BOD AR_dO2_BOD_soils+solids.5, carbonate_lab_01_E

Application report soil respiration in carbonate-rich soils

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Matrix: Soils and solids

Analytical applications
No. 5

Manometric determination of soil respiration with OxiTop® Control in carbonate-rich soils

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Note: This report was made by using OxiTop® Control. All measuring procedures can easily be transferred to the OxiTop®-IDS system.

Area of application

Determination of the respiratory activity in soils with a high level of calcium carbonate. The manometric determination of the respiratory activity is insensitive to falsification due to the high level of calcium carbonate. The negative pressure arising through the absorption of carbon dioxide which was formed from oxygen by respiration processes is measured here. If carbon dioxide is now formed from carbonate-containing soils, this is in fact absorbed but there is no resulting negative pressure.
The titrimetric procedure is different from this. Here it is not possible to differentiate whether the absorbed carbon dioxide originates from biochemical reactions or from inorganic sources because the concentration of the absorption material is measured.
Fields of application:

- Agricultural examinations
- Contaminated land examination
- Refuse examinations
- Fundamental research

Further information and references on this subject can be found in the bibliography list [1] - [8].

Measurement principle

Manometric measurement of oxygen consumption with simultaneous absorption of CO_2 in caustic soda solution and titrimetric determination of the absorbed amount of carbon dioxide.

Material

OxiTop® -Control measuring heads (WTW, Weilheim, Germany)
Oxitop®OC 110 Controller (WTW, Weilheim, Germany)
ACHAT OC PC communication software (WTW, Weilheim, Germany)

Data transmission cable, type AK 540/B for RS 232 (WTW, Weilheim, Germany)
Measuring vessel MG 1.0 with lid clip DV/MG (WTW, Weilheim, Germany)
Temperature-controllable room or thermostat cabinet in variants TS606/2... TS606-G4/Var (WTW, Weilheim, Germany)
Personal computer, minimum configuration: 80486 processor, 16 MB RAM, RS232 interface
Windows 3.1 or 3.11 operating system
EXCEL® software (Microsoft, USA)
Vaseline
Laboratory scales (reading accuracy: min. 0.1 g)
Measuring container (50 mL)
Temperature-controllable room or thermostat cabinet in variants TS606/2... TS606-G4/Var

Volumetric pipette, 50 mL

Measuring cylinder, 50 mL
Burette (50 mL)
Erlenmeyer flask 300 mL
Magnetic stirrer
Magnetic stirrer bar
Caustic soda solution (1 mol/L)
Hydrochloric acid (1 mol/L)
Barium chloride solution (0.5 mol/L)
Phenolphthalein reagent solution (0.1% in 60% ethanol)

Conducting the measurement

Con-sec. no.	Workstep	explanations, comments, notes
1	The preparation and execution of the manometric and titrimetric measurements were carried out as described in [1] and [2].	
2	A calcium carbonate-free soil (reference) without and with the additive of calcium carbonate and sodium hydrogen carbonate was examined. Pure calcium carbonate and pure sodium hydrogen carbonate were also examined.	
3	At the end of the measurement the data were transferred to a PC using the ACHATE OC software and prepared with EXCEL.	

Examples of measurement results

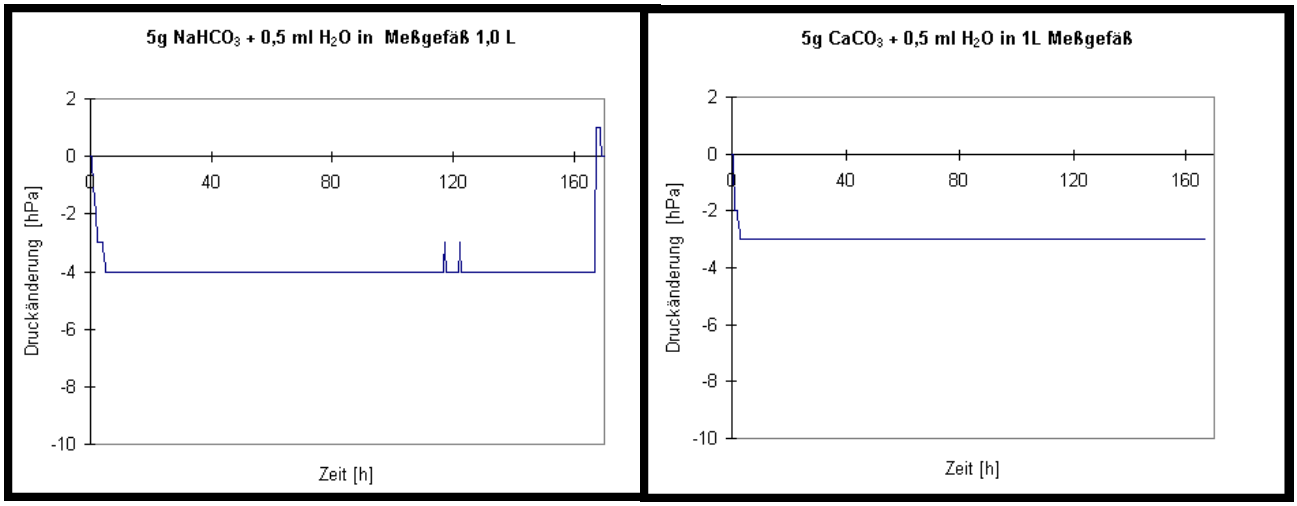


Figure 1:

Control measurement: Measurement of the change in pressure in a 1-litre measuring vessel with moistened sodium hydrogen carbonate or calcium carbonate (5 g of each substance moistened with 0.5 mL water).
After a short adaptation phase, the pressure in the vessels remained constant over the entire measurement period. The sodium hydroxide in the vessel was examined titrimetrically when the measurement was finished (Table 1).

