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On

APPLICATIONS OF RADIOISOTOPES TO ANALYSIS

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INTRODUCTION

This article reviews the publications in nucleonics from late 1955 to late 1957 and follows without overlapping the material presented in the previous review (757). During this period there has been a large expansion in the number of papers published on radioisotopes. This increase appears to have been fostered by the International Conference on the Peaceful Uses of Atomic Energy held at Geneva in August, 1955 and to have been abetted by the recent declassification in the United States of a wealth of analytical information.

The Geneva Conference made available information on many different phases of radioactivity and has resulted directly in publication of articles in many languages and in many different journals. Sixteen volumes of the proceedings of the conference have been published (1208), volumes 7, 14 and 15 being of particular interest to analysts. A new conference will be held at Geneva in September, 1958 and it will be interesting to see the progress made in the field during the three year period.

In September, 1956 the Analytical Section of the International Union of Pure and Applied Chemistry held a conference at Lisbon in which some 34 papers were presented on the subject of radioisotopes applied to analysis. In September, 1957 an International Conference was sponsored

by UNESCO in Paris to report on the use of radioisotopes in scientific research. Abstracts of the 225 papers presented at this meeting have been published (1207) and publication of the entire group of papers is anticipated.

The new declassification policy of the United States Atomic Energy Commission has resulted in the release for general use of a large number of documents dating from as early as 1942. Most of these reports will not be published in journals but many have been included in this review as valuable source material in planning analyses.

The format and purpose of this review follow closely that of the previous one (757). Again it was necessary to omit many worthwhile related papers because of limitations of time and space. In most cases where it was impossible to cite an original article, a Nuclear Science Abstracts or Analytical Abstracts reference is given.

General references are again given at the beginning of each section and are followed by specific references to papers of more limited application. Since it has not been possible to cross index all the methods listed in the general papers and books, it is important that these general survey papers as well as the specific individual papers be read for a complete picture of progress in the field.

I. GENERAL

A large number of papers have been published on the application of radioactivity to analysis. The techniques described in these papers, however, often remain laboratory curiosities of use only to the group that developed them. Catch (199) has noted that the biologist considers tracers to be as necessary and common as his microscope and balance but that the average analytical chemist has shied away from tracer techniques. He feels that exaggerated estimates of the danger and cost of tracer work are the two biggest factors behind the analyst's lack of interest in radioactive tracers. Other items that also contribute are "ignorance of the possibilities and perhaps a measure of conservatism". It is hoped that the following review may show the broad applications of tracers in analysis and encourage the practicing analytical chemist to overcome his conservatism.

A. Review Papers

A number of excellent general review papers in this field have appeared during the past two years. Alimarin (13), in a Geneva paper, has given an excellent summary and discussion of different applications of radioisotopes in analysis. He includes 85 references, most of which are to the Russian literature. Belcher and coworkers (80) reviewed radiochemical methods of analysis to 1955. Broda and Schoenfeld (141) discuss the application of radioactivity to chemical analysis in an Italian article with 200 references. Götte (408) has made an excellent review in German of the

uses of radioactivity in analytical chemistry. Lavrukhina (665) made a review with 86 references of the use of radioactive isotopes in quantitative analysis while Leljaert (688) has also summarized this information.

Cabell and Smales (172) have made a good short general review of radiochemical methods in analysis while Winteringham (1257) discusses radioisotopes as an analytical tool in one of a series of practical articles. Shorter general articles have been written by Willard (1251), Fisher (332) and Weiss (1243).

Popjak (939) discusses isotopic tracer techniques as applied to chemistry and biochemistry, and Talibudeen (1171) reviews the use of isotopes in soil and fertilizer research (66 references).

Several of the books listed below under reference material also contain comprehensive review sections of the general field of application of radioisotopes to analysis.

Kelley (587) has written an excellent discussion of the role of the analytical chemist in nuclear technology as samples of higher radioactivity make remote control instrumentation essential. Schönfeld (1050) discusses the contributions of radiochemistry to the theory of chemical analysis while Herrington and Monk (481) describe the application of microchemical methods to radiochemical analysis. Wilson (1254), in a good general discussion relates radiochemical methods to ultramicroanalytical methods and makes some predictions for the future. A discussion (759) of nuclear techniques training

for analytical chemists has also been published.

B. Nuclear Data

The General Electric Chart of the Nuclides (1134) has been revised to April 1956 and now includes 1,388 nuclides in an easy to use form. The Sullivan Trilinear Chart of Nuclides (1154) has also been revised to January 1957. Both charts furnish up-to-date information on the major decay data for all radioisotopes. For those interested in more elaborate decay scheme information, the National Research Council Nuclear Data Group continues to make available their nuclear data cards (757) and their cumulated yearly data in Nuclear Science Abstracts. The first of the revised National Research Council compilations of nuclear level schemes was published at the end of 1955 (1238). This volume covers the elements between calcium and zirconium ($Z=20$ to $Z=40$). Unfortunately, the next volume for $Z=40$ to $Z=60$ has not yet been issued. A new revision of the Hollander, Perlman and Seaborg Table of Isotopes (496) is being prepared under the authorship of Strominger, Hollander and Seaborg and should be available in 1958.

Clark and Neil (213) have made an arrangement of known radionuclides according to their half-lives. The Oak Ridge group (1265) reports half-life values for some 30 radionuclides which they chemically purified after bombardment and followed for several half-lives. Some of their values are significantly different from earlier ones while others merely confirm those already established. Lockett and Thomas (705) report improved

half-life values for cobalt-60, thallium-204 and europium-152. Blomeke (107) lists the nuclear properties of uranium-235 fission products while Bolles and Ballou (114), in a large compilation report calculated activities and abundances of uranium-235 fission products.

C. Radioisotope Production

The Isotope Index (543) is an invaluable help in locating suppliers of all commercially available stable and radioactive isotopes and many hundreds of labeled compounds. Fifty three United States, Canadian, British, French and German suppliers are listed.

The Oak Ridge National Laboratory continues to supply most of the isotope needs of the United States (868) although recently the cyclotron production of isotopes has been transferred to a private industrial firm (193). Techniques whereby isotopically enriched targets are used for production of carrier-free manganese-54, iodine-125 and tellurium-130 have been reported (845). A summary (839) of suppliers for labeled compounds has been published and an extensive bibliography (826) of syntheses with carbon isotopes has recently been made available.

In the United States, considerable impetus has been given to the use of small amounts of labeled material in isotope dilution and radiometric procedures by the new decision of the U. S. Atomic Energy Commission to make available small (microcurie) amounts of most radioisotopes license free (316). A number of U. S. suppliers now furnish license-free amounts

of many carbon-14 and tritium labeled compounds as well as other radioisotopes.

Radioisotope production in the United States has been described in an article by Rupp (1016), that in Great Britain by Shaw (1079), and that in Russia by Nesmeyanov and coworkers (824).

Stang and coworkers (1127) described their work on production of 21-hour magnesium-28, 2.33-hour iodine-132, 21-hour iodine-133 and 112-minute fluorine-18, while Cook, Payne and Scargill (221) reported use of the Szilard-Chalmers reaction to produce iron, cobalt, copper, zinc, gallium and palladium activities.

Calculations governing isotope production by neutron irradiation (1230,347,828) and fission product build-up in reactors (987) have also been published.

D. Reference Material

References in this field have become difficult to trace because they are spread through a large number of journals. "Nucleonics" (837) continues to be a major source of pertinent articles, although the editorial policy in 1957 appears to be shifting away from nuclear techniques towards applied radiation and atomic power. "The International Journal of Applied Radiation and Isotopes" (530) is a new techniques journal which is of definite interest in this field. "Nuclear Science Abstracts" (835) continues to abstract most of the information in this field and has been used along with "Current Chemical Papers" and "Analytical Abstracts" as a source of references

for this review. "Nuclear Science Abstracts" is the primary source of information on the many documents available as unpublished literature in the United States. A number of other new journals in related fields have appeared during the past two years. They include "Journal of Inorganic and Nuclear Chemistry" (563), "Journal of Nuclear Energy" (564), "Nuclear Science and Engineering" (836), "Radiation Research" (962), "Nuclear Physics" (834), "Nuclear Instruments" (833), "Geochemical et Cosmochemical Acta" (373), "Atomkern Energie" (46) and "Energia Nucleare" (301).

The large amount of information recently declassified by the U. S. Atomic Energy Commission (197) accounts for the flood of document references in this review. Yound (1275) discusses some of the problems involved in making this information accessible.

One of the major problems in any phase of the nuclear field is the location of project literature that does not find its way into regular publication channels. A booklet available (544) from the U. S. Atomic Energy Commission describes special sources of information on isotopes and one available from the U. S. Government Printing Office (1158) lists documents on atomic energy. An AEC document (873) also gives an accumulated numerical list of available unclassified U. S. AEC reports while another publication which is revised semi-annually (876) gives a price list for current AEC research reports. A third publication (975) issued monthly by the U. S. Government lists all government

research reports (including AEC) as they are made available.

Complete files of AEC reports are located in some 70 repositories in this country and 60 repositories outside of this country, as listed on the covers of each issue of "Nuclear Science Abstracts". Individual copies or files of complete categories of these reports are available in micro-card (776) or microprint (966) form for a nominal charge.

An excellent summary (with a large bibliography) of isotope distribution and utilization up to 1955 is available from the AEC (1209). An Atomic and Industrial Forum report (44) discusses the utilization of radioisotopes from 1946 to 1956, while special NUCLEONICS reports (842,838) discuss industrial applications.

The Pergamon Press is initiating a series of books on "Progress in Nuclear Energy" scheduled to appear in the near future. The volumes on "Analytical Chemistry" (588) and "Process Chemistry" (153) should be of particular interest. A Russian book (1218) discusses the use of labeled atoms in analytical chemistry.

Several German books discuss in detail the techniques of radiochemistry. Broda and Schönfeld (142) describe radiochemical methods for microchemistry as well as (143) industrial applications of radioactivity. Both are excellent summaries with a large number of references including many from the U.S.S.R. Weiss (1244) describes in detail the preparation and calibration of radioactive standards for a number of isotopes. Schmeiser (1046) has written a book on radioactive

isotopes and Haissinsky (440) on nuclear chemistry and its applications.

Sections of the instrumental analysis books by Delahay (257) and Berl (84) contain discussions of these techniques. Evans' book (308) is an expansive treatise on nuclear theory which makes a good reference work. Hughes (516) has written a new book on neutron cross sections. Two books discuss the effects of nuclear weapons (386) and other effects and influences of the nuclear bomb test explosions (217).

Aronoff (38) has written an excellent radioisotope techniques book slanted towards use in biochemistry and Winteringham (1257) has written a series of articles describing use of radioisotopes. The revision of Kamen's book (577) and a chapter in "Methods in Enzymology" (1136) should be extensive introduction for the use of tracers in the biological field. Books by Fields and Seed (326), Smullen (1118) and Kinsman (604) may also be of interest. Three Russian books (822,144, 1125) and a French book (303) complete the list of books in this category.

The American Society for Mechanical Engineers (20) has issued a booklet on terms in nuclear science technology which may prove useful to some.

II. RADIOISOTOPES AS SOURCES

The uses of radioisotopes can be divided into two principal classifications: those dependent upon the radioactivity as a source of ionizing radiation, and those dependent upon the tracer characteristics of radioactive material. The first category, considered here, represents by far the most concentrated industrial use of isotopes to date in terms of curies and in number of users.

Two general papers (186,926) discuss the production of radioisotope sources. Several authors (105,381) have devised techniques for fusing radioisotopes into enamel sources. Other authors have discussed the preparation and use of the beta sources yttrium-90 (694), silver-111 (762), promethium-147 (367) and the fission product gas krypton-85 (1167,507); and the X-ray or gamma-ray sources of iron-55 (721), cobalt-60 (658), lanthanum-140 (448), cerium-praseodymium-144 (457), cesium-137 (796) and americium-241 (780,777). The yield of X-rays from beta-ray excited sources has also been discussed in detail (1129). Some of these sources have been designed for clinical and radiographic work rather than analytical purposes, but may well find analytical application in the future.

A. Transmission

Beta ray thickness gages continue to fill a great need in industry and at present are installed in several hundred

plants in the United States. Foster (340) and Ohmart (877) describe some of the uses of these gages in the United States while Gagne (362) discusses their use in French industry. In a symposium on non-destructive testing, Taylor (1173) has described a radiation system for measuring either thickness or density which is sufficiently stable to resolve changes in gamma radiation level of 0.03%. A machine using cobalt, cesium or sodium-22 sources for thickness measurements (310), a beta gage for localized measurements on thin films (25), and an alpha thickness gage using a polonium source (302), are also reported.

Radiation transmission measurements have been applied to many different determinations. Lefort (684) used the alpha rays from a sealed source of 0.6 millicuries of radium chloride to measure two component mixtures of hydrogen-nitrogen, hydrogen-oxygen, hydrogen-carbon dioxide, nitrogen-nitrogen dioxide, or hydrogen-ethane. He reports that as little as one cubic millimeter of gas can be analyzed with a precision of 10%, improving to 1% with 20 cubic millimeters. Nitrogen and oxygen cannot be distinguished with this device. Shteinbok (1088) gives a general discussion of the principle of this type of analysis and its application to practical systems.

Further work has been done on the analysis of gas mixtures by beta-ray transmission. Norhagen and Odeblad (829) used the beta particles from sulfur-35 to measure transmission in mixtures of helium-oxygen, helium-air, helium-nitrogen, and oxygen-nitrogen and found approximately linear relationships

between the gas partial pressures and the transmissions. Jacobs, Lewis and Piehl (546) describe an instrument for determining percent hydrogen in hydrocarbons by measuring beta ray absorption. Corrections must be applied for elements other than hydrogen and carbon, but samples may be analyzed in less than 20 minutes and replicate determinations on six hydrocarbons showed a standard deviation of $\pm 0.03\%$ of the hydrogen present. Rowan (1003) has also described a meter of this sort while Feuer and coworkers (325) discuss beta-ray absorption and scattering in relation to gas analysis.

