Improved Microcombustion Technique for Carbon-Hydrogen Estimation in Perhydro-polycyclic Compounds. I
Steroids and Triterpenoids*

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(Received November 17, 1967)

Introduction

Polycyclic compounds are known to give low carbon values by the conventional microanalytical method. During routine analysis over the last 15 years it has been found that perhydropolycyclic compounds, particularly those having angular methyl groups, give erroneous results using copper oxide or copper oxide-lead chromate filling.

Analytical data of steroids, triterpenoids and similar compounds of natural origin reported in the literature frequently show more than 0.5 per cent deviation in carbon values. In general the values are less for carbon than the calculated ones. It was, therefore, of interest to improve upon the existing methods and the present communication deals with such an attempt.

Experimental

Equipment and Reagents

Microcombustion tube, Vycor, with side arm, from Arthur H. Thomas Co., Philadelphia, U. S. A.
Asbestos, platinised 30%, Arthur H. Thomas Co.
Silver metal wool for microchemical procedures, Arthur H. Thomas Co.

* Communication No. 1223 from the Central Drug Research Institute, Lucknow, India.
Lead dioxide, C.P., from Coleman & Bell Co., Norwood, U.S.A. 

*Procedure*

*Procedure A.* The combustions were carried out on a microcombustion assembly for carbon-hydrogen estimation [Arthur H. Thomas Co., Philadelphia, U.S.A., Catalogue No. 6447-C (1965), p. 693]. The "Combination filling" consisted of a silver plug at the capillary end followed by a lead dioxide layer 3 cm long, a 4.5 cm layer of silver metal wool, a 11.5 cm layer of copper oxide and a 3-cm roll of platinum gauze (the different layers were separated from each other by a pad of 1 mm thick platinized asbestos) and the combustion furnace and kept at 700 ± 25°. The rate of flow of oxygen was regulated at 5 ml per minute and the microboat was kept at a distance of 5 cm from the stationary furnace. The temperature of combustion in the electric sample heater was 900 ± 25°. The analysis was carried out as described by *Niederl* and *Niederl* 12.

*Procedure B.* The "simple band filling" was used; it consisted of three layers of copper oxide alternated with two layers of platinized asbestos each 2.5 cm long, followed by a 0.5-cm layer of silver wool and a 2-cm layer of platinum tetrahedra preceding the bands. Heraeus' Microcombustion Furnace (Type F, No. 3-14) was used in which it was possible to attain a temperature up to 1200°. The temperature of the stationary furnace (Langbrenner) was maintained at 900 ± 25° and the rate of flow of oxygen was regulated at 4 to 5 ml per minute. The sample heater (Kurzbrenner) was driven at 15 mm per minute and its temperature was kept over 1000° by adjusting the needle of the "Indicator Gauge" at mark 8. Other details of the experiment were exactly the same as in Procedure A.

*Results and Discussion*

The investigation was carried out by systematically varying the rate of flow of oxygen, time of combustion, range of temperature and the nature of filling, one at a time. It has been observed that the nature of the filling and temperature of the combustion played a significant role for better analytical data. The substances combusted on "simple band filling" at the conventional temperature 700 ± 25° did not yield good results. The results improved significantly (see Table I) by raising the temperature to 900 ± 25°. Temperatures higher than 900 ± 25° created practical difficulties due to clinkering of the filling. Contrastingly, the "combination filling" used in Procedure A did not yield better analytical values even by raising the temperature to 900 ± 25°. Table I shows the analytical data obtained from the "combination filling" (Column A)