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Application report Oxygen consumption and carbon dioxide generation

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Matrix: soils and solids

Analytical applikations
No. 4

Measurement of oxygen consumption (manometric with OxiTop® Control) and carbon dioxide generation (titrimetric)

1st edition, July 1999

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 of soils and other solids with and without substrates in the measurement range of 3.0 to 1800 mg O_2/[kg*d] (manometric) or 20 to approx. 10000 mg CO2/[kg*d] (titrimetric) in the following sphere of activities:
- Agricultural examinations
- Contaminated land examinations
- Refuse examinations
- Fundamental research

Further information and references on this subject can be found in the bibliography list [1] - [12].

Measurement principle

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

Material

OxiTop®-C measuring heads (WTW, Weilheim, Germany)
OxiTop® OC110 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 vessels MG 1.0 and 1.5 L with DV/MG lid-locking device (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).
Glass beakers (50 mL)
Temperature-controllable room or thermostat cabinet in variants TS606/2....TS606-G4/Var
Volumetric pipettes, 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 test solutions are prepared and the manometric measurement is performed using the method described in Application no. 2 (work steps 1 - 13). As a further control, a measuring vessel is prepared with 50 mL of the same sodium hydroxide added as in the measuring solutions. This is also titrated at the corresponding measurement intervals (recording the blank value of the caustic soda solution).	For this application it is important to note that the caustic soda solution used has a precise concentration of 1 mol/L and that exactly 50.0 mL of this is used as an absorbent; this is a precondition for accurate titrimetric determination of the CO_2. A possible blank value can arise because the sodium hydroxide has already absorbed CO_2 from the air during storage. For this reason, the caustic soda solution should have as little contact with air as possible (seal vessels immediately). The natural CO_2 content of air (approx. 0.03% to 0.04%) corresponds to 0.012 mmol/l.
2	In addition to the manometric oxygen consumption measurement, the caustic soda solution is immediately titrated according to the following protocol (from Point 3) each time the reaction vessel is opened (process based on Isermeyer [1]). The titration must be carried out quickly after opening the reaction vessel so the caustic soda solution does not absorb noticeable amounts of CO_2 from the laboratory air.	The theoretical maximum CO_2 absorption capacity of 50 mL 1.0 mol/L NaOH is 25 mmol: $2\ NaOH + CO_2 \longrightarrow Na_2CO_3 + H_2O$ Based on experience, this should only exhaust up to 50% and, hence, not limit the absorption rate. Because the ratio of CO_2 generation to O_2 consumption in many cases is about 1:1, this means approx. 200 mL of CO_2 is generated for a 1 L reaction (8.3 mmol, 365 mg CO_2); hence, on the one hand, the absorption capacity is sufficient and, on the other hand, it can be titrated with adequate precision. In the original work [1] NaOH or HCl with 0.1 mol/L is used which allows a more sensitive measurement. The aborption capacity is then so low, however, that the manometric measurement is restricted to a small range.
3	All the aborption solution is transferred into a 300 mL Erlenmeyer flask and 20 mL of a $BaCl_2$ solution is added (0.5 mol/L)	Barium ions bond the carbonate ions: $Ba^{2+} + CO_3^{2-} \rightarrow BaCO_3\downarrow$ hence guaranteeing undisturbed titration of the surplus base.
4	A few drops of phenolphthalein solution are added to the mixture.	Transition point at approx. pH = 8.0: alkaline: red; neutral/acidic: colourless

Con-sec. no.	Workstep	explanations, comments, notes
5	The solution is titrated with HCl (1 mol/l) up to the colour transition from red to colourless.	
6	The amount of CO_2 absorbed is calculated as follows: (50 mL – a mL) x 22 mg/mL CO_2 a = HCl consumption (in mL)	
7	The volume of oxygen consumed [2] is calculated from the change in pressure and information on the free gas volume	
8	To calculate the respiratory quotient (RQ), the CO_2 generation is divided by the oxygen consumption.	

Examples of measurement results

Tabel 1

Titration results and calculation of the respiratory quotient (RQ) of the measurement solutions according to Figure 1. In accordance with the chemical equation for the aerobic glucose depletion ($C_6H_{12}O_6 + 6\ O_2 \rightarrow 6\ CO_2 + 6\ H_2O$), a theoretical RQ of 1.38 (based on the mass) can be expected. The results obtained here (RQ = 1.58) show that less oxygen was consumed than would have been expected on the basis of the chemical equation. This also depends on the fact that, in the breakdown of materials, the microorganisms require electrons from the oxidation of glucose which are, hence, not available for the reduction of oxygen. Further supporting measurements are required for the final evaluation of the measurement result.