Kannuna and Cameron (579,578) describe a procedure in which tritium bremsstrahlung is used for the determination of sulfur and tetra-ethyl lead in hydrocarbons. The sensitivity and accuracy compare favorably with other methods, particularly those using iron-55 X-rays, and the cheapness of the tritium source is an advantage. They report that 0.033% thiophene by weight can be determined in benzene with a 0.3% standard deviation and that 0.004% tetra-ethyl lead by volume can be determined in heptane. Seaman and coworkers (1063) have developed techniques whereby chlorine and bromine can be determined in solid samples of organic compounds by absorptiometry using the X-rays from iron-55. Leveque and Martinelli (693) also describe industrial uses of bremsstrahlung radiation.

Gamma-ray sources have been used to study the expansion of gas-liquid systems (378) and the pressure of saturated steam (823), and to give a continuous measure of the dryness

of steam at the output of a liquid metal-water, heat-exchanger (864). The rate of settling of solid uranium compounds in a liquid sodium system (360) was determined with the low energy gamma-ray emitter selenium-75 to discriminate between uranium oxide and sodium at concentrations in the range of 5 to 30 weight percent. Miller (780) has used the weak gamma-rays from americium-241 to determine uranium or plutonium in aqueous solutions at concentrations greater than 100 grams per liter.

A measure of the thickness of cladding of uranium fuel elements (128) and of the density of uranium metal (1044) has been obtained by this method. Other gages control cigarette density (717) and predict the life of filter cloth (170). Deal and coworkers (252) describe a radiological detector and a strontium-90 source for measurement of effluents from gas chromatography. Another article (528) describes a similar type of detector. An apparatus for measuring solid levels in tanks (417) and a radioactivity flow meter employing a differential manometer with a floating radioactive pellet for high temperature and pressure systems (471) are described. Husain and Putman (527) studied the absorption of beta particles as a function of atomic number.

B. Backscattering

R. H. Müller (811) has given an excellent detailed summary of the types of measurements that can be made and the general relationships that can be shown between the backscattering of beta particles from matter and its composition. D. C. Muller

(809) has extended this work to organic compounds. Danguy recently described some of his experiments on determination of concentration by beta backscattering (244) and also some of his measurements on film thickness by beta backscattering (243).

Bogdanov and Funke (112) have used the reflected radiation from thallium-204 for analysis of chromium-niobium alloys containing greater than three percent niobium. The analysis time is about three minutes and the accuracy about two percent. They recommend the procedure for analysis of other binary alloys when there is a substantial difference between the atomic numbers of the components.

Several Japanese workers have used beta-ray backscattering in the investigation of antique relics (39) and in the identification of pigments (1273), the thickness of gold plating on old images and the chemical composition of glasses, bronzes, and glazes (860).

Jesse and Sadauskis (555) have discussed the backscattering of beta particles from gases as a source of distortion in absolute beta-ray measurements. Another group (956) determined tube wall thickness by backscattered gamma radiations.

C. Miscellaneous Effects

Ljunggren (703) describes a blower for transporting ionized air from the vicinity of a shielded radioactive source towards electrostatically charged areas for the elimination of static electricity. D'Eye (263) has studied the problem of displacement of polonium atoms from sources by aggregate recoil from the alpha emission. Beynon and Nicholson (89) describe

the application of a sealed radium source and ion gage to the measurement of latent heats of vaporization or sublimation of organic compounds. Calorimetric measurement and comparison of sources are described (406).

III. RADIOISOTOPES AS TRACERS

The bulk of this review is again concerned with use of radioisotopes as tracers extending from determination of minute amounts of elements by activation with nuclear particles, to isotope dilution and radiometric analyses, the assay and separation of radioactive materials, the specialized procedures required for analysis of highly radioactive materials, and the use of radioisotopes as tracers in evaluating or developing analytical procedures.

A. Activation Analysis

Interest in this field has increased considerably during the past two years. A number of general review articles have appeared in different languages. With the advent of more nuclear reactors and higher level neutron sources it is expected that many of the methods mentioned in the last review and those included in this review will finally be put into operation.

Two excellent review articles have come out of the Harwell laboratories, one by Jenkins and Smales (551) in 1956 including 72 references and several summary tables, and a second with 118 references by Loveridge and Smales (707) which discusses the application of activation analysis to biochemistry. Yakovlev (1270) continues some of the discussions of his Geneva paper in a more recent publication on the determination of impurities in high purity metals by this method.

Morrison (800) and Plumb (935) have discussed in general the problems and procedures for measuring trace elements in

metal samples. A number of general reports on this method have also come out of the Oak Ridge laboratories; references (149) and (671) are the most readily available. An excellent discussion of activation analysis with 15 pages of tables citing many recent examples and giving many cross references can be found in the chapter by Taylor and Havens (1176) in Berl's book. Saito (1027) has reviewed the field in Japanese.

Tables of approximate sensitivities for this method have been included in a number of these general references.

Meinke and Maddock (761) have plotted the most recent thermal neutron activation cross section values against the half-life of the radioisotope produced to give a rough over-all picture of the elements that can be detected with high sensitivity. They include the error limits on the cross section values. Unfortunately no attempt was made to include counting efficiency values for the resulting radioisotopes, a factor which must be considered a part of sensitivity from the practical point of view.

Geiger and Plumb (371) give a useful table, listing in order of increasing half-life the isotopes known to be procured by slow neutron capture, and Bopp and Sisman (119) describe methods for calculation of gamma radiation induced in reactor materials. Mellish and coworkers (767) have investigated the occurrence of (n,p) and (n,alpha) reactions with fast neutrons in a reactor and discuss the effect of such reactions on activation analysis. Fink (327) also discusses the relationship of these fast neutron reactions to analysis for trace

amounts of Z-1 and Z-2 elements by this method.

1. Sources of Neutrons. An excellent summary (745) has been made of neutron irradiation facilities available or planned in the United States up to April 1957 while another report (850) describes reactors which U. S. manufacturers offer for export. Several other articles (1184,770) describe in more detail specific research reactors available at non-AEC installations.

In addition to the new, large reactors being planned throughout the world there have been a number of encouraging developments along the lines of small reactors. Several small low-power reactors are now available (91,849,856) and come completely set up for use without additional building facilities. Operating at 5 watts, a reactor of this type can attain a flux of about 10^8 neutrons/cm²sec. The resultant sensitivity is good enough to measure microgram amounts of elements such as sodium and manganese by activation and p.p.m. of boron by absorption (126). Small reactors of this type have already been installed in some 10 to 12 research facilities.

A dozen or more van de Graaff generators which can produce around 10^8 to 10^9 neutrons/cm²sec are also in use around the country for projects including activation analysis (861,805). Other machines which can provide neutrons include the Cockcroft-Walton generator (201,901).

Although these small reactors and accelerators do not give the versatility and high flux of the large reactors at the National Laboratories, they do now bring the technique of

activation analysis closer to the industrial laboratory. The more than 25 of these facilities now scattered throughout the country give medium sensitivity activation analysis at a cost approximately twice that of a nuclear magnetic resonance apparatus or three times that of a mass spectrograph. While some of the instruments require considerable additional housing, others come ready for operation.

Progress is still being made in the neutron source field. Californium-252 spontaneously fissions, giving off neutrons such that a 30 mg source could provide 10^{11} neutrons/sec (844). Actinium has been used with beryllium to make neutron sources of 10^6 - 10^7 neutrons per second (863). These sources have a longer half-life than the conventional polonium-beryllium sources, while still having low gamma radiations levels. Work on the preparation and properties of polonium-beryllium (806) and plutonium-beryllium (1083) neutron sources has also been recorded.

2. Analysis Schemes. A number of groups have developed entire schemes for determining impurities in a particular material by activation analysis. For example Iredale (531) has described an analysis for impurities in aluminum using radioactivation and scintillation spectrometry with no chemical separation. Morrison and Cosgrove have also avoided chemical separations by using scintillation spectrometry to determine the trace impurities in germanium (801) and tungsten (227). They claim sensitivities of 0.001 + 1 microgram for most elements with a standard deviation of the precision of 3% and of the

accuracy of about 30%.

Albert (9) in his detailed study of the purification of aluminum and iron describes chemical separations from many of the impurity elements. Kant and coworkers (580) have devised a separation scheme for the determination of phosphorus, iron, copper, zinc, gallium, arsenic, silver, cadmium, indium, antimony, thallium and bismuth in high purity silicon by this method. Another group (1182) has combined gamma spectrometry with chemical separation and beta counting to study the determination and sensitivity limits of 29 elements in ultra-pure silica. Brooksbank and coworkers (151) describe radio-chemical procedures for tungsten, chlorine, vanadium, nickel, copper, manganese and silicon present in titanium in amounts of 10^{-4} to 100 micrograms.

Smales (1101) has compared emission spectroscopy, mass spectroscopy (vacuum spark and isotope dilution), polarography, vacuum fusion, colorimetry and fluorimetry, the use of radioactive tracers and radioactivation for the determination of impurities in semiconductors. He also reports (1100) on some trace element determinations in two standard rocks by neutron activation.

This method has been used to analyse antiques and art objects (299), to distinguish between neoprene and natural rubber (70), to determine feldspars (129), to determine active impurities in luminescent minerals (422) and sulfides (56) and to analyse for decay products of naturally radioactive elements in minerals (479).

3. Special Systems. The past two years have seen much exploratory work on extending the realm of activation analysis beyond the usual lengthy activation by thermal neutrons. Turner (1204) has used the 14-Mev neutrons from a van de Graaff accelerator with a zirconium-tritium target to produce measurable quantities of 2.3-minute aluminum-28 by the (n,p) reaction on silicon and 15-hour sodium-24 by the (n,alpha) reaction on aluminum. The method is accurate to within 5% for samples containing 0.4 gram or more of silica and 0.5 gram or more of aluminum oxide. He finds that phosphorus, praseodymium and chromium interfere with the determination of silica while magnesium and iron interfere with the determination of aluminum. Faull (315) has studied the fast neutron reactions on oxygen in water.

Atchison and Beamer (43) have used their van de Graaff accelerator to produce 2-Mev neutrons which in turn are slowed to thermal energies for rapid analysis of milligram amounts of fluoride and microgram amounts of the other halogens. They also describe an apparatus for automatic irradiation and measurement of radioactivity having half-lives of the order of 10 seconds. Lévêque (691,692) has explored the determination of elements such as oxygen, fluorine and hafnium giving short half-lived activities by neutron activation. Sûe (1149,1150) has used deuterons from a cyclotron to produce short-lived activities from boron, oxygen, nitrogen, sulfur and potassium by the (d,n) and (d,alpha) reactions and has applied this technique to analysis.

Edwards (286) measures boron-10 by the (n,alpha) reaction. Pieces of tissue containing boron are placed in contact with a photographic emulsion and are irradiated with neutrons. A count of the alpha particle tracks in the nuclear emulsion gives an idea of the cellular distribution of the boron-10. Along the same lines two German workers, Wänke and Monse (1234) have used scintillation counting to determine the alpha particles from the (n,alpha) reaction on lithium-6 and boron-10 or the fissions from the (n,fission) reaction of uranium-235. They obtained 300 counts per minute for an infinitely thick layer of lithium fluoride, 135 counts per minute for boron and 6 fission counts per minute for uranium oxide by using a radium-beryllium neutron source.

Gold (394) has used the (alpha,n) reaction on beryllium as a means of detecting hazardous airborne beryllium dust collected on filter paper. Exposure to polonium-210 alpha particles indicates the beryllium concentration.

Chauvin and Lévéque (192) have used an impulse discriminator attached to a gamma scintillation detector to cut out the low energy gamma rays from contaminating material and to permit the detection of radioisotopes of sodium or aluminum possessing high energy gamma rays without elaborate pulse analysis equipment.

Putman and Taylor (958) report the possibility of electronically subtracting gamma-ray spectra from two counters, one measuring a sample plus contaminant and a second without the contaminant. In this way it should be possible to suppress

unwanted spectra in activation analysis by introducing successive pure samples in the balancing counter.

A group of French workers (102) have studied the homogeneity of charcoal samples by activation of the mineral matter contained in them. U. S. workers have used the method to show trace metal segregation in metallurgy processes (672) and to determine particle size (853,1).

4. Carbon, Oxygen, Sodium and Aluminum. Point (937) describes a method for determination of carbon in iron by proton activation while a Berkeley group (335) uses proton activation of oxygen-18 to the 1.9-hour fluorine-18 isotope to determine fractions of a microgram of oxygen-18 in photosynthetic studies.

Smales and Loveridge (1104) determine as little as 2×10^{-9} grams of sodium in lithium metal when they purify the sodium-24 by radiochemical separations. A note in "Nucleonics" (859) describes the use of activation to determine the sodium to manganese ratio in a number of ancient potteries in archeological studies. Reiffel and Stone (971) have developed a procedure to determine sodium in milligram or submilligram samples of tissue to within 5%. They report that the activity induced in the tissue is almost entirely due to sodium-24, potassium-42 and phosphorus-32 and can be resolved by simple gamma counting without spectrometry. Nuclear constants of these activities are such that mail service to a reactor suffices for irradiations.

Odeblad (871) uses a polonium alpha source to determine aluminum but finds interferences from boron and appreciable

amounts of magnesium.

