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Portable High-Resolution NMR Sensor Unveiled at Berkeley

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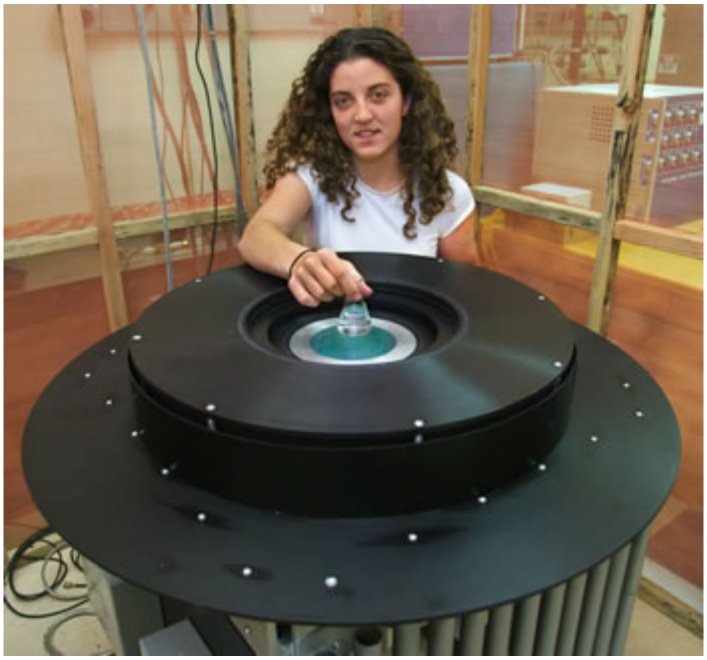
BERKELEY, CA – Homeland security experts may soon be getting a valuable new tool for identifying the chemical constituents in suspicious substances. A portable device makes it possible for the first time ever to take high-resolution NMR spectroscopy — one of the principal tools for chemical analysis — out of the laboratory and into the field for use on samples of any size. This portable NMR sensor was developed by a collaboration of researchers with the U.S. Department of Energy's Lawrence Berkeley National Laboratory (Berkeley Lab), the University of California at Berkeley and the Institute for Technical Chemistry and Macromolecular Chemistry in Aachen, Germany.



Vasiliki Demas loads a sample for analysis inside a low-field electromagnet currently being used as a testbed for obtaining high-resolution NMR spectra without the constraints of having to place samples inside the bore of a high-field magnet.

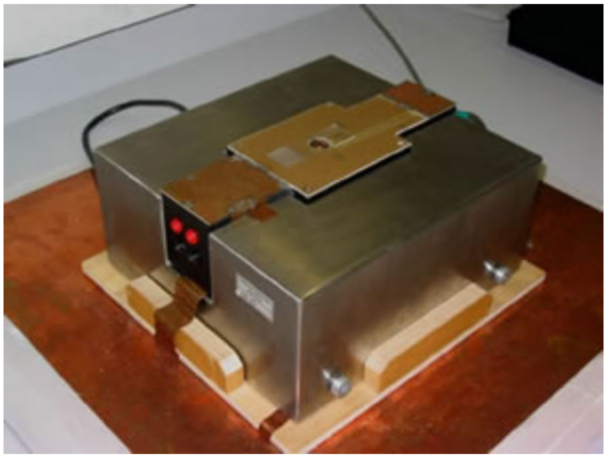
"Our device does not compete with the superconducting magnets that are used to study proteins, but there are many applications, besides homeland security, where you can't bring samples from the field to the laboratory, including medical diagnosis, archaeological analysis, or the exploration of objects in space, like planets or moons," said chemical engineering graduate student Vasiliki Demas, one of the co-authors of a paper describing the portable NMR device, which appears in the April 8, 2005 issue of the journal *Science*. Demas is a member of the research groups of Jeffrey Reimer, who holds joint appointments with Berkeley Lab's Energy and Environmental Technologies Division and UC Berkeley's Chemical Engineering Department, and Alexander Pines, a chemist with joint appointments at Berkeley Lab's Materials Sciences Division and UC Berkeley's Chemistry Department.

Other principal authors of the *Science* paper were Bernhard Blümich, Federico Casanova and Juan Perlo, of the Aachen institute, and Carlos Meriles, a physicist now at City College of New York, in addition to Reimer and Pines. Blümich is the co-inventor of the NMR Mobile Surface Universal Explorer, Reimer is an expert in

NMR technologies for materials engineering, and Pines is one of the world’s foremost authorities on NMR spectroscopy.

