

STANDARD OPERATING PROCEDURE

***Instructions for Calibration and Use of Total Organic Carbon (DC-85A) Instrument***

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**KEY WORDS**

soil, water, organic carbon, TOC

**APPROVALS**

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#### **1.0 INTRODUCTION**

The DC-85A total organic carbon (TOC) instrument can analyze both solids and solutions for TOC. It is not a total carbon analyzer because it does not reach the high temperatures required for total carbon analysis (1200°C). It is, instead, used to determine the TOC in 1-100 mg solids, slurries, and 40 uL solutions depending on the organic carbon content present in the sample. The fundamental principle of the instrument is as follows; the sample is combusted to CO<sub>2</sub> and the combustion gases swept by a carrier gas (O<sub>2</sub>) to a non-dispersive infrared (NDIR) detector after they are cleaned up via an acid bath (removes corrosives) and water trap (removes moisture). The detector provides a response value at the end of the analysis that is a time integrated measure of mean CO<sub>2</sub> concentration on a v/v basis in the combustion gases. Simple calculations allow for a weight integrated calculation of TOC in the sample. More specifically, the instrument readout provides TOC in ppm on a volume basis which must then be converted to ppm C (by weight) of the sample. The conversion relies on the calibration results obtained using a known standard. This standard operating procedure (SOP) discusses the initialization, calibration, use, and shut-down procedures for the DC-85A TOC instrument.

#### **2.0 MATERIALS**

- 2.1 DC-85A total carbon analyzer
- 2.2 2000 mg/L potassium hydrogen phthalate (KHP) standard
- 2.3 pH paper and glass wool
- 2.4 85% strength phosphoric acid (H<sub>2</sub>PO<sub>4</sub>)
- 2.5 Gloves and eye protection
- 2.6 Analytical balance (accuracy = 10<sup>-5</sup> g)
- 2.7 Spatula and tweezers

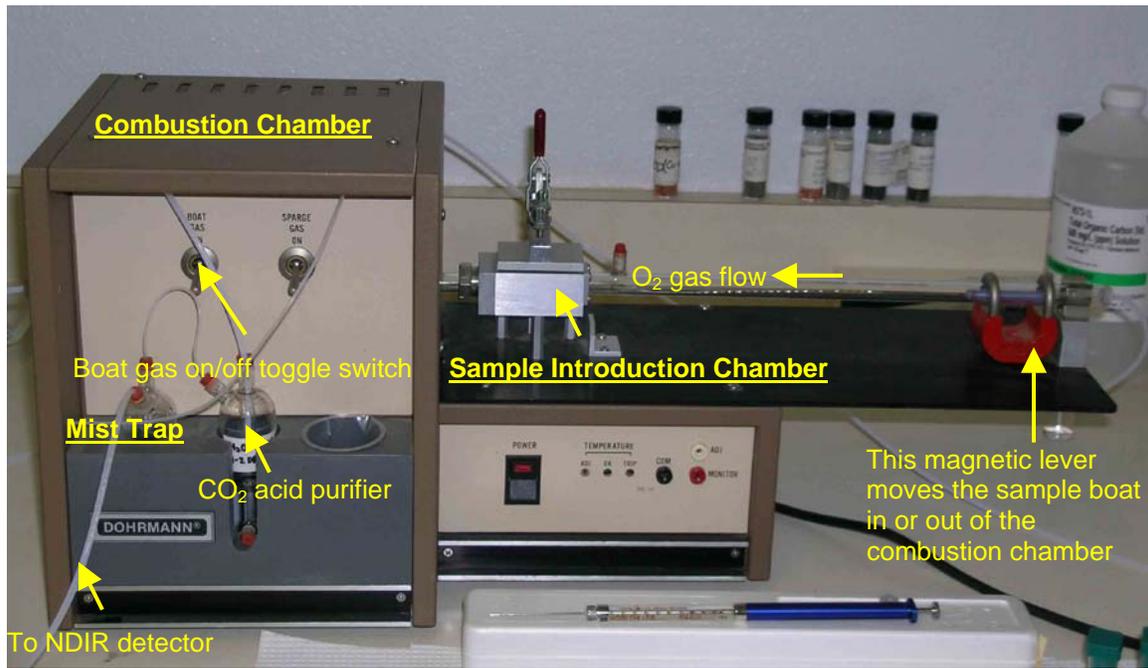
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## 3.0 PROCEDURES

### 3.1 Instrument Initialization

3.1.1 Observe and identify the following components of the instrument. Major instrument components referred to in the text are underlined.



