Gas chromatography fraction collector and transfer system

This note describes a method for the collection of fractions from a gas chromatograph and a unique method for their subsequent introduction into other systems without contamination.

Fig. 1 depicts an array of Hoke TY 440 valves which constitutes one collection station of a manifold attached to the effluent stream of a chromatograph. In order to minimize absorption of materials on the inside surfaces of the manifold the valve

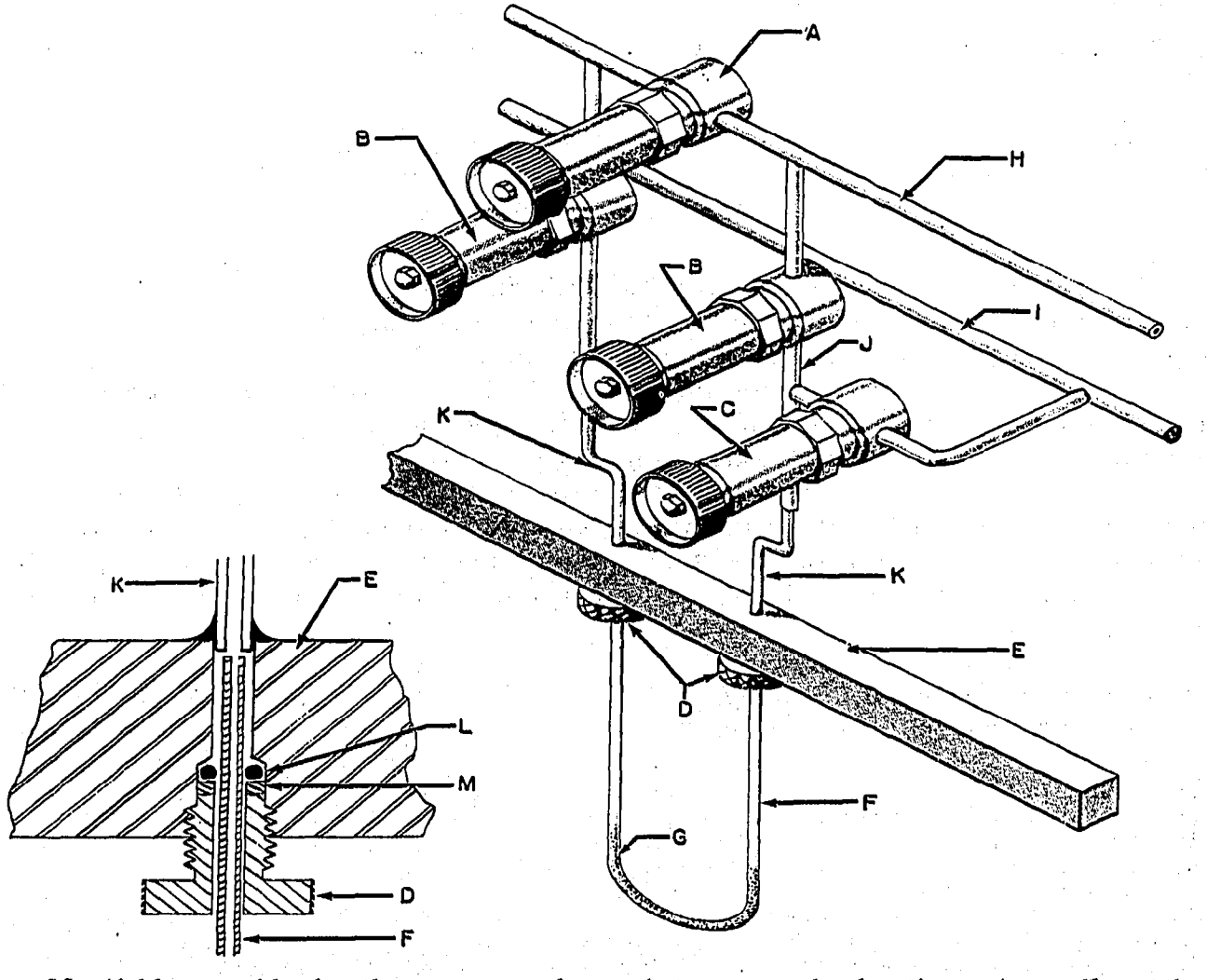

Fig. 1. Manifold assembly for the recovery of gas chromatography fractions: A = effluent by-pass valve; B = sample loop valves; C = vacuum exhaust valve; D = knurled compression nuts; E = stainless steel 1/2 in. × 1/2 in. bar; F = glass U tube 4 mm o.d. nominal wall pyrex; G = silica sand; H = effluent line 1/4 in o.d. × 1/16 in. i.d.; I = vacuum line 1/4 in. o.d. nominal wall; J = loop return line 1/4 in. o.d. nominal wall; K = 1/8 in. o.d. nominal wall; L = "O" ring; M = thrust washer.

and tubing assembly is maintained at a temperature of about 100 °C by a heating tape. The direction of flow is from left to right via line H. By means of valves A, B, the

carrier gas stream can be diverted through the glass sample loop F. Valve C is opened to remove the carrier gas via vacuum line I.

