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A Gas Chromatographic Capillary Column with Negligible Pressure Drop

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NOTE

A Gas Chromatographic Capillary Column with Negligible Pressure Drop

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Summary

Extremely high resolution can be attained with correspondingly long columns made practicable by propelling the carrier gas with a driving force other than inlet pressure.

The analytical resolution in experimental gas chromatography is often limited because it is not practical to use columns that are long enough. The reason is that due to the gradual pressure drop in the carrier gas, the length of the column is utilized ever less efficiently the longer the column becomes. At present, it is rarely worthwhile to make a column longer than 100 m.

In this paper, an open tubular column is proposed which can be made as long as desired because the pressure drop in it remains insignificant. The carrier gas is pushed through the column by a series of droplets of liquid metal which act as pistons while being propelled through the column. An extremely long column of this type can easily be disassembled for renewed coating with stationary phase.

The novel type of capillary column is illustrated in Fig. 1. Its outer shape resembles a ribbon, while its cross section contains a rectangular opening. It is made of a strip of an insulator, such as silicone rubber, sandwiched between two electrodes consisting of strips of nonferromagnetic metal whose outer surfaces are coated with insulating lacquer. Through the insulator strip, which is attached to one of the metal strips, a lengthwise slit is cut, which is covered by pressing the

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FIG. 1. A section of the column described in the text.

other metal strip against it. The metal strips are pressed together by, for example, two zippers (not shown in Fig. 1), each fastened along an edge of the column.

The inside of the column consists of the rectangular channel formed between the strips. Within the channel, the surfaces of the metal strips are of bare metal, while the surfaces of the insulator strip are coated with stationary phase.

The coated column is wrapped around a long, narrow cylinder (e.g., of hollow aluminum), forming a coil which can have many layers of loops wound in the same direction.

When in use, the coiled column is inserted into a solenoid built into the thermostat of the gas chromatograph, and both the solenoid and the column are connected in parallel to a source of dc. The metal droplets which periodically enter the column are moved through it by the principle of a simple dc motor. The electric current flows through every metal droplet in a radial direction, perpendicular to the direction of the magnetic field of the surrounding solenoid. The electrodynamic force acting on the current-bearing droplets is perpendicular to both of these directions and propels the droplets along the circular loops of the column with a speed that tends to be constant.

The solenoid should be made of thick copper wire and bear a dc of constant intensity which should be so high that the solenoid helps to heat the thermostat. Thus, a powerful, homogeneous magnetic field is set up inside the solenoid.

The column, when in use, becomes part of an electric circuit in which it is connected in series with a resistor and a rheostat. The electric resistance of the column must be negligible compared to those of the other two components across which almost the entire potential drop occurs. Under this condition the potential difference across the

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metal droplets will be nearly the same everywhere in the column. The rheostat makes it possible to adjust the flow rate of the carrier gas in the column by controlling the electric current that flows through the metal droplets in the constant magnetic field.

The metal droplets, being liquid, serve as pistons which fit closely everywhere within the column without requiring precision in its construction and without damaging its layer of stationary phase. In most cases it is possible to use mercury, intercepting its toxic vapor chemically as it leaves the detector. For corrosive samples, some low-melting alloys can be used instead; they are nontoxic and so unreactive that they have been utilized to make ampoules for gas chromatography samples. An example of such an alloy is a quinternary eutectic (41.0% Bi, 22.1% Pb, 18.1% In, 10.6% Sn, 8.2% Cd) which melts at 47°C.

Metal droplets of adjustable, uniform size should be produced at regular intervals by an automatic injector located before the column. The droplets introduced into the column should form pistons that are as short as possible and spaced far apart.

Very little electric current needs to flow through the metal droplets in the column to urge them through the highly intense magnetic field. Therefore, a liquid alloy will not be appreciably unmixed by the dc as long as the droplets are permitted to flow. Moreover, the small amount of heat that the current-bearing droplets release will be promptly dissipated through the metal strips. It is important that the temperature of the metal droplets does not rise noticeably so that their passage does not disturb the establishment of gas–liquid equilibria.