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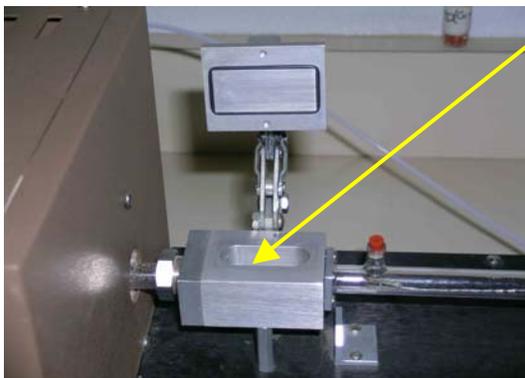
- 3.1.2 Power on combustion unit (detector unit is always on). When combustion chamber is ready, green light on combustion chamber control panel will be illuminated (circled in image below).



- 3.1.3 Turn on gas flow. Verify pressure is set to 30 psi.



- 3.1.4 Insert a blank sample boat into the sample introduction chamber and close chamber door. Make sure chamber door is sealed.



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- 3.1.5 Check acid trap with pH paper; pH should be between 1 and 2. Acid trap consists of water with two drops of 85% strength phosphoric acid ( $H_2PO_4$ ). Water level is indicated by black line on glass container.



- 3.1.6 Connect the gas flow tube to the sparge gas acid trap.



- 3.1.7 Check the mist trap (glass container next to the acid sparge gas wash). It should be filled approximately half of the total volume of the container with water.

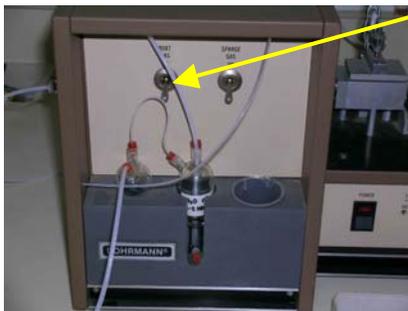
- 3.1.8 Check the gas scrubber (see image below) unit located on the side of the detector to determine if it is clean. Its main purpose is to dry the gas before it reaches the detector. Dirty units can be identified by the blackening of the glass wool at the two ends of the glass tube.



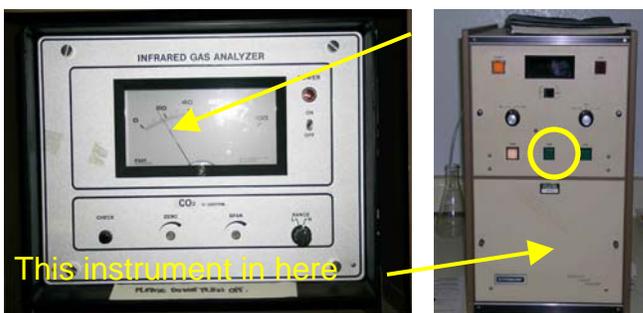
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- 3.1.9 Toggle combustion unit BOAT GAS ON switch to on. Observe flow of oxygen through acid trap. Wait one hour for detector to stabilize.



- 3.1.10 Note detector stabilization on NDIR (should be close to or at zero) and digital display reading on detector; should be low, less than 0.05. If these conditions are met, then an audible sound will be heard as well as the initiation of the READY light (circled on image below) on the detector. If the READY light is not on, consult the instrument's manual.



- 3.1.11 Toggle combustion unit BOAT GAS ON switch to off.
- 3.1.12 Open boat sampler door and place a small amount of glass wool on a sample boat. Close sample introduction chamber door.
- 3.1.13 Toggle combustion unit BOAT GAS ON switch to on and observe gas flow through acid trap.
- 3.1.14 Wait one minute to allow for CO<sub>2</sub> that was introduced into the sample chamber when door was open, to be removed by carrier gas (O<sub>2</sub>).
- 3.1.15 Advance the boat into the combustion chamber by moving the magnetic device towards the combustion chamber (see first image of SOP) to clean the boat. Do not initiate any controls on detector (although you might hear several audible sounds from the detector). Observe needle

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change on detector and its return to zero. Leave boat in combustion chamber for approximately one minute. If using more than one sample boat, all of the boats require combustion to remove dust or other sources that might amplify the sample's TOC signal. Avoid handling the boats with ungloved fingers (use tweezers) as hand oils can artificially increased instrumental response and hinder the accuracy of the instrument.

3.1.16 Return boat from combustion chamber with magnetic lever after two minutes but do not remove from sample introduction chamber. Keep sample introduction chamber door sealed.

3.1.17 Toggle combustion unit BOAT GAS ON switch to off.

3.1.18 Perform calibration; calibration should be performed if samples have not been run in three days or longer. Alternatively, a calibration "check" can be initiated to determine if the instrument is properly calibrated. In the latter case, follow procedure 3.2.16.

