Physical Sciences

Improved Thermal Modulator for Gas Chromatography

Varying temperature can be controlled precisely at relatively low power demand.

Goddard Space Flight Center, Greenbelt, Maryland

An improved thermal modulator has been invented for use in a variant of gas chromatography (GC). The variant in question — denoted as two-dimensional gas chromatography (2DGC) or GC-GC — involves the use of three series-connected chromatographic columns, in the form of capillary tubes coated interiorly with suitable stationary phases (compounds for which different analytes exhibit different degrees of affinity). The two end columns are relatively long and are used as standard GC columns. The thermal modulator includes the middle column, which is relatively short and is not used as a standard GC column: instead, its temperature is modulated to affect timed adsorption and desorption of analyte gases between the two end columns in accordance with a 2DGC protocol.

In general, what is required of a thermal modulator is to vary the temperature of the middle capillary tube in the following cycle:

1. Maintain the tube at a specified low temperature — typically between –10 and –40 °C for a specified time (typically between 1 and 10 seconds);
2. Heat (within tens of milliseconds) the tube to a specified high temperature (typically between 180 and 350 °C) and maintain this temperature for a specified time (typically between 10 and 200 milliseconds); then
3. Cool (preferably within 200 milliseconds) the tube back to the low temperature.

The degree to which this heating-and-cooling profile can be exactly controlled can have significant effects on performance, because of an exponential dependence of gas-elution speed on modulator temperature.

What distinguishes the present thermal modulator from prior thermal modulators is an improved design that enables the required rapid cyclic heating and cooling with greater precision of temperature control and less power demand. The capillary tube is made of metal coated on its inner surface with a protective layer and a suitable stationary phase. Along most of its length, the tube lies in a cylindrical cavity in a housing (see figure). Rapid cooling is achieved through contact between the tube and a coolant liquid that is continuously pumped through the cavity. Rapid heating is achieved by passing a controlled electric current along the tube.

Because of the large radial temperature gradient occasioned by the narrowness of the capillary tube (typically no more than 2 mm wide) and the presence of coolant liquid in contact with the tube, it is difficult or impossible to measure the temperature of the tube accurately by use of a thermocouple, thermistor, or other conventional temperature
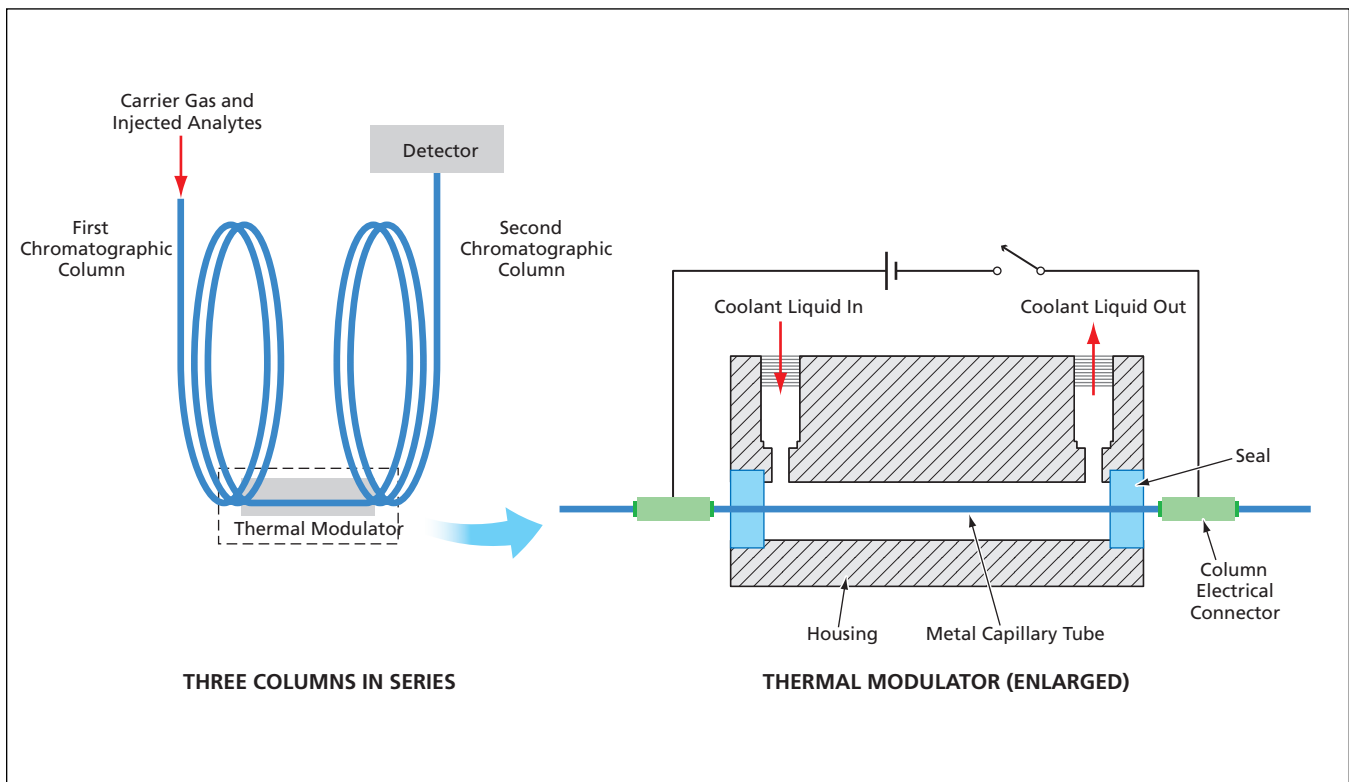
In the Thermal Modulator, the capillary tube is heated ohmically and is in direct contact with a circulating coolant liquid. The electrical resistance of the tube is used as temperature feedback for controlling the applied voltage or current to obtain the desired temperature-versus-time profile.

sensor. In the improved design, no attempt is made to use a conventional temperature sensor. Instead, for purposes of monitoring and feedback control of the temperature, the electrical resistance of the tube is measured as a function of time and the temperature is computed in real time by use of temperature-vs.-resistance data obtained in prior calibration measurements on the tube.