5. Phosphorus, Sulfur, Potassium and Vanadium. James and Richards report a technique for estimating the submicrogram quantities of phosphorus in silicon (548) and the equally small amount of phosphorus in iodine (549). Herr (478) gives a good review of activation analysis in his discussion of the determination of phosphorus in iron, while Reiffel and Stone (971) describe its use in the analysis of tissues.

Sûe and Albert (1151) have used the (d, alpha) reaction to determine microgram amounts of sulfur with an error of about 15%. However, the method requires use of a cyclotron and counting of the 2.5-minute phosphorus-30.

Potassium in tissue has also been analyzed by the method of Reiffel and Stone (971). Smales and Mapper (1105) report a procedure for determining vanadium without radiochemical separation by counting the 3.8-minute vanadium-52 formed by activation.

6. Manganese, Cobalt, Nickel and Copper. Bowen (123) has devised a method for the determination of manganese in biological material, particularly blood. He chemically purifies the manganese to eliminate the sodium, phosphorus and iron backgrounds which interfere in the gamma spectrum and can analyse eight samples containing as little as 0.024 micrograms of manganese in 2.5 hours. Manganese procedures for clays are also reported (859).

Smales, Mapper and Wood (1106) describe in considerable detail activation procedures, including radiochemical separations for the determination of microgram or smaller amounts of nickel,

cobalt and copper in rocks, marine sediments and meteorites.

7. Arsenic, Technetium, Palladium and Indium. A determination of arsenic in tungsten by James and Richards (547) involves separation of the arsenic by distillation with a minimum decontamination factor of 2×10^4 per step. Gauthier (370) has determined arsenic in pyrites by neutron activation while three other groups (409,775,618) have determined as little as 10^{-9} grams of arsenic in teeth and bone by this method with a maximum error of about 10%.

The technique of neutron activation analysis was utilized (23) in the search for technetium in minerals. Vincent and Smales (1217) describe their determination by radioactivation of palladium and gold in igneous rocks and set their detection limit at 0.01 and 0.0005 p.p.m. respectively.

The Harwell group has also described in detail (1107,535) their determination of indium in rocks and minerals. They give (535) an excellent discussion of the results obtained by using two different radiochemical procedures, gross decay curve resolution, and gamma spectroscopy. Hoste and van den Berghe (504) describe their determinations of microamounts of indium in zinc and in gallium by activation with a one gram radium-beryllium neutron source. As little as 0.004% of indium in a 10 gram sample of zinc could be determined with a mean error of 2.3% because of the very high activation cross section of indium.

8. Strontium, Barium, Rubidium and Cesium. Hummel and Smales (522) compare the determination of strontium in sea

water by neutron activation analysis with that by isotope dilution using stable isotopes, and find good agreement. Bowen and Dymond (124) describe their analysis for strontium and barium in plants and soil extracts while Harrison and Raymond (452) in another article discuss in detail their procedure for barium and strontium analysis in biological waste materials. These analyses are accurate to within 5% for levels of 1-50 micrograms.

Cabell and coworkers (173,171) have revised and extended previously published methods of the Harwell group and describe in detail the determination of rubidium and cesium in rocks, minerals and meteorites as well as sodium-potassium alloys. Their chemical procedures include cobaltinitrite precipitations, ferric hydroxide scavenges, cation exchange chromatography, and final counting as the chloroplatinates. The samples examined contained about $10^{-6}\%$ of these elements; replicate determinations usually agreed within 5% and often within 2% for rubidium while cesium was not as good. Gordon, Friedman and Edwards (401) have also used activation analysis to determine cesium in stony meteorites.

9. Rare Earths and Tantalum. An excellent report by Cornish (226) describes in detail the determination of submicrogram quantities of individual rare earths by radioactivation using ion exchange separations, while a Russian article (121) describes the solution of some problems of rare earth analysis. Eichholz (289) has used this method for assaying tantalum in tantalum ores but finds that a correction to his procedures

is necessary if uranium is present.

10. Rhenium and Gold. Herr and Merz (480) have used this method for determining the age of rhenium containing minerals by measuring the amount of the radiogenic osmium present in the minerals. Hummel (521) has studied the determination of gold in seawater and found a variation between 15 and 400 micrograms per cubic meter in various waters. Muller (810) has used this method in medical isotope dosimetry to analyze for inactive colloidal gold particles dispersed in the body from gold-198 treatment. Vincent and Smales (1217) report detailed procedures for analysis of gold in igneous rocks as described above.

11. Thorium, Uranium and Plutonium. The Argonne group (74) has recently reported activation analyses for thorium in stone meteorites, while a group at the University of Chicago (969,445) has analyzed for as little as 10^{-9} grams of uranium in iron and stone meteorites by this method.

Several groups (990,509,753) discuss the analysis of uranium for U-235 by the activation method and McKay (729) reports a procedure for determination of plutonium-239 by this method rather than by direct alpha counting.

B. Nuclear Absorption, Scattering and Reactions

A number of groups have recently attempted to apply other nuclear characteristics to analysis. Since boron gives no radioactive isotopes on irradiation, use is made of its high neutron absorption for analysis. Khristianov and Panov (592) describe a method whereby the boron in rocks can be analyzed

with a relative accuracy of $\pm 3\%$. Braman devotes his entire thesis (131) to a study of neutron absorptiometry using a 10 millicurie radium-beryllium neutron source and describes in detail the application of this method to boron (254).

Simons (1096) has devised a method of measuring the glass content of glass-reinforced plastic laminates by the neutron absorption of the boron in the glass while Hamlen and Koski (446) describe methods for isotopic analysis of di-borane and penta-borane which agree to within 1% of mass spectroscopic values. Boyle and coworkers (126) have determined a few p.p.m. of boron in samples by using the reactor "danger coefficient" method on the 5-Watt AGN-201 reactor. Ford and Picciotto (338) have applied the neutron absorption technique to a study of rare earth minerals.

Anderson (28) has devoted his entire thesis to a study of chemical analysis by resonance absorption of neutrons. He has studied analysis of indium in tin, hafnium in zirconium, and gold in copper and claims a precision of about 5% for amounts of indium ranging from 0.01 to 0.5%. He suggests the method is good for elements with large resonances such as indium, hafnium, gold, gadolinium, cadmium, europium, dysprosium, cobalt, iridium, manganese, rhodium, samarium, silver, uranium and tantalum.

Boyle and coworkers (126) suggest use of the 5-Watt AGN-201 reactor to determine the ratio of hydrogen to carbon in a liquid petrochemical. The method is based on the increase in reactivity of the reactor because of the large neutron moderating power of

hydrogen relative to carbon.

A group at the Stanford Research Institute has studied the application of nuclear scattering and nuclear reactions induced by high energy protons and deuterons to the analysis of solid surfaces (1005,53,858). They have determined oxygen, aluminum, silicon, sulfur, calcium, iron, copper, silver, barium and lead by the scattering method and carbon, nitrogen, oxygen, fluorine and sodium by nuclear reactions other than scattering. Their methods are applicable to the detection of all elements to a depth of several microns with sensitivities in the range of 10^{-8} to 10^{-6} grams per cm^2 . The technique appears to be well worth pursuing further. Holmes (499) has used the neutron scattering effect to measure soil water content.

Recently several groups (1111,195) have been making use of the neutrons available from a small portable van de Graaff generator to explore the interface between oil-saturated strata and brine-saturated strata in an oil well. The prompt gamma rays resulting from neutron capture in these different strata depend upon the elements in the strata, the counting rate in the brine strata exceeding that in the oil strata by about 10% over a range of gamma energies from 5 to 7 Mev. Although this application is at present limited to field use in oil well logging it might well be adapted to analytical procedures, particularly the continuous analysis of processes. Putman (954) has received a British patent on a similar method using a portable neutron source and a scintillation counter to analyze for specific elements in inaccessible substances.

The recoil (1004) and exchange (1254) labeling of organic compounds with tritium may contribute in the future to analytical techniques. Similarly the resonance radiation absorption method (882) for determining mercury-202 may have more general applicability.

C. Isotope Dilution

In this technique, radioactive materials essentially measure the yield of a separation procedure from a mixture of residue where a quantitative recovery is not feasible (757). Sorensen (1122) has recently written an excellent monograph on the use of chlorine-36 to determine organic compounds by this method and gives 123 pertinent literature references as examples of the technique. Rosenblum (994) gives a short summary of the use of the four types of isotope dilution methods in improving quantitative analysis and Trenner (1198) reports on the role of this method in analysis. Semenow and Roberts (1068) include some general discussion of this method in their review article on uses of isotopes in organic chemistry while Ropp (992) discusses some errors which might occur in this type of procedure.

1. Inorganic Applications. Leliaert and Eeckhaut (689) determine both strontium and calcium in mixtures of about 20 mg. of these two elements by isotope dilution using strontium-89 and calcium-45. Salyer and Sweet (1035) have used this method in the determination of small amounts of cobalt in steels and nickel alloys by plating the cobalt (1034) as hydrated cobaltic oxide at an anode after a known specific

activity of cobalt-60 has been added. They can determine less than 1% of cobalt with a maximum error of 0.02% and a standard deviation varying from 0.005% to 0.025%. Considerably less time is required than for the quantitative procedure.

Reliable results have been obtained by Geldhof and coworkers (372) in the determination of from 1 to 20 milligrams of molybdenum in steel using the 67-hour molybdenum-99 tracer. El-Guebely (293) compares an isotope dilution determination of ruthenium in dilute solution with a spectrophotometric procedure while Von Buttlar (1222) reports analysis of lead by the isotope dilution method using RaD as tracer. Gauthier (368) determines the amount of orthophosphate in industrial triphosphates by using paper chromatography and phosphorus-32.

Isotope dilution is also used to measure total blood volumes or total body content of certain materials (1117, 1116, 970). Although they do not at first glance appear to be applicable to analytical chemistry, the techniques involved might well be adapted to analysis of certain closed systems.

2. Organic Applications. The use of isotopic dilution analysis for certain organic compounds has become routine enough that a procedure for assay of vitamin B₁₂ can be found in the first supplement to the most recent U. S. Pharmacopeia (929). A joint panel found (154) vitamin B₁₂ assay by this method to be specific for cobalamins on four commercial type samples. They also report that biologically inactive "red pigments" which might occur in commercial products do not interfere.

Numerof (866) and others (92) discuss the determination of vitamin B₁₂ by this method while Tabern and Storey (1168) discuss the techniques for pernicious anemia diagnosis using the cobalt labeled vitamin B₁₂. It might be noted that the Abbott Pharmaceutical Laboratory makes available pre-assayed capsules of labeled vitamin B₁₂ as well as other labeled compounds for this medical diagnosis program. In some cases these same pre-assayed capsules could be used in isotope dilution analysis procedures to help eliminate some of the inertia involved in initiating a new technique of this sort.

As an alternative to spectrophotometry, Ashton (40) has developed an isotopic dilution assay using chlorine-36 for griseofulvin in fermentation samples. Sorensen (1121) reports on dilution techniques for determination of carboxylic acids, acid chlorides, and anhydrides, using the chlorine-36-labeled chloroanilide of the relevant acid.

Peterson (925) has used carbon-14 labeled corticosterone in the determination of this material in plasma. The method is particularly useful because the plasma extract can be considerably purified without concern about minor losses of the steroid. Berliner and Salhanick (85) couple isotope dilution with chromatography in the microchemical determination of as little as 20 micrograms of non-radioactive steroids. Similarly Wada (1226) reports determination of about one micromole of amino acids with an error of about 10%. Boos and Trenner (117) describe an isotope dilution-static combustion method for organic carbon in submilligram specimens.

Lubran and Moss (711) use isotope dilution in determining serum albumin concentration with iodine-labeled albumin. By using carbon-14-labeled stigmasteryl acetate for determination of stigmasterol in soya sterols a group at Upjohn (270) obtains reproducibilities to within 5% on natural samples. Recovery is usually greater than 97%. Craig (230) measured the active ingredients in an insecticide by this method. Dewitt and coworkers (262) report the application of this technique to studies of the effect of diene structure on the rate of the Diels-Alder reaction.

D. Radiometric Analysis

In this paper we shall again give the term radiometric analysis a limited definition indicating procedures by tracer methods for elements or materials which are not themselves radioactive. In certain types of organic analyses, the borderline between isotope dilution and radiometric analysis is rather hazy.

A number of very interesting methods have been reported during the past two years in this field, especially by the Russian groups. Alimarin and coworkers (17) give an extensive discussion of this technique with 34 references in an English journal and have also written summaries in Russian journals (625,15). Feitknecht and Buser (318) have discussed the identification of stable substances by radioactive indicators in a German article while Duncan and Thomas (276) also give a very good discussion of the radiometric titration of 10^{-7} to 10^{-5} gram amounts of transition metals.

1. Inorganic Determinations. Beryllium in beryl has been determined (104) by using radioactive iron. Two methods for determining fluorine have been reported. Peixoto-Cabral and Götte (904) used the reaction of fluorides with zirconium phosphate labeled with phosphorus-32 to yield an equivalent amount of radioactive phosphorus as phosphoric acid. They find no interference from sodium, potassium, magnesium and calcium but iron prevents the full liberation of phosphoric acid by fluorine. Onstott and Ellis (881) titrate as little as 38 micrograms of fluoride ion with samarium (III) containing europium (III) as a tracer and report less than 2% error.

Two papers (772,938) discuss the radiometric determination of traces of magnesium with phosphorus-32 labeled phosphate and report that in this way 0.5 to 2.5 milligrams of magnesium can be determined with an error of about 1%. Tananaev and Efremova (1172) used the same type of procedure to determine aluminum, indium, lanthanum, cerium, and zirconium by precipitation with labeled phosphate.

Moeller and coworkers (787) used radioactive sulfur and silver to develop specific procedures for determination of traces of sulfates and chlorides in drinking water. Hein and McFarland (467) also describe this type of analysis for chloride ions.

