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safety in the chemical laboratory

edited
MALCOLM M. RENFRE
University of Ida
Moscow, Idaho 838

Procedures for Laboratory Destruction of Chemicals

Blaine C. McKusick
1212 Bruce Road, Carcroft, Wilmington, DE 19803

All laboratory work with chemicals eventually produces chemical waste. Those who have generated the waste have moral and, increasingly, legal obligations[1] to see that such waste is handled and disposed of in ways that minimize potential harm to health and the environment.

A recent National Research Council (NRC) report[2] summarizes what laboratories need to know about the regulations of the Environmental Protection Agency (EPA) and the Department of Transportation (DOT) that apply to laboratory waste. The report provides guidelines for establishing and operating waste management systems for laboratories. It also gives specific advice on the collection, storage, and transportation of laboratory chemical waste and its ultimate disposal in sanitary sewage systems, incinerators, or landfills.

It is feasible to reduce or eliminate the hazard of many hazardous chemicals by chemical reaction in the laboratory. Although this is not a practical way for disposing of all laboratory wastes, it is very useful for some wastes. Examples are substances that are explosive, such as organic peroxides, and substances that react vigorously with air or water, such as sodium, metal hydrides, and acid halides; the transportation of such substances requires special precautions and their use in landfills is prohibited. Chemicals that present a high potential hazard because of their toxic properties, such as alkyl halides, nitrosamines, and hydrogen cyanide, are also good candidates for laboratory treatment, as are chemicals with an objectionable odor, such as mercaptans.

Blaine McKusick received a BS in chemical engineering from the University of Minnesota in 1940 and a PhD in organic chemistry from the University of Illinois in 1944. He was with the Du Pont Company from 1945 until his retirement in 1982. Among his posts there were Associate Director of the Central Research Department, Director of the Laboratory for Agricultural Chemicals Research, and Assistant Director of the Haskell Laboratory for Toxicology and Industrial Medicine. He helped prepare the National Research Council report on disposal of chemicals from laboratories referred to in this article.

It is not new for chemists to destroy such chemicals in the laboratory. For example, chemists have been destroying scrap sodium with ethanol for well over a century. However, they have not always destroyed their chemicals in a well-controlled way, and they have seldom published as detailed directions for destroying a substance as they have for synthesizing it. Too often, the chemist who carefully sets up equipment for preparing a hazardous substance will be comparatively casual in destroying it. Thus, sodium should not be treated with ethanol in the time-honored way, that is, in an open beaker, a procedure that may get out of control and even cause a hydrogen-oxygen explosion. Instead, the decomposition should be done under nitrogen in a three-necked flask equipped with a stirrer, a condenser, and a dropping funnel through which the ethanol is added, and the flask should be in a pan that can be filled with ice and water if the reaction starts to get out of control.

The NRC report includes a chapter of procedures for laboratory destruction of many common classes of hazardous chemicals. The procedures were found widely scattered through the literature. The Safety Column of THIS JOURNAL[3] is one of the best sources. The procedures may not all be the best possible and do not cover all hazardous chemicals, so the chapter is no more than a start. It would be helpful to the chemical community if chemists were to work out additional destruction procedures, write them up in the style and detail of *Organic Syntheses*, and publish them in the abstracted literature (in this Safety Column, for example) or include them in papers describing the research in which such procedures are applicable. Because the successful use of a destruction procedure, like a synthesis procedure, may depend on seemingly minor details, it would be desirable to have the procedure checked experimentally in another laboratory before publication.

As an example of what is needed, a procedure for hydrolyzing alkyl halides and other alkylating agents is reprinted from the NRC report. Many alkylating agents are fairly toxic, and some are carcinogenic. In general they are readily hydrolyzed by hot ethanolic potassium hydroxide to much less toxic products, principally alcohols, but also alkenes, ethyl ethers, and others. A 20% excess of potassium hydroxide is used, based on the equation

$$RX + KOH \rightarrow ROH + KX$$

An additional equivalent of potassium hydroxide must be used for each additional hydrolyzable group in the compound being treated.

The procedure is applicable to most primary, secondary, and tertiary chlorides, bromides, and iodides. It can also be used for allyl and benzyl halides, including fluorides, and for compounds that have two or even three chlorine, bromine, or iodine atoms on one carbon. It is not useful for vinyl or aryl halides or for most alkyl fluorides.

The procedure can be used to hydrolyze the following classes of alkylating agents, all of which contain members that are human or animal carcinogens: dialkyl sulfates, alkyl alkanesulfonates and arenesulfonates, chloromethyl ethers, epoxides, and aziridines. Although some of the stronger alkylating agents can be hydrolyzed effectively by hot aqueous 10% sodium hydroxide, the present procedure is more broadly applicable.

Procedure for Hydrolyzing 1.0 Mole of an Alkyl Halide. Place 79 g (1.20 mol) of 85% potassium hydroxide pellets in a 1-L three-necked flask equipped with a stirrer, water-

(*Continued on page A154*)

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[1] U.S. Environmental Protection Agency (EPA) regulations under the Resource Conservation and Recovery Act (RCRA), 45 Federal Register, 10 May 1980, and subsequent amendments; Title 40 Code of Federal Regulations, Chapter I.

[2] National Research Council Committee on Hazardous Substances in the Laboratory, "Prudent Practices for Disposal of Chemicals from Laboratories," xii + 282 pp., $16.50, 1983, National Academy Press, 2101 Constitution Ave., NW, Washington, DC, 20418.

[3] The Safety Columns for the period 1964–1980 are collected in "Safety in the Chemical Laboratory," (4 vols.), Division of Chemical Education, American Chemical Society, Easton, PA.

cooled condenser, dropping funnel, and heating mantle or steam bath. With brisk stirring, 315 mL of 95% ethanol is added rapidly. The potassium hydroxide dissolves within a few minutes, causing the temperature of the solution to rise to about 55°C. The solution is heated to gentle reflux, and the liquid alkyl halide (or its solution in 95% ethanol if it is a solid) is added dropwise. The rate of addition and the heat input are adjusted to maintain gentle reflux. The dropping funnel is rinsed with a little ethanol, and stirring and reflux are continued for 2 hr (stirring is essential to prevent bumping caused by the potassium halide that often precipitates). If the reaction products are water soluble, the mixture is diluted with 300 mL of water, cooled to room temperature, neutralized, and washed down the drain with about 50 volumes of water. Otherwise, the mixture is neutralized and sent to an incinerator or landfill.

Destruction procedures such as the above should be carried out with the same care and safety precautions that are used in synthetic procedures, and only by or under the direction of a trained professional who understands the chemistry involved. Such procedures are intended only for laboratory quantities, and not more than a few hundred grams at a time. Because hazards tend to increase exponentially with scale, scaling up should be done cautiously, and only by a professional.