

Notes

Production of pure helium carrier gas by diffusion

The successful use of a helium ionization detector for permanent gases in gas chromatography requires the production of a very pure carrier gas containing no more than 1 p.p.m. (by volume) of impurities in total. The impurities in commercial helium amount to 10 p.p.m. (by volume) approximately, excluding water, and are principally neon, nitrogen, hydrogen, oxygen, argon and carbon dioxide.

The methods of purifying helium proposed so far have been the use of traps cooled in liquid helium, traps containing molecular sieve cooled in liquid nitrogen¹, or a chemical purification train². This last method is the most commonly used. The train is composed of absorbers containing molecular sieve at both room and liquid nitrogen temperatures and furnaces containing titanium at 600° and Hopcalite (mixed copper and manganese oxides) at 350°. We have used such a purification train and find it difficult and expensive to set up as it requires meticulous attention to cleanliness and a long period of preliminary operation before stability is reached. The life of absorbers is limited and the train requires periodic replacement. Although BOURKE *et al.*³ have proposed a simpler chemical system the same criticisms may be applied.

The principle of stress enhanced diffusion of helium through quartz, investigated by MCAFEE⁴, has not so far been proposed as a means of preparing a pure carrier gas for helium ionization chromatography. We have found that a commercially available diffusion cell* will deliver helium of acceptable purity within hours of starting the flow (the impurities are stated by the manufacturers to total less than 0.5 p.p.m. (by volume)). The construction of this cell is similar to that of MELNYK AND HABGOOD⁵. It consists of a steel tube containing a bundle of quartz capillary tubes, which are individually sealed at one end. The sealed ends are exposed to the impure helium at high pressure. The tubes pass through a resin seal, with their open ends terminating on the low pressure side, into which they release the diffused helium. This cell was connected directly to the helium cylinder by means of the hose and pressure regulator supplied by the manufacturers. The diffusion area of the cell was wrapped with heating tapes dissipating a maximum of 500 W and supplied by a variable transformer. The seal area of the cell was water cooled. A pressure release valve** set at 35 p.s.i. was inserted downstream of the cell to protect the chromatograph.

Maximum temperature obtainable in the diffusion area was 600° and at 1,500 p.s.i. inlet pressure 450 ml/min of pure helium could be obtained at atmospheric pressure. Normal operation at 375° and 500 p.s.i. at the inlet provided 70 ml/min.

When the output from this diffusion cell was substituted for the output from a chemical purification train supplying a chromatograph, the standing current in the ionization detector of the chromatograph fell markedly.

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Some comparative values of detector current are given in Table I, the last figure of which obtained by BOURKE *et al.*⁶, probably represents the best obtainable by chemical means.

TABLE I
COMPARATIVE VALUES OF DETECTOR CURRENT

Instrument No.	Carrier gas purifier	Radiation source (mC)		Detector	
		³ H	⁹⁰ Sr	Volts	Amps
1	chemical	100		500	1.1 · 10 ⁻⁸
1	diffusion	100		500	4.2 · 10 ⁻⁹
2 ²	chemical	100		750	1 · 10 ⁻⁸
			10	—	1 · 10 ⁻⁸
3 ⁶	chemical		10	1,000	1.8 · 10 ⁻⁹

Several cylinders of helium have now been processed by this method without difficulty. Rapid venting of the high pressure gas in the diffuser when changing the supply appears to be sufficient to prevent excessive accumulation of impurities and there has been no indication that the life of the system will be limited in any way.

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1 J. E. LOVELOCK, *Res. Develop.*, 2 (1962) 36.

2 R. BERRY, in M. VAN SWAAY (Editor), *Gas Chromatography 1962*, Butterworths, London, 1962, p. 231.

3 P. J. BOURKE, M. D. GRAY AND W. H. DENTON, *J. Chromatog.*, 19 (1965) 189.

4 K. B. MCAFEE, *J. Chem. Phys.*, 28 (1958) 218 and 226.

5 N. MELNYK AND H. W. HABGOOD, *Can. Mining Met. Bull.*, (1961) 768.

6 P. J. BOURKE, R. W. DAWSON AND W. H. DENTON, *J. Chromatog.*, 14 (1964) 387.

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R_M values in simplified four-component systems of the type: ternary mixed solvent/pure solvent

Multi-component solvents, which are more versatile for the choice of optimal conditions of separation, are often used in paper and thin-layer chromatography. It is easier to analyse the effect of phase composition on the partition chromatographic behaviour of solutes when the two liquid phases are essentially immiscible, that is, when the stationary phase held by the supporting material is insoluble in the developing solvent (*e.g.*, the system carbon tetrachloride-benzene-chloroform/formamide¹). Nevertheless, even in these cases, the R_F -composition relationships for four-

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