A method of determining (432) chromium in aluminum black treated with chromic anhydride and of measuring (624) 0.1 milligram amounts of potassium with cobaltinitrite labeled with cobalt-60 has been reported. Korenman and coworkers (623)

report the radiometric determination of zinc and copper by titration with potassium ferrocyanide or ammonium mercurithiocyanate in the presence of zinc-65. Another group (607) reports a radiometric method using iodine-131 in a solution of mercury-iodide complex for precipitating copper as the orthophenylenediamine complex. With 0.05 milligrams of copper the results are accurate to within $\pm 3\%$. Bradhurst, Collier, and Duncan (127) give a good discussion of radiometric titrations with silver and cobalt tungstates.

Alimarin and Gibalo (16) determine the zirconium in alloys by a radiometric titration with 0.1 M phosphoric acid containing a small amount of phosphorus-32. Ishimori and Ueno (541) have determined antimony by the formation of a cobalt labeled complex of the antimony.

Thallium has been determined by radiometric titration with sodium tetraphenylboron (1098) or with hexa-amino cobalt using either cobalt-60 or thallium-204 as the indicator (540). Ishimori and Takashima (539) have determined lead by the use of thallium-204 tracer.

2. Organic Determinations. Italian workers (1203,1060) report the determination of C-terminal amino acids in polypeptide chains using ammonium thiocyanate labeled with sulfur-35 while a group from Portugal (912) describe a similar determination of quercitin and rutin using cobalt-60. The use of carbon-14 tagged aldehyde in the trace analysis of monomeric vinyl chlorides has also been reported (683).

A new and rapid technique for the determination of

microgram quantities of protein by precipitation with radioactive tungsten as the tungstate (1082) should prove useful. Brante (133) uses iodine-131 labeled protein or fat in the determination of proteolytic and lipolytic enzymes. Leegwater (682) has been able to determine as little as 0.3 micrograms of estrogens by reacting them with "pipsyl" chloride labeled with iodine-131 and Bojesen (113) has determined microgram amounts of cortisol in plasma with a variation of about 6%. In the same way Fresco and Warner (351) have determined microgram quantities of pyrimidines by treatment with labeled "pipsyl" chloride. They give 15 pertinent references.

Paikoff and Christian (884) report a study of the analysis of phenobarbital using mercury-203 as the indicator. Levels as low as 12.5 micrograms were analyzed with errors around 7% while levels of 1 microgram gave errors of about 25%. The method is applicable only to samples that are free from impurities and that react with mercury to form insoluble substances.

Other papers report the determination by gamma-ray counting of the equivalent weights of iodine-131 labeled derivatives (1146) and the determination of unsaturation in butyl-rubber using carbon-14 labeled isoprene (733).

E. Assay of Radioisotopes

Before a tracer experiment of any kind can be brought to a successful conclusion the radioisotopes used must be reproducibly assayed. Radioisotopes used in tracer experiments vary so considerably in their half-lives, types of radiation

and strength of radiation that often the assay is the most critical aspect of the experiment.

Many assay procedures have already been included in the previous sections on activation and isotope dilution analysis as part of the general methods used. In this section, assay methods including both sample preparation and measurement techniques will be described. References dealing only with radiochemical separation will appear in a later section.

Several counting manuals of National Laboratory groups have been made available recently. The manual by Steinberg (1137) on the interpretation of counting data discusses many general considerations of radioisotope assay and is an excellent summary with 156 references. Manuals are also available from groups at Hanford (1058), Mounds Laboratory (240) and Oak Ridge (1263). Riedel (985) gives a good discussion of the different errors including self absorption inherent in assay procedures.

A number of manuals and compilations of analytical and radiochemical procedures have also been made available, including several manuals of process and recovery laboratories (959,885,1084,1085,187), bio-assay groups (1053,501), and others (5,75,76). The Los Alamos radiochemistry group has again revised its compilation of procedures (609,608) and the Oak Ridge group has recorded some of its procedures (979,977).

Radiochemical analysis and assay of the "Bikini ash" has been discussed in detail by Japanese workers (598,217,597,599) and Thomas (1180) outlines a procedure for analysis of the

"Bravo" shot soil samples. Crouch and Cook (233) give detailed procedures and flow diagrams for analysis of fission products contained in soil and rocks or neutron irradiated plutonium or uranium.

Thorburn (1186) discusses the fission product analysis of urine while Heath and Passell (466) describe a system of radioanalysis for the MTR process water off-gas stream. Meinhard (756) reports a method for estimation of fission product beta activity in uranium. Foss (339) discusses separate radioassays of two isotopes in doubly tagged samples. Weygand (1248) and Winteringham (1257) discuss the assay of labeled compounds in organic materials while two other groups (621,1000) discuss the measurement of weak beta emitters. A bibliography on analysis of certain radioactive elements in biological materials is available (976) and a summary has been made of a symposium on the use of radioactive materials in biological assay (92).

Many of the general references above (13,142,143,1244,1046,38) also present good summaries of general assay procedures.

1. Sample Preparation Techniques. A number of interesting techniques to produce even samples, often the key to reproducible assay, have been reported. Carswell and Milsted (185) make very thin alpha sources by ejecting an organic solution from a fine capillary by the application of an electric field, collecting the material on a metal plate. Tuck (1202) reports a technique for direct evaporation of organic solutions for alpha counting.

Cranston and McCreary (231) have found that beta-ray sources can be made by electrophoretic deposition of material

on a fine wire while another group (653) has made thin layers of electrophoretic boron. Several groups, (1144,832,831,96,769) have described in detail specific procedures for preparing thin beta-ray sources by high temperature evaporation, sputtering or other techniques. Although these sources have been prepared for beta-ray spectroscopy, the same techniques could easily be applied in standard radioassay.

The preparation of uniform films of uranium, plutonium and neptunium has been described by Pepkowitz (906) and Brodsky and coworkers (145). Greene (418) describes a method for electropolishing nickel discs for precision alpha counting while Tait and Townshend (1170) discuss a device for producing a uniform distribution of radioactive material over an area as large as 50 cm².

2. Sample Preparation Equipment. Several pieces of equipment to improve assay procedures have been devised. Bloom (108) describes a filter paper support for the mounting and assay of radioactive precipitates while Jarvis (554) uses millipore molecular filter discs for this purpose. Bronner and Jernberg (147) use a simple centrifical filtration assembly.

Dauby (248) discusses assay and counting techniques. A modified Joliot apparatus for study of the electrodeposition of radioactive materials is described (941) and the application of the ring-oven technique to radioactive substances is reported (1246).

3. Tritium. This radioisotope has now become the least expensive and hence a very popular tracer. A group at Hanford (984)

prepared a tritiated water standard and determined its activity in three different ways, with an error in the final result of about 2%. They discuss their techniques in detail and give 20 references.

Tritium makes an excellent tracer from the point of view of its long half-life, recently determined to be 12.262 ± 0.004 years (560) but its weak beta particles present problems in assay. Consequently, many types of systems have been devised for measuring tritium, extending from the very simple to the rather complex and expensive. Jackson and Lampe (545) used a windowless gas-flow counter for direct counting of solid and liquid labeled tritium samples, with a standard deviation of 4 and 6% for infinitely thick and infinitely thin samples respectively. Banks and coworkers (61) discuss several simple techniques for tritium assay and include a number of good references and diagrams.

Interest in the liquid scintillation counting technique has grown considerably. Okita and coworkers (878) describe measurements with a commercially available Tri-Carb liquid scintillation spectrometer giving results with a standard error of 3.7% for compounds doubly labeled with tritium and carbon-14. The same group (879) and a Los Alamos group (657) report assays for tritium in urine using this type of instrument. Kinard (602) reports a new liquid scintillator for analysis of tritium in water and a British group discusses (284) their tritium measurements. Haigh (439) described a simple liquid scintillation counter for measuring tritium while another

group (1212) reports counting of tritium labeled amino-acids and proteins by this method.

Many variations are reported on the technique for converting tritium in liquids to hydrogen gas for counting. Brown and Grummitt (152) use zinc fusion and Geiger counting of tritium while Christman (208) uses the same preparation but is able to eliminate memory effects by counting in glass proportional tubes. Several groups (312,465,719,720) use calcium metal to decompose urine or water and measure the hydrogen evolved with ion chambers or counters. Healey and Schwendiman (465) report aqueous samples containing as little as 0.01 microcuries per milliliter can be analyzed with an accuracy of about 4% and that about 8 samples per hour can be run with their apparatus.

Another group (1165) reduces the sample in a borosilicate glass bomb with magnesium amalgam at 400° C. and obtains reproducibilities of $\pm 2\%$ over the range of 10^{-9} to 10^{-6} curies per millimole. Glascock (384) converts tritiated water to butane with n-butyl magnesium bromide and uses it as a self-quenching filling gas in a Geiger counter.

Bradley and Bush (130) utilize a technique in which the tritium of water exchanges with the hydroxyl hydrogen of alcohol. The labeled alcohol is then introduced into the Geiger tube as a quenching agent for internal counting. Finkelstein and Lesimple (329) use calcium carbide to convert tritiated water into acetylene which is then counted in an ionization chamber.

Cameron (178) describes a system in which small amounts of water vapor containing tritium are used as part of the filling of an internal gas counter. Butler (168) counts in the Geiger region with much the same type of system while Drever and Moljk (273) discuss the problem of absorption effects in the Geiger or proportional counter assay of tritium.

Robinson (939) describes an improved proportional system for counting tritium as methane with an efficiency of about 11% and low memory. Several modified ion-chamber systems are also discussed. Mattraw (750) describes a system for the determination of as little as one part in 10^9 of tritium in other gases. Ryder (1025) describes an explosion resistant ion chamber for tritium measurements at 10^{-5} microcuries per cc. Tolbert (1189) gives an excellent review of procedures and techniques for ion chamber assay of tritium.

Gracheva and Kusainova (415) determined tritium in liquids while two other groups (1215,1205) discuss their methods for measuring tritium gas. An article by Matsukawa and Eaborn (749) describes simple measurements of the relative activities of tritiated organic materials. Finnigan (330) has applied the Regnault method to tritium purity determinations.

The weak radiations of tritium introduce special problems in safety and monitoring techniques. Barker (67) gives an excellent discussion of the problem of tritium protection and includes 45 references. Techniques and equipment for monitoring tritium in air have also been described (1276,1078,304,274).

Certain special equipment developed for use with tritium

includes a tritium handling system (748), a tritium gas dispenser (72), and a thermal diffusion column for concentrating tritium in tritium-hydrogen mixtures (19). The reaction of tritium with stopcock grease has also been investigated (724).

4. Carbon-14. This radioisotope continues to occupy a prominent position in the organic field. Both wet and dry combustion have been used extensively to decompose samples of organic matter for radiocarbon assay. Glascock (383,384) mixes labeled samples with copper oxide and burns them in oxygen at 700° C. to convert the radiocarbon into carbon dioxide. This gas is mixed with carbon disulfide and counted in a Geiger counter. Christman and coworkers (210,209) use diagrams and pictures to describe their dry combustion apparatus and proportional counting system for assay of micro samples of organic compounds. Vasil'ev (1211) discusses radiocarbon assay by dry combustion and barium carbonate counting.

Rabinowitz (961) describes an all glass apparatus for the wet oxidation of organic samples and subsequent carbon dioxide trapping in preweighed centrifuge tubes for barium carbonate assay. Adcock (4) gives recovery data for a number of biological materials analyzed by the Van Slyke wet oxidation procedure. Rutschmann and Schöniger (1020) give a good discussion of the wet combustion technique while Duff and Knight (275) describe a simple apparatus for handling radioactive barium carbonate obtained in the wet combustion.

Das-Gupta and Nair (247) describe counting of radiocarbon as solid barium carbonate and give self-absorption curves while

Banks and coworkers (60) describe a windowless flow counter for this type of measurement. Moyer and Isbel (808) convert carbon-14 labeled cyanide into carbon dioxide, which is absorbed in alkaline ethylene glycol to then be counted directly in a proportional counter.

Burr and Wiggans (167) determine carbon-14 in blood by direct plating and discuss self absorption corrections for the method. They report results in agreement with those obtained from an oxidation and barium-carbonate precipitation method. Pearce and coworkers (900) dissolve tissues and organs in hot formamide and measure the radioactivity of an aliquot dried on filter paper in a planchette. They also give self absorption curves for their experiments. Garrow and Piper (366) homogenize biological samples and then precipitate the proteins with trichloroacetic acid. The defatted, dried sample is dissolved in sodium hydroxide and transferred to a circle of lens paper for counting. They report results reproducible to within 5%.

Many groups have turned to gas counting to avoid the self absorption problem of the weak beta particles of carbon-14. Christman and Wolf (211) in an excellent article discuss the errors and lower limits of activity detection in the gas-phase proportional counting of carbon-14. Sinex and coworkers (1097) have used a 90% argon, 10% methane counting gas mixture in the Bernstein-Ballentine technique to reduce the voltage required for proportional counting of carbon-14 from 3800 volts to 2000 volts. Apelgot and coworkers (33) describe a system whereby radiocarbon is measured in a counter containing only

carbon dioxide, while in another article (34) they compare gas counting with a barium carbonate-Geiger Mueller counter assay.

Several other articles (1205,1019,1164,258,140) describe equipment and techniques for measuring radiocarbon as carbon dioxide in Geiger or proportional counting systems. Tolbert (1189) has made an excellent practical review of the ionization chamber assay of radioactive gases. In another article (1190), he describes a system using both ion chambers and infrared absorption whereby continuous labeled and unlabeled carbon dioxide records are kept in animal excretion studies.