## 3.2 Calibration

3.2.1 CALIB button light should be off. If not, press calibration button; light goes off.



3.2.2 Adjust detector switchers to the following settings;  
Set detector mode = TOC  
Set detector scale = ppm  
Set sample size = 40 uL.

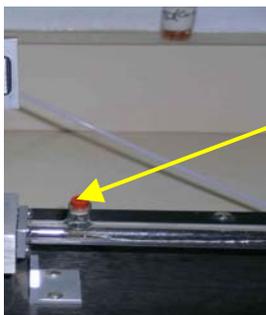
3.2.3 Make certain that sample introduction chamber door is sealed.

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- 3.2.4 Toggle combustion unit BOAT GAS ON switch to on. Observe gas flow through acid trap.
- 3.2.5 Obtain 40 uL of 2000 ppm KHP standard (purchased from a lab supplier and pre-acidified). Check expiration date on standard. Details of standards can be found in the instrument manual.
- 3.2.6 Sample boat must be cooled to room temperature so that the standard will not evaporate prior to analysis.
- 3.2.7 Move sample boat so that it is located under the syringe port (in the sample chamber).
- 3.2.8 Remove septum covering syringe port and Inject 40 uL of the KHP standard into the sample boat.



- 3.2.9 Close syringe port and let system equilibrate for one minute. This allows for any CO<sub>2</sub> introduced as a result of opening the syringe port to move past the detector.
- 3.2.10 Press START and immediately advance the boat into the combustion chamber. Note that the READY light goes off and the START light (circled in image below) comes on.



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- 3.2.11 Note changing digital panel display. When the sample analysis is complete, the READY light will come back on. An audible sound is also an indication that the sample analysis is complete.
- 3.2.12 Return the sample boat from the combustion chamber to the sample introduction chamber when analysis is complete and let it cool to room temperature.
- 3.2.13 The panel will display a four digit number. The number obtained should be between 1125 and 1875. Record display reading.
- 3.2.14 Repeat 40 uL addition of 2000 ppm KHP at least three more times for a total of four analyses. Repeating the measure several times is important because the final calibration is based on the average of the analyses; the more calibration runs, the better the average.
- 3.2.15 When four calibration runs have been successfully completed, initiate (press) the CALIB button (circled in figure below) and keep depressed for at least one full second. The detector will then calculate a final output number and this number will be digitally displayed. Record value. When the CALIB light comes on, the DC-85A is calibrated.



- 3.2.16 Following calibration, Inject 40 uL of 2000 ppm KHP on the sample boat through the syringe port, close syringe port and let system equilibrate for one minute.
- 3.2.17 Initiate START and immediately advance boat into combustion chamber.
- 3.2.18 Note digital display reading in ppm scale (should be close to 2000 ppm). If it is not within 200 ppm of 2000 ppm (10% deviation) then the calibration procedure must be repeated.

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#### **3.3 Sample Analysis**

- 3.3.1 Toggle combustion unit BOAT GAS ON switch to off.
- 3.3.2 Open the sample boat chamber door and remove boat.
- 3.3.3 Weigh out 1-100 mg of sample directly into a boat using the mg scale balance. Record weight of sample. The more organic carbon content in the sample, the less sample must be weighed into the boat and vice-versa. Combusting too much sample volume will result in detector saturation. See figure 1 below for an approximate sample weight based on sample carbon content; the recommended minimum sample size is 9 mg. However, the figure below is only an approximation and sample weights less than 9 mg may have to be used for samples with high carbon content.

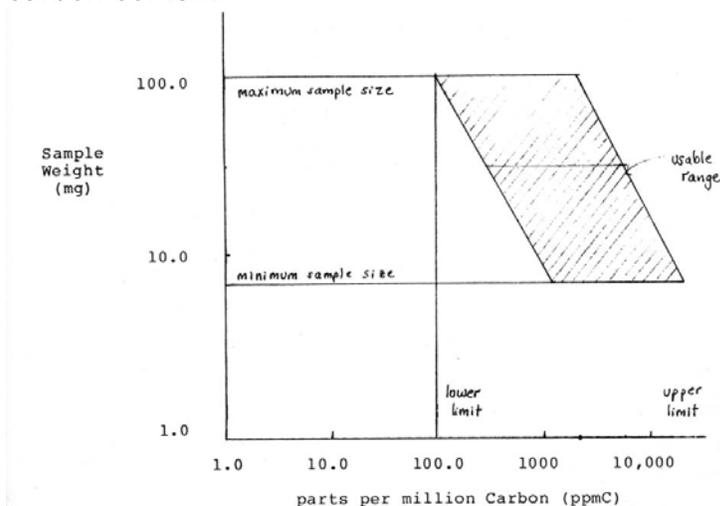


Figure 1. The influence of carbon content on sample weights.

- 3.3.4 Insert boat into sample chamber and seal the door.



