separate, drain off and discard the lower aqueous layer. Drain the toluene layer into a 50-ml. beaker. Determine the absorbance of this solution in a 10-cm. cell (Pyrocell Mfg. Co., Cell S25-220, 10 mm. in O.D., capacity about 4.5 ml.) at 420 mμ using a Beckman DU spectrophotometer with toluene in the reference cell.

Prepare the calibration curve (0 to 6 μg. of selenium) as follows:

Pipet 0, 1, 2, 4, and 6 ml. of a standard sodium selenite solution (1 μg. of selenium per ml.) into a series of 200-ml. beakers and make up with water to a total volume of 150 ml. Proceed as in the analysis of samples beginning with the addition of hydroxylamine solution. The calibration curve follows Beer's law from 0 to 6 μg. of selenium; the 6 μg. standard corresponds to a reading of about 25% transmission.

Results and Discussion

The method was used to recover selenium added as sodium selenite from oats. The recoveries are shown below:

Added, P.P.M.	Recovery, %
0.5	95.0
0.5	122.0
0.5	108.0
2.0	87.5
2.0	80.5
2.0	89.0

In recovery studies, the oat pellet is fortified after it has been placed in the platinum holder. The 2.0-p.p.m. level is attained by pipetting 0.1 ml. of a sodium selenite solution (20 μg. of selenium per ml.) onto a 1-gram pellet of ground oats. This volume of liquid does not prevent combustion and the sample may be burned immediately without drying. The 0.5-p.p.m. level is attained by pipetting 0.1 ml. of a 5 μg. per ml. selenium solution onto each of two 1-gram pellets which are burned successively in the flask; the same 100 ml. of absorbing solution is used for each burning.

The method was used to determine the selenium content of three oat samples from South Dakota in which selenium had been determined by the method of Klein (*4*). The results are given below:

	Selenium, P.P.M.	
Code No.	Klein method	Flask combustion
1	0.9	0.86
2	2.1	2.06
3	4.7	4.23

The check value for six analyses averaged 0.10 p.p.m. of selenium. The method will detect 0.25 p.p.m. of selenium in a 2-gram oat sample. The colorimetric procedure is an adaptation of that of Cheng (*1*). The selenium stock solution (50 μg. of selenium per ml.) is stable for about 1 week. After this, precipitation takes place and the solution must be freshly prepared. The aqueous solution of 3,3′-diaminobenzidine darkens rapidly during storage. The rate of darkening can be greatly reduced by purging the headspace of the container in which the reagent is stored with nitrogen before letting it stand for prolonged periods. Store the reagent in a refrigerator. When measuring absorbance, rinse the cuvet with about 1 to 2 ml. of the solution whose density is to be determined.

This method may be applicable to the determination of selenium in other biological material, since the flask has been used to burn cabbage, cherries, onions, and potatoes prior to determination of arsenic, bromine, chlorine, and manganese (*3*), and to burn organic soils for determination of organic carbon content.

Acknowledgment

The authors thank Oscar E. Olson for the oats and selenium analyses.

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Received for review February 20, 1961. Accepted May 31, 1961. Division of Agricultural and Food Chemistry, 140th Meeting, ACS, Chicago, Ill., September 1961.

SOIL EFFECTS ON PESTICIDES

Determination of Carbon in Organic Soils by Oxygen Flask Combustion

WALTER H. GUTENMANN and DONALD J. LISK

Entomology Department, Cornell University, Ithaca, N. Y.

A method is described for determining organic carbon in organic soils by match-produced combustion of a pelleted sample of dry soil in an oxygen-filled flask. Excellent agreement is obtained by comparing carbon analysis by this method and the electric furnace combustion procedures. Soils containing 14% of organic carbon burn spontaneously. The method is simple, inexpensive, and requires about 30 minutes per sample.

Dry combustion is considered the most accurate method for determination of organic carbon in soils. The most common of these methods employs an electric furnace in which the soil is burned in a stream of oxygen in the presence of one or more catalysts. Carbon dioxide may be absorbed with Ascarite and weighed, collected for Orsat analysis, or measured by other means. The equipment, however, is expensive: difficulties arise from leaks in the system and plugging of absorbers.

In the course of a study of the disappearance of pesticides in soils as related to their organic matter content, a much simpler method was devised. In this method, the dry soil sample is pelleted and burned in an oxygen-filled flask. Evolved carbon dioxide is absorbed in sodium hydroxide, magnetically stirred, and contained in the flask. Carbon is determined as bicarbonate by titration of the base with standard acid between the phenolphthalein and methyl orange end points.

Procedure

Weigh approximately 0.5 gram of well mixed, dry soil accurately and place in a Paar pellet press having a 0.5-inch diameter bore. Pelletize and transfer the resulting disk of soil gently into the platinum holder of the 5-liter combustion flask (*1*) using forceps. Place a fuse (1 mm. wide and 8 cm. long) cut from Whatman No. 42 filter paper in the basket touching the sample. Pipet exactly 100 ml. of approximately 2*N* sodium hydroxide in the flask for absorption of carbon dioxide. Place the magnetic stirring bar in the flask and purge the flask with oxygen. Ignite the fuse and gently insert the platinum holder in the flask. After combustion, allow the magnetic stirrer to mix and splash the solution vigorously inside the flask for 10 minutes.

Transfer exactly 4 ml. of the absorbing solution to a 125-ml. Erlenmeyer flask. Add about 5 ml. of water and a few drops of 0.1% phenolphthalein in methanol. Titrate the solution with approximately 0.25*N* hydrochloric acid

until the end point is approached (pink color begins to fade). Continue titration with approximately 0.1N hydrochloric acid until the last trace of pink color disappears. Add 2 drops of 0.1% of methyl orange in methanol to the solution and titrate to the methyl orange end point with standard 0.1N hydrochloric acid.

Calculate the per cent of carbon in the soil sample as follows:

$$\%C = \frac{(V - 0.3)(N)(12.01)(25)(100)}{500}$$

where V and N are the volume in milliliters and normality, respectively, of the standard acid required for the titration. The fraction 0.3 is the volume (milliliters) of 0.1N acid needed to titrate 4 ml. of the absorbing solution when the fuse alone is burned.

Results and Discussion

The method was used to determine the carbon content of 10 organic soils covering a wide range of organic matter content. Results of analyses of several soils by flask combustion and the electric furnace procedure as described by Piper (*2*) are shown below:

Soil Code No.	Carbon, % Electric furnace	Flask combustion
1	34.2	33.6
2	15.1	14.8
4	35.9	36.7
5	34.9	35.0
6	26.6	26.6
9	43.8	42.6, 43.1, 42.3
10	33.8	31.6
13	40.4	40.7
14	32.0	31.4
16	22.0	21.6
20	42.6, 41.6	41.5

Flask combustion enables determination of the carbon content of organic soils with little investment in special equipment. The technique is simple, rapid, and not subject to many of the errors common to other methods. Care is required when handling the soil pellet with forceps. Some soils do not pellet well: more pressure is required during the pelletizing operation. Also place the platinum holder in the flask gently to avoid jarring loose soil fragments before they ignite. Further work is in progress to make the technique applicable to the determination of the organic carbon content of mineral soils.

Acknowledgment

The authors thank T. Greweling for furnishing the soils and the electric furnace combustion analyses.

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Received for review February 17, 1961. Accepted May 31, 1961. Division of Agricultural and Food Chemistry, 140th Meeting, ACS, Chicago, Ill., September 1961.