The measurements of the release of carbon dioxide showed that $CaCO_3$ released comparably less CO_2 during the examination period; this was also shown in soil mixed with $CaCO_3$ (Table 1, lines 1 and 4). In contrast, $NaHCO_3$ released comparatively high amounts of CO_2 which was also the case in the appropriately mixed soil (Table 1, lines 2 and 5).
In the titration the soil mixed with calcium carbonate resulted in respiratory activity increased by about 112% compared with the negative pressure measurement where only an increase of about 41% was recorded. After adding the hydrogen carbonate, the titration actually resulted in a value that was higher by 700% compared to an increase of about 24% in the manometric measurement method.
The results confirm that soils with high carbonate or hydrogen carbonate content can release measurable amounts of CO_2, particularly if acid forms in the soil. This leads to results that are too high in the measurement of respiratory activity through the formation of CO_2. The moderate increase of the oxygen consumption rate can be related with more favourable conditions for microorganisms. However, no check was made of whether the values lay outside the normal range of fluctuation for multiple measurements.

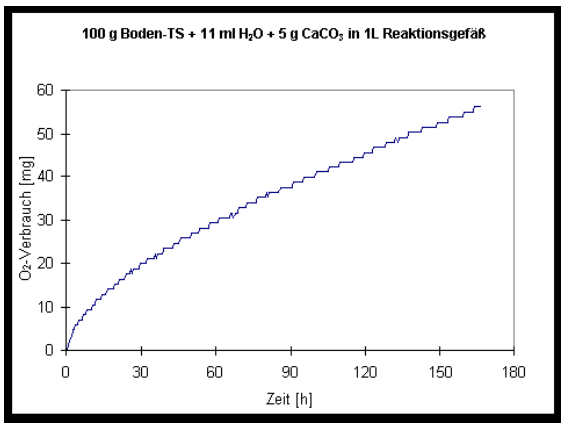
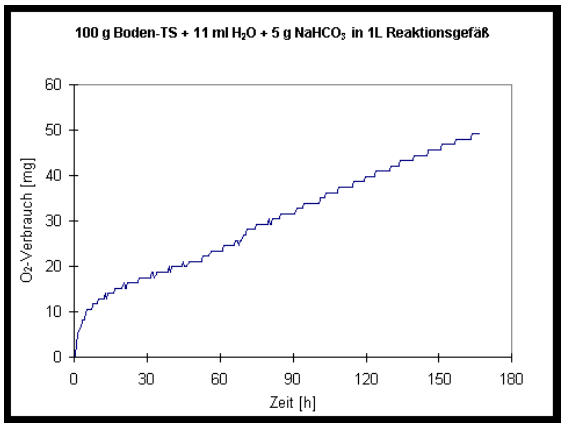


Figure 2:

A soil sample was divided into three parts and, as well as the control with no additives, mixed with sodium carbonate or calcium carbonate (5 g per 100 g soil). The respiratory activity was recorded over 7 days.
The basic respiration without any additive gave a value of 42.3 mg O_2/[kg*d]. The soil samples to which carbonates were added showed activities of 54.8 mg CO_2 [kg*d] ($NaHCO_3$) and 59.0 mg CO_2 [kg*d] ($CaCO_3$) and, thus, the activity was 30 to 40% higher than the untreated soil (within the scope of these measurements, no statistic examination was made as to whether the increase of the activity was significant).



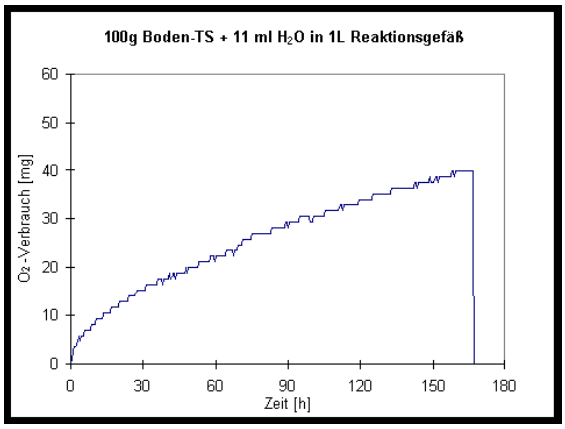


Table 1:

Measurement of released quantities of CO_2 after 7 days from the reference soil, from calcium carbonate and sodium hydrogen carbonate and the soils mixed with them (see Fig. 1 and Fig. 2).

Sample	Titration result [mL]	CO_2 formed [mg]	Decrease in pressure [hPa]	O_2 consumption [mg]
$CaCO_3$	48,0	44,1	0,0	0,0
$NaHCO_3$	41,9	178,0	0,0	0,0
soil	46,5	77,0	-34,0	39,8
soil + $CaCO_3$	42,6	163,0	-48,0	56,2
soil + $NaHCO_3$	19,8	665,0	-42,0	49,2

Bibliography

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Note

The information contained in our application reports is only intended as a basic description of how to proceed when using our measurement systems. In isolated instances or if there are special general conditions on the user side, exceptional properties of the respective sample can, however, lead to a change in the execution of the procedure or require supplementary measures and may, in rare cases, lead to a described procedure being unsuitable for the intended application.

In addition, exceptional properties of the respective sample such as special general conditions can also lead to different measurement results.

The application reports have been prepared with the greatest possible care. Nevertheless, no responsibility can be accepted for the correctness of this information.

The current version of our general terms of business applies.

Any further questions? Please contact our Customer Care Center:

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