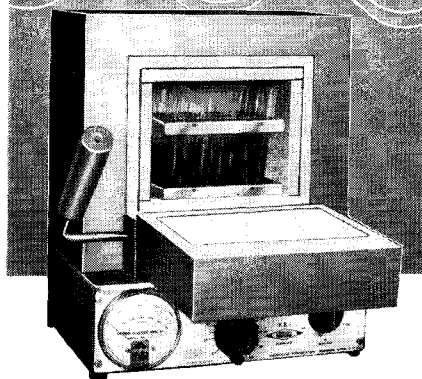


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**NEWS**

**Homogeneous Precipitations.**

Adding a precipitating reagent as compound instead of as itself has several special advantages, according to Dr. Hobart Willard. Adding the reagent as a compound avoids any excess and maintains a homogeneous region around the particles of the precipitate. Adsorption and coprecipitation are less. Larger crystals form, making easier filtering and washing to achieve more complete separation.

There are several methods for generating suitable reagents and for varying conditions in the solution. Those given special emphasis by Dr. Willard include pH control, changing oxidation state, and others. Hydrolysis of urea or acetamide to precipitate basic salts or hydroxides will increase the pH of the solution. Hydrolysis of esters, such as  $\beta$ -hydroxyethyl acetate, lowers the pH and will decompose the silver-ammonium chloride complex to produce crystalline silver chloride, for example.

A typical way to change the state of oxidation would be to use oxidation of chromic nitrate to chromic acid to precipitate lead chromate.

The physical properties of precipitates obtained from organic reagents by usual procedures often leave much to be desired, says Dr. Willard. These precipitates are improved by use of homogeneous solutions. Excellent large crystals of nickel dimethylglyoxime result when nickel is precipitated by the reaction between biacetyl and hydroxylamine.

**O, N, H by Emission Spectrography.** Oxygen, nitrogen, and hydrogen have now joined the list of elements which can be determined in metals by emission spectrographic methods, Dr. Velmar A. Fassel of Iowa State University told the symposium. First a technique had to be devised to overcome problems associated with "sopping up" of excitation energy of oxygen, nitrogen, and hydrogen by metal atoms in the discharge zone of the arc. By use of a carbon supporting electrode and an argon atmosphere, much of the problem sopping up of energy is solved.

Environmental conditions strongly affect the rate and degree of evolution of gaseous elements. Dr. Fassel and coworkers have employed a variety of electrode geometries and assemblies to get optimal evolution of gaseous elements. Key to success remains in getting the anode spot of the arc discharge to rest directly on the molten globule of sample. Resulting in a large temperature gradient and efficient convection stirring, this technique causes high

reaction rates and rapid gas evolution.

With these techniques, Dr. Fassel reports that oxygen and nitrogen in various steels, and oxygen in niobium, vanadium, tantalum, copper, and nickel have been determined in the concentration range of 0.005 to 0.5 weight %. To handle metal samples such as zirconium, titanium, and thorium, an electrode assembly is used that provides a molten platinum bath after the arc is initiated. Most metal systems used in these analyses need time for the metal to alloy with the platinum before any of the metal sample reacts with the graphite electrode.

Dr. Fassel also described techniques and recent refinements in spectroscopy of the rare earth elements.

**Stripping Analysis.** Using the group of techniques known as stripping analysis, some metals may be determined in solutions as dilute as  $10^{-9}M$ , with a limit of detectability of about  $10^{-11}M$ , Dr. Irving Shain of the University of Wisconsin said. Many forms of stripping analysis have been reported. But the one most applicable involves the use of fast-sweep polarography and stationary electrodes. However, if the unknown will form an amalgam, the hanging mercury drop electrode is most suitable.

Some equations have been derived to guide in selecting optimum conditions for electrolysis. These equations are based on considerations of the diffusion processes within a hanging mercury drop electrode. The equations allow ready detection of intermetallic compound formation, precipitate formation, and irreversibility. Correlation between experiment and theory has proved excellent, Dr. Shain says, for determination of tin as an example.

The technique has been extended to determination of halides using a silver microelectrode. In the pre-electrolysis step, controlled potential oxidation of the electrode produces an insoluble silver halide precipitate on the electrode surface. The precipitate is then analyzed using fast-sweep polarography for cathodic stripping.

Most laboratories have used home made equipment for fast-sweep polarography to make use of these methods and the hanging mercury drop electrode. However, a commercial instrument has been developed by E. H. Sargent Co., and is expected to be introduced soon, according to Dr. Shain.

**Ion Exchange.** New information on the physical chemistry and on analytical applications of ion exchange resins and ion exchange processes was covered by Dr. Harold F. Walton of the University of Colorado. When