

CHROM. 9260

Note

Improved Hall conductivity detector: system without solvent interference

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(First received January 27th, 1976; revised manuscript received April 5th, 1976)

In 1974, Hall¹ published a description of a selective microelectrolytic conductivity detector with a sensitivity of 20 to 50 times that of the Coulson detector², introduced in 1965. The Hall detector is now being produced commercially, instead of the Coulson detector, by Tracor (Austin, Texas, U.S.A.). It operates on the same basic principles as the Coulson detector and is considered a refinement over the Coulson detector.

The commercial detector must be mounted as a separate unit external to the chromatograph via heated transfer tubing. The transfer tubing is connected directly to the quartz reaction tube, therefore, allowing all the column effluent to pass through the reaction tube. Hall¹ reported the use of a 0.5–1.0 mm I.D. quartz reaction tube which allowed this direct connection without apparent contamination to the reaction tube. The effects of operating in this configuration are described in this paper.

EXPERIMENTAL

Reagents

The compound for this study, lindane, was selected because its chromatography and detection in chloride-specific detectors are well documented in the literature. A series of lindane solutions at a concentration of 1 $\mu\text{g/ml}$ were prepared. The different solvents used were *n*-hexane, pentane, petroleum ether, 2,2,4-trimethylpentane, toluene, cyclohexane, acetone, ethyl acetate, and benzene. Lindane (purity 99.9%) was obtained from Perrine Primate Lab. (Perrine, Fla., U.S.A.), and the pesticide grade solvents were obtained from Mallinckrodt Chemical Works (St. Louis, Mo., U.S.A.) and Burdick and Jackson Labs. (Muskegon, Mich., U.S.A.).

Experimental conditions

A Tracor MT-220 gas chromatograph equipped with a Hall conductivity detector, as shown in Fig. 1, was operated in the reductive mode. The chromatograms were recorded with a Westronics MT-22 strip chart recorder. The following instru-

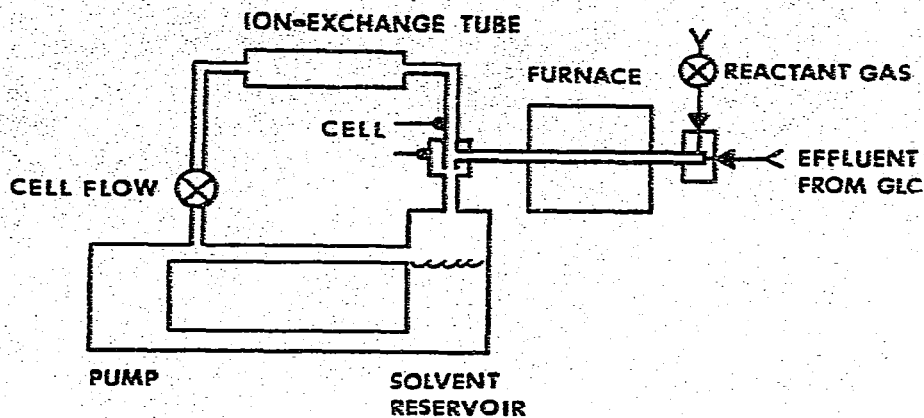


Fig. 1. Hall detector.

