AN AUTOMATIC MICROMUFFLE FOR THE DETERMINATION OF ASH IN CARBONACEOUS MATERIAL

By Robert Meyrowitz and C. J. Massoni

Trace Elements Investigations Report 412

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ABSTRACT

An automatic micromuffle for the determination of the ash content of small samples of carbonaceous material is comprised of a furnace of the radiant-heating type with a direct reading pyrometer for temperature control. This furnace can be assembled with a minimum of labor using parts available from scientific laboratory apparatus companies.

INTRODUCTION

A study of the mineralogy and geochemistry of carbonaceous rocks is being carried out as part of a program of research on the geochemistry of uranium that the Geological Survey is conducting on behalf of the Atomic Energy Commission. To help solve certain phases of the problem, an organic microanalytical laboratory has been organized. The purpose of this paper is to describe an automatic microcombustion apparatus which has been designed and used in this laboratory for the determination of the ash content of small amounts of carbonaceous materials. Very often the amount of material available for analysis is small, necessitating the use of micromethods. Norton, Royer, and Koegel (1940) have shown that there is a saving of time without any loss of precision when using the micro-technique for ash determinations.

Two automatic micromuffles have been described (Norton, Royer, and Koegel, 1940; Steyermark, 1951). These micromuffles have been made to order and require a great deal of machine-shop work in their construction. The micromuffle described by Norton, Royer, and Koegel (1940) has platinum heating coils and the temperature is regulated by varying the voltage using a variable autotransformer. Either one or two samples can be burned at one time; the furnace is stationary, and the tubes are drawn through the furnace. There are two rates of travel, the slowest speed being 2.5 cm per 10 min. Steyermark's micromuffle (1951) is heated by means of small nichrome wire and temperature regulation is by means of a variable autotransformer. Two samples can be burned at one time. In this apparatus the tubes are stationary and the furnace is moved. There is one rate of travel which is 2.5 cm per 10 min.

The advantage of the micromuffle described here is that it can be assembled with a minimum of labor using parts available from scientific laboratory apparatus companies.

DESCRIPTION

The micromuffle pictured in figure 1 consists of two parts: (1) a furnace drive, single speed (2.0 cm per 10 min.), electric, having adjustable limit stops to arrest automatically the advance of the furnace; Arthur H. Thomas Co., Philadelphia 5, Pa., Catalog No. 5683K, and (2) the moving, short-furnace section of an automatic, micro- and semimicro-combustion apparatus, Catalog No. S-21580, available from E. H. Sargent and Co., Chicago 30, Ill.

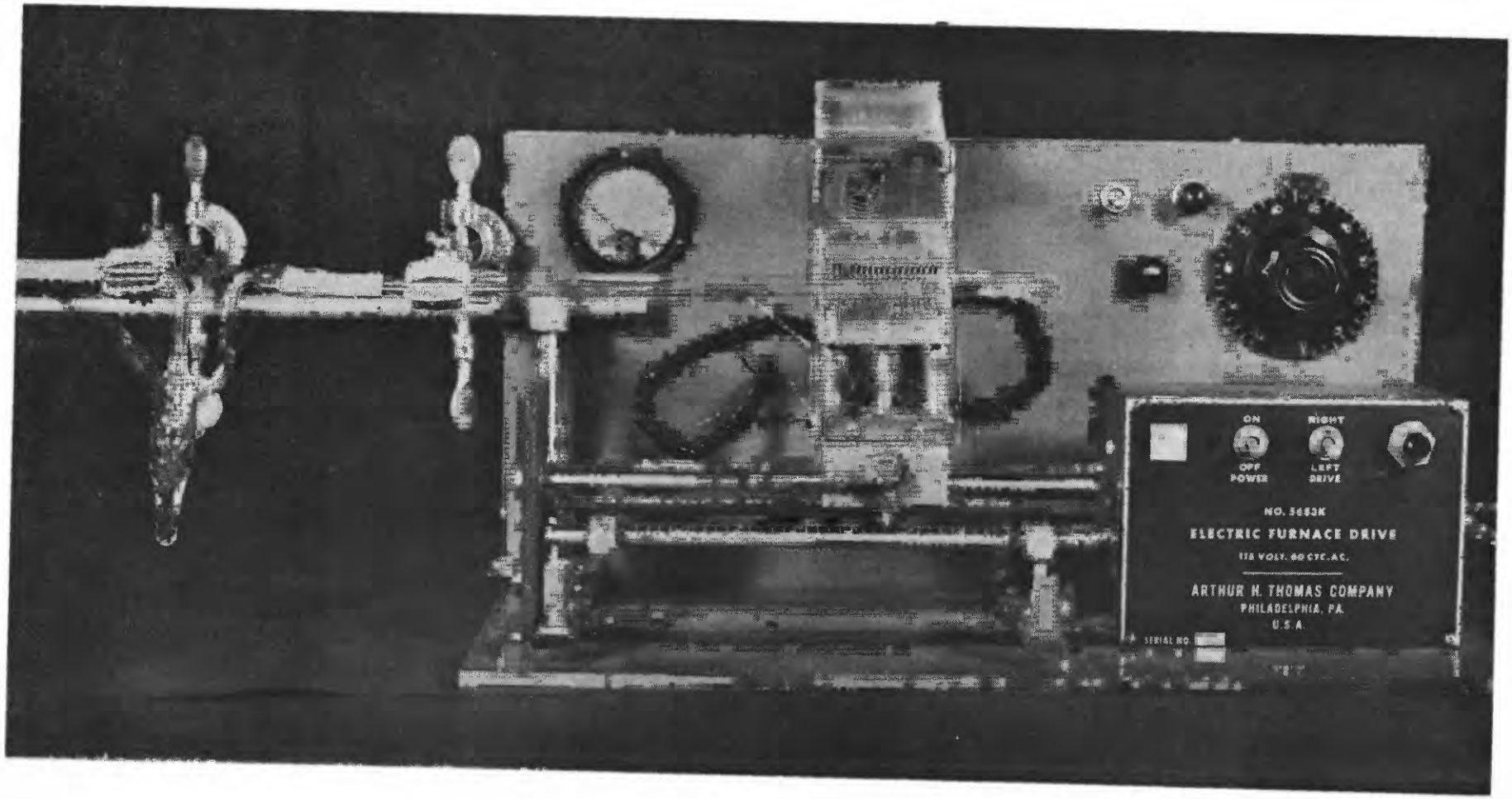

Figure 1.--Front view of apparatus.

This furnace is of the radiant-heating type, with hinged shell and open-sided heating elements and operates on 115 v, 60 cycle, a-c circuit. The furnace unit consists of six parts: (1) voltage reducing transformer, designed to operate on 100 v, 50/60 cycle, single phase service, the secondary is rated 10 v at 28 amps; (2) variable auto-transformer; (3) thermocouple and direct reading pyrometer for temperature control; (4) fuse; (5) pilot light; and (6) switch. The maximum temperature for continuous operation is 900 C.

The pyrometer is calibrated in the following manner. The thermocouple of a second pyrometer is inserted into the combustion tube until it reaches the position to be occupied by the combustion boat. While a stream of oxygen is flowing at the same rate to be used in the ash determination, the position of the thermocouple in the furnace is adjusted so that the reading of the pyrometer of the micromuffle is the same as that of the second pyrometer.

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Norton, A. R., Royer, G. L., and Koegel, R., 1940, Ash in organic compounds. Determination by microtechnique with automatic combustion: Anal. Chemistry, v. 12, p. 121-123.

Steyermark, Al, 1951, Quantitative organic microanalysis, Philadelphia, The Blakiston Company, p. 48-49.