Self absorption problems are also avoided by assaying carbon-14 in a liquid scintillation counter. The radiocarbon beta particle is energetic enough that simple liquid scintillation techniques requiring no refrigeration can be used for its assay. One literature report (1212) discusses the use of this method for counting carbon-14 and tritium labeled amino-acids and proteins. Two other articles (439,878) give consideration to much the same type of double tracer problem. Dowben (271) describes conversion of carbon-14 samples to benzoic acid while Williams and coworkers (1252) discuss the use of this material as a standard for liquid scintillation counting. Helf and White (469) discuss the problem^{of} using this technique to count organic compounds with nitro groups. These nitro groups tend to quench the scintillation but secondary solutes can be used to increase counting efficiencies.

Several articles (1249,357,462) are devoted to recently

developed techniques for measuring compounds that are insoluble in the liquid scintillator solvent as suspensions or gels.

Funt and Hetherington (358) describe the suspension counting of carbon-14 in scintillating gels.

Passmann and coworkers (893) dissolve labeled carbon dioxide directly in the scintillating solution without recourse to intermediate compounds. Leger and Pichat (685) use paraldehyde for incorporation of large amounts of labeled carbon into the liquid scintillator. An Oak Ridge group (1240) has employed automatic sample alteration in liquid scintillation counting to count sample, background and standard successively.

Schneider (1048) has published results of an investigation measuring carbon-14 with a scintillation detector in various arrangements. Schram and Lombaert (1051) describe the continuous determination of carbon-14 in aqueous solution by a plastic scintillator while Stranks (1147) uses a glass chamber having a plastic scintillator as one wall for assay of radioactive gases.

Götte and coworkers (410) discuss a simplified determination of carbon-14 compounds and a Russian group (628) describes methods for determining the activity of organic compounds containing radioactive carbon.

Specialized low level measurement techniques are required for radiocarbon dating. Several authors use proportional counting of carbon dioxide (132) or methane (165,265), while others use liquid scintillation counting techniques (953,952, 359,65). May (752) gives a chemical procedure for isolation

of organic carbon from bones for radiocarbon dating.

Mamul (739) describes a quantitative autoradiographic method for assay of carbon-14 in histological samples.

Tolbert and Lemmon (1191) give a good review with 58 references of the radiation decomposition of pure organic compounds.

5. Phosphorus-32. Silker (1093) reports a yield of 86% for radiochemical assay of this isotope in a mixture of radio-nuclides and Heald (464) describes an analysis for radiophosphates in extracts of cerebral tissues.

deLoose (260) has made a comparative study of methods for the determination of phosphorus in plant tissues. He includes self absorption curves for the phosphorus-32 samples. Another article (1148) describes a method for determining total and radioactive phosphorus in tracer experiments on small plant samples. In this procedure, self absorption corrections are eliminated before radioactivity measurement by equalizing the amount of phosphorus in each sample. An appendix to this article describes a method for estimating the technical error originating from experimental procedures as distinct from the error caused by random fluctuations in radioactivity.

Schweitzer and Eldridge (1055) discuss the reproducibility of samples of phosphorus-32 prepared by direct evaporation. Meissner (763) reports a paper chromatographic separation and procedure in which as little as 10^{-9} grams of phosphorus may be detected.

6. Sulfur-35. Because of the similarity of the radiations emitted by sulfur-35 and carbon-14, many of the techniques applicable to the latter can also be applied here. Articles mentioned above suggest assay for sulfur-35 with plastic scintillators (105), and with nuclear emulsions (739,269).

The articles by deLoose, (260) comparing various methods of sulfur assay and Dohlman, (268) describing procedures for assay on small amounts of sulfur-35 in tissue, should provide a good background for this isotope. Walkenstein and Knebel (1231) discuss routine analysis of sulfur-tagged drugs. Poddar (936) and Burr and Wiggans (167) have determined sulfur in dried blood samples and have applied known self-absorption coefficients to keep errors down to about 5%.

Götte and Theis (412) discuss the assay of this isotope in thiourea while Helf and coworkers (468) report two different radioassay methods for sulfate impurities in cellulose nitrate. The latter group count their samples in a liquid scintillation counter. A paper by Mitchell and Sarkes (785) describes detection of sulfur-35 with a plastic scintillator and compares this with detection by Geiger tubes. Garrow and Piper (366) describe assay procedures for proteins containing this isotope.

7. Silicon-31, Sodium-24 and Potassium-40,42. Rothbuhr and Scott (997) determine silicon-31 in biological material with an accuracy of 0.5 microgram in studies of silicon uptake by wheat plants. Blum (110) uses a single gamma sensitive detector to determine mixtures of short-lived sodium-24 and potassium-42 in body electrolytes. Assay of the long-lived,

naturally-occurring, potassium-40 by scintillation spectrometry is described by Hurley (525). Other articles describe the determination of this natural potassium in glass (529) and various salts (627).

8. Calcium-45, Chromium-51, Iron-59 and Cobalt-60.

deLoose (260) has developed an optimum procedure for assaying calcium-45 while Mitchell and Sarkes (785) use a plastic scintillator for its assay. The general problem of counting chromium-51 has been considered (1195). Price and Healy (951) discuss measurement of this isotope in the presence of large gamma backgrounds. Libby and Hand (697) show that differentiation of iron-59 and chromium-51 in mixtures can be made with a scintillation detector and a simple lead absorber when no pulse analyzer equipment is available. Odeblad and coworkers (870) analyze mixtures of cobalt-60 and iron-59 by a coincidence technique while Helmholtz (470) describes an instrumental method in which cobalt-60 in fission product waste streams can be determined with a special coincidence spectrometer.

9. Krypton-85, Rubidium-87 and Xenon-133. Tobin (1188)

has assayed radiokrypton and radioxenon with a gamma-ray spectrometer in the course of his diffusion studies. Libby (698) has developed a special technique for assaying the natural activity of rubidium.

10. Strontium-89,90 and Barium-140. Widespread alarm over the radioactive fall-out problem has resulted in the development of a number of strontium assay methods. Martell (744) discusses in detail the absolute assay of strontium in biological

materials, soils, waters and air filters. Use of a special counter with a background of 0.15 counts per minute allows high precision determination of total strontium-90 activities as low as 1 disintegration per minute. Hahn and Straub (438) have determined radiostrontium and radiobarium in water by a combination ion exchange-precipitation process while Stanley and Kruger (1128) used ion exchange entirely.

British procedures for radiostrontium determination in many kinds of biological materials have been reported (158,159). Milton and Grummitt (783) discuss ion exchange methods for determination of strontium-90 in milk ash and Fresco, Hardy and Welford (350) report development of procedures for isolation of strontium-89 and-90 from large samples of bone, soil, vegetation, water, urine and milk. Farabee (314) reports a procedure for analysis of radiostrontium and barium in human urine. Dalton and Welch (242) describe a rapid routine separation and determination of strontium and cesium from fission products.

11. Zirconium-95, Niobium-95, Molybdenum-99 and Technetium-99.

Brennan and Flagg (135) report a determination of fission product zirconium based on TTA extraction while another paper (437) describes a precipitation technique and assay. Morris and Scargill (799) describe a rapid (half hour) procedure for niobium in fission products. Fudge (353) uses paper chromatography with gamma scintillation counting to simultaneously analyse up to 20 samples for niobium in about two and one half hours. The results agree with standard procedures to within 3%.

Rider (983) also uses spectrometry for niobium-95 assay.

McIsaac (725) has used scintillation spectrometry to analyze for molybdenum-99 and technetium-99 with an accuracy of about 10%.

12. Ruthenium-106, Iodine-131 and Astatine-211.

Barraclough (69) describes the determination of radioruthenium in seaweed ash. Camilli and Flagg (179) worked out an electrolytic method for separating and determining ruthenium in redox solutions while Shannon (1074) used an extraction method on organic samples.

Leboeuf (669) and Jones (558) describe methods for determination of radiiodine in vegetation. Pinajian and Christian (934) discuss its assay for medicinal purposes and include 35 references. Gevantman and Pestaner (375) consider losses in counting iodide and iodine on paint and metal surfaces while another group (559) discusses the general counting of radiiodine in chemically separated samples. Jensen (552) compares the scintillation counter and scintillation spectrometer for measurement of iodine-131 uptake.

Basson (73) discusses the absolute counting of the alpha-emitting astatine-211 in a liquid scintillation counter.

13. Cesium-137 and Cerium-144. Procedures have been reported that determine cesium by cobalticyanide precipitation (656), hexanitrodiphenylamine precipitation (436), chloroplatinic acid precipitation (309), a combination of anion exchange and gamma-ray spectrometry (1264), and anion exchange-precipitation (921). Kahn, Smith and Straub (569) describe three procedures

for determining radioactive cesium in water in concentrations as low as 10^{-4} to 10^{-8} microcuries per milliliter. Yamagata (1271) uses ion exchange for this same type of determination. Crouthamel and Turk (235) use a standardized assay of cesium-137 as an indication of fission burn-up for pile constant measurements. Dalton and Welch (242) describe a rapid routine determination of cesium from fission products.

Smith and Moore (1113) use liquid-liquid extraction to assay radiocerium, with a yield of about 80% and a precision within 2%. McIsaac (725) has used scintillation spectrometry for this isotope.

14. Iridium, Lead, Bismuth and Polonium. Campbell and Nelson (180) applied rapid ion exchange techniques to identification of a lead isomer with a 0.8-second half-life and an iridium isomer with a 4.9-second half-life. King and coworkers (603) describe procedures for assay of relatively long-lived lead-210 (RaD), bismuth-210 (RaE) and polonium-210 in the atmosphere.

Spoerl (1126) reports procedures for assay of polonium in urine. Black (97) has assayed polonium in soft tissue and bone, as well as in urine. Smith and others (1112) describe autoradiographic methods for determining polonium in biological materials. Ishimori and Hataye (538) determined polonium in radioactive spring waters. de Benedetti (253) made measurements of polonium in infinitely thick samples.

15. Emanation and Radium. Determinations of radon have been made on air (79,259, 229), on expired radon in health tests (713), and on the air of uranium mines (174). Other

determinations have been made on water (349), on radioactive springs (1274,189) and on airborne dust particles (649).

Two groups (1069, 932) have counted radon at low temperatures to avoid diffusion. Lucas (712) describes an improved low-level alpha scintillation counter for radon capable of detecting as little as 10^{-16} curies of radon per liter of air while Burke (164) uses a cylindrical ionization chamber for this type of measurement.

Barker and Thatcher (66) detail a procedure whereby as low as 0.1 micro-microcuries of radium can be detected in water while Kirby and Brodbeck (606) report assay procedures for radium-226, actinium-227 and thorium-228 in urine. Gubeli and Jucker (427) compare emanation methods for radium determination with direct radiation counter measurements and get good agreement. Several methods (510,77) have been reported for determining radium in ores, including an elaborate study by Rosholt (995). May (751) used a gridded ion chamber for radium and radiothorium analysis.

16. Actinium and Protactinium. Perey and coworkers have reported in several publications (917,916,913,914) a method for determining actinium by its daughter francium. This method is sensitive enough to detect as little as 0.003 microcuries of actinium. Actinium has been determined in urine by Kirby and Brodbeck (606) and in ores by Becquerel (77). Foster and Stites (341) report a novel method in which the neutron emission from actinium fluoride is used for a quantitative assay. A one-curie source of this type

theoretically should give 1.07×10^6 neutrons per second and experimentally it was found to give 1.21×10^6 . They suggest the possibility of extending this method to other alpha-active elements.

Salutsky and coworkers (1028) determine protactinium-231 by gamma spectrometry while a Russian group (1131) reports a new method of determination. Fudge and Woodhead (354) describe the isolation and determination of protactinium-233 and include 25 references. Moore and Reynolds (790) also describe its determination while McIsaac and Freiling (726) evaluate methods for determining its counting efficiency.

17. Thorium. Scintillation spectrometry has been applied to thorium analysis (585,524,525) as has alpha spectrum analysis (311,751). Silker (1094) determined thorium by counting the thoron daughter produced. Several groups have worked on assay procedures for thorium in rocks and ores (886,1076,8,77). Thorium isotopes have been determined in sea water (615) and in urine (606). Starik and coworkers (1132) report an assay procedure for ionium (thorium-230) while Picciotto and Wilgain (933) use photographic emulsions for thorium assay at the millimicrogram level. Campbell and Carl (181) combine a radio-metric and fluorescent X-ray method of analyzing for thorium and uranium. The natural gamma activity associated with thorium at different times after purification has been calculated (978).

18. Uranium. Assay procedures are reported for uranium isotopes in rocks (886,8,77,1070) in mud (596), in precipitated phosphate materials (1007), in leach solutions (887), in

irradiated thorium (883), in natural waters (1178), and in human urine (334). Several groups have used gamma spectrometry (525,524,585,779,228), another alpha spectrometry (311) and still another has determined content of radioactive minerals by autoradiography (1206). Stewart (1141) describes the beta counting of uranium-237 and -239, Rubino (1006) reports an alpha counting assay for uranium in ion exchange effluents, and a third group (1266) describes a gamma count estimation of uranium-235 concentration. Campbell and Carl (181) combine radiometric and X-ray fluorescent methods for uranium while Lovett and Roberts (708) compare different methods for the isotopic analysis of uranium in a good summary article. Isolation and measurement of uranium at the microgram level has been studied (1015) and special techniques required to get very thin films of uranium have been reported (614,495,388).

19. Neptunium, Plutonium and Americium. Moore (790) describes a separation and determination of neptunium based on extraction into TTA, while Lyon (714) and Wish (1258) have determined the counting efficiencies for neptunium-239. McIsaac (725) uses scintillation spectrometry to measure this isotope.

