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Weighing Reproducibility

Researchers confront an array of issues and questions when experimental work cannot be reproduced

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It's a frustrating reality in chemistry and other disciplines that hard and thorough scientific work sometimes cannot be reproduced, for a variety of reasons. When that happens, months or years of effort behind exciting results can be negated.

And if irreproducible results have been published, the tangle of working with other labs and reworking experiments to try to find out what has gone wrong and why can mean even more time and effort spent in what may turn out to be a blind alley.

In the past year, an unprecedented five papers and parts of a sixth were retracted from the *Journal of the American Chemical Society* by **Dalibor Sames**, a professor of chemistry at Columbia University (*C&EN Online*, **Latest News, June 16**). He also withdrew a paper from *Organic Letters*, another ACS journal. (ACS is the publisher of C&EN.)

Sames says the work by coauthor and former Ph.D. student Bengu Sezen cannot be reproduced in his lab or other labs. He has refused to speak further with the media because Columbia has been investigating the matter.

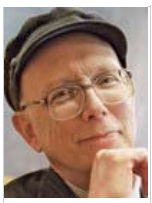
Sezen, on the other hand, has cried foul over the retractions and insists that the work can and has been reproduced (*C&EN Online*, **Latest News, March 23**). What had been a successful collaboration between a doctoral student and a professor is now shattered, and they no longer maintain direct contact. Sames has moved on with his work at Columbia, and Sezen is pursuing another Ph.D. at the University of Heidelberg in Germany.

Although Columbia is conducting an investigation, Sezen says university officials have had minimal contact with her and have given no timetable for completing their review. In the meantime, she has waged a one-woman e-mail campaign to have her published work reinstated. But it remains unclear when and how this situation will be resolved.

Most cases of irreproducible results don't lead to such controversy. And as many researchers point out, there is something to be learned even when experiments fail or fall apart under greater scrutiny.

"If it's science, the work has to be reproducible," says Harvard University chemistry professor **George M. Whitesides**. But in reality, he notes, the issue of reproducibility varies from field to field. In the life sciences, for example, valid work may be irreproducible because of the unique nature of living experimental materials or subjects, including human subjects.

In most of physics and chemistry, however, "you should be able to reproduce everything," Whitesides says. "Chemical experiments are intrinsically more reproducible than some biological and biomedical experiments." If results or reactions cannot be reproduced, "you must understand why. Is it by error or by intent? And you can't say somebody's work is irreproducible without being sure of it."



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"If it can't be reproduced, it should be retracted," says University of Utah chemistry professor C. Dale Poulter. "Some techniques are hard to reproduce, and that's okay for a finite period of time. But at some point, if the work can't be reproduced, then clearly something is wrong."

As for the Sames/Sezen case, Whitesides cautions about drawing any premature conclusions. He says he doubts that scientific fraud is an issue, because the retractions dealt with "an interesting synthetic reaction" with no obvious payoff from fraud. "I assume both parties are right, but I wonder why they haven't gotten together to resolve the matter. Cases of fraud in chemistry are pretty rare."

Whatever the cause of any irreproducible result, Whitesides continues, "if work cannot be reproduced, then the principal investigator is ethically constrained to say that. You have to retract. But you want to make sure that retraction doesn't add to the confusion."

Michl
Credit: Courtesy of Josef Michl

Many factors may account for work in chemistry that cannot be repeated. "My initial conclusion is that I am doing something wrong," says **Josef Michl**, professor of chemistry at the University of Colorado, Boulder. "I may have a solvent or a chemical that is impure or contains a different kind of impurity than the authors had since it comes from a different vendor. Or, I may wash my glassware in a different way, or I may have a systematic error in my instrumentation. I may have different humidity, or it may be more subtle: I may be at a different altitude, and therefore my liquid nitrogen boils at a slightly different temperature."

"Sometimes part of the art of chemistry doesn't get included in published papers," Whitesides says.

And even though cases of fraud in chemistry might be rare, they do happen.

"I kept thinking this is just too good to be true," says University of California, Berkeley, chemistry professor **Richard Saykally** of work from his lab by a postdoc that was being prepared for publication some 15 years ago. The results of the study concerning the surface of liquids, he says, jumped out because they were so remarkable.

Saykally followed his gut feeling about the postdoc's work. "I started getting suspicious of his experiments," he says, "and had another student work with him to reproduce the results."

In the end, Saykally says, "it was all junk. The guy had faked every bit of the data." The postdoc had apparently used a software program to massage his data far beyond the actual experimental results.

"We were about to submit this to a journal," Saykally says, adding that he considers himself lucky to have caught the fabrication prior to publication. He notes that software programs to massage data, such as the one used by his former postdoc, have become more sophisticated in the years since the incident in his lab.

