Note

Chromatographic alarm system for aqueous and organic solvents

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(First received August 9th, 1985; revised manuscript received October 17th, 1985)

Preparative chromatographic columns that are not constantly monitored throughout sample application will eventually run dry. Repacking them is time consuming, often expensive, and always frustrating. To avoid this problem, one may employ a commercially available photoelectric liquid level monitoring device[1]. Unfortunately, this unit is expensive, can only be used on columns with external diameters between 10 and 30 mm, requires a 120-V power supply, and can not be used with opaque columns. We therefore developed a high impedance solid state alarm system. The unit is portable, durable, and less expensive than the photoelectric device. The alarm can also be used to estimate the composition of two-component solvent systems, a feature which may obviate the need for NMR spectroscopy in some chromatographic applications.

MATERIALS AND METHODS

The electronic components of the alarm unit (Fig. 1) are mounted on an aluminum front plate which is enclosed in a Bakelite® box. The probe's electrodes are $2 \times 2 \times 0.01$ cm gold-plated brass (Fig. 2); for small columns, the electrodes may be reduced in size. If the solvents permit, the electrodes are separated by a 0.5-cm
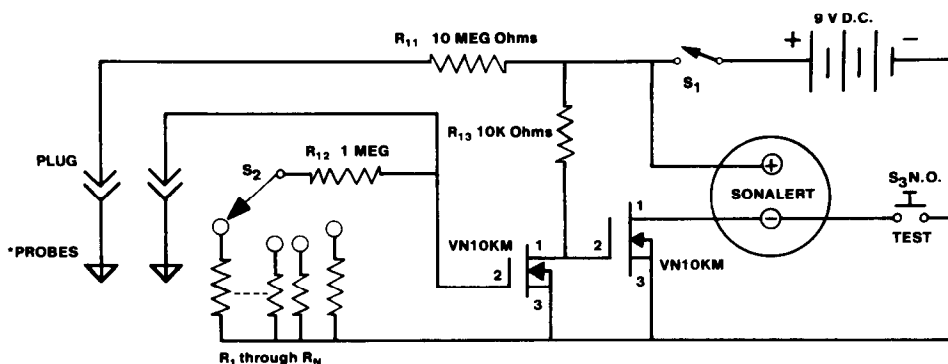

Fig. 1. Schematic of the alarm. R_1–R_N are fixed or variable resistors. Useful values for R_1–R_N are: 2.12, 2.13, 2.16, 2.19, 2.23, 2.25, 80.0, 10.0, 102.0 and 10.36 MΩ. S_1 = Single-pole, single-throw switch; S_2 = rotary switch; S_3 = momentary switch; VN10KM = a VMOS device (may be replaced with an SK9155).
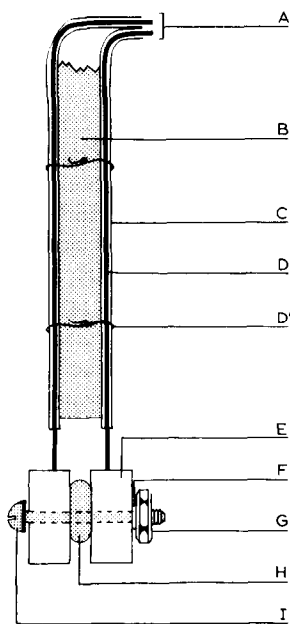
Fig. 2. Diagram of the probe. A = Connection (via vinyl-coated copper hook-up wire) to the alarm; B = 0.635 cm PTFE rod; C = 0.2 cm I.D. PTFE tubing; D = 0.102 cm I.D. stainless-steel wire; D' = stainless-steel tie wires; E = gold-plated electrode; F = stainless-steel lock-washer; G = No. 6 stainless-steel nut; H = 0.5 cm nylon washer; I = No. 6–24 nylon machine screw.

nylon washer (H) which is retained by the nylon screw (I) and the stainless-steel lockwasher (F) and nut (G). Alternatively, the stainless-steel wire is rigid enough to maintain the interelectrode distance in most applications. The alarm may be mounted outside the laboratory if additional wire is interposed between the probe and the box. Interested readers may contact the authors for additional details regarding the construction of the electronic unit and the probe.

