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Note

Temperature stabilization of a commercial photoionization detector

Performance evaluation

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The photoionization process is a temperature-independent phenomenon over the temperature range normally employed in gas chromatography (GC). However, a commercial photoionization detector (HNU Systems Model PI-52-02) shows short- and long-term baseline disturbances directly attributable to changes in detector temperature.

Because of their potential for high sensitivity, photoionization detectors are often used in trace and ultratrace analyses. This use requires the operation of the detector under conditions leading to a maximum signal-to-noise ratio (S/N). To maximize S/N, we found that it was necessary to control the temperature of the detector.

In this paper we describe a simple modification to the HNU photoionization detector to achieve thermostatic temperature control. In addition, we compare the capillary column performance of the stabilized detector with that of the flame ionization detector.

EXPERIMENTAL

A Perkin-Elmer 3920 gas chromatograph, equipped with a quartz injector/trap[1], was modified to accept a PI-52-02 HNU photoionization detector (HNU Systems, Newton, MA, U.S.A.). The 10.2-eV source was used throughout this work.

A 1:1 splitter, constructed from equal lengths of 0.25 mm I.D. fused-silica lines, was installed in the detector manifold section of the chromatograph. One branch of the splitter was connected to the photoionization detector, while the second was connected to the flame ionization detector. Make-up gas (helium) was added at the end of the column to maintain a flow-rate of *ca.* 20 ml/min through each detector.

Warm-up, short- and long-term stability studies were done after removing the detector from the chromatographic system. This was done to eliminate the possibility of introducing column- or chromatograph-related artifacts while performing these studies. A stream of clean helium (10 ml/min) was used to simulate the chromatographic effluent. A Model 7132A dual-channel recorder (Hewlett-Packard, Palo Alto, CA, U.S.A.) was used to display detector and thermocouple outputs.

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Thermostatic control of temperature was achieved by the use of an inexpensive proportional controller (RFL Industries, Booton, NJ, U.S.A.). The controller was mounted in a BUD minibox® (Newark Electronics, Chicago, IL, U.S.A.). A schematic diagram is shown in Fig. 1. Pin numbers 12 and 13 on the power cable connector of the HNU detector (Fig. 1B) were disconnected. Two leads were soldered to the wires leading to the detector heater. These leads were connected in series with a 200-W light bulb as shown in Fig. 1. A 1-MΩ, 61A1 glass thermistor probe (Victory Engineering, Springfield, NJ, U.S.A.) was used as the sensor. The sensor was inserted through the opening provided in the detector head for the cables carrying the polarizing voltage, collector signal and thermocouple output. The detector head was shielded from ambient air by a loosely-fitting plastic cover made by cutting a plastic bottle. The cover was wrapped with aluminum foil and grounded.

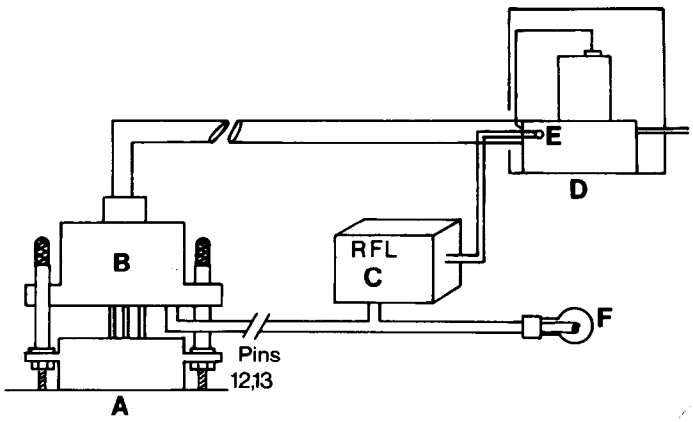

Fig. 1. Diagram of thermostatic controller for a HNU photoionization detector. A = Back panel of electrometer/power supply module; B = connector of detector power cable; C = RFL controller; D = photoionization detector (PI-52-02) housing; E = thermistor probe (1-MΩ); F = light bulb (200 W).

RESULTS AND DISCUSSION

The detector output follows changes in detector temperature as shown in Fig. 2. We measured a temperature coefficient of *ca.* 2.8 pA/10°C, when the temperature changed from 205 to 195°C, as indicated in Fig. 2. Note that the new detector temperature (195°C) is constant within 4 min after the onset of the change. However, the detector output required over 15 min to establish a new baseline. In addition, the short-term noise increases, and shifts in the baseline appear, long after the temperature disturbance is over and the detector block is at the new equilibrium temperature. The biggest factor affecting drift was reported to be poor temperature stability of the detector cell, while the source of sporadic high noise was reported to be contaminants in the insulating material of the cell[2]. We believe that both short- and long-term stability problems are likely to be caused by small shifts in the position of the energy beam caused by shifts in the window and gasket positions. Therefore, minimizing rapid temperature changes should reduce these problems.