"Ultimately, we have to trust our coworkers," Saykally continues, "but every now and then, some bad apple comes along." He worries that today there is perhaps tremendous economic pressure for foreign students and postdocs to commit fraud in the interest of advancing their careers. He says most foreign students face tight job markets at home and in the U.S. and might be pressured to look for an edge, however that might be obtained.

"Recent history points to that direction," Saykally says, meaning cases of fraud that have involved foreign students or postdocs.

If a student's or a postdoc's work cannot be reproduced, "I talk to the student or postdoc who did the original work, or I write or call if they are no longer in my group," Michl says. "I have them communicate directly with the person who has problems repeating the work. I follow copies of the communication until the work is reproduced. This has not happened to me more than two or three times, and we were always able to reach a satisfactory conclusion.

"It is usually a matter of a compromise between the tersest possible description of experiments, which saves journal space, and the most detailed possible description, which makes it easiest to repeat the work," Michl continues. "Some people are unfamiliar with procedures that others may consider standard practice, requiring no description."

Michl says he always assumes the best about people when confronted with these situations. "It would take several labs not being able to reproduce the results or the original lab not being able to reproduce them before I would seriously entertain a suspicion of fraud." He adds that "certain procedures acquire a reputation for being hard to reproduce, and work from certain labs may acquire such a reputation, without necessarily being outright fraudulent. Some synthetic procedures are capricious and give variable yields yet may still be needed. Good authors note that fact in their papers.

"We have just submitted a review paper," Michl continues, "in which we point out that the yield originally reported in one of our papers as 40% when working on small scale is more commonly only 20 to 25% on a larger scale. In the same review, we note that another author's synthesis of another material could not be reproduced in at least two other labs. I doubt that it's fraudulent—it is just very hard to repeat and therefore not useful."

An interesting question related to reproducibility, Whitesides says, is "Do academic labs maintain adequate records? Can one go back to lab notebooks? University labs are much less careful about keeping lab notebooks" than industrial labs are, for example.

Whitesides says he has become an advocate of better recordkeeping, because "the issue of the integrity of data is one of growing concern throughout science." He points out that chemistry is becoming more interdisciplinary, and interdisciplinarity means that different ideas about experimental data and reproducibility have to be reconciled.

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Sometimes, the reason that work cannot be reproduced remains a puzzle. A research group at Boston's Northeastern University published two papers in 1998 and 1999 reporting that graphite nanofibers could store huge amounts of hydrogen (*J. Phys. Chem. B* **1998**, *102*, 4253; **1999**, *103*, 10572). Subsequent efforts to reproduce these results were not successful, at least not in terms of the magnitude of hydrogen storage capacity in the original claim (*J. Phys. Chem. B* **2005**, *109*, 14979).

The original result "was a big deal, but ultimately it was wrong," says **George C. Schatz**, a professor of chemistry at Northwestern University who is the editor of the *Journal of Physical Chemistry B*. "It wasn't intentional. It was a case in which people didn't get their measurements right."

One of the Northeastern researchers, Terry Baker, now working at Catalytic Materials, Holliston, Mass., says, "We never again got the high numbers we first got, and the outcome is we have given up work in that area." He says the graphite nanofibers are made from ethylene, and "you get a polymerization that tends to coat the fibers. Polymerization results from uncatalyzed olefin decomposition and collects on the nanofiber edge regions.

"It's difficult to remove that polymer," Baker continues. And although he's not entirely sure, he says he thinks that the polymer probably blocks hydrogen absorption. "We've never been able to get a growth process worked out where we get 'clean' nanofibers. I think we got unbelievably lucky the first time around."

Michl suspects that only a small fraction of the published chemical literature ever gets reproduced. "I bet many of the new compounds we have reported over the past 20 years have not been attempted again, and most of the spectra we reported have never been remeasured. Yet unpredictably, some may become important 10 years from now and will be reproduced by many labs. We published some work with pleiadene in the early 1970s that probably did not get repeated for two decades, and then we got a flurry of inquiries about it when somebody repeated it early in this century."

Schatz points out that in his journal, there is a comment section where, occasionally, researchers have reported that another group's results were not reproducible or that they were difficult to reproduce. He thinks that such comment-and-response mechanisms are a good way to make transparent to other investigators all that might be involved with a given body of experimental work.

When work appears to be irreproducible, Whitesides says, claims and counterclaims that are published in a journal might be okay—to a point. But if the issue is primarily experimental, he believes, it's not the journal's responsibility to sort out what may be going on. Rather, it's the responsibility of the people who are doing the work. "That's the whole essence of science," Poulter agrees. "That it can be reproduced."

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