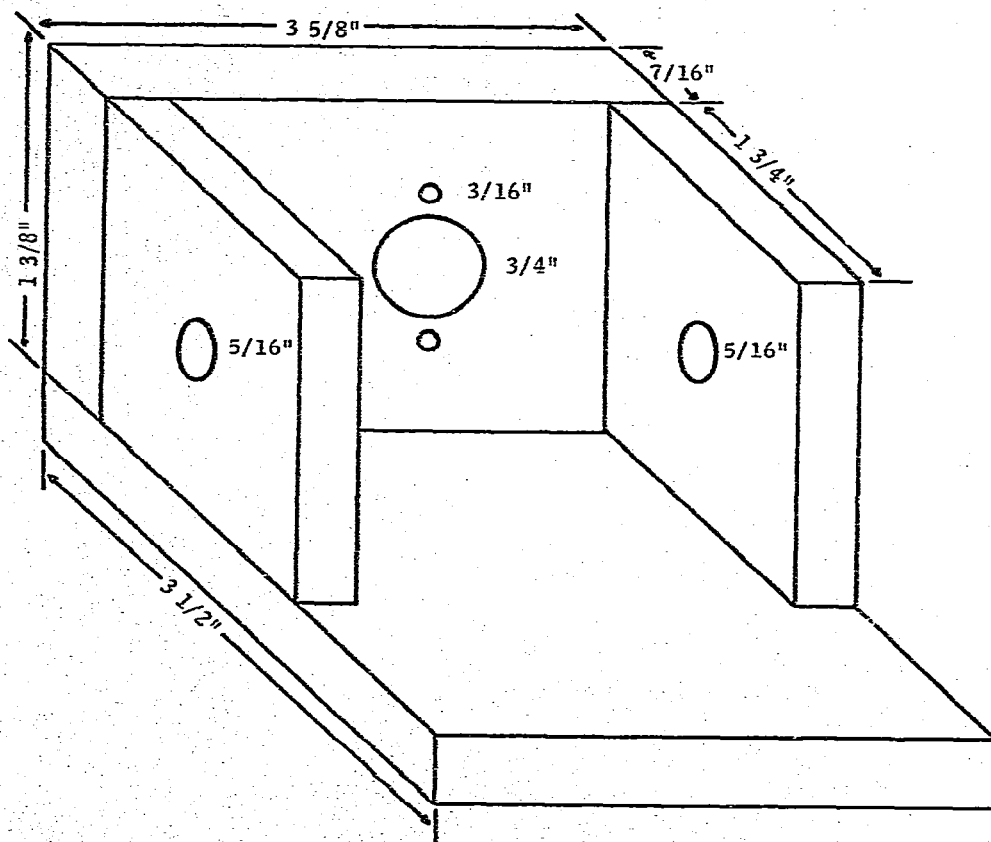


Fig. 2. Valve block.

mental operating conditions were used initially in this study: a 6 ft. \times 4 mm I.D. glass column packed with 3.0% OV-17/3.9% OV-210 on 80-100 mesh Gas-Chrom Q operated at 225°; helium carrier flow-rate, 60 ml/min; hydrogen reactant gas flow-rate, 7 ml/min; cell liquid flow-rate, 1 ml/min; pyrolysis furnace temperature, 900°;

inlet transfer line temperature, 250°. The conductivity cell was operated at ambient temperature.

To eliminate solvent effects the strip heater and heater block for the sample inlet of the Hall were removed and discarded. A valve block, as shown in Fig. 2, and a single transfer line were installed between the gas-liquid chromatographic (GLC) effluent and the pyrolysis oven. The $\frac{1}{4}$ -in. adapter was replaced with a $\frac{1}{16}$ -in. low dead volume union. A Nupro fine metering valve was added to control make-up gas flow.

After installation of the high-temperature vent valve (valve block temperature 240°), helium make-up gas was added at a flow of 60 ml/min. The valve vent time was 1.5 min.

RESULTS AND DISCUSSION

The basic Hall conductivity detector¹ (Fig. 1) consists of a heated transfer line, pyrolysis furnace, quartz reaction tube and PTFE transfer line to conductivity

TABLE I
TIME REQUIRED TO RETURN TO ORIGINAL BASELINE BEFORE VENT VALVE

Solvent	Solvent tailing time (min)	
	5% original baseline	1% original baseline
Pentane	2	4
2,2,4-Trimethylpentane	5	16
Petroleum ether	3	6
Hexane	3	5
Cyclohexane	14	40
Toluene	10	20
Benzene*	∞	∞
Acetone	12	42
Ethyl acetate	54	>360

* Detector contaminated beyond use.

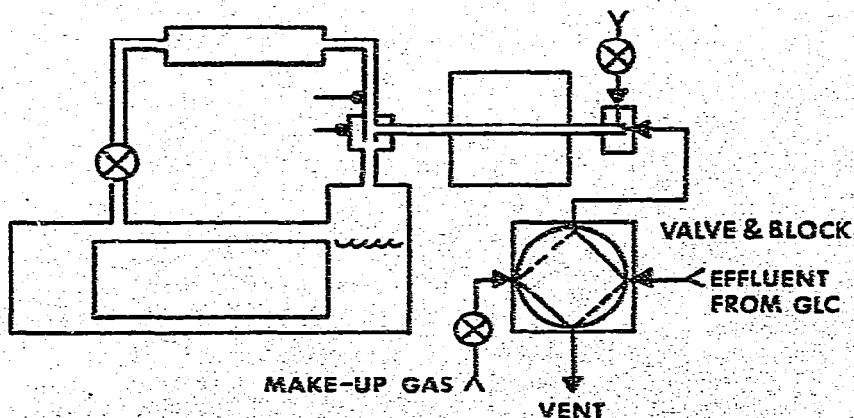


Fig. 3. Modified Hall detector.

cell. The hydrogen combustion gas is mixed with the helium carrier in front of the pyrolysis furnace. This configuration allows the column effluent to continually enter the pyrolysis furnace. Using the detector under these conditions, many solvents were found to elute from the quartz reaction tube slowly, masking early eluting compounds or reducing the efficiency of pyrolysis, as seen in Table I.