The warm-up, long- and short-term detector stability, and lamp warm-up time

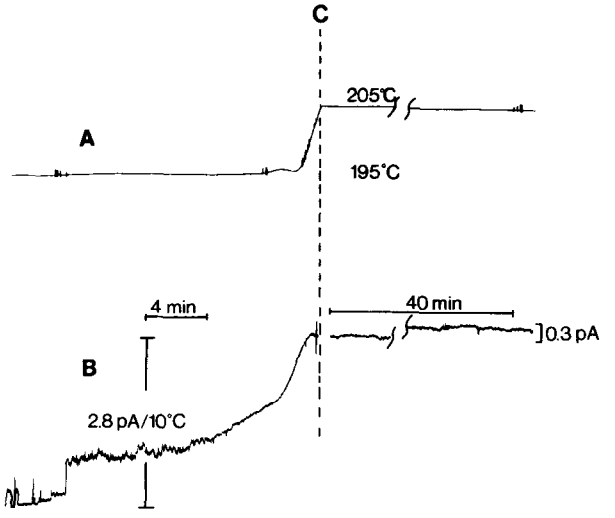

Fig. 2. Effect of temperature change on the current output of a HNU photoionization detector. (A) Temperature. (B) Detector output. (C) Onset of temperature change.

of the temperature-stabilized detector, are illustrated in Fig. 3. The detector block is within 1°C of its equilibrium value in less than 1 h. After the warm-up period, the long-term temperature stability is better than 1°C/h. The detector long- and short-term current stability are *ca.* 0.5 pA/h and 0.03 pA/min, respectively. Once at temperature equilibrium, the lamp requires less than 30 sec to produce a stable baseline current after turn-on, as shown in Fig. 3.

The chromatographic performance of the temperature-stabilized detector is

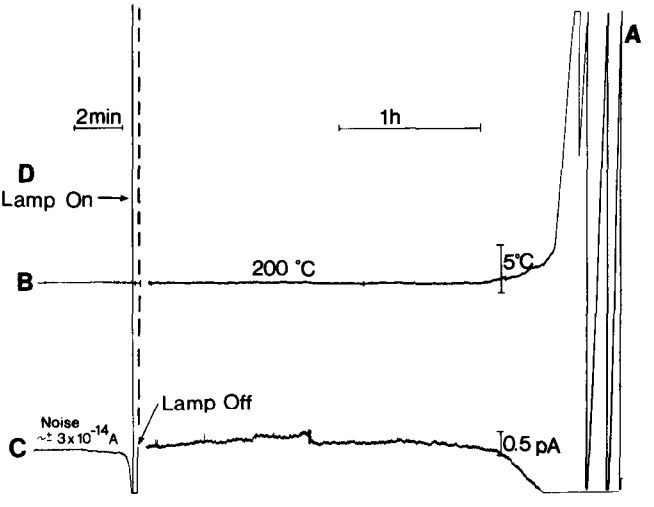

Fig. 3. Long- and short-term stability of a temperature-stabilized HNU photoionization detector. (A) Starting point: controller and lamp turned on. (B) Temperature. (C) Detector output. (D) Lamp on.

illustrated in Fig. 4. The full-scale sensitivity of the flame ionization detector channel (Fig. 4A) was adjusted to have a comparable noise level to that observed in the photoionization detector channel (Fig. 4B). In general, the S/N of the two traces is comparable when the peak-widths of the photoionization detector approach those measured with the flame ionization detector. Although the ionization efficiency of the photoionization detector is larger than that of the flame ionization detector (0.3 Coul/g C *vs.* 0.02 Coul/g C), there is no significant gain in detectability over the flame ionization detector when the photoionization detector is used with capillary columns. This is in spite of ,larger peak areas measured for the photoionization detector. Comparable results were obtained by Jennings *et al.*[3].

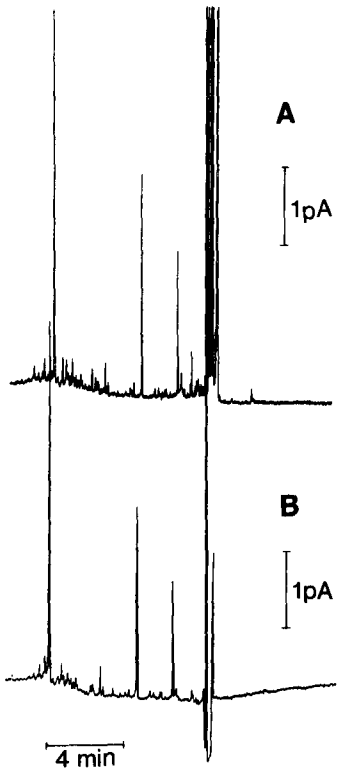

Fig. 4. Comparison of flame ionization detector and photoionization detector outputs at comparable noise levels and split ratio. (A) Flame ionization detector. (B) Photoionization detector. OV-101 fused-silica column, 50 m × 0.32 mm I.D. (Hewlett-Packard, Palo Alto, CA, U.S.A.). Programmed 50 to 150°C at 4°C/min. Analysis of a 10-ml sample of ambient air. Baseline disturbance on B was caused by water in the sample.

In conclusion, the output of the temperature-stabilized detector shows excellent short- and long-term stability. Although the detector may not offer increased detectability compared to the flame ionization detector in capillary GC, the combination of detectors (photoionization detector–flame ionization detector) may be used to establish identity or purity of materials by means of area ratios. In addition, because of its non-destructive character, the photoionization detector can be used as a sniff-port in the analysis of odorous compounds.

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