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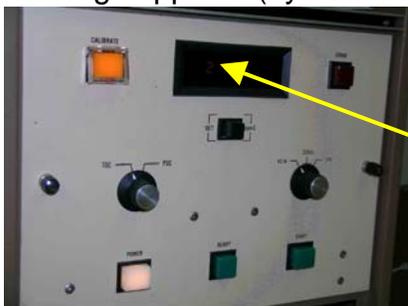
3.3.5 Toggle combustion unit BOAT GAS ON switch to on. Observe gas flow through acid sparge wash.

3.3.6 Allow two minutes for CO<sub>2</sub> introduced to sample chamber, when door was open, to go through detector.

3.3.7 Press START on detector and immediately advance the sample into the combustion furnace. Make sure detector switch (circled in image below) is on ppm.



3.3.8 Wait for the analysis to finish and confirm by audible sound. Note panel reading in ppm C (by volume) and record.



3.3.9 Retract sample boat from combustion chamber to the sample introduction chamber.

3.3.10 Toggle combustion unit BOAT GAS ON switch to off.

3.3.11 Let sample boat cool for at least one minute.

3.3.12 Open sample chamber door and remove boat.

3.3.13 Wash sample boat with water.

3.3.14 Reintroduce boat to sample chamber and seal sample chamber door.

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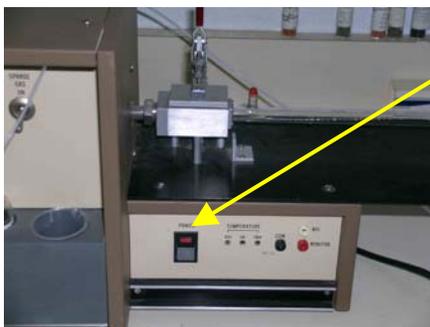
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- 3.3.15 Move sample boat into combustion chamber for one minute. Do not initiate any buttons on detector and do not toggle BOAT GAS ON switch to on. The purpose of this step is to clean the boat so it can hold the next sample.
- 3.3.16 After one minute, retract sample boat from combustion chamber to the sample introduction chamber.
- 3.3.17 Let sample boat cool for at least one minute.
- 3.3.18 Open sample chamber door and remove boat.
- 3.3.19 Repeat from step 3.3.3. At least three replicates of the same soil sample should be run so that statistical analysis can be used to determine the mean and variation in the data (i.e., standard deviation).

### **3.4 Instrument Shut-down**

- 3.4.1 After sample analysis is complete, toggle combustion unit BOAT GAS ON switch to off.
- 3.4.2 Power off combustion unit.



- 3.4.3 Do not turn off detectors. Detector POWER button and infrared gas analyzer power light should be continuously illuminated (circled in images below).



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- 3.4.4 Remove tube from acid sparge gas unit to prevent moisture backflow into detector and combustion chamber.



- 3.4.5 Turn off gas.

## 4.0 CALCULATIONS

The following example calculation can be used to calculate carbon in terms of weight.

Information required;

Amount of 2000 KHP standard used (40 uL)

Amount sample used = 35 mg

Detector response for 35 mg (0.035 g) sample = 1450 ppm C by volume

Detector average response for 40 uL KHP standard obtained in section 3.2.18  
(here assumed to equal 2000 ppmv)

$40 \text{ uL standard} \times 2000 \text{ ng C/uL} = 80 \text{ ug C}$

$80 \text{ ug C} / 2000 \text{ ppm C by volume} = A / 1450 \text{ ppm C by volume}$  (solve for A)

$A = (80 \text{ ug C} / 2000 \text{ ppm C by volume}) \times 1450 \text{ ppm C by volume}$

$A = 58 \text{ ug C}$

$58 \text{ ug C} / 0.035 \text{ g} = 1657 \text{ ug C/g soil} = 1657 \text{ ppm.}$

## 5.0 REMEDIAL ACTION IN CASE OF MALFUNCTION

Consult the instrument manual or contact Amrith Gunasekara  
(amrithsrg@yahoo.com).

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#### **6.0 SAFETY**

Good laboratory practices should be used at all times during instrumental operation. Wearing gloves and eye protection during experimental analysis is recommended. Extreme care should be taken when handling any acids as part of this instrumental procedure. Use hood space when using concentrated acids. Gas cylinders must be properly secured.

#### **7.0 REPORTING REQUIREMENTS**

Record the date, user, samples analyzed, and instrumental response for the 2000 ppm KHP standard in the instrument log book.

#### **8.0 REFERENCES**

DC-85A TOC Analyzer Systems Manual. 1<sup>st</sup> Ed. Rosemont Analytical Division, Dohrmann, Santa Clara, CA. Part no. 915-239.