Technical documentation

shortened version as a method description
for scientific purposes only

Conductivity meter Black Gauss I

shortened version for method description for scientific purposes

Content

1 Introduction

1.1 Product name and type designation

Product Name:	Black Gauss I
Item number:	20B1000-00

1.2 Manufacturer details

Name:	Eurofins Environment East Ltd.
Address:	Lindenstraße 11, 09627 Bobritzsch-Hilbersdorf
Email:	info_freiberg@eurofins.de
Phone:	+49 3731 2076500

1.3 Warranty

shortened

2 Security

2.1 Intended use

The measuring device is designed to compress a powdery, carbonaceous material under a pressure of up to 50 kN and enables the conductivity to be determined with the associated external components. Essential external components are both a device for pressure build-up such as a hydraulic press or a toggle press and a multimeter with which the 4-wire resistance measurement can be carried out. The intended use of all external components can be found in the operating instructions of the individual devices.

2.2 Potential misapplication

The measurement setup must not be used for purposes other than intended. The following foreseeable misuses are not permitted:

- Processing of matrices not intended for the measuring device that could lead to damage of the integrity
- Excessively long dwell times under applied high pressure
- Independent conversion or repair measures
- Etc.

2.3 Symbols and notes

The manual uses symbols, signal words and notes to warn of hazards and to ensure safe operation. The symbols are shown and explained below.

CAUTION
This signal word indicates an imminent danger.
Failure to do so may result in minor injury.

NOTE
This symbol indicates an imminent danger.
Non-observance may result in damage to property.

2.4 Safety instructions

Read and follow the instructions in this operating manual to operate this measuring instrument and its external attachments (pressing device and multimeter) safely. Do not carry out any independent conversion measures, modifications to the setup or repairs to any of the devices.

2.5 Personal protective equipment

Protective gloves
Wear protective gloves.

Industrial safety shoes
Wear protective work shoes.

Safety goggles
Wear protective goggles.

2.6 Special safety instructions

In addition to the safety instructions described below, which apply to the measuring system defined here, the safety instructions for the individual external system components and their manuals must also be observed.

2.6.1 Transport, assembly, commissioning

The transport of the measuring device as well as the named external components is carried out by order of Eurofins Umwelt Ost GmbH Freiberg branch. The Black Gauss I measuring instrument is shipped in assembled condition with all associated components and is fully ready for use without any further work steps. No special safety measures have to be taken. In case of independent assembly of the individual parts (e.g.: after cleaning), the assembly instructions must be followed.

CAUTION
With a weight of about 24 kg, the supplied toggle press sometimes exceeds the permissible limit lifting load and must be handled accordingly.

2.6.2 Operation

During the time of use of the measuring system, make sure that the necessary safety equipment is worn. Furthermore, no force greater than 50 kN is to be exerted on the measuring device. The supplied toggle press operates with a pressure point of 12 kN well below the permissible total load, which means that there is no potential hazard when using it.

CAUTION
If the maximum permissible pressure is exceeded, individual measuring components are in danger of bursting.

2.6.3 Cleaning, maintenance, servicing

As part of a daily measurement check, the integrity of the system must be ensured. The measuring device is considered to be maintenance-free. For external system components, the maintenance measures and precautions specified by the manufacturer must be observed.

3 Product description

3.1 General function Black Gauss I

The method for the determination of conductivity in coals and (bio)chars is a newly developed procedure, which was tested and verified in cooperation between Eurofins Umwelt Ost GmbH and the Ithaka Institute.

The Black Gauss I measuring instrument and its external components make it possible to indirectly determine the conductivity on a carbonaceous matrix. First, the electrical resistance within the matrix is measured. The measurement is carried out under a defined pressure using the 4-wire measuring principle on a suitable multimeter. The position of the electrodes in the measuring device itself is at the top and bottom of the test specimen, whereby the resistance is measured vertically through it. Taking into account the geometry created in each case, the electrical resistance is converted to resistivity. Finally, the conductivity can be represented by the reciprocal of the resistivity.

Depending on the press equivalent used, pressures of up to 50 kN can be investigated. The use of a multimeter with the possibility of 4-wire resistance measurement is essential for the documentation of exact results. The complete package includes both a toggle press with a total pressure of 12 kN (or hydraulic manual press up to 50 kN) and a corresponding multimeter. Corresponding connection cables are included.

The presented package contains all necessary contents for the determination of conductivity based on the European Biochar Certificate (EBC).

