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SECURING THE FUTURE OF NMR

With the **HELIUM SHORTAGE** in the past, scientists seek to ensure supplies of the cooling element and keep legacy instruments running

MARC S. REISCH, C&EN NEW YORK CITY

IN GOOD TIMES, problems that seemed important not very long ago are often forgotten. That is not the case for scientists who use nuclear magnetic resonance spectroscopy in their research. They still recall the pain of recent helium shortages and the trauma of Agilent Technologies' exit as a maker of high-field NMR instruments.

The helium shortage is over, but a number of scientists are making plans in case of another one. Without helium, NMRs are unable to perform their analyses of small molecules, natural products, proteins, and advanced materials. No other substitute will do to keep the machines' magnets at their optimum operating temperature of –269 °C.

A committee formed by three scientific societies—the American Chemical Society, the American Physical Society (APS), and the Materials Research Society—seeks to ensure the long-term availability of helium. It plans to issue a report later this year containing policy recommendations meant to avoid future shortages and help reduce helium costs, which are more than four times what they were a decade ago and can consume as much as one-third of research grant funds.

A second initiative by ACS, which publishes C&EN, and APS will enlarge a pilot helium-buying consortium. Physicists use helium to conduct low-temperature experiments.

Separately, operators of Agilent NMR systems anxious to keep their legacy instruments viable can now count on free access to the software and source code that govern their machines' operation. A deal recently signed by Agilent and the University of Oregon will allow scientists to update the formerly proprietary software to serve their research needs.

Scientists who use NMR have been through a lot in the past few years. Helium shortages in 2007 and then again in 2012 and 2013 deprived them of the element, which is needed to cool the superconducting magnets in their instruments. During the shortages, prices spiked, and in some cases machines that cost as much as $1 million were damaged when scientists could not get what they needed.

Researchers received another blow in late 2014 when instrument maker Agilent decided to close its NMR business and limit support of its old product line.

But things are looking up again. The opening of a helium plant in Qatar's Ras Laffan Industrial City in 2013 brought more than 37 million m^3 per year of the element to the market. The new source, equivalent to almost 40% of current U.S. production, effectively ended the last shortage, notes Maura Garvey, marketing research director at consulting firm Intelligas Consulting.

The new source and a moderation in demand should keep supply and demand in balance through 2018, Garvey notes.

However, beginning with 2019, all bets are off. Helium is mainly recovered from natural gas and carbon dioxide wells, Garvey notes. Should a warm winter or a cool economy cause gas demand to fall, helium supply will fall as well. The logistics of transporting helium from distant and sometimes volatile regions of the world could also impact availability, she points out.

Helium users also have to consider that the U.S. government's program to sell its helium reserves will end by 2021, Garvey notes. Sales from the reserve at one time accounted for about one-third of global supply but now account for less than 15%. The program had "acted as a flywheel" to stabilize prices and supplies for years, she says.

The possibility of future shortages is what motivated the three scientific societies to form their committee. Heading the 12-member group, four from each society, is Simon Bare, an R&D fellow and expert on heterogenous catalysis at UOP, a process technology and catalysis firm owned by Honeywell.

"Each time there is a helium crisis, it's the small researcher that takes the brunt of a shortage," Bare says.

SONNY AN/U OF ILLINOIS, URBANA-CHAMPAIGN

GREAT ESCAPE Plumes of escaping helium surround Olson as he prepares to insert liquid helium into a superconducting magnet.

"We have to think of ways to conserve and recycle helium."

Helium is a slippery devil. It sneaks past even the tightest seals closing off superconducting NMR magnets. Unrestrained, it bolts off into the atmosphere and then into space. "Once it's gone, an element that took millions of years to make through radioactive decay and was sequestered in deep underground wells is gone for good," Bare says.

U OF OREGON

GOING STRONG Open source software will keep this old Varian NMR, operated by Strain, useful to scientists.

As it stands now, manufacturers offer NMRs that can capture and recycle most of the helium that boils off during use. But not everyone pays for the upgrade, and thousands of older instruments are still in use. Depending on how many instruments are in place at an institution, users consume anywhere from a few hundred to thousands of liters of helium per year.

Some larger NMR facilities have found other ways to recycle helium. At the University of Illinois, Urbana-Champaign, Dean Olson, who is responsible for the chemistry department's 11 NMRs, expects that a recently installed capture system linked to the physics department's liquefaction facility should bring his annual helium cost down to about $48,000 from $80,000.