Procedural step/ Measurand	Endogenous respiration	Substrate-induced respiration	Corrected values for glucose
Titration result of the 1st titration	46,5 mL	37,0 mL	-
CO_2 generated up to the time of the 1st titration	77 mg	286 mg	-
Titration result of the 2nd titration	-	45,9 mL	-
CO_2 generated up to the time of the 2nd titration	-	90 mg	-
Total volume of CO_2 generated	77 mg	376 mg	299 mg
Total volume of O_2 consumed	40 mg	229 mg	189 mg

Mass ratio $CO_2 : O_2$	1,93	1,64	1,58

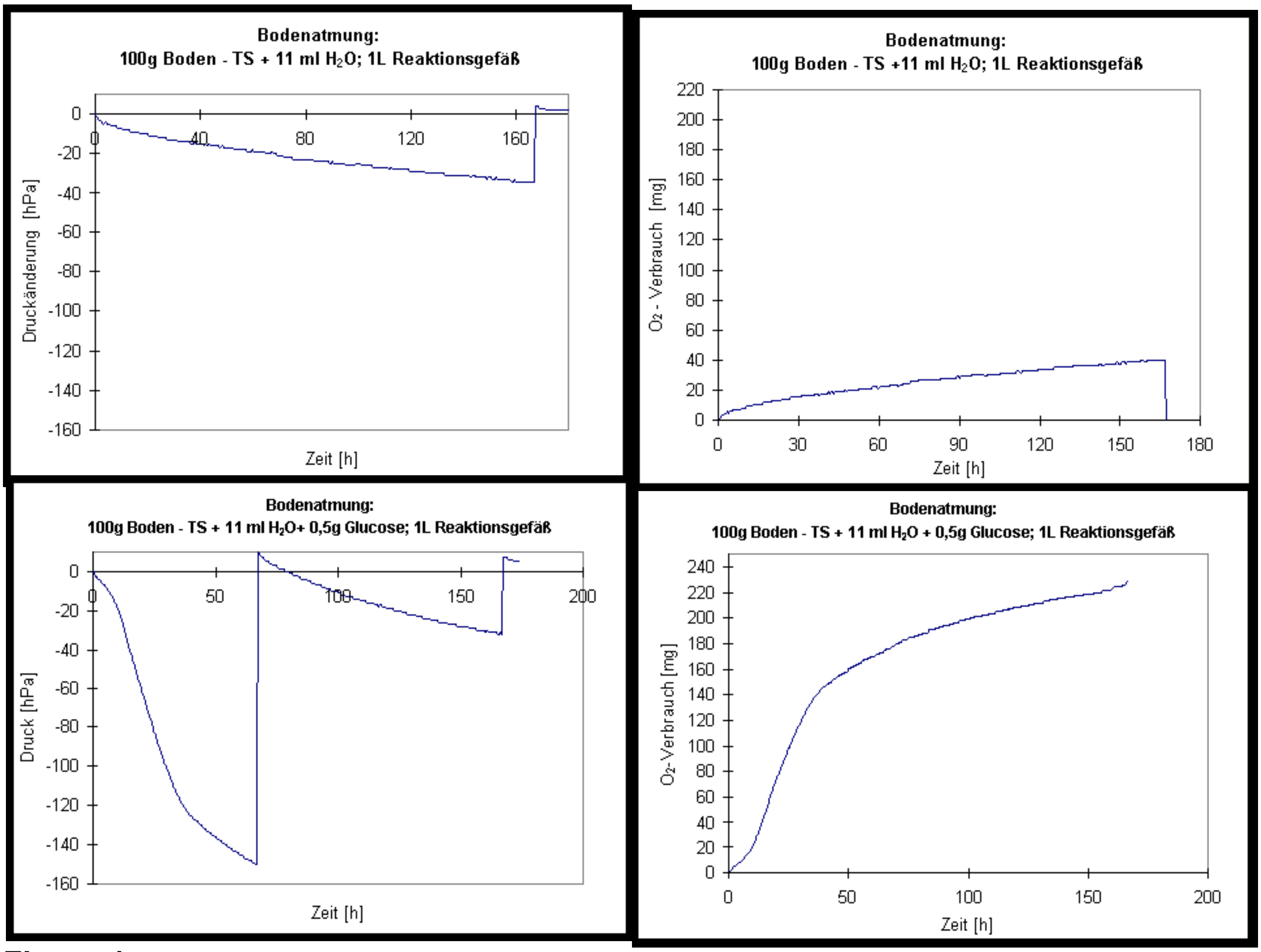

Figure 1:
Endogenous respiration and substrate-induced respiration of a soil sample with ½ WK_{max} (10% water content) at 20°C: Left: Progress of pressure decrease in the reaction vessels, right: derived oxygen consumption. After applying the tangents for the times t = 24 h and t = 144 h, the resulting respiration rates are listed in Table 2.

Tabel 2
Respiration activities of the untreated soil (endogenous respiration) and the soil mixed with glucose ("substrate-induced respiration") at two different points in time. In this soil inside one day glucose caused very high respiration rates which dropped again to the values at the start of the measurement in the control preparation (endogenous respiration) after 6 days.

Time after beginning [days]	Endogenous respiration mg O_2/[kg*d]	Substrate-induced respiration mg O_2/[kg*d]	Difference in respiration rates (factor)
1	73,5	1230	17
6	23,2	69,5	3

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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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