

Note

A controlled atmosphere sample holder for dynamic reflectance spectroscopy

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INTRODUCTION

The technique of dynamic reflectance spectroscopy (DRS) was first introduced by Wendlandt¹ in 1963. With this technique, the reflectance change of the sample is determined as a function of temperature. The usual procedure is to heat the sample and record the change in its diffuse reflectance, at a given wavelength, against temperature as the temperature is increased at a slow, linear rate. Numerous applications have been made of this technique to the field of coordination chemistry and other areas^{2,3}.

In the past, no attempt has been made to control the atmosphere surrounding the sample while it is being heated. A cover plate of Pyrex glass or quartz was employed but this was to prevent the sample from accidentally falling into the integrating sphere of the spectroreflectometer. In order to control the atmosphere surrounding the sample, a new sample holder was designed and constructed and it is described here.

EXPERIMENTAL

Sample holder

The sample holder is illustrated in Fig. 1, while the wiring diagram is given in Fig. 2.

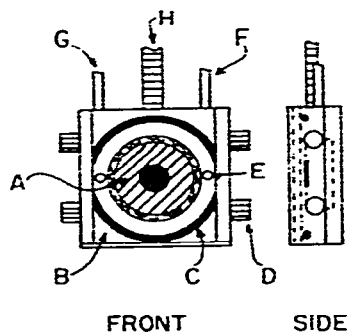


Fig. 1. Schematic illustration of the heated sample holder. (A) Silver sample block and heater; (B) glass or quartz cover plate; (C) "O" ring; (D) thumb screw (one of four); (E) gas inlet to sample chamber; (F) gas outlet tube; (G) gas inlet tube; (H) connecting cable.

The sample is contained in a 1-mm by 10-mm in diameter indentation machined in the surface of a silver heater block. The circular block is 25 mm in diameter and it is heated by two 2.6- Ω Nichrome wire heaters. It is contained in a enclosure, 55 mm square and 13 mm thick. The heater is thermally insulated from the main body of the sample holder by a thin layer of ceramic fiber insulation. The sample side of the holder is enclosed by a quartz plate, 50 mm on an edge by 2 mm thick, which is held firmly in place by two metal strips. Each metal strip is fastened to the holder by two thumb screws; they can easily be removed and hence the cover plate, to facilitate sample loading and removal. A gas tight seal between the cover plate and the sample holder is provided by a 44 mm internal diameter "O" ring. Two 0.125-inch diameter aluminum tubes, located at the top of the holder, are used to control the gas inlet and outlet to the sample chamber.

The circuit diagram (Fig. 2) for the sample holder shows the two 2.6- Ω heaters, connected in series, and two Chromel-Alumel thermocouples. One thermocouple is used to control the temperature programmer and also to provide the temperature readout on the Deltatherm III panel. The other is to detect the sample block temperature; an external recorder is connected to it through the adapter box.

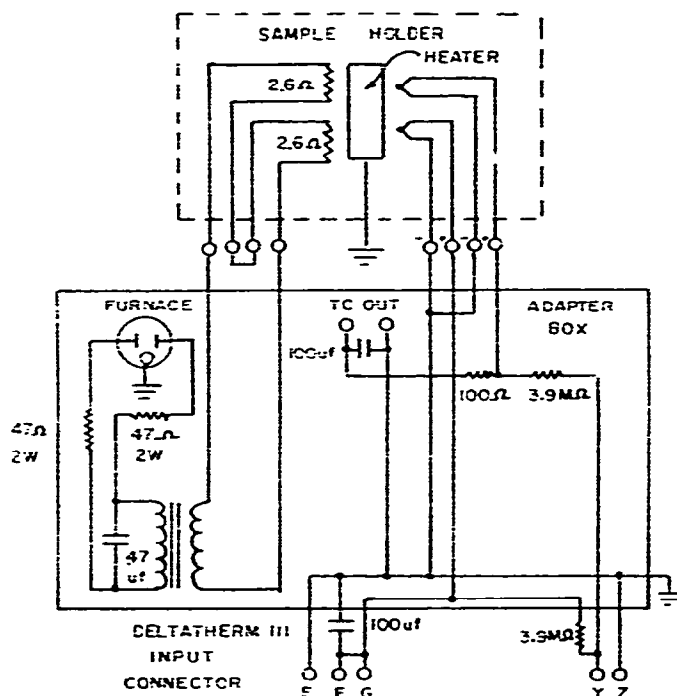


Fig. 2. Schematic circuit diagram of heated sample holder and adapter box.

The sample holder is connected by means of a multi-conductor wire cable to an adapter box which is connected to the Deltatherm III differential thermal analysis instrument. The DTA furnace and sample holder are removed from the instrument and the adapter box plugged into its place. In this manner, the temperature program-

mer and temperature readout of the instrument can be used with the sample holder. Sample heating rates from 0.625 to 10°C/min are possible. Or, by placing the instrument on the "Hold" position, the temperature of the sample may be kept constant for any length of time.

Operating procedure

A typical operating procedure for obtaining the DRS curve of a sample is as follows: the sample is placed as a thin layer on the indentation in the silver heater block. To provide a smooth surface, it is lightly tamped in place with the end of a small, smooth surfaced metal rod. By means of the two metal strips and thumb screws, the quartz cover is attached to the sample holder and the entire assembly placed on the sample port of the integrating sphere. Due to the weight of the sample holder, the spring loaded sample holding clip was replaced by a more robust metal clamp. The sample holder was positioned over the sample port and held in place by a small thumb screw on the clamp. The gas inlet and outlet hoses are attached to the holder and a stream of gas (usually nitrogen) is allowed to flow through the chamber. Dynamic or static atmospheres may be employed during the heating cycle. A small slot was cut at the top of the sample enclosure door to provide an opening for the control cable and gas hoses.

The sample heating rate, from 0.625 to 10°C/min, was adjusted on the Delta-therm III programmer and the heating cycle begun. The sample temperature could be read to $\pm 1^\circ\text{C}$ using the indicator on the instrument. The DRS curve was recorded on one channel of a multipoint strip-chart potentiometer recorder while the temperature was recorded on another channel.

To obtain diffuse reflectance curves at a constant temperature, the Delta-therm III programmer was placed on the hold position at any temperature desired and the reflectance curve recorded as a function of wavelength.

The sample holder is capable of operation from ambient room temperature to about 500°C. For use at temperatures above 300°C, an external cooling fan must be employed to keep the main body of the assembly at a reasonable temperature and also to prevent damage to the rubber "O" rings.

ACKNOWLEDGMENT

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