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 SUBJECT: Abstract Summary and Subject Index of
 Preliminary Reports, Analytical Chemistry
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TO: See Distribution Below

FROM: M. T. Kelley

This is a compilation of brief abstracts of the Preliminary Reports on work done in the laboratories of the Analytical Chemistry Division at the X-10 Site during the months of January through June, 1956. A subject index of these reports is also included.

The opinions and conclusions given in these reports are tentative and are subject to revision at any time prior to their publication in more formal reports. These Preliminary Reports are primarily intended as a means of increasing the communication of information among the various groups with the Analytical Chemistry Division.

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ABSTRACTS OF PRELIMINARY REPORTS

ANALYTICAL CHEMISTRY DIVISION

X-10 SITE

JANUARY - JUNE, 1956

IONIC ANALYSES RESEARCH AND DEVELOPMENT

P. F. Thomason

A. D. Horton

Sodium Azide as a Reductant for Excess Ceric Ion Used to Oxidize Chromium(III to VI) and Colorimetric Determination of Chromium(VI) by Diphenylcarbazide (ADH-2)

Sodium azide has been used successfully to reduce excess ceric ion that was used to oxidize chromium(III) to chromium(VI) prior to the colorimetric determination of chromium(VI) by diphenylcarbazide. Uranium does not interfere, and this procedure can probably be used in the coulometric titration of chromium(VI) with electrically generated ferrous ion.

A Sampling Manifold for Uranium Hexafluoride and Fluorine Gases (ADH-3a)

A sampling manifold for fluorine and uranium hexafluoride gases has been completed and is being tested for leaks under vacuum. Methods are presented for the sampling and analysis of fluorine that are in accord with accepted methods of the K-25 Works Laboratory. Uranium hexafluoride flow will be measured by a thermoelectric flowmeter, and the gas will be collected in a cold trap. Analysis of uranium hexafluoride will be made by methods presently in use.

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H. Kubota

Determination of Free Acid (HK-1)

The method of conductance measurement was evaluated for determining free acid in the presence of hydrolyzable ions. Synthetic solutions that contained uranyl sulfate, copper sulfate, and sulfuric acid were used. The results of the study indicated that empirical standard curves can be prepared from which free acid determinations sensitive to small changes in acid concentration can be made rapidly.

F. J. Miller

Sulfate Determination (FJM-1)

A method was developed for the automatic photometric titration of sulfate. It has been used successfully by the Reactor Analyses Group for the remotely controlled titration of as little as 0.5 mg of sulfate in radioactive samples with a relative standard deviation of less than 1%.

Anodic Dissolution of S.S. Sandwich of UO₂ (FJM-2)

A method was developed for the anodic dissolution of stainless steel sandwiches of uranium dioxide and for the separation of the uranium by the electrodeposition of other constituents of the sample into a Melaven mercury cathode cell. The method is especially suitable for "hot cell" work.

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Anodic Dissolution of Hg-Ti Alloys (FJM-3)

A satisfactory procedure was developed for the anodic dissolution of mercury-titanium alloy in 10% sulfuric acid solution.

Conductometric Titration of Sodium Acetate (FJM-4)

A conductometric method was evaluated for the titration of sodium acetate at a concentration of approximately 2×10^{-4} M in the presence of 1 M sodium sulfate. The method was satisfactory for synthetic samples but not for the actual samples of interest.

Conductometric Measurements on ThO₂ Slurries (FJM-5)

Conductance measurements were made on thorium oxide slurries. The results were used as an indication of the mode of preparation and the temperature of calcination of the thorium oxide.

P. F. Thomason

Precise Determination of Aluminum and Silver in Pure Solutions (PFT-1a)

Aluminum and silver were determined in solutions made from reagent grade chemicals with a relative standard error of $\pm 0.3\%$ and $\pm 0.8\%$, respectively. The gravimetric 8-hydroxyquinoline method was used to determine the aluminum, and the automatic potentiometric titration was used to determine the silver.

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ANALYTICAL INSTRUMENTATION

D. J. Fisher

L. C. Bate

Motorized Arc Stand (LCB-3)

An Iowa State College model arc stand was motorized for remotely controlled use by the Spectrographic Analysis Group in the High-Radiation-Level Analytical Facility. It was raised upon legs, and the motors to the various drive shafts were located under the arc stand. A control box having interlocked circuits was designed and provided for the stand. Tantalum inserts were fabricated for the electrode holders.