This work was done by Ernest Frederick Hasselbrink, Jr.; Patrick J. Hunt, and Richard D. Sacks of the University of Michigan for Goddard Space Flight Center.

In accordance with Public Law 96-517, the contractor has elected to retain title to this invention. Inquiries concerning rights for its commercial use should be addressed to:

Nuclear-Spin Gyroscope Based on an Atomic Co-Magnetometer
Sensitivity to magnetic fields is eliminated.

John H. Glenn Research Center, Cleveland, Ohio

An experimental nuclear-spin gyroscope is based on an alkali-metal/noble-gas co-magnetometer, which automatically cancels the effects of magnetic fields. Whereas the performances of prior nuclear-spin gyroscopes are limited by sensitivity to magnetic fields, this gyroscope is insensitive to magnetic fields and to other external perturbations. In addition, relative to prior nuclear-spin gyroscopes, this one exhibits greater sensitivity to rotation. There is commercial interest in development of small, highly sensitive gyroscopes. The present experimental device could be a prototype for development of nuclear-spin gyroscopes suitable for navigation. In comparison with fiber-optic gyroscopes, these gyroscopes would draw less power and would be smaller, lighter, more sensitive, and less costly.

The co-magnetometer (see figure) includes a spherical aluminosilicate glass cell containing potassium vapor, several atmospheres of helium-3, and a small quantity of nitrogen (which serves as a buffer gas). The cell resides in a small oven, which is used to maintain the cell contents at a temperature of 170 °C. The oven is located within a housing that includes several layers of magnetic shielding.

Potassium atoms are polarized by optical pumping, and the polarization is transferred to the helium by spin-exchange collisions. A high-power diode laser generates the pump beam, which passes through holes in the magnetic-shielding layers and oven and through the cell along the z axis of an *xyz* Cartesian coordinate system. Another, lower-power diode laser generates a linearly polarized probe beam, which similarly passes through the cell along the z axis. The probe beam is used to measure the direction of polarization of the electrons in the potassium atoms, which is coupled to the nuclear polarization of the helium due to the imaginary part of the spin-exchange cross-section.

For sufficiently high buffer-gas pressure in a spherical cell, this coupling can be represented by an effective magnetic field that each spin species (K or He) experiences from the average magnetization of the other.

It has been shown that the relationships among the electron polarization of the potassium atoms, the nuclear polarization of the helium atoms, the magnetic fields, and the mechanical rotation of the magnetometer are described by a system of coupled Bloch equations. The equations have been solved to obtain an equation for (1) a compensating magnetic field, automatically generated in the magnetometer, that exactly cancels other magnetic fields; and (2) a gyroscope output signal that is proportional to the rate of mechanical rotation about the y axis and independent of magnetic fields. In experiments, the gyroscope-output equation has been verified to within a calibration error of 3 percent, and the expected insensitivity to rotation about the x and z axes was confirmed. In a future version, sensitivity could be increased by substitut-
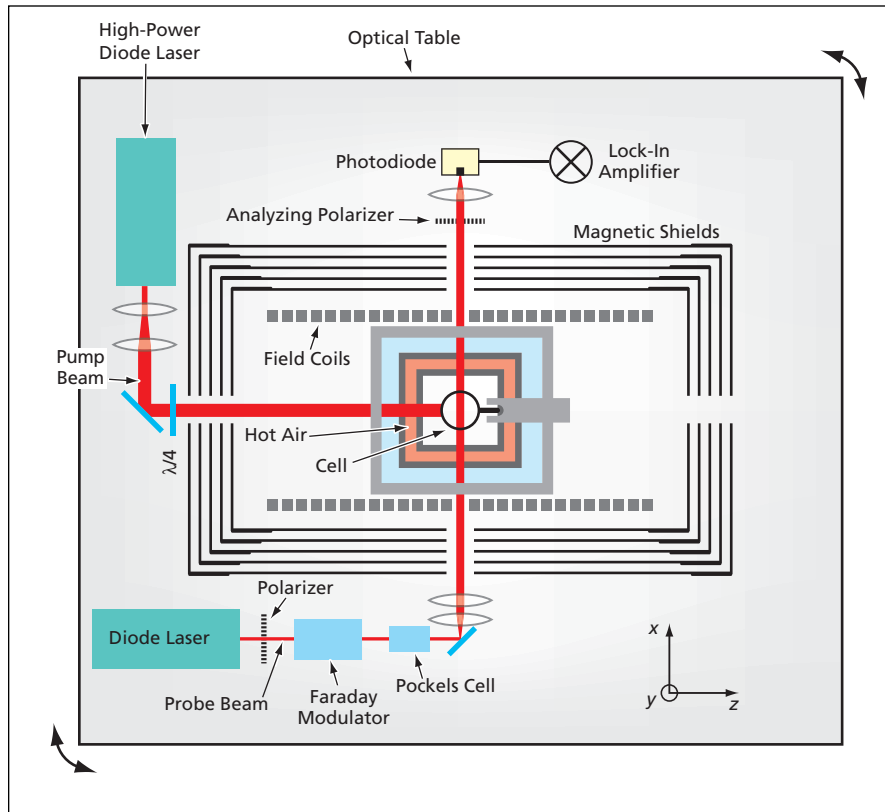
This **Atomic Co-Magnetometer** generates an output signal proportional to the rate of rotation about the y axis.