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Note

AN APPARATUS FOR THERMOPHOTOMETRY

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Collins and Wendlandt [1] have previously described an apparatus to detect the ignition temperatures of various materials. It consisted of a Vycor tube furnace in which a photomultiplier (PMT) tube was attached to one end via a Pyrex glass window. Any light emission by the sample, which would be indicative of ignition, was detected by the PMT and recorded as a function of temperature on an X—Y plotter. Controlled static or flowing gaseous atmosphere could be maintained in the furnace chamber.

A more sensitive light detection system using a photon counter was employed in an apparatus described by Wynne and Wendlandt [2]. The PMT was mounted directly on the glass plate cover of the sample-holder enclosure of the Perkin-Elmer DSC-1B calorimeter. Using this arrangement, the DSC curve as well as the light emission curve could be recorded simultaneously.

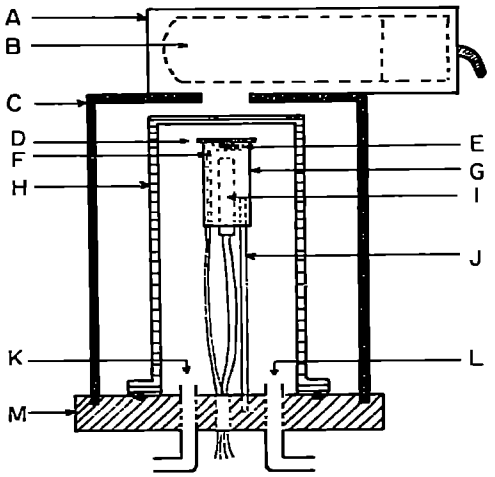
Neither of the above systems could be used very satisfactorily under reduced pressures (<760 mm Hg). A new apparatus, which incorporated this feature as well as others, was designed and constructed and is described here.

EXPERIMENTAL

Apparatus

The thermophotometry apparatus is illustrated schematically in Figs. 1 and 2.

The cylindrical furnace, G, 18 mm × 30 mm, was heated by a small stainless steel heater cartridge of 50-W capacity. It was supported by a ceramic rod 3.0 mm in diameter, which was attached to an aluminum base. The sample was contained in a 1.0 mm × 7.0 mm in diameter disposable aluminum crucible. To prevent spattering by the sample on the Pyrex glass enclosure, H, a circular microscope coverglass (15 mm) was placed on the top of the furnace. The temperature of the furnace and sample was detected by a chromel—alumel thermocouple, F, which was positioned as close as possible to the sample cavity in the furnace. The glass enclosure was covered by a light-tight metal cylinder, C, which contained the 1P28 PMT, B, and enclosure, A. The furnace chamber could be flooded with various gaseous atmospheres as well as operated at reduced pressures via the two inlet and outlet ports, K and L.



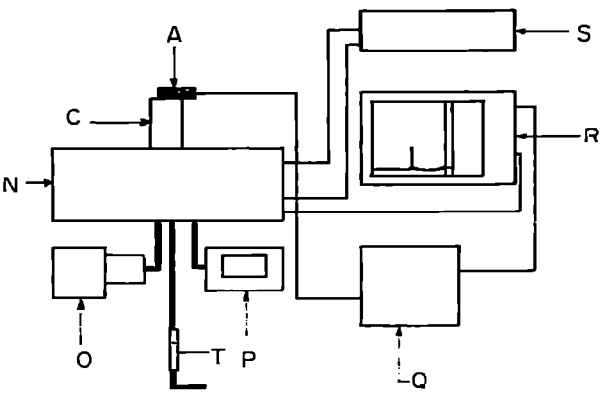


Fig. 1. Schematic diagram of furnace and enclosure. A, photomultiplier tube housing; B, photomultiplier tube (1P28); C, aluminum enclosure; D, cover glass; E, sample chamber; F, sample thermocouple; G, aluminum furnace; H, glass enclosure; I, heater cartridge; J, Ceramic support rod; K, gas inlet port; L, gas outlet port; M, aluminum base.

Fig. 2. Schematic diagram of apparatus. A, photomultiplier tube housing; C, furnace enclosure; N, cabinet; O, vacuum pump; P, digital manometer; Q, photometer; R, *X*—*Y* plotter; S, temperature programmer; T, flowmeter.

The PMT signal was processed by the photometer, Q (American Instrument Co., Silver Spring, MD) whose output was recorded on the *Y*-axis of an *X*—*Y* plotter, R (Model 7035B, Hewlett-Packard Corp., Pasadena, CA). Furnace temperature, as detected by thermocouple F, was recorded on the *X*-axis. A pump, O, was used to evacuate the furnace chamber whose pressure could be determined using the digital manometer, P (Model CD23, Validyne, Northridge, CA). A temperature programmer, S (Model TP-2000, Theall Eng. Co., Oxford, PA) was used to program the furnace at heating rates from 0.1 to 100.0°C min^{-1}.

Method

The sample is weighed out into aluminum crucibles (usually 5—10 mg) and placed in the sample cavity of the furnace, G. It is enclosed with the coverglass and then the glass enclosure, H, and furnace enclosure, C, are fitted to the metal base, M. The furnace chamber is flushed with a controlled gas or evacuated and operated at a reduced pressure. Temperature rise of the furnace is controlled by the temperature programmer, S; a heating rate of 13°C min^{-1} is usually employed. Output from the photometer, Q, is recorded as a function of furnace temperature on the *X*—*Y* plotter, R. The maximum temperature of the furnace is about 500°C.

RESULTS AND DISCUSSION

This apparatus has been used to determine the light emission of selected coordination compounds containing reducing and oxidizing groups. The results of this investigation are reported elsewhere [3].

ACKNOWLEDGEMENT

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REFERENCES

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