3.2 Components

The entire measuring system has the following components:

- Black Gauss I
  - Stamp
  - Sleeve
  - Floor

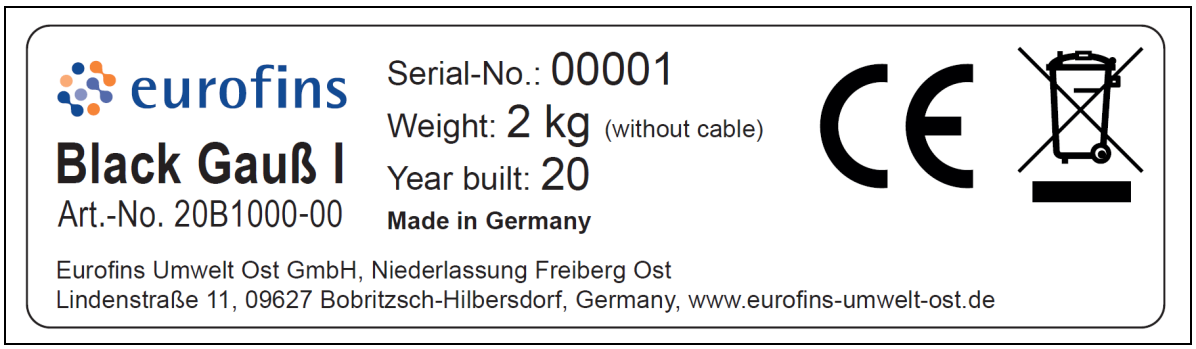
- Toggle press EP 1200-60, Fa. Mäder (or hydraulic manual press)
- Multimeter 2110 5 ½ Digit Multimeter, Fa. Keithley

3.3 Technical data

Toggle press EP 1200-60, Fa. Mäder
For complete technical data, refer to the relevant manufacturer's data sheets.
Multimeter 2110 5 ½ Digit Multimeter, Keithley Company
For complete technical data, refer to the relevant manufacturer's data sheets.

3.3.1 Nameplate

The nameplate is clearly visible on the outside of the case. A respective serial number has been applied to both the base and the stamp.



3.3.2 Dimensions and weight

Parameter	Unit	Value
Base height*	[mm]	108
Maximum test chamber height*	[mm]	138
Total diameter	[mm]	65
Diameter electrodes	[mm]	20
Maximum sample quantity	[cm³]	~ 10
Maximum load	[kN]	50
Weight	[kg]	2

* in compliance with the manufacturing tolerance

4 Installation and assembly

4.1 Requirements for the personnel to be performed

The installation of all individual components can be carried out without restrictions and independently using the included installation instructions.

4.2 Requirements for the installation site

The measuring system incl. all components may only be installed in closed rooms with a level and solid base. Furthermore, the following specifications must be met.

Parameter	**Unit**	**Value**
Max. Temperature	[°C]	40
Min. temperature	[°C]	0

4.3 Mounting of the measurement setup

Perform the following steps:

1. Assembly Black Gauss I

First, the three individual components already named must be assembled. The individual components are the base, sleeve and punch. Basically, they all consist of a structuring stainless steel coating and an insulating PET layer. An electrode made of a copper alloy is installed in the base and the stamp. The connection of the components takes place on the part of the soil by means of 3 screws within a quick-locking device and forms the sample chamber in the inside. The stamp is only inserted into the sleeve.

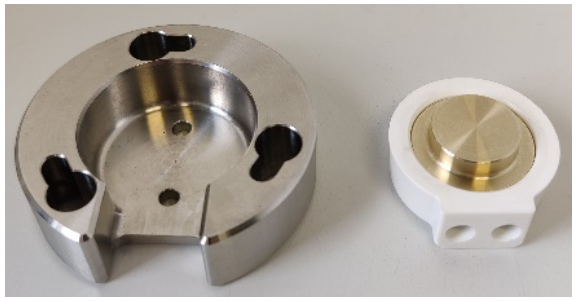
Base:
- components: Metal base, insulation base, electrode base with cable enclosure
- The individual components are plugged into each other according to the recesses provided, only one position is possible in this case
- Sequence: metal base - insulation - electrode
- Then the cables can already be inserted into the electrode (fixes connection electrode-insulation)
- in the metal base are included 3 twist locks

