Production of Metals and Compounds by Radiation Chemistry

Experiments have been conducted that demonstrate the preparation of metals and compounds by radiation-induced chemical reactions. The method involves irradiation of metal salt solutions with high energy electrons. Because the preparations may be carried out at low temperatures (limited by the freezing point of the solution), this technique offers a method for the preparation of high purity metals with minimum contamination from the container material or the cover gas. Unlike electrochemical methods, the radiation method affords a means of producing metals and metal compounds in an inert solvent such as toluene, and eliminates the possibility of contamination of the product by a reactive electrolyte.

Thus far, emphasis has been on reducing reactions, especially the formation of free metals from their aqueous salt solutions. The mechanism involves the decomposition of water by the ionizing radiation into

(continued overleaf)
hydrogen atoms and hydroxyl radicals. This is followed by the scavenging of the hydroxyl radicals by an oxidizable organic compound (e.g., primary and secondary alcohols). The remaining hydrogen atoms then act as the reducing agent.

In a typical experiment, an aqueous solution of 0.2 molar copper(II)sulfate containing 1.0 molar methanol (OH radical scavenger) was irradiated with 2 MeV electrons and 10 micro-amperes beam current. This produced a copper yield of about 1 gram per hour. Thus far, the radiation method has been used to prepare copper, nickel, cadmium, zinc, lead, thallium, tin, and antimony from aqueous systems. In addition, high purity anhydrous ferrous chloride has been prepared from irradiation of a solution of ferric chloride in toluene.

The irradiation of the solution was accomplished in the reaction vessel shown in the figure. The vessel is made of glass and has a volume of about 500 ml. After the solution is poured into the vessel, it is covered with titanium foil mounted in a brass ring. The foil serves as the inlet window for the vertical electron beam. The electron beam was furnished by a linear accelerator; an arbitrary selection of 2-million-volt electrons was used for these experiments. Helium passing through a glass frit at the base of the vessel agitates the liquid during irradiation. The gas is exhausted through the outlet tube. The reactants are cooled by immersing the vessel in a water bath. The actual reaction zone is about 1 centimeter deep (range of penetration of the 2 MeV electrons) and about 4 centimeters in diameter (width of beam); essentially, no reaction takes place once the products leave this zone.

Notes:
1. By variation of certain parameters, metal powder can be obtained of variable particle size extending down to the micron range. The very fine active powders may be useful as catalysts.
2. Under some conditions, adherent metallic mirrors were formed on the glass reaction vessel.
3. Documentation is available from:
   Clearinghouse for Federal Scientific and Technical Information
   Springfield, Virginia 22151
   Price $3.00
   Reference: TSP69-10123
4. Technical questions may be directed to:
   Technology Utilization Officer
   Lewis Research Center
   21000 Brookpark Road
   Cleveland, Ohio 44135
   Reference: B69-10123

Patent status:
No patent action is contemplated by NASA.

Source: W. H. Philipp and S. J. Marsik (LEW-10231)