Assay procedures have been reported for plutonium in air-borne dust (1169), in uranyl solutions (629), in sodium and potassium salt solutions (556), and in urine (1103,1038,313). A special procedure using nuclear emulsions for very low levels of plutonium in urine has been reported (1059). A solvent extraction method using TTA is described (792). Several

articles (388,292,612,1267) describe preparation of infinitely thin sources of plutonium and other actinide elements. Another reference compares direct evaporation and lanthanum fluoride methods for plutonium analysis (83).

An assay for americium has been described (1047) and a method for the electrolytic deposition of this element reported (611).

F. Separation and Preparation of Radioisotopes

These papers differ from those in the previous section in that in general their primary scope is the separation of a radioactive material, although in some of the general compilations of procedures the assay may also be discussed. So many articles have appeared on procedures for carrier-free separations that they have been grouped into a separate section.

Fission product schemes reported include separations by electrolytic precipitation (702), electrodialysis (280), adsorption from organic solvents (1052) and concentration by zone melting (1063). Other procedures for these elements include separation from aluminum waste solutions by ion exchange (100), leaching from soil samples (566), concentration (455), fuel element processing (972,1072), and other chemical operations (391,342,281,568,1043).

A number of groups (803,298,709,663,650,1102) have been interested in decontamination and clean-up of water contaminated with radioisotopes. Other general articles include ones on isotope production and process development (1061), an excellent review by Cook and Seligman (223) on chemical treatment of

isotopes produced in a reactor, a survey (21) of masking agents used in quantitative analytical chemistry and several books on process chemistry and transuranium elements (731,153,584,416).

1. Fluorine to Calcium. The production procedure for fluorine-18 has been described by Banks (59). Hollbach and Yaffe (497) have developed a process which recovers 99.8% of sodium-22 in high specific activity, and Perkins (922) reports a procedure for the separation of trace amounts of sodium-24 from reactor effluent water. Production of magnesium-28 both in a reactor and in a cyclotron is described (765,857). For reactor production, an alloy of magnesium and lithium enriched in lithium-6 is used, and the magnesium-28 produced by the (t,p) reaction. Kohman and coworkers (517) describe the production of long-lived aluminum-26,

Three patents (723,118,306) and a report (1036) describe different methods for recovering phosphorus-32 from irradiated sulfur. Fogelström and Westermarck describe (336) the production of phosphorus-33 by neutron irradiation of sulfur and discuss (1247) the problems in millicurie production of this isotope. Two other papers describe the preparation of colloidal suspensions of chromium phosphate (29) and zirconium phosphate (755) labeled with phosphorus-32. Sunderman and Meinke (1157) have developed optimum procedures for radiocalcium separations and Nervik and coworkers (821) discuss the separation of calcium from milk with Dowex-50 resin.

2. Chromium to Strontium. Smith and coworkers (1115) give detailed procedures for minute amounts of chromium, iron,

nickel, and cobalt corrosion products of stainless steels present in a gross fission product mixture. A separation for manganese in reactor effluent water has been reported (922). A method for preparation of radioiron of high specific activity (1166) and a quantitative electrodeposition procedure (738) for iron have been described.

Schneider (1049) describes the separation of radiocobalt from aqueous waste solutions and Caldwell and Graves (176) discuss the preparation of secondary standard cobalt-60 sources by electrodeposition. Maletskos and Irvine (738) describe the quantitative electrodeposition of radiocobalt and radiozinc on copper planchettes. Copper separation from reactor effluents has been described (922).

The separation of strontium and cesium from calcined metal oxides has been described by Abriss and coworkers (3) and a rapid separation of strontium-89 by ion exchange is reported (1054). A strontium separation procedure involving precipitation techniques which gives optimum yields and minimal contamination from representative tracers has been described (1157), and the separation of strontium from milk by ion exchange has been studied (821).

3. Zirconium to Silver. Moore (789) uses a TTA extraction to separate zirconium from other elements. A report (487) and a patent (485) describe solvent extraction separations of niobium using methyl isobutyl ketone and diisopropyl ketone respectively. Two separation procedures for molybdenum have also been reported (1040,889). Krohn (639) discusses a survey

of separation processes for technetium. Gresky (421) has prepared ruthenium-106 tracer from waste metal solutions while Haissinsky and coworkers (442) have used electrochemical methods for preparing ruthenium sources. The Harwell group report (204) a procedure for isolating the 36-hour rhodium-105 tracer from mixed fission products.

Sicilio and coworkers (1091) describe a precipitation procedure for separation of radioactive silver from pile-irradiated palladium. Sunderman and Meinke (1157,762) discuss the separation of radioactive silver by the isotopic exchange technique and Olcott (880) describes the temperature dependence of this exchange. Sunderman and Meinke (1157) also describe an optimum radiochemical procedure for silver based on an evaluation of precipitation, electrodeposition, and isotopic exchange procedures. Joshi and Thosar (562) use a conveyer belt for separating and studying very short-lived silver activities.

4. Indium to Cesium. Chemical separations reported for indium include two extraction procedures with the acid bromide (533,534) and one method using concentrated sulfuric acid (867). Sunderman (1156) has studied yields and decontaminations for the acid bromide extraction, and for an anion exchange, and a precipitation separation of indium. Antimony has been concentrated (819) by recoil using triphenylantimony difluoride.

Two papers (220, 36) and a patent (1256) describe the production of iodine-132 from the tellurium-132 radioactive parent, while two other papers (305,590) describe the production

of iodine-131 by neutron irradiation of stable tellurium. Another reference (1017) describes the production of fission product iodine-131. Papers have discussed the removal of iodine-131 by isotopic exchange (718,1156) and by molecular sieves (1229). The separation of organic and inorganic iodine has been effected by anion exchange (101) and by radiochromatographic techniques (1145,993). Others utilize isotopic exchange (249) or precipitation (399) reactions for this type of determination. Procedures are reported for preparing the 12-day xenon-131 isomer (722).

Separation of cesium has been made from urine (1064), from macro quantities of sodium and potassium salts (1185), and from high-level calcined metal oxides (3). Chromatographic separation of cesium-137 on cellulose is reported (343) while two other papers describe industrial scale separation of cesium-137 (333,463).

5. Barium to Rare Earths. Kruger and Coryell (640) describe an ion-exchange separation of barium-140 from its daughter lanthanum-140. Other groups report the separation of barium and radium (942), and barium and natural uranium (643). An isotope "milker" which supplies 2.6-minute barium-137 from the parent cesium-137 has been designed and is commercially available (827). An optimum barium procedure has been developed after testing a number of precipitation procedures with many representative radioactive tracers (1157). A pilot plant process for separating barium-140 from uranium fuel elements is also described (686).

Kirby (605) describes the preparation of radiochemically pure cerium by solvent extraction. Three papers (207,239,948) consider the separation of short-lived rare-earth fission products by ion exchange techniques while two other papers (910,907) discuss extraction of these elements in tributyl-phosphate-nitric acid systems. Stewart and Faris (1141) describe a good technique for concentrating rare-earth materials from large volumes of eluate. Chromatographic separation of the yttrium group (943), and paper chromatographic separation of the gadolinium group^(b7b) of rare-earths have been reported. Several other procedures for these elements have also been described (679,498,946,355,162).

6. Tantalum to Polonium. Tantalum has been separated by solvent extraction (487,485). Mitchell and Martin (786) use the Szilard-Chalmers process for preparing osmium. Methods are reported for preparation of radioactive colloidal gold (473) and for recovery of thallium-204 from old plating baths (1108).

Harrison and coworkers (451) describe an electrolytic separation of bismuth-212 and lead-212, while another article (376) describes the influence of surface state on the cathodic deposit of bismuth-210. A paper by Ishimori (537) gives an anion exchange separation for lead-210, bismuth-210 and polonium-210. Separation of polonium has been made by distillation (300), solvent extraction with diisopropylketone (175) and electrochemical exchange with silver (1197,51). Precision plating of polonium is discussed (319) as are its colloidal

properties (1130). Rapid separations of polonium from cyclotron targets are described in detail by Oelsner and Forsling (872). Several summary articles (50,49,45,807) with many references on the chemistry and separations of polonium are also available.

7. Emanation to Thorium. Kusaka (644) reports a new method for collecting thoron from thorium oxide. A laboratory scale ion-exchange separation of radium and barium has been reported (942) while Salutsky and Stites (1032) have developed a larger scale radium-barium separation process based on precipitation of the chromates from homogeneous solution. Glover and O'Connor (389) report a preferential deposition of radium-224 on stainless steels from thoria slurries.

Salutsky and Kirby (1029) precipitate actinium oxalate from homogeneous solution while Tsong (1201) uses ion exchange to separate traces of actinium from large amounts of lanthanum. Another article (991) describes the separation of actinium and its daughters with lead sulfate.

Separations of long-lived thorium-232 by anion exchange (184), tributylphosphate extraction (908) and by coprecipitation (1177) have been described. Procedures for other thorium isotopes include an extraction of thorium-228 from bone (1135), an extraction of ionium (thorium-230) (198), and a separation of UX₁ (thorium-234) (291,261,630).

8. Protactinium and Uranium. Two detailed papers describe the separation of protactinium (395,1031). Individual separation steps reported include concentration of protactinium by anion exchange (68), solvent extraction (392,908), manganese

dioxide precipitation (583) chromatography and paper electrophoresis (680), and electrolytic deposition (323,324).

Ricci (982) describes a new method for the separation of protactinium-234 (UX_2).

Solvent extraction separations of uranium with tributylphosphate (322,267,420,908,1163), diisopropylether (320), methyl isobutyl ketone, dibutyl cellusolve (321), and ethyl ether (55,981) have been reported. Kienberger describes (595) an automatic solvent extraction apparatus for uranium. Other methods include ion exchange (184,869,1161,1259), and precipitation (1177). Two articles describe micro separations of uranium (297,960) and another discusses the recovery of uranium from biological slimes (390).

9. Neptunium to Berkelium. Two American papers (490,736) and two Russian papers (642,1089) give methods for separating neptunium. Kooi (620) describes tracer experiments on the solvent extraction of neptunium, while another paper (922) gives details of the separation of neptunium from reactor effluent water.

Plutonium separations that have been reported have involved solvent extraction (236,397,620), cation exchange (266,701,1092) and precipitation (642,1183) methods. Two tracer methods are described (930,1021) in addition to a larger scale separation (1024). Electrolytic oxidation of plutonium prior to deposition is described (610).

Ion exchange separation of gram amounts of americium has been described (35,216) and a procedure for milligram

amounts is also reported (477). Coprecipitation separations (1269) and anion exchange separations (385) for americium have been discussed. Gift (379) compares analytical methods for americium. A solvent extraction separation for berkelium (911) is reported.

G. Carrier-free Separations

A specialized category for carrier-free separations has been included in this review since so many separations recently have been designed with this idea in mind. This list supplements and does not overlap the list presented above in the general separation section.

Murin, Nefedov and Yutlandov (813) give an extensive review with 128 references of the methods of production and separation of carrier-free isotopes. Several authors (898, 1155, 1260) describe the application of nuclear recoil in the separation of carrier-free isotopes. Evans and Willard (307) discuss the separation of carrier-free substances by gas phase chromatography, and Samsahl and Taugböl (1037) describe this type of separation by a diffusion method. Preiss and Fink (944) consider the practical problem of adsorption of carrier-free radioisotopes on glass storage vessels.

1. Beryllium to Ruthenium. Preparation of carrier-free beryllium-7 from lithium by extraction with acetyl acetone is described (115). Simultaneous production of carrier-free aluminum-26 and sodium-22 is reported by Rightmire and coworkers (986), while Hudis (511) describes production of magnesium-28 from aluminum. The short-lived phosphorus-30 has been prepared

by recoil (897) and phosphorus-32 by solvent extraction (632). Mellish (764,766) describes the production of carrier-free manganese-54, cobalt-56 and cobalt-58 by anion exchange. Passell (892) suggests use of the (n,p) reaction on nickel-58 to produce cobalt-58.

Preparation of carrier-free nickel-63 by the (n,p) reaction on copper-63 is reported (945) and copper-62 can be prepared without carrier by recoil techniques (1152). Mellish (764) prepares zinc-65 by anion exchange.

Carrier-free yttrium-90 has been prepared by paper chromatography (593,594), by electrolysis (654) and by other methods (125). Zirconium and niobium activities can be prepared carrier-free from fission products by cupferron-chloroform extraction (743) while niobium has also been separated by anion exchange (764). Kambara (575,576) describes distillation procedures for radioruthenium.

2. Indium to Bismuth. Russian workers isolate indium isotopes without carrier (820,1010,1012) and Lawson and Kahn (667) use absorption and solvent extraction procedures for separating indium from cadmium. An Indian paper (90) gives details of the separation of tin-113 from indium by extraction of the stannic cupferrate complex with carbon tetrachloride. Another paper (377) describes the separation of carrier-free tellurium tracers by paper chromatography.

Perkins (920) uses a filtration-precipitation separation of barium-140 from lanthanum-140. Two groups (1087,1262) describe the carrier-free separation of cesium-137, while two other groups (215,891) report carrier-free separation and purification of rare

earth fission products. Rudenko (1008) prepares radiochemically pure thorium-B (lead-212) and thorium-C (bismuth-212) by a dithizone extraction procedure.

H. Tracer Applications

Only a part of the many applications of tracers in analysis are outlined below. As mentioned in the previous review, the extent of the use of tracers in analysis seems to be limited only by the ingenuity of the research worker. Small amounts of radioactivity are ideal for tracing in chromatographic or electrophoretic apparatus, whether it be columns of cellulose or ion exchange resins, or papers with one or two way flow. Many other types of analytical separations such as precipitations, extractions and electrodepositions have also been studied with tracers.