Sleeve:
- Components: Metal sleeve, insulation sleeve, retaining ring sleeve system, 3 hexagon socket screws, retaining ring, 3 hexagon socket screws for connecting sleeve - base.
- The combination of metal sleeve and plastic insulation is a dimensionally accurate interference fit, which is pre-assembled accordingly
- If the parts are to be reconnected independently, the insulation must be inserted accordingly so that the fitting shapes match
- Fixation of the system metal sleeve and insulation is made by a retaining ring with 3 screws
- The retaining ring must always be fixed accordingly after mounting this system
- 3 threads are pre-drilled on the underside of the sleeve into which 3 screws are inserted accordingly
- By means of the 3 screws in the sleeve and the associated quick-release fasteners in the base, the lower part of the measuring system can be completely assembled

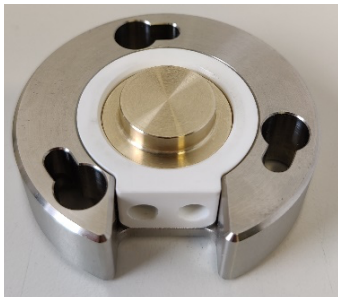
Stamp:
- Components: Metal stamp, electrode stamp with cable enclosure, cover metal, insulation cover, 4 hexagon socket screws for connection stamp system
- First, the electrode is inserted into the metal socket, making sure that the recesses for the cables are in line in both the metal socket and the electrode, as there is no fixed point.
- Fixation is performed using the covers (metal and plastic) by means of the 4 enclosed screws, with the middle screw fixing the electrode rod in the sleeve.

The following pictures serve to illustrate the described processes .

