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Dosing of carbonates by mass loss

Introduction

1. Theory

Carbonates are an abundant group in the biosphere. However, a fraction of carbonate minerals dominates: the forms linked to calcium CaCO₃, magnesium MgCaCO₃, sodium Na₂CO₃ and iron FeCO₃. However, there are many intermediate species. These are the result of a large number of pedogenic and biogeochemical processes and can provide valuable information depending on the desired observations. For more information on carbonate determination methods, refer to Pansu and Gautheyrou (2003).

2. Principle

In this case, the determination of the carbonates is carried out by measuring the loss of weight on a sample of soil previously sieved at 2 mm and crushed. The addition of HCl initiates the decarbonation process according to a reaction of the type:

$$CaCO_3 + 2 HCl \rightarrow Ca^{2+} + 2 Cl^- + H_2O + CO_2$$

Following decarbonation of the sample, it is then possible to measure the difference of mass between the initial sample and the final sample and to deduce the carbonate content.

Safety Equipment







Equipement/ Reagents

- Tube for the centrifuge (50mL).
- Ultrasonic bath.
- Centrifuge.
- Balance.
- Oven at 105°C.
- HCl 10%.
- Distilled water.

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Method

- 1. Weigh the 50mL centrifuge tube with its cap and record the exact mass.
- 2. Tare the centrifuge tube and weigh 4g of the sample into the tube, note the exact mass.
- 3. Wet the sample with a little of distilled water.
- 4. Add 10mL of HCl 10%.
- 5. Once the reaction is complete (no more foaming), put the samples in ultrasonic bath for 2 minutes.
- 6. Shake horizontally for 15 minutes at 120 rpm on the shaking table. (**Be careful** not to do this until almost all the carbonate have been removed. With a lot of carbonated samples, the cap may leak /jump or the tube can explode).
- 7. Centrifuge for 10 min at 4000 rpm. Discard the supernatant, being careful not to lose any material. Take care to balance the tubes!
- 8. Check that all the carbonates are destroyed by adding a few drops of 10% HCl. If this is not the case, start again from point 4, otherwise continue.
- 9. Complete the centrifuge tube to 40mL with distilled water.
- 10. Shake horizontally for 10 min at 120 rpm.
- 11. Centrifuge for 20 min at 4000 rpm. Discard the supernatant, being careful not to lose any material.
- 12. Repeat step 9.
- 13. Once the supernatant has been discarded, place the samples in an oven at 105°C for one night.
- 14. Remove the sample from the oven, put the cap on and allow them to cool.
- 15. Weigh the samples.

The carbonate content in weight percent (wt %) is the difference in weight between the original sample and the sample after acidification divided by the weight of the original sample, multiplied by 100.

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References

Pansu, M. et Gautheyrou. J. (2003). *L'analyse du sol : minéralogique, organique et minéral*. Springler-Verlag France, p.993.