Catch (188) presents a good general discussion of the use of radioisotopes as tracers and has included 43 references. Rudenko (1009) describes methods depending upon complex formation for isolation of radioactive isotopes while Specker and Hartkamp (1123) give a good discussion with 129 references of the aims and methods of concentrating trace elements. Other general articles on tracers have also appeared (431,794, 974,1033).

1. Chromatography (Including Ion Exchange). Use of ion exchange resins to concentrate radionuclides for subsequent analysis is described (567). Several general articles on ion exchange (641,673,1045) have sections on radioisotope separations and an extensive bibliography on ion exchange

resins is now available (570). Elovich and Prusakov (296) describe a radiometric study of the chromatography of bi- and ter-valent ions over a concentration range of 10^{-1} to 10^{-9} .

Tracer studies on cation exchange resins have included rapid separations with alpha-hydroxy-isobutyric acid (206,782,1114), with ethylenediaminetetra-acetic acid (282), and with other miscellaneous complexing agents (295,435,1193). Bonner and Smith (116) report a selectivity scale for some divalent cations and Surls and Choppin (1159) discuss the equilibrium sorption of certain lanthanide and actinide tracers. Stewart (1139) has studied the problem of controlling rare-earth separations by means of varying resin column operating temperatures.

Further tracer studies include ion exchange separations of trace impurities (150), fission products (784), polonium (245,1192) and rare earth and transplutonium elements (1140). Tracers aided in anion exchange studies of thiocyanate complexes of the actinides and lanthanides (1160) and other studies (99,947).

Kraus and his coworkers have continued their work on anion exchange with several tracer studies (634,635,1018). Hicks and coworkers (486) have issued a good summary report on qualitative anionic behavior of a number of elements with Dowex-2 resin. Herber and coworkers used tracers to explore the effect of cross-linkage on the elution of transition elements (475) and on the separation of nickel (474). The anion exchange separations of zirconium and protactinium (571,734),

of molybdenum and technetium, tungsten and rhenium (515) and of tellurium, ruthenium, cesium, and the rare earths (1162) have been studied. Vick and Leddicotte describe an ion exchange separation procedure (1216).

Radiotracers have been a boon to paper chromatography. General discussions include those of Lederer (678), Nakano (815), Guedes-de-Carvalho (428), and a Japanese group (536). Warren and Fink (1236) have modified the method so that separation of microgram quantities of radioactive materials can be made in 2 to 20 minutes. Götte and Pätze (411) summarize the identification of uranium fission products by paper chromatography while another article (166) describes the use of radioisotopes in paper chromatographic studies of inorganic compounds.

Other tracer experiments with paper chromatography include separation of alkalies, alkaline earths and rare earths (344), non-complexed rare earths (675), niobium and tantalum (156), germanium (674), beryllium, germanium, indium and gallium (429), and technetium (695). Troitsky (1199) has modified this technique for the identification of short half-lived radioisotopes.

Tracers have been used in paper electrophoresis (677), electrophoretic separation of niobium and tantalum (155), chromatographic separation of hafnium and tantalum (1011), electrochromatographic studies of phosphates labeled with phosphorus-32 (1086), equilibrium studies on natural ion-exchange minerals (22), paper electrophoretic studies of radioactive chromium salts in blood (1279), and chromatographic separation of iodine labeled derivatives of sterols and amines (1145).

Other articles describe a study of adsorption columns for effecting precipitation separations (482), and the concentration of isotopes by electromigration on asbestos impregnated with fused salts (203).

2. Solvent Extraction. Tracers have played an important part in many of the solvent extraction studies reported in the new book by Morrison and Freiser (802). An interesting study was made with labeled tributylphosphate (589), while a number of tracer experiments have been run with organophosphorus compounds (488,732,1250). Other studies include extraction with acetylacetone (638), isobutylmethyl ketone (1023), and long chain amines (791). Studies of the extraction of individual fission products from process wastes (1026), the concentration of radio-elements (565) and the dependence of the distribution coefficient on the metal ion concentration (264) have been described.

Tributyl phosphate has been used in tracer studies as an extracting agent for nitrates of the actinide elements (728), for yttrium and the lower lanthanide nitrates (1041), for zirconium nitrate (11), for the plutonium nitrates (87), and for thorium nitrate (483). Another paper discusses the solvent extraction behavior of scandium, thorium and zirconium in tributyl phosphate systems (909). An interesting early summary, recently declassified, by C. A. Kraus (633) reviews the extraction of iron and uranium from aqueous solution. Solvent extraction studies with tracers have been made of niobium and tantalum (157), arsenic and germanium (138),

protactinium (636), astatine (825), polonium (52,245,246), neptunium (619) and certain fission products (730).

Tracer studies of the extraction of styrenated oil acids (111) and DDT metabolites in house flies (923) have been reported. Tracers have been applied to studies of the extraction of niobium, tantalum, and titanium cupferron (14), mercury iodide (804), and niobium rhodanide (1200) complexes, a correction for the colorimetric determination of niobium in the presence of tantalum (160), and the partition coefficient of ferric chloride between organic solvents (797). Solvent extraction studies of metal halides at low concentrations (940), use of acetylacetone extractions in iron analysis (727), and separation of microamounts of indium from zinc by liquid-liquid extraction (631) have also been reported.

Several articles also deal with the use of radioactive indicators in the study of fused salts and mixtures (746,1153,1219) and high temperature separation processes (798). The problem of radiation damage to processing reagents has been discussed (840). A continuous ether extractor (553) and a miniature mixer-settler for laboratory-scale, continuous, countercurrent solvent extraction (109,1232) have been described.

3. Precipitation. The Russians have been particularly instrumental in developing techniques for use of organic coprecipitants in analytical chemistry. Kuznetsov has written a book (645) as well as a general report (646) on the subject. Others report the coprecipitation of cadmium (626), cesium (622), thallium (647,1223), tungsten (648), cerium and europium

(737), and cobalt (1056) with organic coprecipitants.

A study of coprecipitation of trace ions with ferric hydroxide (1081,1157), and aluminum hydroxide (1157) has been made. Mashima (747) measured the amount of zinc coprecipitated with ferric hydroxide while two other papers (666,965) discuss the mechanism of coprecipitation of radioisotopes with hydroxides.

A number of tracer studies have been made of coprecipitation from homogeneous solution. These include studies of rare earth iodates on thorium iodate (1077), radium on barium sulfate (405), thallium on silver chloride (404), and zinc and yttrium on ferric periodate (403). Gordon and coworkers also discuss coprecipitation in binary systems of rare earth oxalates (317) and concentration of microgram amounts of rare earths in thorium (402). Studies have been made of the experimental conditions for coprecipitation of phosphorus-32 on copper sulfide (581) and of the use of surface active agents to prevent coprecipitation of ions with sulfide precipitates (1013).

Also studied with tracers were anodic coprecipitation of manganese and silver with lead dioxide (169), coprecipitation of antimony with manganese dioxide (48), coprecipitation of manganese, lanthanum and titanium with thorium oxalate (573), coprecipitation of transuranium elements with rare earth oxalates (476) and removal of fission products from an acid aluminum nitrate solution by coprecipitation (637).

The separation of rare earths from thorium nitrate (690), the determination of thorium in mineral spring waters (542),

the separation of magnesium from sodium and potassium (484), and the effect of iron on the determination of tin in brass and bronze (661) have also been studied with radiotracers.

4. Miscellaneous. Lavrukhina (664) reports on the behavior of ultra small amounts of elements and gives 23 references. Tracer studies of the low concentration chemistry of yttrium (1057) and cesium (1272) are also reported.

Simnad (1095) gives an extensive review on the use of nuclear irradiation and radioisotopes in metals research. Two Geneva papers (519,1237) and a report (450) describe applications of radiotracers in the petroleum industry. Other papers (177,561,582,1042) describe general uses of tracers in the chemical industry and in process and quality control.

Thomas (1181) uses tracers in gas mask canister studies, while another paper (843) describes their use to measure the absorption of cosmetics. Carbon-14 has been used (225) to study the distribution in an engine of fuel additives while cobalt-60 and sulfur-35 have been used to study engine deposits (352). Radioisotopes have been used to find water main (851), gas storage (848), and nuclear plant leaks (1214), to determine the behavior of argon in vacuum systems (183), and to leak test sections of the transatlantic cable (846).

Radioactive inks (855) have been found to make excellent "photocopies". Low energy beta sources provide long life-time self luminous surfaces for light calibration standards (862). Putman and Smith (957) used tracers to determine sand and silt

movements under water while Begemann and Libby (78) report the use of tritium in studies of ocean mixing rates and circulation patterns. Baranov and Kuzmina (63) discuss analysis of deep sea deposits in relation to sediment accumulation rate.

Tracers have been used in many kinds of mixing problems. Bromine-82 as brom-methane vapor has been used to study turbulent diffusion and mixing in coal mine ventilation (494) while other tracers have been used to study mixing in surge tanks and stills (520). Tracers measure wear in piston rings (854), ball bearings (651), machine parts (425) and tools (768,927). Tracers locate entrainment in distillation units (1227), trace catalysts in refinery crackers (433) and study industrial flow patterns in plants (518).

Tracers can be used in surface studies. Caro and Hill (182) determine the surface area of dicalcium phosphate by isotope exchange with phosphorus-32, while Lewis and Plumb (696) determine the specific surface area of aluminum metal surfaces with tracers. Another group prepares "built-up" monomolecular layers of cobalt-60 stearates (456) while two other articles describe measurements of monlayer adsorption (224,550). Haissinsky (441) uses tracers to study the heterogeneity of electrode surfaces.

The Hahn emanation technique using iodine-132 has been used for study of changes in the solid state (222). In washing tests, zirconium-95 labeled colloidal carbon dispersions compare favorably with the reflectance method (287,928). Washing coefficients for solids have been studied with phosphorus-32 (773).

Methods for generating radioactive aerosols (924) and preparing air-borne simulated nuclear fall out (214) are described.

Govaerts uses radioisotopes to study the chemical state of phosphate and calcium ions in plasma (414) and the fixation of phosphate in the earth (413). Radioactive tellurium has been used in fluoride volatility studies (331) and sulfur-35 in sulfate studies (369). Other papers describe the determination of the different chemical forms of sulfur contained in mucines (42), the use of phosphorus-32 in the assay of vitamin D (866), and a comparison of radioisotope technique and flame photometry in the bioassay of mineral-corticoids (704). Discussion of radiochemical purity as a new specification for purity of an analytical reagent of cerium is presented (1030).

IV. INSTRUMENTATION AND MEASUREMENT

The field of instrumentation continues to progress rapidly with innovations in design and scope. The guide to instrumentation literature issued in December 1955 by the National Bureau of Standards (146) gives an excellent introduction and source list of over 1200 references. Other general discussions include those in the Handbook of Microchemical Methods (86), a book by Sharpe (1075), and two summary articles (407,430). Parish and Kohler (899) give an excellent review of the use of counter tubes in X-ray analysis, much of which is generally applicable to counting equipment. Several general articles on counting techniques are available in the literature (444,506,1230,1245), including the excellent one by Steinberg (1137). An index of suppliers of the latest instrumentation and measuring equipment can be found in the Nucleonics annual buyers' guide (852).

A. Scintillation Counting

This counting method continues to be increasingly popular. Several general discussions of scintillating materials and equipment have been given (148,532,655) while other articles discuss scintillation counting of beta emitters (1067) and detection of very low energy gamma rays (508).

Many articles on solid scintillators can be found in recent issues of "Nucleonics" and "Reviews of Scientific Instruments", but there will be space here to mention only a few representative and summary articles. Sangster and Irvine

(1039) report an extensive study of the scintillation counting behavior of a large number of organic scintillators. Several articles attack the problem of crystal efficiency (668,1001), resolution (841) and background (778). The high cost of halide crystals has brought the development of large plastic scintillators (212) which are particularly adaptable to well counters (491,774). Other articles discuss the optical mounting of a scintillator (616), a well-bottom container to improve gamma counting (272) and a special scintillation counter for paper chromatograms (1133).

Many developments have been made in liquid scintillation counting because of its usefulness in measuring weak beta emitters. Some techniques of this type have already been mentioned in the sections on assay of tritium and carbon-14. Hayes (458) has written an excellent general discussion of the attributes and applications of liquid scintillators and has co-authored papers on pulse height comparisons of primary solutes (461) and secondary solutes (460) and on quenching problems (591) of liquid scintillators as well as their application in nuclear medicine (459).

Davidson and Feigelson (250) give an excellent discussion of the practical aspects of internal-sample liquid-scintillation counting with commercially available equipment. Seaman (1062) describes a simple liquid scintillation counter for chemical analysis with radioactive tracers. Several authors (47,574, 1277) discuss ways of increasing the efficiency and extending the application of liquid scintillators. Still others (357,

462,1249) describe techniques for counting as a gel or suspension materials that are insoluble in the scintillator.

Special liquid scintillation techniques have been developed for measurement of low-energy beta radiations (999), for application to radiocarbon dating (952) and for measurement of weak beta emitters on filter paper (998). Three other short techniques papers (7,62,103) may be of interest. Brennan and Landecker (135) describe a large area liquid scintillation counter of high uniformity.

Kinard (601) discusses the temperature dependence of photomultiplier gain for scintillation counting systems and gives a number of pertinent cross references. Ball and coworkers (54) also discuss this subject. Other reports discuss the dependence of counting rate on the voltage of the detector (139), the reduction of photomultiplier thermal noise (400), and the fatigue of photomultipliers at high counting rates (1220).

B. Spectrometry

During the past two years, single channel gamma-ray spectrometers have become readily available and multichannel analysers have become more versatile and gradually less expensive. In measurements described in the previous section, all pulses greater than a certain level are counted. In contrast, this section deals with situations where an energy spectrum that is characteristic of a nuclide can be obtained.