Overview individual parts base



Composition measuring system base



Complete base

Overview individual parts sleeve

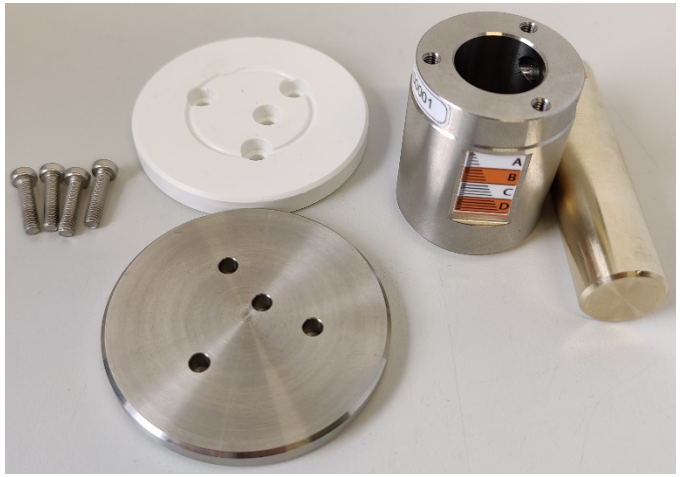
Mounting screws

Insulation insert

Complete sleeve

Measuring unit sleeve bottom

Area above electrode = sample chamber



Overview individual parts stamp

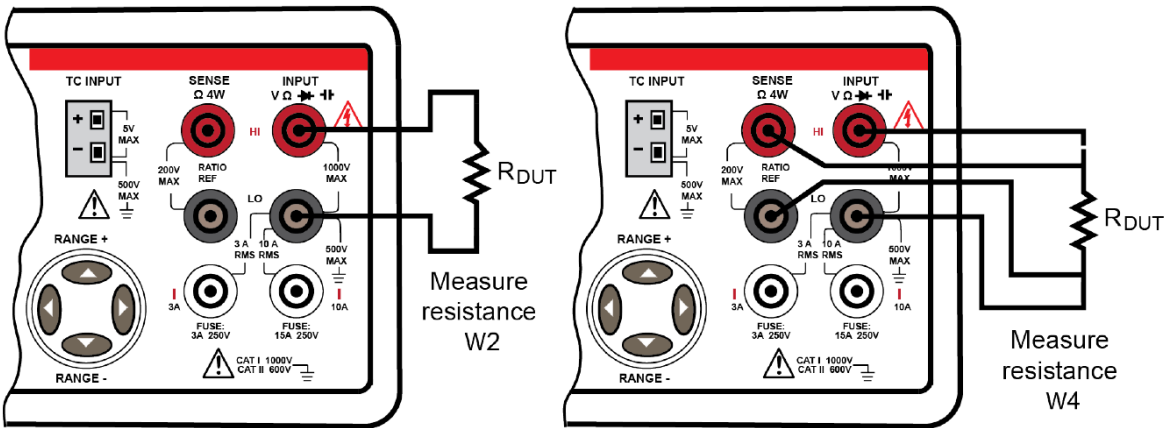
Mounting measuring system punch

Full stamp

2. Installation of the measuring stand

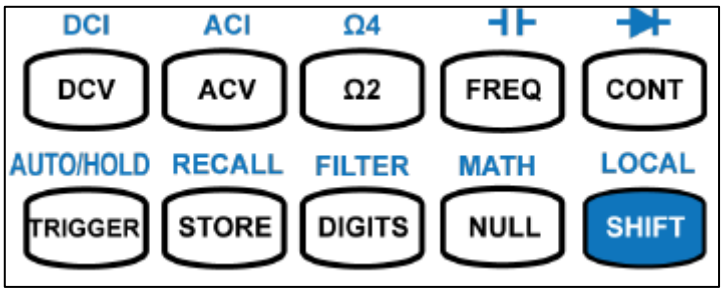
The entire measuring stand consists of all the components shown, i.e. Black Gauss I, toggle or hydraulic press and multimeter. An exemplary arrangement is shown in the following figure:

The wiring between the Black Gauss and the multimeter shown in the figure is based on the following circuit diagram and can be found accordingly in the manual of the device manufacturer.

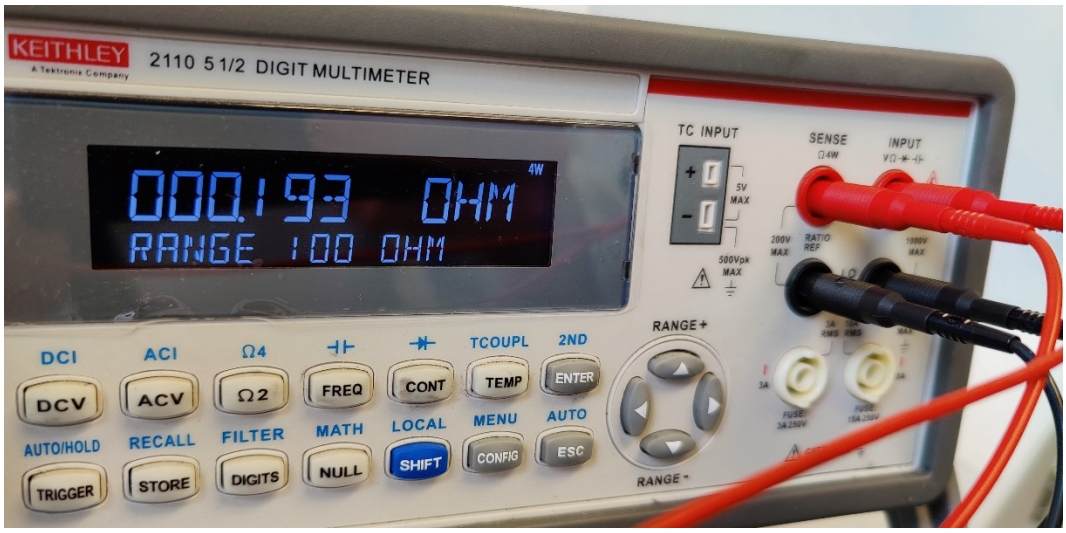


Source: Keithley Instruments Inc.: *Reference Manual 5 ½ Digit Multimeter*, August 2013

The 4-wire measurement is to be used at every measuring point, for which both the wiring shown and the correct setting for the resistance measurement are essential. It is important to note that the 4-wire measurement is programmed on the Ω2 key in secondary assignment as Ω4 by means of the Shift key. The measurement setting can be read in the top line of the display in the right-hand corner (correct designation: "4W"; see illustration).



Source: Keithley Instruments Inc.: *Reference Manual 5 ½ Digit Multimeter*, August 2013



5 Operation

5.1 Measurement

The basis for operation is a detailed knowledge of the presented basics of this manual as well as the successful completion of the bullet point 4. Installation and assembly.

The sample matrix to be analyzed must be transferred to the sample chamber above the bottom electrode in the analysis-fine state according to the specifications of the EBC. A balance can be used for this purpose. However, the use of this is not mandatory. If used, a sample weight of around 1.4 g is suitable. The influence of the initial weight on the measuring system is included via the parameter of the height (see below).