NMR is a phenomenon involving the atomic nuclei of molecules in which at least one proton or neutron is unpaired. The imbalance causes such nuclei to spin on an axis like miniature tops and gives rise to a magnetic moment, which means the nuclei act as if they were bar magnets with a north and south pole. When a sample is exposed to a strong external magnetic field, these spinning "bar magnets" attempt to align their axes along the lines of magnetic force. The alignment is not exact, resulting in a wobbly rotation about the force lines that is unique for each type of nuclei. If, while exposed to the magnetic field, the nuclei in a sample are also hit with a radiofrequency (rf) pulse, they will absorb and re-emit energy at specific frequencies according to their individual rates of rotation. These frequencies show up in an NMR spectrum as distinct peaks of varying height that, like a set of fingerprints, can be used to identify the sample’s constituent nuclei.

Because the rate at which resonating nuclei realign themselves with magnetic field lines is heavily influenced by their neighboring nuclei, NMR can also be used to provide detailed information on the structural, dynamic, and spatial relationships of atoms in a sample. Deviations from reference peaks on the NMR spectrum, called "chemical shifts," reflect different concentrations of a sample’s constituent nuclei and can be used to positively identify the molecular composition and chemical nature of the sample.



This portable device utilizes a unique “ex-situ” NMR technology to make it possible to obtain a high-resolution NMR spectrum outside of the laboratory and from samples of any size.

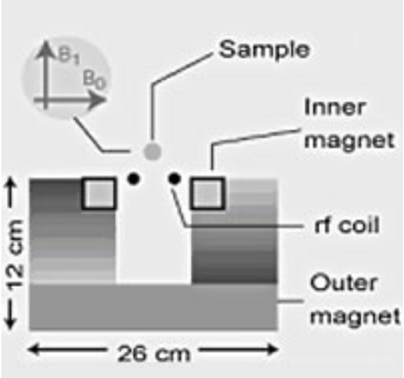
Until recently, high resolution NMR spectroscopy could only be done by placing a sample inside the bore of a very large stationary magnet that produces a strong, uniform magnetic field. Portable NMR systems with open, single-sided probes, have been built, but the lack of uniformity in their magnetic fields limit them to low resolution.

“The variations within the magnetic fields of previous portable NMR devices are usually orders of magnitude too large to detect chemical shifts,” said Demas. “These devices mainly yield relaxation times as a crude estimate of a sample’s composition.”

In 2001, an effort led by Pines, Meriles, and Dimitris Sakellariou, formerly with the UC Berkeley chemistry department, demonstrated a technique for obtaining high-resolution NMR spectroscopy data from samples in a grossly nonuniform magnetic field. In this “ex situ" method, the lack of uniformity in the magnetic field is compensated for by zapping a sample with a series of rf pulses that are precisely varied in energy, duration and timing.

Said Meriles at the time this technique was announced, "We have demonstrated that high-resolution NMR spectra can be recovered even with a strongly inhomogeneous magnetic field, which means it may be possible to develop a mobile magnet that can be scanned over otherwise inaccessible objects to get magnetic resonance information."

The authors of this *Science* paper applied the ex-situ technique in a portable single-sided sensor device, which, for these experiments, was configured for high resolution NMR spectroscopy. Two



In this conifguration for ex-situ high-resolution NMR spectroscopy, an inner magnet opposes the field created by the outer magnet to induce a sweet spot 7 millimeters above the magnet surface where a sample can be placed for analysis. An rf coil is positioned to optimize its interaction with the static magnetic field.

concentric U-shaped permanent rare earth magnets (Neodymium-Iron-Boron) were arranged to create a magnetic a "sweet spot" about seven millimeters above the active magnet surface. In this sweet spot, the static magnetic field is parallel to the plane and reaches a field strength of 0.2 Tesla. The rf pulses were generated through a rectangular rf coil that is shaped and positioned with the inner of the rare earth magnets to optimize the interaction between the static magnetic field and the magnetic fields generated by the rf pulses. When a sample is placed in the sweet spot and hit with a carefully orchestrated series of rf pulses the result is an NMR spectrum with a sharp enough resolution to reveal chemical shifts of eight parts per million within three minutes after sample testing begins.

"Eventually, we think we can attain a better than one part per million resolution, that is our goal for when this device becomes commercially available," said Demas, who recently returned from Germany where she had been participating in tests of the new NMR sensor. In the proof of principle testing, Demas and her colleagues were able to acquire high resolution NMR spectra of fluorine nuclei in several types of liquid fluorocarbons.

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Berkeley Lab is a U.S. Department of Energy national laboratory located in Berkeley, California. It conducts unclassified scientific research and is managed by the University of California. Visit our Website at www.lbl.gov.

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