The increasing importance of scintillation spectrometry in analysis has brought forth a number of excellent review articles. These include articles by Peirson (902,903), Cook

(219), and Connally (218). Other general articles on the subject have also appeared (234,345,670,754). A simple recording gamma-ray spectrometer (1213), and two spectrometers with photographic recording (205,735) have been described. Barchuk and coworkers (64) discuss the resolving power of scintillation spectrometers. Scintillation spectrometry of low energy gamma rays (120) and bremsstrahlung (288) is discussed. One article (454), describes the distortion of spectral intensities because of finite channel width, while another (134) gives a mathematical treatment of spectrometer statistics. Garlick (365) discusses the physics of the scintillation counter, particularly as applied to the statistical fluctuations in the peak spread.

Davis and coworkers (251) describe the response of "total absorption" spectrometers to gamma rays. Plastic phosphors have been used to surround the detector to reduce the Compton effect (1002). Spectrometers have been used in fuel burnup determinations (337), in physico-chemical studies (1100), and in analysing mixtures of isotopes (31). Crasemann and Easterday (232) describe ways of locating Compton edges and backscatter peaks in gamma scintillation spectra. Tove (1194) has studied the distortion of spectra by long light guides while another group (88) has compared cesium iodide with sodium iodide for scintillation spectroscopy.

Lyon and Reynolds (715) have assayed electron capture nuclides with a proportional counter spectrometer. A cylindrical proportional counter applicable to X-ray spectrometry has been

described (137) and some corrections for its use discussed (328). Solid scintillators have been used for beta spectrometry (95) with good results. An Argonne group (346) discusses a backscattering correction required for these measurements. Gardner (364) has developed a hollow plastic scintillator for routine beta-ray spectrometry.

Successful spectrometry depends upon the ability of the system to divide the energy spectrum into equal segments. This can be done either by a continuous scanning technique with a single channel analyser or by multichannel equipment. Higinbotham (489) gives an excellent review of the pulse height analyser methods currently used for this purpose.

A British report (387) discusses some types of problems to which pulse analysis is applicable. Koch and Johnston (613) have made an excellent review of multichannel pulse height analysers. Putman and Taylor (958) propose the subtraction of gamma-ray spectra as a tool in the analysis of nuclide mixtures. Chauvin and Leveque (192) use a discriminator instead of a shifting window to distinguish between certain nuclides, thus eliminating the need for pulse analysis in simple situations.

C. Nuclear Emulsions

Lauda (662) describes photographic methods of radiochemistry in a section in the "Handbook of Microchemistry". Several groups (2,290,931,1206) have applied autoradiographic techniques to the determination of radioactivity in rocks. Gomberg and Schlesinger (398) describe high resolution autoradiographic techniques and Mamul (739) gives his method of quantitative

autoradiography using a radioactive wedge. Pelc (905) has developed a stripping film technique of autoradiography. Smith and coworkers (1112) have used nuclear emulsions in the assay of polonium and uranium. Skillern (1099) uses film to measure the beta activity of fission particles and Domingues and coworkers (269) describe a simplified method for quantitative autoradiography. Meulemans (771) has made a study of the development of nuclear emulsions while Goldsack and Van der Raay (396) have designed an automatic scanner for nuclear emulsions. LaPorte (659,660) reports further development and application in "kinetic-nucleography".

D. Absolute Counting.

The majority of analyses made with radioisotopes compare the unknown with a standard of the same material, thereby eliminating problems of geometry, scattering, absorption, mounting, etc. When this is not possible, one must resort to absolute counting. Grimeland (423) uses a scintillation counter for absolute measurements of both gamma rays and beta particles while Gabbard and coworkers (361) discuss the absolute beta measurement of mixed nuclides. Pate and Yaffe (894,895) have continued their series on aspects of 4 pi counting. The Bureau of Standards group (742,1110) has written two articles discussing the efficiency of 4 pi scintillation counting and problems of dead time and coincidence. Steyn (1143) compares the liquid scintillation counter with other methods for the absolute standardization of beta-emitting isotopes. Several other authors (24,443,919) apply 4 pi counting techniques to

specific assay problems.

Standard end-window Geiger tubes and other regular counting equipment can be standardized for "absolute assay". Assays of this sort have been reported by several groups (161, (419,424,968,1228). Leistner (687) gives a mathematical derivation for the absolute efficiency of a counting tube. The coincidence method of absolute counting has been used by Vuccino (1224) for calibration of radiation sources, by Putman (955) for measuring beta-gamma emitters, and by Lyon and Reynolds (716) for flow counter calibrations.

E. Standards

Several excellent survey articles on this subject have appeared during the last two years. Mann (741) discusses the preparation and maintenance of radioactivity standards for national and international comparison and Seliger (1065) describes some applications of these standards. In his book Weiss (1244) treats the preparation and calibration of a number of radioactive standards. Aglintsev (6) describes the standardization of radioactive preparations and Allen (18) describes the standardization of electron-capture isotopes. The status of international comparisons of radioactive standards is discussed in two articles (41,202). A private firm, Nuclear-Chicago is now taking over from the National Bureau of Standards the preparation and distribution of radioactive standards in the United States (200). Comparisons of national radium standards (706), and neutron source calibration procedures (255,256) and services (865), have been described.

F. Measurement Techniques

Many measurement techniques have been reported as short notes in techniques journals such as "Nucleonics", "International Journal of Applied Radiation and Isotopes", and others. Since in most cases these notes are also abstracted in "Nuclear Science Abstracts" only those of particular interest will be reported below.

1. Geiger and Proportional Counters. Many articles still appear about Geiger counters in spite of the fact that much of the precision detection has shifted to proportional and scintillation counters. Softky and Nather (1119) describe a low-background Geiger counter for solid beta-emitting materials. Weiner and Peterson (1241) use a Geiger well counter for large volumes of active material. Ward and Krumbein (1235) have written a long article on the characteristics of chlorine-quenched Geiger tubes while Reddie and Roesch (967) discuss ways to determine Geiger tube age, and Weaver and Morgan (1239) consider potential sources of error in beta counting with Geiger tubes. A review of the literature on Geiger counters has been published (758).

Houtermans and Oeschger (505) describe a proportional counter for measuring soft beta rays while Lang (652) has published notes on the design and performance of X-ray proportional counters. Hendee and coworkers (472) discuss gas flow proportional counters for soft X-ray detection while another group (788) describes the operation of proportional counters at high temperatures.

2. Counting Corrections. Many factors can influence the counting rate of a detector. The self absorption and window absorption corrections in 2 pi beta proportional counters for certain fission products have been discussed by Cuninghame and coworkers (237,238). Siba (1090) considers effects of various geometrical factors on gamma ray counting and Chappell (191) published a monograph showing gamma-ray scattering from thin scatterers. deBenedetti (253) discusses the analysis of thick samples of alpha emitting polonium. Measurements of the deadtime of a counting system have been made by Peacock (899) with 2.57-minute barium-137, by Myers (814) with a double source method, and by Mann and Seliger (742) for scintillation systems.

Hanson (449) records decay curves for 23 different fission products. Ellis (294) shows how the log-log scale of a slide rule can be used for decay calculations. Two authors (348,918) discuss the resolution of complex decay curves while another (492,493) reports on the determination of radioelements by the distribution of intervals between successive disintegrations. Toballem (1187) discusses the accurate measurement of radioactive periods while Zilversmit and Orvis (1278) describe methods for calculating average counting rates. Shaw (1080) gives a good treatment of the statistics of nuclear measurements.

3. Absorption Measurements. Duncan and Thomas (277) compare three absorption measurements for beta energy determination while Barreira and Laranjeira (71) present a graphical absolute method for range determination of beta particles. Two groups of

authors (915,1225) describe the use of beta absorption curves for determination of the energy of constituents of a sample. Seliger (1066) has described his experiments on transmission of positrons and electrons.

4. Low Level Counting. In a review article Anderson and Hayes (26) summarize low level counting techniques used up to December 1955 while Johnston (557) has made a somewhat similar review up to 1956. Anderson and coworkers (27) summarize their measurements of the radioactivity of food and people. Setter and Goldin (1071) have measured low level radioactivity in water. Grummitt and coworkers (426) report recent developments in low background Geiger counters while Volchok and Kulp (1221) have developed a low-level beta counter for routine radiochemical assay. Hughes (517) discusses reduction of the counting time in low activity assays while two other articles (1179,1196) make suggestions for the selection of the best counter for low level measurements.

5. Solution Counting. Albert and coworkers (10) have used a plastic coil to simplify counting of radioactive liquids. Walton and Thom (1233) used an immersion counter for beta and gamma assay in solution while Mandleberg and Henderson (740) designed an electroscope for this purpose. Another article (30) gives a formula for calculating the counting rate of a detector immersed in an extended volume of liquid.

6. Chromatography and Automation. Bangham (57,58) has used a collimated windowless Geiger counter for scanning chromatograms. Fuller (356) and Lowenstein and Cohen (710)

have also used this type of instrument, while Steenberg and Benson (1133) have used a scintillation counter for soft-beta paper chromatograms.

Aronoff (37) describes a two dimensional scanner for radiochromatograms. Müller (812) describes an isodose recorder which has been designed for a medical problem but which may have applications in analysis. Other automatic scanning devices include a scintillator grid for localizing gamma emitters (586), a scanner for carbon-14 labeled materials (988), and a device for quantitative estimate of protein spots from radio-electrophoresis (1138). Van der Does de Bye (1210) discusses the scintiscanning problem. Gilbert and Keene (380) have set up an array of Geiger counters for measuring chromatograms.

Rydberg (1022) and Rankin and Harvey (964) describe automatic sample changers for radiometric work. A system for counting thousands of samples in 12 hours automatically (973), an inexpensive rapid automatic recorder (980) and other automatic devices (194) are also reported. Gevantman and coworkers (374) have developed a device for regulating small flow rates in chromatographic columns while Ginsberg⁽³⁸²⁾ describes a simple large volume fraction collector for column chromatography. Rossi (996) writes about the measurement of radio-paper chromatograms and Blake (98) describes a chromatograph monitor which can stop the progress of chromatographic formation automatically at any stage. Preuss (950) describes a humidity and temperature control unit for a counting room.

G. Chemical Techniques and Effects

The presence of radioactivity in a solution is a handicap rather than an asset to a certain group of analysts. Kelley has written a good summary of this problem (587). Several authors have discussed the problems of spectrochemical analysis of radioactive solutions (93,94,122,434,523). Two papers describe apparatus for flame photometric determinations of radioactive materials (285,500) while another paper (681) discusses pyrohydrolytic separation and spectrophotometric titration of fluorides in radioactive samples. Others (600,1242) have used radioactivity in a study of electrode surfaces.

Decontamination of equipment that has been used with radioisotopes is an important facet of the field and Huff (512,514) has discussed the effectiveness of different decontaminants for various materials. Huff (513) and Bennett (81) also give electrodecontamination procedures for metals while Bennett (82) describes some absorption and desorption experiments on stainless steel surfaces. The problem of analysis of massive amounts of platinum used for alpha sample plates for tracer amounts of radioactivity has also been discussed (241).

There is a large area having to do with the effects of radiation on all types of materials that has not been mentioned in this report. "Nucleonics" (847) published a long summary article on this problem. Hammond (447) describes the problems of chemical processing in intense radiation fields. Harteck and Dondes (453) describe experiments used in producing chemicals with reactor radiations, methods which might eventually have some kind of analytical significance. Horne, Coryell and

Goldring (502) give an excellent discussion of generalized acidity in radiochemical separations.

H. Hot Lab Design and Techniques.

Many articles have appeared in the literature on various aspects of this subject. Reports summarizing the annual Hot Lab Symposia held in 1955 (874,875) and in 1957 (279) are available. Several published papers (283,503, 896,1073) have discussed the problems of remote automatic analysis in highly radioactive solutions. Several Geneva papers (363,393, 1124,1268) also describe hot lab facilities and philosophies. Millett (781) has compiled a bibliography of articles published between 1946 and 1956 on laboratory design. Two other recent articles (760,1120) also discuss this problem.

Two papers report gamma-ray attenuation and shielding calculations (190,1014) while others describe a dispenser for radioactive solutions (963), a microdistillation apparatus for radioactivity procedures (106) and simple remote tongs (949).

V. SAFETY

The utility and application of all the techniques described above are predicated upon their safe use. Millett (781) has published a selective bibliography on radiological protection during the past 10 years and Parker (888) has given an excellent 10-year review of radiation protection in the atomic energy industry. Morgan (795) gives an extensive review of the permissible internal dose of radionuclides and Taylor (1175) reviews the current situation with regard to permissible radiation exposure levels.

The Bureau of Standards has issued three more of its series of handbooks. One is on X-ray protection (816) one on radiological units and measurements (818), and the third on legislative regulation of radiation exposure (817), which is also treated by Taylor (1174). To date most of the control of radiation has been on the national level, but certain states, taking the government recommendations as their guide, are beginning to set up their own atomic codes (196). Novak (830) has written a radiation safety guide discussing some of the problems and precautions involved in working with radioisotopes.

Radioactive fallout has evinced considerable discussion during the past two years and Libby (700) and Dunning (278) report on analyses involved in this problem. Libby (699) puts the fallout problem in its proper scientific perspective by comparing it with dosages from natural radioactivity and cosmic rays.

An article issued in 1956 describes disposal of radioactive wastes in the U. S. Atomic Energy Program (1261).

Several continuously recording instruments which have been developed for radiological survey work might be applicable to analysis. Three articles discuss instruments for monitoring low level radioactivity in liquid streams (526,572,1255).

Other articles describe air monitoring procedures (32), tritium survey instruments (274,304,1078,1276) and an iodine-131 scintillation monitor (1109).

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