

TABLE II

Separation	Separation factor	H ₂ SO ₄ -EtOH concentrations
Nd-La	1.9	0.01-0.001 N H ₂ SO ₄ -80% EtOH
Y-La	1.6	0.01-0.001 N H ₂ SO ₄ -80% EtOH
Nd-Y	1.8	0.01 N H ₂ SO ₄ -80% EtOH
Pr-Y	1.2	0.01 N H ₂ SO ₄ -80% EtOH

Separations would be expected to be slow because of the strong adsorption of rare earths by the anion exchanger.

The distribution coefficient data plotted in Fig. 1 offer little prospect for an effective anion exchange separation of the neighbouring rare earths Nd and Pr.

Dilute sulphuric acid-ethanol eluants are unsuitable for the separation of macro amounts of rare earths owing to the limited solubility of rare earth sulphates in these media.

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A fog precipitator for gas chromatography

The flow of hot carrier gas from a gas chromatography column usually results in the sample emerging as a fog. Flow rates commonly used make efficient collection using conventional cold traps almost impossible, yields often being as low as 60%. The apparatus described below makes possible recoveries of over 98%.

In this laboratory, eluting material is collected from columns of between 1/4 and 1 in. diameter, with nitrogen flow rates of between 20 ml/min and 2 l/min, and temperatures from 20 to 220°.

The apparatus consists of a glass cold trap and a metal precipitator containing an electric field supplied from a source of constant current sufficient to develop a field strength of from 5,000 to 12,000 direct volts.

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Fig. 1 shows a cold trap designed to collect from a 1-in. diameter column which often produces 2 g of sample during a period of 5 min, with a nitrogen flow of 1 l/min. Smaller traps may be used for the collection of smaller quantities, but care should be taken to see that the trap size is reduced to scale, because the ratios of the tube diameters are selected to assist condensation by causing turbulence of the fog in the correct places. It has been found convenient to construct a simple jig to enable any number of cold traps to be made, each with two B₁₀ sockets spaced the same distance from each other and at the same height, so that they may be exchanged quickly. This method is satisfactory since peaks do not emerge very quickly from our columns and the work is too varied to justify the construction of automatic collection apparatus.

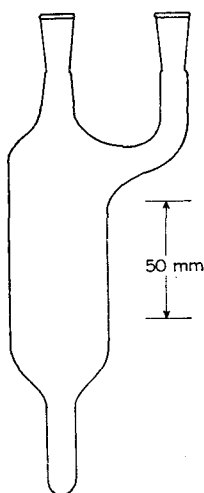


Fig. 1. Cold trap, with two standard B₁₀ sockets.

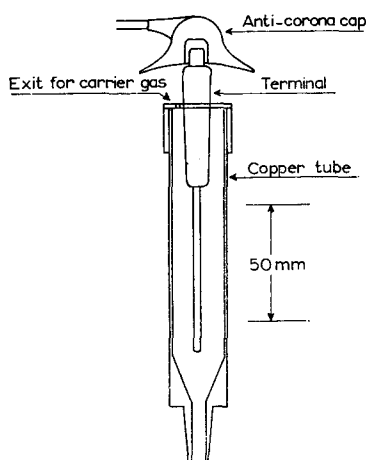


Fig. 2. Metal precipitator. With anti-corona cap., 15 kV (Radiospares Ltd.).

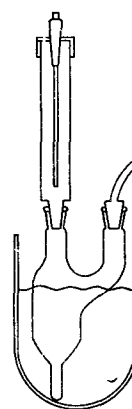


Fig. 3. The assembled apparatus. Ceramic to metal lead through sealed terminal (K.L.G. type CS. 115/2). Copper tube 25 mm in diameter.

The metal precipitator (Fig. 2) does not always require cleaning after each peak of eluted material because the geometry of trap and precipitator and the shape and extent of the electric field are such that only a small percentage of unusually high vapour concentrations ever enter the precipitator. Fig. 3 shows the assembled apparatus. It should be noted that gas enters the side arm of the trap.

High voltage for the precipitator is derived from a radio frequency oscillator, the output of which is rectified to direct voltage by a valve (type U25), and taken by a very well insulated cable to the central electrode, as shown in Fig. 4. This method of generating a high voltage offers advantage over mains frequency circuits, since the energy content is less at source and therefore the power available is limited if the output should be presented to a low resistance such as the human body, although of course every effort should be made to avoid receiving a shock.

