

CHROM. 3929

Liquid level detector for column adsorption chromatography*

The sensing of liquid levels in fraction collectors is commonly accomplished in a variety of ways such as light absorption, change in refractive index, conductivity, and high frequency oscillator cells. We now describe a very simple, inexpensive, versatile, and trouble-free liquid level detector for the monitoring of eluents in chromatography. This device alerts (by means of a buzzer) the operator when the desired volume of eluent has been collected. This "buzz box" innovation allows the chromatographer to perform other duties while a chromatogram is in progress.

This detector functions by a very simple principle. A schematic for its construction is shown in Fig. 1. The level sensing probe is a thermistor, which is used in a self-heating mode. It senses the presence of eluent by a change in the thermal conductivity of a liquid as compared to the thermal conductivity of the atmosphere. The eluent, when in contact with the probe, conducts more heat from the thermistor, cooling it. This results in a change in resistance of one leg of a balanced Wheatstone bridge circuit. The resultant error signal is amplified by transistors Q_1 and Q_2 to energize the relay R_1 . The thermistor used was a glass-coated probe whose two leads were isolated from each other by a piece of small diameter glass tubing. Both leads, housed in a slightly larger diameter glass tube, were then sealed in epoxy cement, after an appropriate length of connecting shielded cable (to amplifier) was attached. The

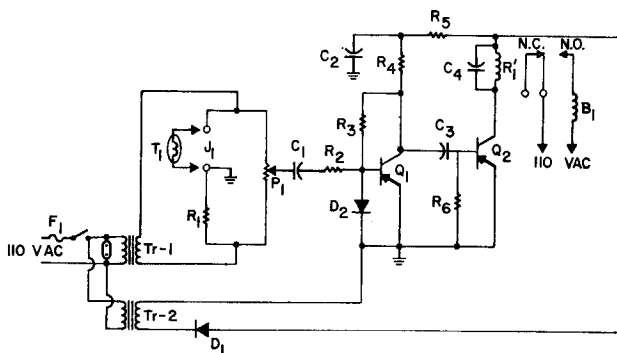


Fig. 1. Schematic for liquid level detector. (T_1) $3k\Omega$ at 25° glass probe thermistor; (P_1) $1k\Omega$, 2 W; (R_1 , R_2) 500Ω , 1 W; (R_3) $100k\Omega$, $1/2$ W; (R_4) $5.2k\Omega$, $1/2$ W; (R_5 , R_6) $1.8k\Omega$, $1/2$ W; (C_1 , C_2) 5 MFD, 10 V d.c. electrolytic; (C_3 , C_4) 50 MFD, 50 V d.c.; (Q_1 , Q_2) 2N1376; (Tr-1) 12 V secondary, 1 A; (Tr-2) 24 V secondary, 1 A; (R_7) 1500Ω relay, 2 A contacts; (B_1) 110 V a.c. buzzer; (D_1) IN1692; (D_2) IN60; (J_1) phono jack; (F_1) 1 A fuse.

entire detector was conveniently housed in a portable metal cabinet (ca. $15 \times 15 \times 10$ cm) with a panel-mounted fuse box, pilot light, on-off switch, and connector jack to accept the connecting cable from the thermistor probe.

The control of eluent volume was simply determined by the depth to which the thermistor was immersed in the receiving vessel. For our purposes, the thermistor

* Contribution No. 536 of Central Research Laboratories, Minnesota Mining and Manufacturing Company.

was attached (either by a piece of Scotch brand tape or a rubber band) to the drip-tip of the chromatographic column with the precaution that the drops of eluent from the column did not impinge directly on the thermistor (prematurely tripping the buzzer relay).

The normally open relay contacts of the detector were used, in our case, to energize a buzzer. This relay can be employed in a variety of other ways. For example, it may be used as an eluent control by tripping a solenoid-operated plunger valve affixed to the chromatographic column (replacing the stopcock), and thus shutting off the flow of eluent when the desired volume has been collected. In another manner, this device can be positioned to the top of a chromatographic column. It will then warn the operator when the liquid level approaches the stage where the column would go "dry".

With such an easily constructed, inexpensive, and portable instrument, chromatographic operations may become as sophisticated as the operator desires.

*Central Research Laboratories,
Minnesota Mining and Manufacturing Company,
St. Paul, Minn. (U.S.A.)*

ARTHUR MENDEL
DONALD F. HAGEN

Received December 31st, 1968

J. Chromatog., 40 (1969) 303-304

CHROM. 3912

Combination of thin-layer chromatography and the ring-oven method for the semi-quantitative estimation of some herbicides

The importance of the herbicides for the pre- and post-emergence control of weeds has increased in recent years. At present, many of these compounds are officially approved for such use. With the wider application of pesticides in general, the problem of residue determination grows.

The analytical problem presented by pesticide residues in food and other materials has been briefly defined¹⁻⁴ and is essentially unchanged. In order to study the relative retention of pesticides (herbicides) in samples of this nature, various methods are required for their microdetermination. MAJOR⁵ proposed a paper chromatographic method. ABBOTT *et al.*⁶ used a paper chromatographic determination using reflectance densitometry for quantitative measurement, and also thin-layer chromatography^{7,8}. The application of the ring-oven technique to herbicide analysis has also been described⁹. Advances in gas chromatographic methods have lately included pesticide residue analysis though the application of gas chromatography to the analysis of these residues was initially confined to chlorinated hydrocarbons and related compounds; however, herbicides are now included¹⁰⁻¹².

The combination of thin-layer chromatography and the ring-oven technique on a semi-quantitative basis was thought to offer a simple, rapid and inexpensive

J. Chromatog., 40 (1969) 304-307