

## **BOD** AR\_BOD\_soils+solids.3, low activity\_lab\_01\_E

## Application report Low respiraton activities in soils





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Matrix: Soils and solids Analytical applications Nr. 3

Determination of low respiraton activities in soils with the OxiTop<sup>®</sup> Control measurement system

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

## Area of application

Determination of the respiratory activity of soils and other solids in the lower measurement range up to  $1.5 \text{ mg O}_2/[\text{kg}^*\text{d}]$  in the following sphere of activities:

- Agricultural examinations
- Contaminated land examination
- 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<sub>2</sub> in caustic soda solution.

Sensitive measurement by choosing a small measurement vessel (550 ml) with greater amount of soil (up to 400 g).

## **Material**

OxiTop®-C 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) DV/MG lid-locking device (WTW, Weilheim, Germany) Measuring vessel 0.55 L volume (Weck, Wöhr-Öflingen, 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) Laboratory scales (reading accuracy: min. 0,1 g). Absorption vessel(50 mL) Volumetric pipette, 50 mL Measuring cylinder, 50 mL Caustic soda solution (1 M) Vaseline



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## Conducting the measurement

Con- sec. no.	Workstep	explanations, comments, notes
1	Adjust the soil sample to be examined in sufficient quantity for optimum water content for the purpose of the examination. Reserve part of the material for a dry substance determination [2].	For soils: normally the adjustment is made to half of the maximum water capacity [1]. De- pending on the question, adjustment can be made to other water contents.





Con- sec. no.	Workstep	explanations, comments, notes
2	Determine the suitable amount of sample for the test (100 to 400 g)	As standard the DV/DG lid-locking device is designed for vessel sizes 1.0 and 1.5 L. After removing the holder for the absorption ves- sel, the lid can also be used for smaller vessels.
	Preserving jars from Weck (0.55 L) are used as measurement vessels; the holder for the absorption vessel is screwed off the DV/MG lid-locking device.	
3	Place the absorption vessel with 50 mL caustic soda solution (1 M) in the measurement vessel.	
4	Place the soil loosely shaken into the mea- surement vessel around the absorption ves- sel.	The absorption vessel should not be set on the soil so that it does not compact it.
5	Apply a thin coat of vaseline to the sealing ring of the lid-locking device (without holder for the absorption vessel)	Caution! Do not use silicone grease as this can cause damage upon contact with the OxiTop® measuring heads.
6	Put on the lid and affix with 4 clamps (4x 90°)	
7	Fit rubber sleeve.	Do not grease the joint! If necessary, trim the rubber sleeve as a seal!
8	Tightly screw on the Oxitop®-C measuring head	Do not use force!
9	Place the test preparation into the constant temperature room or thermostat cabinet	All materials should be preheated in order to avoid incorrect measurements.
10	Start recording the measurements with the OC110 controller (Pressure p mode).	The total measurement time depends on various factors. The lower the respiratory rate, the longer the measuring time should be. 360 measuring values are recorded at equidistant time intervals over the entire measuring period.
11	To prevent oxygen-limiting measuring condi- tions arising, the measurement data of the measuring heads are transferred to the cont- roller at regular intervals. If the defined pres- sure value is undercut or the selected war- ning pressure of -100 hPa, for example, whi- le processing the affected measuring ves- sels/samples by ventilating, addition of nutri- tive solution, sampling, refilling of the CO <sub>2</sub> absorber etc., for instance, call up the in- stantaneous value before starting and after ending the action and save in the controller (max. 10 instantaneous values, M01 M10)	In the case of manometric/respirometric de- termination of the respiratory activity, the partial pressure of oxygen in the sealed measuring vessel decreases during the measurement. If a minimum partial pressure of oxygen is maintained, the biological activity of the micro organisms is not affected. The Oxitop® Control measuring system docu- ments the entire pressure progression or the oxygen consumption as a graphic function.





Con- sec. no.	Workstep	explanations, comments, notes
12	If the above-mentioned pressure range is undercut, the measuring vessel should be opened and the caustic soda solution re- placed.	This procedure can be repeated as often as necessary during the specified time period or over 10 storable instantaneous values with- out having to restart the measuring head.
13	At the end of the measurement the data are read into a PC using the ACHAT OC soft- ware and prepared with EXCEL for presen- tation of the data.	The stored instantaneous values are listed at the end of the value table with M01 to M10 and can be displayed/evaluated as a con- stant function using Excel (differential forma- tion).

## **Examples of measurement results**



**Figure 1:** Determination of the respiratory activity of a soil at 20°C in the 0.55 L measurement vessel.

Left column: Measurement value recording by the OxiTop® Control measuring system. Right column: Data prepared by Excel.



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From the top downwards, increasing amount of soil weight (100, 200, 300 and 400 g DS, each with 10% water content). See text for further explanations.

All figures show that the activity of the soils is higher immediately after starting the measurements than after 50 to 100 h, where the respiratory activity approximates to a constant value (see 11). After this time, the respiratory activity is determined by applying the tangent. The specific respiratory rate referred to the amount of soil results in 31.8, 40.6, 42.4 and 45.0 mg  $O_2/[kg^*d]$  respectively for the measurement solutions. Apart from the first measured value, these lie quite close to one another. The measurements reveal a linear relationship between the amount of soil and the respiratory rate in terms of the measurement uncertainty. This shows also that the respiratory rate can be determined independently of the thickness of the layer of soil. Assuming not too much compaction of the soil sample, the gas exchange is not significantly impeded by the thickness of the layer!

In the measuring solution with 400 g of soil, the oxygen was completely depleted after 3 days; after that, there was a slight increase in pressure which points to the formation of a gas. The nature of the gas was not examined (methane formation is unlikely). Immediately after ventilating and exchanging the sodium hydroxide solution the respiratory activity is further measured at the same rate.

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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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