The traps consist of glass U tubes constructed of standard wall 4 mm o.d. pyrex; they are attached to the manifold by compression "O" ring seals illustrated in the cross sectioned detail part of Fig. 1. These seals have proven quite reliable and have been in constant use in this laboratory for over two years. The traps have a section

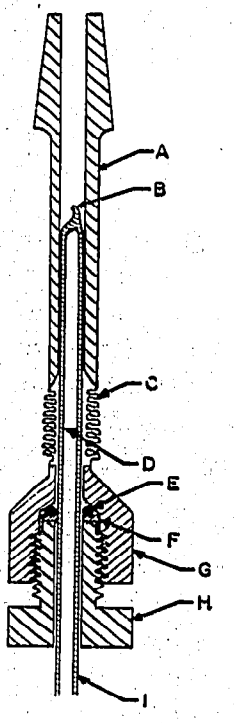

Fig. 2. Device for vacuum transfer of recovered fractions. A = stainless steel male inner connector ground joint; B = seal off tip of glass U tube arm; C = stainless steel sylphon bellows; D = file scratch on glass tubing; E = "O" ring; F = thrust washer; G = knurled body; H = knurled jamb nut; I = glass U tube.

of silica sand in the bottom of the loop retained by plugs of quartz wool. This provides the necessary entrapment for efficient sample recovery. For permanent gases such as methane and carbon monoxide, activated charcoal is substituted for the sand. The trap is held in liquid nitrogen during the collection of a fraction, and while still cold the carrier gas is removed by evacuation through exhaust valve C to a pressure of about 10^{-4} mm Hg. While evacuated and still cold (liquid N_2 temperature), the trap is sealed off at points about 1 inch below the jamb nuts D. A splicing torch has been found highly satisfactory for this operation. The samples are thus sealed off in glass and may be stored indefinitely.

Transfer of the sample to another system by vacuum handling is accomplished by means of the device illustrated in Fig. 2. A small file scratch is made on one of the glass arms about 2 inches below the sealed off end. This is inserted through the "O" ring compression seal E until the scratch is positioned well inside a section of flexible metal sylphon bellows C. The assembly is attached to the system and evacuated. Introduction of the sample is accomplished by flexing the assembly enough to snap the tubing inside at the scratch point.

The above system has been used for collecting several thousand fractions from a chromatograph with subsequent introduction into a mass spectrometer. It has proven to be efficient and trouble-free.

Research Department, U.S. Naval Ordnance Test Station, China Lake, Calif. (U.S.A.)

CHARLES M. DREW
JOSEPH H. JOHNSON

Received March 22nd, 1962

J. Chromatog., 9 (1962) 264–266

Improved sampling valve for gas chromatography

For the gas chromatographic analysis of engine exhaust and air for trace amounts of hydrocarbons, a gas sampling valve with the following features was required: (1) no detectable gas leaks, (2) no contamination or adsorption of the sample, (3) fast switching speed for instantaneous sample injection, (4) interchangeable sample volumes with the smallest about 0.1 ml, and (5) good sampling precision. Five different valves available for laboratory gas chromatographs have been tried, but all failed to meet these requirements.

KARASEK AND AYERS[1] have described a unique gas sampling valve for use with industrial gas chromatographs. One of these pneumatically operated valves, obtained from the Greenbrier Instrument Company, Ronceverte, West Virginia, is shown in Fig. 1 with a four-way solenoid valve (PAL type, Ross Operating Valve Company, Detroit, Michigan). Either 1/16- or 1/8-in. Swagelok fittings may be used and a 1/16-in. tube delivering a sample volume of 0.14 ml is shown.

As received from the manufacturer the valve leaked seriously, but otherwise showed promise of meeting the above requirements. It therefore appeared worthwhile to develop a method for making the valve gastight. This method and an example of the use of the valve in the analysis of highly diluted hydrocarbon mixtures are discussed in this paper.

The disassembled valve is shown in Fig. 2. The stainless steel part (left) is separated from the brass base by a Teflon diaphragm, which opens and closes the valve ports by means of air pressure. In initial attempts to eliminate leaks, other diaphragm materials were evaluated, including Mylar, polyethylene, polyvinyl chloride, and polyurethane. Only with the last material was the valve gastight at 20 p.s.i.g. nitrogen. However, in trials with nitrogen samples containing 0.1 % benzene, the polyurethane adsorbed 10 times as much benzene as did the Teflon diaphragm, so that it was discarded as unsatisfactory for quantitative work.

The surfaces separated by the diaphragm bore the marks (mainly long scratches) of the grinding operation in the manufacture of the valve. It was found that the valve could be made gastight with Teflon diaphragms by polishing both surfaces to a mirror-like finish. The stainless steel part was first polished with 2/0, 3/0, then 4/0 Carborundum emery paper taped to a glass plate. Finer polishing was done with No. 9, No. 6, and No. 1 diamond pastes (Elgin National Watch Company, Elgin,