

# Studying the chemistry as it happens in catalytic reactions

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(PhysOrg.com) -- While retaining their speed, catalysts have lost some of their secrets, thanks to a new probe built by scientists at Pacific Northwest National Laboratory to help clarify the steps catalysts take in promoting reactions. The new device is called a large-sample-volume constant-flow magic angle spinning probe for use in a nuclear magnetic resonance (NMR) spectrometer. With it, scientists can flow a gaseous reaction mixture through a solid catalyst and collect NMR data on the intermediates and products generated during the reaction. In addition, using NMR can provide structural information about the catalyst itself

during the reaction.

"Scientists have been trying for a long time to get something closer to a realistic environment with NMR data. This is the newest approach to doing that," said Dr. Charles Peden, a catalysis researcher in the Institute for Integrated Catalysis at PNNL who worked on the study.

From refining gasoline to manufacturing margarine, catalysts are involved in ~90% of all commercially produced chemical products. Making existing catalysts more effective or devising new ones could reduce costly inefficiencies in current processes, and could enable new commercial processes to produce fuels and chemicals. To improve existing and invent new catalysts and catalytic processes, scientists need data about the steps that occur during the reaction. With this new probe, scientists get that type of detailed information via NMR spectroscopy.

"NMR is a powerful technique. Being able to apply it to catalytic reactions while they are occurring has been a really tough problem. This new in situ NMR probe lets us perform experiments we couldn't do before," said Dr. Jian Zhi Hu, the lead PNNL scientist on this study.

In a reaction that turns chemical A into chemical Z, many intermediates can be formed. Some of these can become undesirable waste products, but others eventually form the desired product.

The challenge with using NMR for catalysis centers on a technique called magic angle spinning. This method requires spinning the [catalyst](#) sample inside the NMR instrument. The scientists want the sample to represent how it exists during the actual industrial catalytic process. They want the reaction of gaseous feedstocks over the solid catalyst to be taking place right when they collect the NMR data.

"You need to spin the sample—often several thousand times a second,

but it's not possible with gas lines directly attached," said Hu.

But, they really wanted the data.

So, working together in EMSL, they devised a new method that allowed them to use magic angle spinning on solid catalyst materials with the desired continuous flow of gaseous materials. They built an NMR probe, containing the mechanisms for magic angle sample spinning and an NMR transmitter and receiver, which fit into the heart of the spectrometer's superconducting magnet. At one end of the probe is a heatable sample cell filled with a solid catalyst. This cell includes some special connections to gas lines that allow for introduction of fresh reactants into the sample chamber. In the chamber, the gas reacts with the catalyst. The gases, now containing varying concentrations of reactants and products, are pulled to a vacuum pump through the tube. The connections of the magic angle spinning cell to the gas lines are made in such a way as to minimize leakage while still allowing for high speed spinning. Another important feature is that the probe allows for the use of a large catalyst sample volume for enhanced sensitivity.

"EMSL has a really fantastic technical team that can construct these probes," said Peden, who has conducted experiments at EMSL since the facility opened in 1997.

Next, the team used the device to follow several different catalytic reactions. For example, they followed a dehydration reaction that removes a water molecule from a 4-carbon alcohol. The team identified the type and number of different isomers of the product that formed during the catalytic reaction.

This new probe will soon be available to scientists through EMSL's user proposal process.

The scientists are conducting experiments on different catalysts and catalyzed reactions, working in collaboration with colleagues from the University of California at Berkeley. In addition, they are considering how to refine the probe, creating a next-generation device that can even more closely match industrial catalysis conditions.

**More information:** JZ Hu, et al. 2012. "[A Large Sample Volume Magic Angle Spinning Nuclear Magnetic Resonance Probe for In Situ Investigations with Constant Flow of Reactants](#)." *Physical Chemistry Chemical Physics* 14, 2137-2143. [DOI: 10.1039/c1cp22692d](#)

Provided by Pacific Northwest National Laboratory

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