When used as a column alarm, the probe is inserted into the column so that the electrodes are 0.5–1.0 cm above the top of the column bed. Switch 2 is set at a value that prevents the activation of the sonalert when the electrodes are in the eluting solvent. Switch 1 is turned to the "on" position, and the unit is checked by momentarily depressing switch 3; the sonalert should sound. The probe is left in place as the sample and wash solvent flow into the column bed.

In operation as a solvent-pair analyzer, the probe is placed into the test solution. The unit is turned on and tested as described above. Switch 2 is rotated until the highest resistance is found which prevents the sounding of the sonalert. The approximate composition of the solvent-pair can be determined by reference to a standard curve prepared with a range of solvent-pairs composed of the two solvents.

RESULTS AND DISCUSSION

Several precautions must be taken in constructing the probe. The stainless-steel lead wires (D in Fig. 2) must be covered with PTFE tubing (C) to prevent the

stainless-steel tie wires (D') from shorting the lead wires. A PTFE washer and screw would be preferable to those of nylon (H, I); unfortunately, most PTFE screws contain plasticizers that are dissolved by some organic solvents.

We have successfully used this column alarm for over a year with the original battery. The alarm can be heard at a distance of 30 m. By dialing in the appropriate resistance with S_2, we can monitor columns employing the following solvents, or solvent-pairs, as eluents: water (2.12 MΩ); methanol (2.13 MΩ); ethanol (2.16 MΩ); nitromethane (2.19 MΩ); acetone (2.23 MΩ); n-propanol (2.23 MΩ); ethyl acetate (80.0 MΩ); acetone–ethyl acetate (50:50) (10.0 MΩ); diethyl ether (102.0 MΩ); chloroform (102.0 MΩ); acetone–light petroleum (b.p. 60–90°C) (50:50) (10.36 MΩ). For some solvents and solvent-pairs, resistors R_1 through R_{10} may be inappropriate and should be replaced with other fixed or variable resistors; variable resistors facilitate adapting the alarm to a specific solvent. Because of the inherent electrical properties of the VMOS (= V-shaped metal oxide semiconductor) devices, the unit can not be operated with solvents less polar than ethyl acetate–light petroleum (1.4:1) nor, with any resistor on S_2 that is greater than 103 MΩ.

In operating the device as a column alarm, several precautions must be observed. If conducting columns are employed, both electrodes must not be in contact with the sides of the column. Very small electrodes may be used provided that a 0.3-cm interelectrode gap is maintained; if the gap is less than 0.2 cm and if the viscosity of the solvent is great enough, *e.g.* water, a drop of solvent may bridge the gap and prevent the system from functioning. We have, however, found that with a 0.5-cm interelectrode gap a column with a diameter of 2.2 cm may be operated with water at speeds as great as 50 ml/min without alarm failure.

This alarm may be safely operated with flammable liquids because, even if a short circuit occurs across the two electrodes, less than $6.9 \cdot 10^{-7}$ A flows through the solvent. This figure is probably less than incidental static electricity.

When the alarm is used with an aqueous solvent, a small amount of electrolysis occurs. With a polar solvent, the current flow would be approximately $6.9 \cdot 10^{-7}$ A. In 20 min the attendant electrolysis would dissolve (or plate out) as much as $8.7 \cdot 10^{-9}$ mol of the material comprising the electrode (or the eluting solvent). If a gold-plated brass electrode were used with a chloride containing buffer, gold would be dissolved as $AuCl_4^-$, eventually exposing the underlying brass. If the sample or the solvent interacts with the copper or zinc thus exposed, either the sample or the column packing material could be damaged. To avoid such problems, one could use platinum electrodes. With these inert electrodes, oxygen gas and hydrogen ions would be liberated at the anode and hydrogen gas and hydroxyl ions would form at the cathode. There would be some formation of the platinum oxides Pt^IOH, $Pt^{II}O$, and $Pt^{IV}O_2$ on the electrodes, but these are essentially insoluble species. An additional theoretical concern is the small potential difference between the electrodes, a condition which could cause the adsorption of small amounts of protein to the electrodes. If the alarm is operated with protein-containing solutions, it may therefore be necessary to clean the electrodes periodically to maintain optimal operation of the alarm system.

tutes of Health and 74-228 from the Children's Hospital Research Foundation. We are indebted to Rita Compston for preparation of the manuscript.

REFERENCES

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2 B. Grob, *Basic Electronics*, McGraw-Hill, New York, 5th ed., 1984.