognized laboratories experiences will be exchanged



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# Thank you

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