Analytical Chemistry in Nuclear Technology

The chemist engaged in analysis of high-purity nuclear and reactor materials, especially for substances present in concentrations of parts per million or per billion, is faced with a number of problems. Among the most important of these is that of knowing exactly what information about the composition of the material in the reactor is needed by the engineer requesting the analysis. Especially difficult is the problem of choosing a method or methods of analysis which will yield information of requisite accuracy and precision in a reasonable time and at reasonable cost.

An outline of these problems and suggestions for solving them were presented by A. A. Smales of the United Kingdom Atomic Energy Authority, Harwell, England, at the eighth Conference on Analytical Chemistry in Nuclear Technology, held 6–8 October 1964 in Gatlinburg, Tennessee. Smales pointed out that the analytical chemist in selecting the most appropriate method of analysis must consider advantages and disadvantages of each possible method. He briefly evaluated a number of different methods, including absorption, fluorescence, and emission spectroscopy, gas chromatography, mass spectrometry, isotope dilution, vacuum spark, resonance spectrometry, and radio activation analysis, with regard to their sensitivity, specificity, accuracy, and freedom from contamination. He stressed that a competent analytical chemist must be quick to learn and evaluate new techniques and be willing to adopt them where suitable. Smales indicated a great interest in the utilization of the techniques of spark-source mass spectrographic instruments of recent design, which he thinks promise to alleviate the problem of trace analysis of high-purity metals. These instruments were discussed earlier in the conference by W. Fletcher (UKAEA, Capenhurst Works) and J. C. Fletcher (Y-12 Plant, Oak Ridge, Tennessee).

To the question of whether he thought the so-called "wet chemist" will disappear from the analytical laboratory, Smales replied that he did not. He commented, however, that this does not mean that the "wet" methods should be retained when new methods and techniques are more adequate.

Smales also pointed out that planning by the analytical chemist and the engineer who will use the results of the analysis can avoid or circumvent many problems, including those of analysis, during the early stages of development or research work.

In the session on burnup of fuel in reactors, the use of both radioactive (^{99}Tc) and stable (^{139}La) fission product nuclides as burnup monitors was discussed. A handicap to the accurate determination of the degree of burnup is the large uncertainty concerning the values for a number of the physical constants, such as fission yields and cross sections, that are needed in calculating the growth or decay of certain isotopes and their relation to the disappearance, or burnup, of fissionable fuel. J. E. Rein described the long-range program at Phillips Petroleum, Idaho Falls, Idaho, which has as its goal a better understanding of these parameters.

At the session on nuclear methods of chemical analysis, the precision, applications, and differences in techniques of radioactivation methods of analysis were described. At another session general applications of liquid scintillation counting were presented, including a description of a method for determination of noble gases by D. L. Horrocks and M. H. Studier (Argonne National Laboratory) and a technique for absolute assay of beta emitters of maximum energy greater than 200 kev by G. Goldstein (Oak Ridge National Laboratory).

New or modified techniques were discussed in the session on the determination of carbon, hydrogen, oxygen, and nitrogen. M. E. Smith (Los Alamos Scientific Laboratory) told of a device for crushing pyrolytic carbon-coated uranium carbide particles in the inert atmosphere of the furnace prior to analysis for oxygen. M. S. W. Webb (UKAEA, Woolwich) described an apparatus with which 27 steel samples could be analyzed spectrographically for oxygen. The apparatus was sensitive to as little as 1 μg of oxygen, and the analysis time was 2.5 minutes per sample.

W. E. Dallman (Iowa State University) presented data from a comparative study on the determination of gases in rare earth metals. Inert gas fusion, hot extraction, vacuum fusion and d-c carbon arc methods were discussed.

A complete session was devoted to the analysis of trace impurities in the alkali metals. The impurities discussed were oxygen, carbon, and hydrogen. Methods discussed for the determination of oxygen were a modification of the amalgamation technique which reduced the time per analysis, a distillation technique, and a neutron activation method. A freezing-point method for the determination of oxygen in rubidium and cesium was presented. Both wet and dry combustion methods for the determination of carbon were discussed, and thermal extraction and isotopic dilution techniques were presented for the determination of hydrogen. Of note was the application of the modified amalgamation technique to the simultaneous determination of oxygen and hydrogen in the same sample.

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Space Simulation: Man-Rated Testing and Vacuum Generation

Human beings have been used as subjects in tests in pressure chambers associated with deep-sea exploration and in altitude chambers for simulation of aircraft flight, but "man-rated" testing in the simulated environment of space is in its infancy. Such testing, performed in space-simulation chambers designed for life-support and safety, is required in the development of manned spacecraft and in training for lunar exploration and other extravehicular space activities. Along with the improvement of vacuum systems for space testing, man-rated testing was a principal topic at the second national Space Simulation Testing Conference (Pasadena, California, 16–18 November 1964), sponsored by the American Institute of Aeronautics and Astronautics.

Space-simulation chambers are needed for man-machine tests and to evaluate support equipment: suits, control systems, portable life-support systems, and the like. A manned chamber requires the combination of a reliable vacuum chamber with its associated systems and controls which, in the event of any type of failure or malfunction, can be made to react im-