Some of the most popular gas chromatography detectors do not respond to the low vapor pressure of the metal droplets, and many others can distinguish the sample from metal vapors. Figure 2 illustrates how dead volume can be avoided in the connection between a column of this type and a detector. The elution time of a droplet is too short to interrupt the recording of a chromatogram.

The proposed column is simple to use. To bring it to the desired temperature, place the column into the solenoid and adjust the controls of the thermostat; turn the carrier gas on to provide a high inlet pressure; switch on the current for the solenoid, for the column, and for the droplet injector. When the column is thermostatted, set the carrier gas to a low inlet pressure and adjust the flow rate with the rheostat.

The useful length of such extremely long columns will be limited by the eventually excessive broadening of the vapor bands due to their

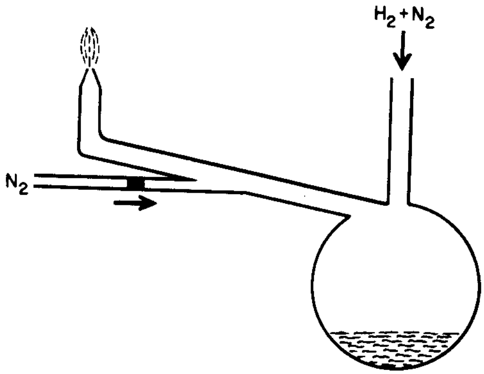

FIG. 2. Connection of a flame-ionization detector to the column described in the text. The carrier gas is pure nitrogen. The liquid metal accumulates in a reservoir.

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diffusion in the gas phase. It is well known that the maximum resolution, within the time allotted by this process, is achieved with monomolecular layers of stationary phase chemically bound to the inert support, as they permit high flow rates. Only with monomolecular stationary phases will the higher expense of this novel column be justified.

The extremely high resolution thus attained should be used for the analysis of isotopically-labeled compounds, of optical isomers, and of natural products such as coffee aroma, etc.

DISCUSSION

In preparative gas chromatography, in which the samples are known and simple, methods for limiting the pressure drop are widely used. Column sections are often connected in series and used consecutively, each with its own carrier gas supply. Circular columns and countercurrent columns are commonly used for binary mixtures.

In analyses with conventional capillary columns, it is customary to diminish the effect of the excessive pressure drop by gradually increasing the inlet pressure during the chromatogram. The relative pressure drop in capillaries can be minimized by maintaining extremely high inlet and outlet pressures, as in gas chromatography of involatile samples with supercritical vapors, but the practicable resolution is decreased by the slower diffusion in the dense gas. Increasing the internal diameter of capillary columns is self-defeating because this lowers the resolution.

Capillary columns that cannot be taken apart are not necessarily advantageous when they are made extremely long, because they become difficult to coat with stationary phase. It is also difficult to cause carrier gas to flow through them with negligible pressure drop, as the following examples show.

Ferromagnetic colloids are promptly removed from a mercury droplet by a magnet. Neither bands of polar or paramagnetic gas, nor droplets of electrically charged or diamagnetic metals can be moved through a capillary by, respectively, static electric or magnetic fields of moderate strength. An alternating magnetic field can repulse a metal droplet, but heats it excessively by eddy currents. Peristaltic pumping, done mechanically, soon damages a flexible column; with electrostriction, the stationary phase decomposes; magnetostriction of presently available materials requires uneconomically large magnets. Metal droplets can be moved by centrifugation through capillaries wound into the shape of a disk or cone. They can also be moved by periodic inertial flow through a capillary coiled around a cylindrical, accelerating rotor. However, these mechanical devices are too unwieldy to fit into a thermostat. The use of a large number of miniature pumps between column sections would be feasible but too expensive.

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