

THE SULFURIC ACID PROBLEM

In 1947 José found himself translating scientific articles on synthetic chemistry from the German. Some of these translations were for Chemical Abstracts, while others became necessary as he considered starting work on his PhD thesis, where he would be seeking to prepare several compounds that might possibly prove to be intermediaries in the synthesis of 4(7)-nitrobenzimidazole (JACS 73, 3030).

One of the German references called for concentrated sulfuric acid to be used as a catalyst in a nitration of a phenolic compound. This article, published in 1909, simply mentioned that a certain amount of concentrated sulfuric acid was to be added. When José followed these directions, the result, to his bewilderment, turned out to be carbonization of the original materials. Puzzled, he repeated the experiment several times, each time adhering strictly to the directions he had translated. Always, the results were the same. Something obviously was wrong, but what?

After mulling the problem over for some time, he suddenly thought of a new possibility. It occurred to him that perhaps concentrated sulfuric acid made in Germany in 1909 might not be of the same strength as concentrated sulfuric acid produced in the United States in 1947. Accordingly, he set about trying to locate some German chemical journals from the early 1900s, particularly those which might contain ads and commercials. Sure enough, with luck he found such an ad, and here the concentrated sulfuric acid mentioned, the best commercially available at the time, was of a 70% concentration. It became apparent that José had been using acid of the wrong strength, since the 1947 American commercial product was over 99% in concentration. When he changed to the new strength, his procedure immediately yielded the desired product.

From this experience, José learned a valuable lesson. Whenever following a recipe, one must check the strengths and names of materials described, especially from very old recipes, many of which might fail to correspond with those of materials currently in use. It was an unexpected but significant step in a PhD candidate's training in the search for accuracy and truth.