

**84-013 TOTAL CARBON, LECO induction furnace****1. Application**

- 1.1 Several quantitative methods are available for the determination of total C in soils. The use of a LECO induction furnace provides an accurate, fast and convenient method of analysing for total C. For samples containing carbonates, organic C can be determined by subtracting carbonate C from total C. The procedures differ depending on the age and model of the instrument and the CO<sub>2</sub> measuring system involved. The following method is suitable for the model 577-100 carbon analyser and requires approximately five minutes per sample. The newer models permit more rapid determinations of total C.

**2. Apparatus**

- 2.1 Leco induction furnace equipped with purifying train, and carbon determinator.
- 2.2 A supply of spare parts that require replacement on a regular basis (combustion tubes, filter cloths, etc)
- 2.3 Crucibles

**3. Reagents**

- 3.1 Red levelling solution - Dissolve 0.4g of methyl orange in 200 mL distilled water, boil, cool to room temperature, and filter. Dilute this solution to 800 mL and add 40 mL concentrated H<sub>2</sub>SO<sub>4</sub>. To this solution add 2 mL LECONAL wetting agent. This solution will last indefinitely and need be changed only when dirty or for some other obvious reason. This solution is poured into the levelling bottle and the bottom of the meniscus in the calibrated stem is adjusted to the zero point by either adding or removing red solution.
- 3.2 Caustic solution - Dissolve 450 g of KOH in 900 mL distilled water. Allow solution to cool to room temperature and pour all of the solution into the absorption vessel.
- 3.3 Manganese dioxide is used in the sulphur trap to absorb sulphur gases which interfere with the determination.
- 3.4 Concentrated sulphuric acid, drierite, ascarite, and glass wool are required to prepare the purifying train.
- 3.5 Iron chip and tin metal accelerators.

#### 4. Procedure

##### 4.1 Blanking the apparatus and testing for leaks

- 4.1.1 Plug in the furnace 15 minutes or so before analyzing a sample. This allows the catalyst furnace, which converts CO to CO<sub>2</sub>, to warm up. Turn on the filament switch (green pilot light) 5 minutes before blanking the apparatus.
- 4.1.2 Turn on the O<sub>2</sub> tank to about 4 psi and close the needle valve on the purifying train. DO NOT apply too much force to the needle valve. Turn the buret stopcock to the exhaust position (down) and raise the levelling bottle to the upper cup until the red solution fills the buret and seats the float valve. Seat the valve SLOWLY BY PINCHING the tubing BEFORE the liquid reaches the valve. This keeps the solution out of the stopcock.
- 4.1.3 Turn the buret stopcock to the furnace position (left) and place a crucible on the pedestal of the raising mechanism. The crucible should contain 1/2 scoop of tin accelerator and one scoop of iron chip accelerator.
- 4.1.4 Open the needle valve and adjust the O<sub>2</sub> flow to 0.5 liters/minute. Close the raising mechanism on the furnace and set the levelling bottle on the base. Lay the tubing on the bench.
- 4.1.5 When the red solution is about 2/3 of the way down the calibrated stem of the buret, open the raising mechanism and turn the stopcock to the exhaust position (down).
- 4.1.6 Allow the buret to drain (about 30 seconds, use a stopwatch). Check the drainage time periodically by turning the stopcock to the lock position (right) and noting if the red fluid rises above zero. If the red liquid is not at zero after drainage, add or remove some red solution from the levelling bottle.
- 4.1.7 Turn the buret stopcock to the caustic position (up) BEFORE (to avoid loss of CO<sub>2</sub>) raising the levelling bottle to the upper cup and above to remove all the gas from the buret. Lower the levelling bottle below the table level until the KOH solution in the absorption vessel rises and seats the float valve (this can be seen through the window).
- 4.1.8 Turn the buret stopcock to the lock position (right) and then place the levelling bottle on the base and lay the tubing on the bench. Allow the buret to drain for 60 seconds and read. It should be 0. Frequently there is a blank reading of .04 or so after the first flushing with O<sub>2</sub>. If this occurs, repeat the blanking procedure.