Olson was able to tie in to the physics department's $600,000 worth of helium compressors and collection pipelines. But not everyone is so lucky.

Sophia Hayes, a professor of chemistry at Washington University in St. Louis and member of the scientific societies' helium committee, says funds for recycling are tight. Her university has a decades-old helium capture system that is leaky and inefficient. And because the captured helium is a low grade of gas, all that it is good for is filling party balloons, Hayes laments.

Hayes would like to have a state-of-the-art recovery system in place for the three NMRs she uses. She has sometimes had to divert funds that might otherwise be used to pay other research expenses to instead pay for helium that now costs more than $17 per L, up from about $4.00 a decade ago, she says.

Were she to have the funding, Hayes could consider some of the new lab-sized helium recycling units. Peter Gifford, president of Syracuse-based Cryomech, says his firm makes a $170,000 system that recovers up to 22 L per day from one or more NMRs. Although physics labs have shown interest in the system, Gifford says he is puzzled that few chemistry labs have installed it.

A recent survey of NMR user spending plans suggests that when shortages abate, users lose interest in helium recycling. The survey, by investment research firm Cleveland Research Co., found that only 3% of 74 respondents ordered an NMR system with recycling capabilities between June and December of 2015, down from 8% in the first half of 2015 and 13% in the second half of 2014, when the shortage still lingered.

Shock at the added cost of systems with such capabilities appears to hold back purchases. Cleveland Research found that respondents figure it would take too long—10 years—to pay off the added cost of a recycling system through helium savings.

Some of those worried about helium costs and availability have already found relief through an initiative of ACS and APS. A pilot program that got under way in 2015 ensures consistent supplies at stable prices to seven institutions: Stanford University, Boise State University, West Texas A&M University, the University of Memphis, the University of New Hampshire, Amherst College, and Worcester Polytechnic Institute.

The buying initiative takes advantage of a provision in the government's helium sales program that gives federally funded researchers priority in obtaining helium. The seven institutions experienced more reliable deliveries and on average saved 15%, according to Ryan Davison of the ACS Office of Public Affairs. Davison and his counterpart at APS, Mark Elsesser, a policy analyst, are counting on all seven institutions to remain in the program this year and 12 new ones to join.

Helium availability hasn't been the only problem for owners of NMR instruments made by Agilent and its predecessor Varian. After Agilent's abrupt exit from the business in 2014, they have worried about being left without the latest operating and analytical software.

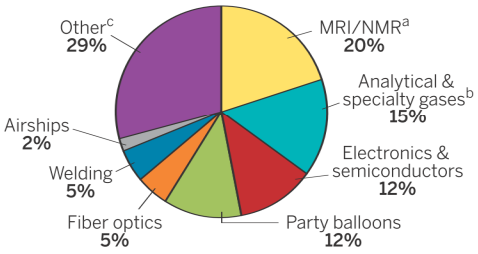
Thanks to the deal between Agilent and the University of Oregon, Agilent is making the software and source code for its legacy NMRs and magnetic resonance imaging instruments available to scientists, according to Michael Strain, who runs Oregon's NMR facility. Agilent has also transferred operating document copyrights to Oregon and has posted some equipment schematics on its Web portal to allow scientists to maintain their equipment.

With the software gift, Strain says, "we'll be able to support our legacy systems for a long time." Programmers scrubbed the software to remove Agilent copyrights and some code that Agilent licensed but did not own. Strain says he has put the open source version of the software, known as VnmrJ, on two Varian NMRs, and "it actually works."

Two of Oregon's Varian NMRs are more than 20 years old, Strain explains, and given the dearth of federal funding, replacing them is a problem. With access to the software, "we'll gain the ability to add new algorithms to manipulate data and affect hardware operations."

Nobel Prizes have been won for the discovery, development, and application of NMR spectroscopy, points out Bare, the helium committee chairman. If NMR is to thrive as an analytical science, he says, scientists "need to keep hammering away" at the problems they face in good times and bad. ■

LIGHT TOUCH Helium has a diverse range of uses.



Global helium consumption in 2015 = 168 million m^3

a Magnetic resonance imaging is a larger use than nuclear magnetic resonance. **b** Includes gas chromatography and mass spectrometry. **c** Includes leak detection, space launch vehicles, and particle collider research. **SOURCE:** Intelligas Consulting