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GRAVIMETRIC METHOD FOR MICRO DETERMINATION OF MOLYBDENUM

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Introduction.—As the necessity arose for determining molybdenum micro-analytically in organic compounds, it has been found that Pregl's method for the quantitative micro determination of metals in organic substances[1] can be used in principle for the gravimetric micro determination of molybdenum as molybdenum trioxide.

Apparatus.—While the apparatus and manipulations in the course of the analysis remained the same, the following changes were necessary in the analytical procedure to give satisfactory results.

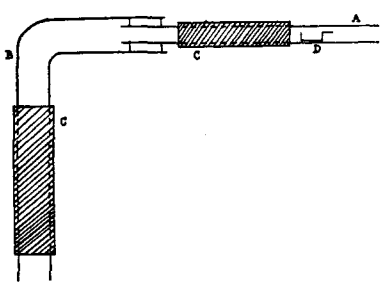

Fig. 1.—Micro muffle, according to Pregl. A, combustion tube; B, bent tube; C, wire gauze; D, combustion boat.

Method.—Three to five mg. of the substance is weighed in a micro porcelain combustion boat (a platinum boat cannot be used since partial reduction of the molybdenum trioxide may occur) on a micro-analytical balance (Christian Becker micro-analytical balance proved to be satisfactory). To this a drop of concentrated nitric acid is added. The boat is then brought into the combustion tube of the micro muffle (as shown in the diagram above) and heating begun over a small wire gauze with a Bunsen burner at about 30 mm. distance from the combustion boat.

Within a few minutes the nitric acid and the fumes of the oxides of nitrogen will be driven off and the wire gauze and flame are moved directly under the boat. After heating the boat in this manner for about five minutes, the wire gauze is removed and the heating continued at the same spot under the combustion boat for exactly five minutes longer. Since the molybdenum trioxide is volatile at temperatures above 450°,[2] the temperature of the flame is important. The flame should be non-luminous and so adjusted that it burns quietly, with an outer cone of about 7 cm. and an inner cone of about 3 cm. in length.

The material in the combustion boat should now appear yellow while

[1] F. Pregl, "Quantitative Organic Analysis," P. Blakiston's Son & Co., Philadelphia, **1924.**

[2] W. D. Treadwell, *Z. Elektrochem.*, **19,** 219 (1913); Brinton and Stoppel, THIS JOURNAL, **46,** 2454 (1924).

hot and grayish-white when cold. At this point the operation is finished and the boat containing the molybdenum trioxide is weighed.

This method, which requires only a short time for completion, is applicable for organic and inorganic substances provided they contain no other non-combustible or non-volatile constituents.

Results.—Some of the results obtained are as follows.

Molybdic Acid.	56.67% of Molybdenum.		Macro Analysis.	
Sample taken, mg.	3.105	2.275	2.442	3.250
MoO_3, mg.	2.642	1.934	2.075	2.758
Molybdenum found, %	56.71	56.68	56.62	56.58
Ammonium Molybdate.	53.87% of Molybdenum.		Macro Analysis.	
Sample taken, mg.	4.155	5.675	3.786	2.667
MoO_3, mg.	3.339	4.576	3.045	2.144
Molybdenum found, %	53.57	53.75	53.53	53.59
Histamine Molybdate.	Calcd., 48.24% of Molybdenum.			
Sample taken, mg.	3.936	5.017	4.406	
MoO_3, mg.	2.852	3.664	3.202	
Molybdenum found, %	48.30	48.60	48.45	

Summary

A rapid gravimetric micro method for the quantitative determination of molybdenum in organic compounds has been described.

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[CONTRIBUTION FROM THE DEPARTMENT OF PHYSICS, UNIVERSITY OF MICHIGAN]

I. PURIFICATION OF METHYL FLUORIDE
II. QUANTITATIVE GAS ANALYSIS BY HIGH DISPERSION INFRA-RED SPECTROSCOPY

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Besides the interest in a ready method for preparing pure methyl fluoride, it is thought that this investigation will be of interest inasmuch as an application of infra-red spectroscopy is made in determining the purity of an organic compound with greater accuracy than is available through the usual chemical methods.

The best method for the preparation of methyl fluoride which a search of the literature revealed was that of E. Moles and T. Batuecas.[1] They prepared silver fluoride by recrystallization and warmed with methyl iodide, purifying the vapors by repeated warming with silver fluoride. Their test of purity consisted of bringing the methyl fluoride gas to constant density by purification. They also tried the method of heating

[1] Moles and Batuecas, *J. chim. phys.*, **17**, 537 (1919).