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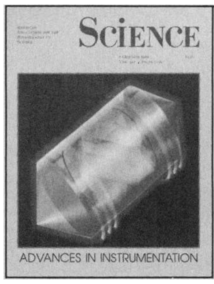

COVER Motions involved in double rotation, a means to obtain sharp nuclear magnetic resonance (NMR) spectra for quadrupolar nuclei in solid materials. The sample is contained in the inner (orange) cylinder, which rotates ~7000 times per second around an axis inclined at 30.6° to the axis of the outer (blue) cylinder. The outer cylinder, which has a diameter of ~1 centimeter, rotates ~1000 times per second around an axis inclined at 54.7° to the magnetic field. See page 71. [Computer-generated image courtesy of Lawrence Berkeley Laboratory]

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Analytical Instrumentation and Measurements

Improvements in analytical instruments and methods continue to expand opportunities for research in many areas of the biological and physical sciences. Some of these advances are described in four articles in the current issue of *Science*.

Gas, liquid, and supercritical fluid chromatography as a group constitute some of the most broadly applicable analytical tools available to the natural sciences. In this issue Novotny surveys recent developments that have increased their usefulness. In gas chromatography, open tubular columns have replaced packed columns, leading to shortened time of analysis and less contamination from packing. Advances have been made in treating the stationary surfaces of the columns with highly selective phases, including chiral substances. Thermal stability has been improved dramatically. Natural petroporphyrins have been separated at column temperatures as high as 420°C. Many interesting advances have been made in detection technology.

Applications of supercritical fluid chromatography continue to increase. The less dense fluids (usually CO_2 or N_2O) permit faster solute diffusion than do liquids. They make possible improved separations of substances that are insufficiently stable or volatile for gas chromatography or are difficult to detect by liquid chromatography. Compounds in excess of 10,000 daltons can be separated and then quantified by flame detectors. Major applications include heavy constituents of fossil fuels, synthetic oligomeric mixtures, polymer additives, food products, and agricultural chemicals.

Kennedy and colleagues in Jorgenson's laboratory describe efforts aimed at exploiting the sensitivity and separative powers that can be achieved by open tubular liquid chromatography (OTLC) and by capillary electrophoresis. The theory of OTLC predicts that a column with an inside diameter of 2 micrometers would generate 1 million theoretical plates with an analysis time of less than an hour. More than 1 million theoretical plates have been achieved in the separation of proteins using capillary zone electrophoresis. Typical injection volumes are on the order of picoliters to nanoliters. The authors point out that the major challenge is quantitating the results of such separations. Detectors are required to be sensitive to femtomole and attomole amounts of analyte. They have successfully made use of electrochemical detectors and laser-induced fluorescence. The resultant capabilities have been applied to analysis of single neurons of the land snail *Helix aspersa*. They were able to obtain semiquantitative values for 17 of the free amino acids in a single cell.

Fenn and colleagues discuss means of achieving multiple ionization of large molecules without fragmenting them. They mention achieving intact ions with 20 or more charges. This permits easier analysis in mass spectrometers. For example, an ionized protein of molecular weight 30,000 could have a mass-to-charge ratio of 1,500. In practice they obtain a coherent series of peaks differing by a unit of charge. Spectra have also been obtained for oligonucleotides. In the electrospray system a sample solution 1 to 20 microliters in volume enters a chamber through a needle maintained at a few kilovolts. The emerging liquid is charged, and this causes droplet dispersion due to Coulomb repulsion. Evaporation of the solvent leads to a further explosion of droplets and ultimately to a field at the surface of the droplets sufficient to desorb ions.

Chmelka and Pines review some of the many developments in the nuclear magnetic resonance (NMR) of solids. In the past the NMR of solids lagged far behind that of liquids. In liquids, high resolution with narrow lines is attained, owing to the rapid isotropic nature of molecular motion. In solids many of the deleterious effects due to the lack of rapid isotropic motion can be minimized by magic-angle spinning of samples. A key improvement is the use of double rotation of the solid materials simultaneously about axes inclined at two magic angles. This technology, which has been demonstrated for oxygen-17 in silicates, makes it possible to obtain spectra of improved resolution from nearly all NMR-active nuclei of the periodic table. In many cases enriched rare isotopes would be used.

The authors provide numerous examples of insights that have been obtained in the study of solids. For example, recent NMR experiments have probed solids and adsorbed molecules in the millikelvin range and at 1500 K. The high-temperature studies have provided information about structure and motion in magmas.—PHILIP H. ABELSON