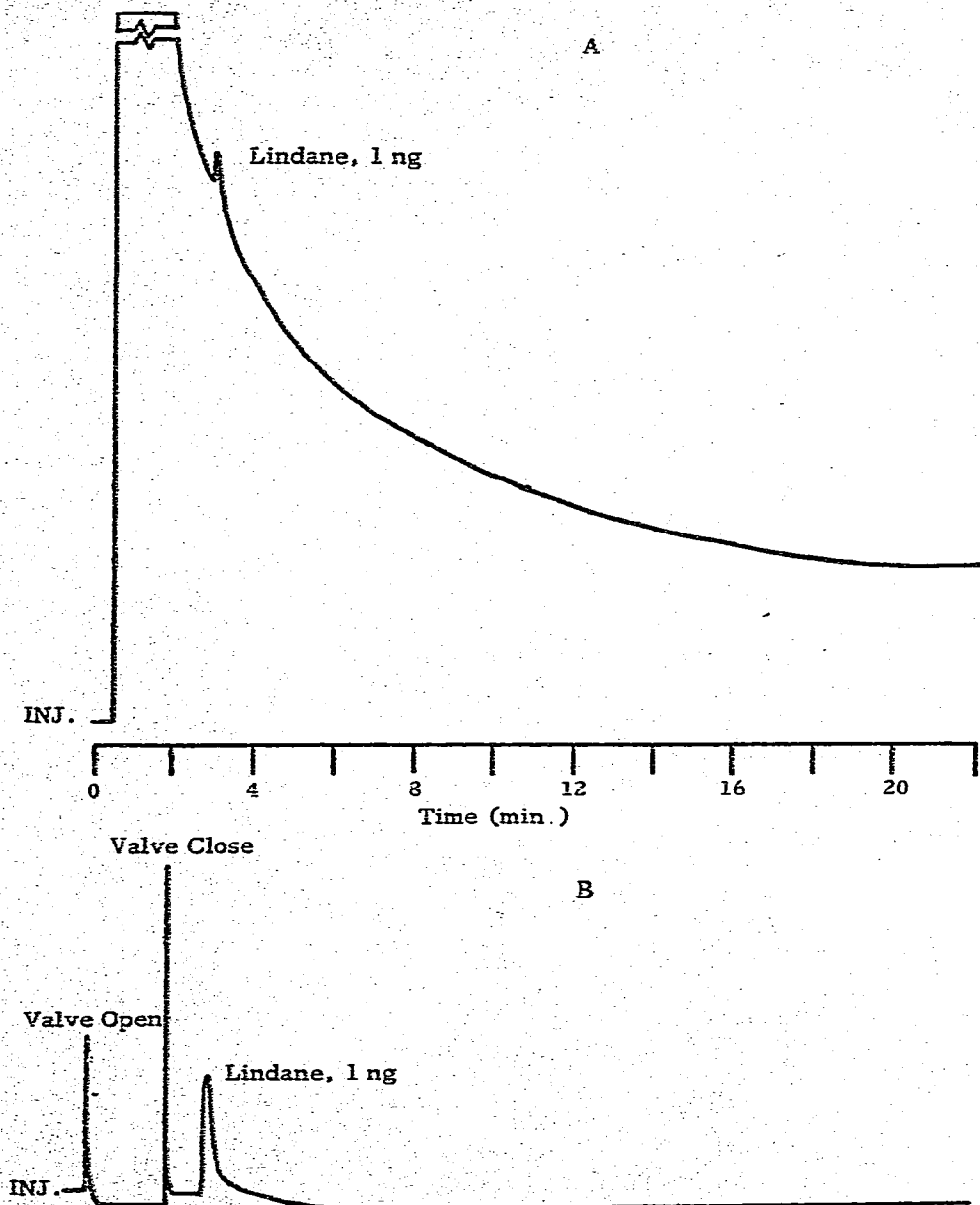


Fig. 4. Gas chromatograms of 1 ng of lindane in ethyl acetate. A, Before installation of the vent valve. B, After installation of the vent valve.

After installation of the vent valve (Fig. 3), the lindane solutions were reinjected. Fig. 4 shows that the chromatographic solvent interferences were eliminated by venting the solvents away from the detector. The times required for return to the original baseline (Table I) were significantly reduced. All the solvents investigated returned to within 5% of the original baseline in less than 0.01 min and to within 1% of the original baseline in less than 1.0 min. In addition to eliminating solvent tailing and peak masking, "down-time" was reduced tremendously by eliminating carbonaceous deposits in the pyrolysis tube. This also improved the reproducibility of repeated injections and improved the signal-to-noise ratio. Increased reproducibility was evidenced by eliminating decreases in response with repeated injections down the pyrolysis tube without the valve.

REFERENCES

- 1 R. C. Hall, *J. Chromatogr. Sci.*, 12 (1974) 152.
- 2 D. M. Coulson, *J. Gas Chromatogr.*, 3 (1965) 134.