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# Analytical chemistry: Ultrasensitive radiocarbon detection

Richard N. Zare<sup>1</sup>

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Radiocarbon is rare, forming no more than one part per trillion of the total carbon content of the atmosphere. An optical method allows radiocarbon to be detected at roughly 25-fold lower levels than this, opening up fresh avenues of research.

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Radiocarbon dating is an invaluable technique for determining the age of carbon-containing samples up to about 50,000 years old. Until now, the only method available for measuring levels of radiocarbon (carbon-14) in a sample has been high-energy accelerator mass spectrometry, but the apparatus involved is bulky, expensive and complex. Reporting in *Physical Review Letters*, Galli *et al.*<sup>1</sup> describe an optical technique for measuring radiocarbon concentration that might overcome these problems. Their approach promises to greatly extend the use of radiocarbon measurements for dating, and as a tracer technique for following the fate of organic compounds in the body. It forms part of a growing revolution that is replacing mass spectrometry with optical methods for isotope analysis<sup>2, 3, 4</sup>.

On Earth, there are three naturally occurring isotopes of carbon. The most abundant of these (99%) is carbon-12, with most of the rest being carbon-13. But carbon-14 also occurs in trace amounts, forming only as much as 1 part per trillion (0.0000000001%) of the carbon in Earth's atmosphere. Unlike <sup>12</sup>C and <sup>13</sup>C, <sup>14</sup>C is radioactive, decaying with a half-life of about 5,730 years — a fact that makes it potentially useful as a radiolabel for several applications.

Carbon-14 is mainly produced in Earth's atmosphere from the bombardment of nitrogen molecules by cosmic rays. Plants fix atmospheric carbon dioxide during photosynthesis, and so the level of <sup>14</sup>C in plants (and in animals that eat plants) when they die approximately equals the level of the isotope in the atmosphere at that time. Because the amount of <sup>14</sup>C in dead organisms subsequently decreases as a result of radioactive decay, the date of carbon fixation (or death) can be determined

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by measuring the amount of the isotope in the remains. This is the basis of radiocarbon dating, the technique that has been a workhorse for estimating the age of organic remains from archaeological sites. In practice, the ratio of the number of  $^{14}\text{C}$  atoms to the total number of other carbon atoms in a sample is measured.

Because of the paucity of  $^{14}\text{C}$ , radiocarbon dating presents a huge technical challenge. Most isotope-ratio measurements are carried out using mass spectrometry, in which ions are weighed by measuring their trajectories in electric and/or magnetic fields in a vacuum. Standard mass spectrometers, however, do not have sufficient resolution to distinguish the small mass difference between  $^{14}\text{C}$  and  $^{14}\text{N}$ , the most common isotope of nitrogen. The abundant presence of  $^{14}\text{N}$  therefore tends to mask any signal from radiocarbon.

High-energy accelerator mass spectrometry can overcome this problem. For this technique, the sample must first be turned into solid carbon (graphite) using a series of chemical transformations. It is then bombarded with caesium ions to produce negatively charged carbon ions, which are accelerated by a positive voltage of millions of volts. The negative ions, by now travelling at a few per cent of the speed of light, are subsequently converted into positive ions by an electron stripper (which consists of a gas or a thin foil), before the ions' masses are determined. Because nitrogen atoms do not form stable negative ions, the resulting data are free from nitrogen interference. But although accelerator mass spectrometers are powerful tools, they are also costly. Establishing and maintaining such an instrument costs millions of dollars, and so they tend to be found only at national facilities.

A much simpler approach is to completely oxidize a sample so that every carbon atom is turned into carbon dioxide<sup>3</sup>. The various isotopic forms of carbon dioxide can then be distinguished from each other because each has a slightly different infrared spectrum (they absorb slightly different frequencies of infrared light). All that is required to determine the ratios of carbon isotopes in a sample of carbon dioxide is to precisely measure the intensities of the spectral lines that correspond to infrared absorption for each isotopic form of the gas. Other compounds, such as water vapour and nitrogen, do not interfere in the infrared spectrum, either because they have different infrared spectra or because they do not have 'allowed' infrared transitions — that is, quantum mechanics prevents the molecules from undergoing energy transitions that would be detected in the infrared.

This optical approach has already been used<sup>3</sup> to determine the carbon-isotope ratio of carbon dioxide containing  $^{12}\text{C}$  and  $^{13}\text{C}$ , but the low concentration of  $^{14}\text{C}$  has made its measurement in carbon dioxide extremely difficult. Using an ultrasensitive technique called saturated-absorption cavity ring-down spectroscopy<sup>5</sup>, Galli *et al.*<sup>1</sup> have now succeeded in measuring the ratio of  $^{14}\text{C}$  to total carbon at values well below radiocarbon's natural abundance in carbon dioxide.

In their technique, the authors placed a gas sample between two or more highly reflecting mirrors that form an optical cavity. Infrared light that is incident on the cavity continually circulates within it, so that it takes many round trips. This effectively increases the optical path length of the light, allowing infrared absorption by the gas to be detected with a sensitivity that vastly exceeds what can be achieved in traditional absorption experiments.

Another feature of the cavity is that, when the infrared light source is interrupted, the radiant energy stored in the cavity 'rings down' — it decreases over time. Using a powerful infrared laser to 'saturate' the vibrational-rotational transitions in carbon dioxide that correspond to infrared absorption, Galli *et al.* used the rate of ring down as an excellent absolute measure of the concentration of absorbers inside the cavity (an approach that has previously been reported for infrared spectroscopy<sup>5</sup>). The authors obtained a linear concentration response down to a detection limit of about 43 parts per quadrillion, which makes their technique quite well suited for radiodating carbonaceous samples. It may also have applications in positron emission tomography (an imaging technique used in medicine for body scans), which often requires<sup>6</sup> monitoring of carbon dioxide labelled with carbon-11, an artificial radioactive isotope of carbon.

Galli and colleagues say that the size of their experimental set-up is roughly two square metres in area, about 100 times smaller than the footprint of typical accelerator mass spectrometers. Furthermore, the equipment costs only about US\$400,000 — many times less than an accelerator mass spectrometer. For widespread adoption of the infrared technology, however, it will be necessary to reduce the cost even further, say by a factor of five or ten. Even so, an infrared method for measuring isotope ratios represents a real breakthrough because of the many possible uses of the technique. And there are other advantages. For example, in mass spectrometry, an ion from a sample is counted only once because its measurement neutralizes it. But infrared-absorption measurements do not destroy the sample, allowing it to be repeatedly

analysed.

With further improvements, the infrared technique may well become the method of choice for measuring the isotope ratios of many common elements. Moreover, if the anticipated cost reductions are realized, the measurement of isotope ratios might become a widely used tool in determining the origins of materials used for a broad range of purposes, from environmental monitoring to medical research.

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### Affiliations

Richard N. Zare is in the Department of Chemistry, Stanford University, Stanford, California 94305-5080, USA.

### Corresponding author

Correspondence to: [Richard N. Zare](#)

### Author Details

Richard N. Zare

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