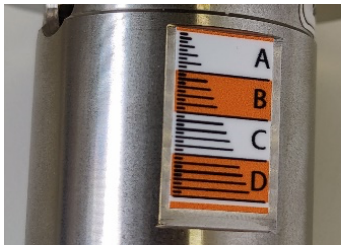
When the sample has been transferred to the sample chamber of the base-sleeve system, the stamp is finally inserted, which contains the counterpart to the measuring electrode in the base. The Black Gauss gauge (see 4.3 Installation), wired accordingly, is inserted into the press (following the press manufacturer's instructions) and fixed accordingly. Then, before starting the measurement, the operating status and the correctly set method of the multimeter are checked.

The actual measurement is then carried out by pressing down the lever of the press - corresponding to the way a toggle press works (by building up the desired pressure with the hydraulic press). At the lowest point of the lever, the defined pressure is reached. Now the displayed electrical resistance of the measuring instrument is read without any time delay. Fluctuations in the displayed value as time progresses are due to the sample matrix as well as the specifics of the measuring system and the measurement inaccuracies of the multimeter. If several pressure levels are to be measured with the hydraulic press, start with the lowest pressure.

The following table is intended to provide some orientation values concerning the associated reasonable reading accuracy of the multimeter.

Measuring range in [Ω]	Decimal places	Influence
0 - 100	0,000	
100 - 500	0,00	
500 - 1000	0,0	
> 1000	0	
>> 1000	0	

In addition to the documentation of the resistance, the reading of the height is essential for the subsequent calculation of the conductivity. The measuring ruler attached to the stamp of the Black Gauss is used for this purpose. The accuracy is in steps of 0.5 mm. The measuring ruler does not have a number scale. However, as an aid, individual segments are color-coded, with one segment (A, B, C, D) corresponding to 5 mm.



A special feature of the measurement setup is the so-called zero height. The zero height corresponds to the base height of the system, i.e. the height that can be read on the measuring ruler when the system is assembled without a specimen. This zero height depends on the production and differs from instrument to instrument. Accordingly, it must always be read individually for your own measurement setup and updated accordingly if individual components had to be replaced.

5.2 Cleaning

Following the measurement and the documentation of the required individual values, the pressure is released. Black Gauss is removed from the toggle press (or similar) and the punch is pulled out. This procedure should generally be easy to perform without any real force being applied. If necessary, it may be useful to turn the punch in on itself or to remove it while turning it. It is not necessary to disconnect the cable connection to the multimeter.

The electrode on the stamp is to be cleaned dry with a lint-free cloth from char particles adhering to it. The bases is turned upside down and can be easily divided into sleeve and base by means of the 3 screws on the base. Here, one turn is sufficient to release the quick-release fastener. The contained carbon can be easily removed or already loosens itself in the course of the disassembly (at low carbon contents of biochars sometimes very hard test specimens arise which must be pressed out). The electrode in the base must be cleaned in the same way as the electrode in the stamp.

Finally, the sleeve containing the char must be cleaned. Water is best suited for this purpose. Other cleaning agents must be checked for their suitability and compatibility with the materials used. Furthermore, a water temperature of 60 °C must not be exceeded. The sleeve should be cleaned accordingly with lint-free cloths and dried. The individual parts are then reinserted and the screws tightened. The system is considered ready for measurement.

5.3 Calculation

Using the determined data ($\Omega_{electric}$, h), the conductivity of the individual specimen is calculated using the following formulas.

$$\Omega_{spec} = \Omega_{electric} * \left(\frac{A}{h - h_0}\right)$$

Ω_{spec} ... resistivity of the specimen in Ohm*cm

$\Omega_{electrif}$ ... measurable electrical resistance of the specimen in ohms

$A_{electrode}$ ... electrode surface area (circular) in cm with $\frac{\pi}{4} * d^2$ [d ... diameter electrode in cm]

h ... Height of the compact during compression incl. h_0 in cm

h_0 ... Zero height of the test setup without specimen in cm [device-specific value depending on the manufacturing tolerances, which can be changed as a result of component replacement].

$$C = \left(\frac{1}{\Omega_{spezifisch}}\right) * 1000$$

With units:

$$[C] = \left(\frac{1}{Ohm * cm}\right) = \frac{S}{cm} = \frac{1000\ mS}{cm}$$

C ... Conductivity in mS/cm

Due to the calculation specifics, there is a large sensitivity of the calculated result, especially for small resistances.

Conversely, with increasing resistances, a reasonable measurement limit is quickly reached. Depending on the height of the test specimen, the resistances differ from each other, but it can be generally formulated that conductivities < 0.01 mS/cm are not meaningful.