Current flowing through the precipitator is developed across "R" (Fig. 4) to provide feedback and give a measure of automatic constant current control. The

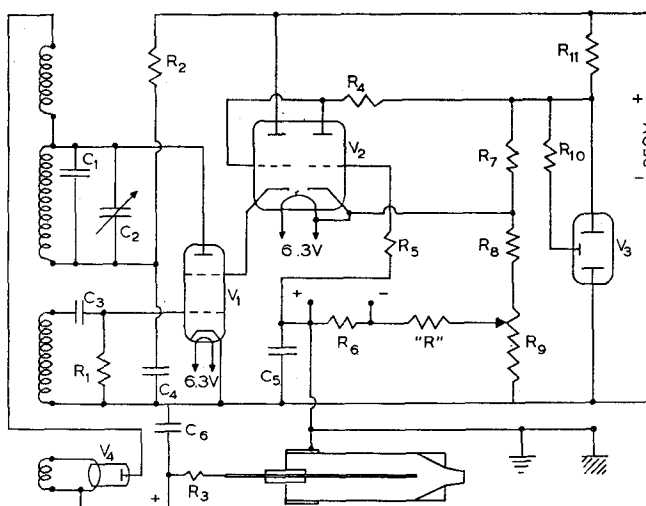


Fig. 4. Circuit diagram. Resistors, all within 10%:

R_1	47	$k\Omega$	1/4	W	R_7	33	$k\Omega$	2	W
R_2	220	Ω	1/2	W	R_8	1.5	$k\Omega$	1/2	W
R_3	470	$k\Omega$	1	W	R_9	5	$k\Omega$	5	W. Wire wound.
R_4	680	$k\Omega$	1	W					"Set Current" control.
R_5	470	$k\Omega$	1/4	W	R_{10}	1	$M\Omega$	1/2	W
R_6	22	$k\Omega$	1/4	W	R_{11}	10	$k\Omega$	5	W
					"R"	220	$k\Omega$	1/4	W

Capacitors, all within 20%:

C_1	0.002	μF	Mica,	1000	V	C_4	0.05	μF	500	V
C_2	0.001	μF	Variable,	1000	V. Frequency control.	C_5	0.25	μF	300	V
C_3	0.001	μF	300	V		C_6	0.001	μF	12	kV

Valves:

V_1	6V6 or 6P25 or 6BW6
V_2	6SN7 or B65 or ECC32 or 12AU7 or B329 or ECC82
V_3	150B3 or QS150/15 or CV287
V_4	U25

Coil:

"NERA" R.F. E.H.T. Coil. 8-12 kV.

(Tyer and Co. Ltd., Merrow Siding, near Guildford, Surrey, England.)

range of current available (approx. 10 to 100 μA) is determined by the setting of the "Set Current" control (R_9), a potentiometer mounted on the front panel, and the reference voltage across this component. If required, current may be further increased by decreasing the value of "R". Automatic control is achieved by using one half of a valve (type 6SN7) to amplify changes of voltages across "R" and applying these changes to the other half which is used to adjust the screen grid of the oscillator valve (type 6V6). The high tension supply of 250 direct volts is required to provide a current which varies between 30 and 50 mA during normal operation. Since the reference tube 150B3 cannot correct the oscillator for mains supply variations it is preferable to supply the H.T. from a stable source within the limits of 230 to 300 V, although the author has found a simple unregulated power supply to be adequate.

When setting up the apparatus, the "Set Current" control is adjusted for maximum current and the oscillator frequency control (0.001 μ F variable capacitor) is adjusted until a flash-over discharge can be heard from within the precipitator, with the carrier gas flowing. This capacitor is then readjusted until the discharge ceases. The "Set Current" control may now be used.

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Comparison of two partitioning phases for gas-liquid chromatography with two types of detectors utilizing four halogenated hydrocarbons

Several halogenated hydrocarbons have been separated by gas-liquid chromatography. PERCIVAL¹ separated trichlorofluoromethane and dichlorofluoromethane using di-*n*-octyl phthalate as the liquid stationary phase and obtained quantitative recoveries of 98-102%. ZWEIG AND ARCHER², ZWEIG, ARCHER AND RUBENSTEIN³, and COULSON, CAVANAGH, DE VRIES AND WALTHER⁴ utilized silicone grease as the stationary phase for the separation of organo-halogenated compounds.

The present work was undertaken to investigate a suitable column packing for the efficient separation of *cis*-, and *trans*-1,3-dichloropropene, 1,2-dichloropropane, and 1,2-dibromo-3-chloropropane. Data are presented on a comparison of two column packings at two temperature levels together with a comparison of the sensitivities of the hot-wire katharometer detector and the microcoulometer cell.

Experimental

Apparatus and procedure. Gas chromatographic data were obtained by two types of apparatus: (a) Burrell Kromo-Tog, Model K-1 equipped with a hot-wire katharometer detector and an 8 ft. \times 0.25 in. o.d. U-shaped glass column, using helium as the carrier gas. (b) Microcoulometer gas chromatograph Model G-100 (Dohrmann Instruments Company, Palo Alto, California) using a 6 ft. \times 0.25 in. o.d. spiral aluminum or stainless steel column and nitrogen as the carrier gas.

Column packings were (a) silicone grease, 20% w/w (Dow-Corning, high methyl polymer) and (b) di-*n*-decyl phthalate, 30% w/w (Eastman Organic Chemicals); each coated on 30-60 mesh acid-washed Chromosorb (Johns-Manville).

Chemical compounds were 1,2-dichloropropane (b.p. 95-97°), *cis*-1,3-dichloropropene (b.p. 104-105°), *trans*-1,3-dichloropropene (b.p. 110-111°), and 1,2-dibromo-3-chloropropane (b.p. 196°).