Analytical Laboratory, Clairol, Inc., Stamford, Conn. 06904, U. S. A.

Semimicro Determination of Carbon in Organic Compounds by Closed Flask Combustion: Evaluation of the Method

By

Stanley Goldstein

(Received February 2, revised April 19, 1967)

Introduction

The problem is to develop a method for carbon determination that involves simple equipment on hand when the microcombustion train is not available. The close flask combustion method was found to be suitable. This laboratory finds the results in three significant figures to be sufficient. A semi-micro balance, weighing to five decimal places, ensures the accuracy of the weighing to 0.1 mg, and it has been found best to weigh in the range of 15 to 20 mg.

A review by Macdonald\(^1\) and a report by Ingram\(^2\) have stated that low results were due to incomplete combustion, some of the sample being vaporized before ignition occurred. Even when ignition is satisfactory, there are often smears of carbon left on the support. An approach to the problem has been described by Cheng and Smullin\(^3\). The sample was placed in a small porcelain boat and closely covered with a fine piece of platinum gauze; the boat was then inserted into the platinum heating coil. Visible amounts of unburnt carbon showed on the boat, however, possibly due to the larger sample size used.

Use of a high melting glass filter paper in this laboratory has given results more satisfactory than those obtained by using the porcelain boat. An improvement of the efficiency of the ignition was carried out by filling the flask twice with oxygen. In attempts to improve the combustion for more difficult oxidizable compounds, such as polyhalogenated types, it was found that a thin platinum helical coil bunched closely together as the sample holder gave improved results. However, it was found quite difficult to clean the high melting fused glass from the coil without taking off the coil, scraping it, rewinding and attaching the coil again.
prior used saw-tooth ignition wire gives compatible results with the helical type on most compounds and is preferred.

In the double indicator method\(^4, 5\) for determining carbon by the half titration of \(\text{Na}_2\text{CO}_3\), difficulties were found at distinguishing both of the end points. At the first end point, when phenolphthalein was used as an indicator, the end point faded too rapidly and gave inconsistent results. Using a 0.1% solution of a mixture of thymol blue and cresol red as the indicator it was found that the violet end point lasted over too wide a range and that the first appearance of the violet was gradual. A fairly good end point was developed by using this same mixed indicator, and titrating to a colorless slightly yellow end point. The over-titration was found from the theoretical first end point using pure \(\text{Na}_2\text{CO}_3\) and a multiplication factor was determined. This factor was found to be constant over a range of 65 ml titration with 0.03 \(N\) HCl in arriving at the amount of overtitration. It was also found that a closed flask and slow titration with a magnetic stirrer for the first end point gave consistent results. This cut to a negligible amount \(\text{CO}_2\) lost during the titration through local excesses of HCl.

The second end point was found to give somewhat inconsistent results using a mixture of methyl red and methylene blue as the indicator. The green to orange end point was over too wide a range (about 0.7 ml). Also the disappearance of the orange and the appearance of a pink color wasn’t sharp. This was improved by adding 2 ml excess HCl, boiling for 5 minutes, cooling, titrating to a green end point with \(\text{NaOH}\) and then titrating back to a pink end point with HCl. The end points were sharp but a greater amount of inherent error was created by the increased buret readings. Extra time was also consumed and added calculations were needed making the overall time for the double indicator method about 50 minutes from start of weighing to final calculation. By comparison, the \(\text{BaCO}_3\) separation method which requires about an hour has less inherent error and the single end point involved is excellent.

**Experimental**

**Equipment**

Carbon Combustion Head, from F & M Scientific Corp.
Fritted Glass Filter Tube, 1” diameter, medium porosity, from Arthur H. Thomas Co.
Wash Bottle, inlet and outlet tube, with 29/42 joint and connecting stopcock.
Magnetic Stirrer.