

Flame Ionization Detector for Gas Chromatography

It has been found that the electrical conductivity of a flame burning a mixture of hydrogen and nitrogen (in air) is very sensitively affected by the vapours of organic substances, and this effect can be used for detection purposes in gas chromatography.

In a simple form of the detector the carrier gas used was a mixture of approximately equal parts by volume of hydrogen and nitrogen and the issuing gas was burnt at a jet made from a 23-gauge hypodermic needle (cut square) which itself served as the positive pole for the conductance measurement. The negative pole consisted of a piece of 30-mesh brass gauze, about 1 cm. above the needle, and the current, driven by a battery of about 300 volts, passed through a 100,000-ohm resistor, across which a Leeds and Northrup 10-mV. potentiometric recorder was connected. The sensitivity of this arrangement to organic vapours was at least as good as reported for the best katharometers.

For the highest sensitivity yet obtained, a double triode 'long-tailed pair' impedance conversion circuit was used (voltage gain about $\times 2$), with a reference jet (burning the same gas mixture) feeding one grid and the detection jet feeding the other. Circuit details are shown in Fig. 1.

The valve is an *E80CC* (Philips), heaters in series with a 12-volt accumulator. The output is filtered with an *RC* circuit with a time constant of about 5 sec. The combustion chamber and collector leads are completely shielded. The combustion chamber is supplied with filtered air since dust particles cause large disturbances.

With the jets connected to short partition columns, injection of 0.01 ml. of a solution of one part of diethyl ether in 10,000,000 of ethyl acetate (by liquid volume) gave an output of about 1 mV. for the ether peak, which was eluted during the passage of 100 ml. of carrier gas. This deflexion was about five times the background fluctuations, and the sensitivity (for twice background) was estimated at not less than 5×10^{-12} mole of diethyl ether, or 4×10^{-10} gm. The sensitivity at the point of detection was about one part in 400 million (by gas volume). From the daily variations in background, it is thought probable that the sensitivity is at present limited by the quality of the insulation and the stability of the grid resistors rather than by the stability of the flame outputs. (Much higher output could be obtained with higher grid resistances, provided background was not proportional.)

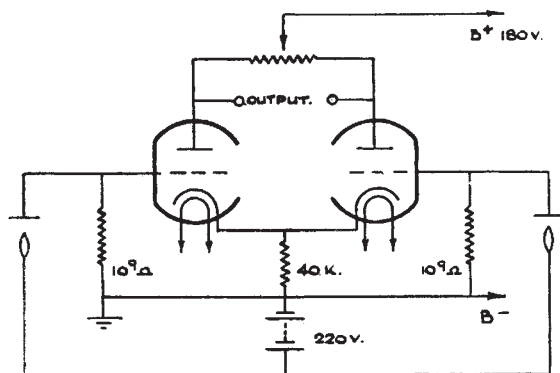


Fig. 1. Circuit of dual-jet detector

With the double-jet system fed with the same gas mixture to both columns, the base line is remarkably insensitive to nitrogen-hydrogen ratio, total flow-rate, vibration, ambient temperature or minor variations in the supply of filtered air. The air supply is passed straight into the bottom of the jet chamber without any device to suppress turbulence.

Other organic vapours so far tried give sensitive responses, including benzene, toluene, xylene, carbon tetrachloride and methylene chloride. Very little if any response is obtained with carbon dioxide.

Preliminary measurements and calculations based on Faraday's law, appear to indicate an ion yield of between one and ten ions per 10,000 molecules of ether, but further investigations, using electrometer valves with very low grid currents, are planned. It is hoped to publish the results at a later date.

A remarkable demonstration of the sensitivity of the apparatus (and of the difficulty of producing known concentrations of vapour by serial dilution) is given by charging an ordinary 2 ml. glass hypodermic syringe with ether vapour, pumping out completely about 100 times, and then injecting a syringe-full of air. A peak of several millivolts is obtained, from ether desorbed from the glass.

The above detector was described at a discussion on gas chromatography held at Cambridge on October 4, 1957.

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Polarization of the Integrated Light of Comet Arend-Roland, 1956h

From a pair of photographs of the comet taken on the night of April 29-30, 1957, through a 'Polaroid' filter with the 50-cm. Zeiss triplet of the Mond Astrograph, percentage polarization in integrated light (spectral range, 4000-5900 Å.) has been measured in the brightest part of the coma, and also at two locations in the tail 4 and 5 min. of arc respectively from the former position. The small scale of the photographs (6.85 min. of arc/mm.) prevented any attempt at a more detailed survey.

The correct position angle of the polarizing filter was determined visually by a preliminary observation of the comet, and the filter was rotated through 90° between the successive exposures, which were given separately on Kodak O 800 plates. It had been intended to give an exposure time of 2 hr. in each instance; but the presence of clouds limited the exposure through the 'crossed' filter to 1 hr. 20 min. The mean altitude of the comet was approximately 20° .

Both plates were microphotometered; and the photographic density measures at each of the three selected locations were reduced by the aid of magnitude-density curves, based on measurements of the trailed images of five field stars, namely, *H D*, 21291, 21389, 21447, 25291 and 25642. No corrections were applied to the observed magnitudes for differential atmospheric extinction.