4.1.9 Test for leaks by filling the buret with the red solution and letting  $O_2$  enter until the red solution reaches some level on the calibrated stem of the buret. Turn off the needle valve. If the solution continues to fall, a leak is indicated. Progressively pinch off sections of tubing between the needle valve and the carbon determinator to find at what point the solution stops falling. The leak is then BEHIND the last pinched off point.

#### 4.2 Selection of sample weight

4.2.1 Sample weight is based upon an estimate of the C present in the soil. The maximum C that can be determined by this model is 15 mg. The samples should be ground to 35 mesh or finer prior to analysis.

4.2.2 A guideline to weight (nearest mg) for estimated C is

C 1%	Weigh 500 mg
C 1-4%	Weigh 250 mg
C 4-8%	Weigh 125 mg
C 8-20%	Weigh 50 mg
C 20-50%	Weigh 25 mg

#### 4.3 Analyzing a sample

4.3.1 Add one scoop of iron chip accelerator, 1/2 scoop of tin accelerator. Include standards and blanks with each set of samples. MAKE SURE THAT THE ACCELERATOR COVERS ALL OF THE SAMPLE. The amount of accelerator required may vary depending upon the furnace.

4.3.2 Repeat steps 4.1.2 to 4.1.8 inclusive. The  $O_2$  flow rate should be 0.5 L/min. The meter above the filament switch should read over 300 milliamperes at the hottest stage of combustion and it should read this level before the red solution is half way down the bulb of the buret. If this does not occur, the results will be low as combustion and sweeping will be incomplete. It is important to lower the raising mechanism before the red fluid reaches the bottom of the buret.

4.3.3 Read the buret by raising the levelling bottle until the liquid in the side arm reading tube is at exactly the same level as the liquid in the buret. Read the right scale of the buret. The right scale is calibrated in % C for 0.250 g samples; the left scale for 1.00 g samples. By weighing the above recommended amounts, calculations of % C are simplified.

4.3.4 When the instrument is not in use, pull out the furnace plug. Fill the buret with red solution to just below the valve. DO NOT SEAT the valve. Turn the stopcock to the lock position (right) and place the levelling bottle on the base.

**NOTES:**

- (1) The combustion tube must be cleaned about four times each day. A brass brush is supplied and the vacuum cleaner is quite helpful. TURN OFF the filament switch before cleaning. A partly plugged tube will result in low temperature and incomplete combustion.
- (2) Replace dust filter cloth periodically. Clean it with a vacuum cleaner at least twice each day.
- (3) Replace MnO<sub>2</sub> periodically when samples high in S are being run.
- (4) The temperature in the tube during combustion is directly related to the amount of accelerator added and the O<sub>2</sub> flow rate. If the previously mentioned values are used, one will seldom have any trouble using this instrument. If the meter above the filament switch should reach 500 milliamps and hold for more than three seconds, IMMEDIATELY lower the raising mechanism. Repeat the sample using less accelerator.

**5. Calculations**

- 5.1 Corrections are made for temperature and pressure by reading the thermometer in the buret and reading a barometer (once a day is usually sufficient). The correction is made with the aid of the factor chart supplied.
- 5.2 For the following conditions: sample weight 0.200 g, correction factor 0.950 and a final reading of 2.40
 
$$\% C = \frac{0.250 \times 0.950 \times 2.40}{0.200} = 2.85$$

**6. Precision**

- 6.1 In the LRRRI analytical service lab the coefficients of variations at carbon levels of 11.2% and 3.3% were 1.8% and 3.1% respectively.

**7. References**

- 7.1 Bremner, J.M. and Tabatabai, M.A. 1971. Use of automated combustion techniques for total carbon, total nitrogen and total sulphur analysis of soils. In Instrumental methods for analysis of soils and plant tissue. p. 1-16. L.M. Walsh, ed. Soil Sci. Soc. Am. Proc., Madison, Wisconsin.
- 7.2 Tabatabai, M.A. and Bremner, J.M. 1970. Use of the Leco automatic 70-second carbon analyzer for total carbon analysis of soils. Soil Sci. Soc. Am. Proc. 34, 608-610.