The 1-ml, Plastic, Remotely Controlled Dilution Pipetter (LCB-4)

The precision of delivery of the 1-ml, plastic, remotely controlled pipetter has been determined. All parts exposed to samples were fabricated from fluorothene or Teflon.

H. C. Jones

High-Sensitivity, Recording, Single-Beam Flame Spectrophotometer (HCJ-1a)

A single-beam flame spectrophotometer was designed for operation with the 16PML, 6217, 1P21, 1P22, and 1P28 multiplier phototubes. The instrument was designed to operate in the same manner as the ORNL model Q-1457 flame photometer when the latter is operated as a single-beam instrument. The range of the voltage supply has been extended to include all values of voltage from 600 to 2000 volts.

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RADIOCHEMICAL RESEARCH AND DEVELOPMENT

S. A. Reynolds

T. H. Handley

Investigation of Cr-55 (THH-1b:SAR-1b)

An investigation of the radiochemical properties of Cr-55 has been completed. Chromium-55 was found to have $< 0.01\%$ gammas associated with its decay.

Radiochemistry of Thorium Series (THH-3b)

Thorium-228 was determined in thorium oxide from the Thorex process by the measurement of Ra^{224} , a daughter of Th^{228} .

Radiochemical Analysis of Special Cyclotron Targets (THH-8c)

Radiochemical methods were developed for N^{13} , Mg^{27} , and Si^{31} activities to be separated from targets bombarded by N particles in the 63-in. cyclotron. A large number of analyses have been performed by use of these procedures.

Study of Induced Radioactivities by Pile Irradiation of Metal Samples Submitted by Lockheed (THH-9)

Metal filings of structural materials for airplanes were irradiated in the X-10 Graphite Reactor. The relative amounts of gamma activities induced into these materials were measured at successive decay times.

Assay Methods for New Radioisotope Products: Mo-99 (WSL-10:THH-10)

See under W. S. Lyon below.

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W. S. Lyon

Determination of Thermal Neutron Activation Cross Sections for a
Number of Elements (WSL-6b)

Data have been obtained on the pile neutron cross sections for (n,γ) reactions on the following target elements: Mg²⁶, K⁴¹, Cr⁵⁰, Ni⁶⁴, Cu⁶³, Zn⁶⁴, Zn⁶⁸, Ga⁷¹, As⁷⁵, Br⁸¹, Rb⁸⁵, Zr⁹⁴, Mo⁹⁸, Pd¹⁰⁸, Ag¹⁰⁹, Cs¹³³, Ba¹³⁰, La¹³⁹, Ta¹⁸¹, W¹⁸⁴, Pt¹⁹⁶.

Assay Methods for New Radioisotope Products: Pd-109 (WSL-8)

Palladium-109 has been examined qualitatively and quantitatively for impurities. The beta absorption extrapolation has been found to be satisfactory for assay of this isotope.

Assay Methods for New Radioisotope Products: Au-199 (WSL-9)

Ion chamber and scintillation counter factors have been determined for the assay of Au-199.

Assay Methods for New Radioisotope Products: Mo-99 (WSL-10:THH-10)

Molybdenum-99 was found to be greater than 99% pure at discharge time. A factor for the scintillation counter was obtained by the beta-gamma coincidence counting of an aliquot of Mo-99 freed from Tc-99.

Assay Methods for New Radioisotope Products: Br-82 (WSL-11)

Bromine-82 was found to be free from contaminants other than 4.6-h. Br-80. The assay may be done by ionization chamber measurement

S. A. Reynolds

Investigation of Cr-55 (THH-1b:SAR-1b)

See under T. H. Handley above.

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ACTIVATION ANALYSES RESEARCH AND DEVELOPMENT

G. W. Leddicotte

W. A. Brooksbank

Activation Analysis: Determination of Selenium, Tellurium, and Sulfur in an Indium-Arsenic Alloy (WAB-21)

Selenium, tellurium, and sulfur have been determined in indium-arsenic by activation analysis. The tellurium content was 43.2 ± 0.2 $\mu\text{g/g}$, and the selenium content was 104.0 ± 7.0 $\mu\text{g/g}$. The sulfur results showed poor precision, possible because of the method of sample preparation.

Activation Analysis: Determination of Aluminum in High-Purity Silicon (WAB-22:RED-1)

Microgram quantities of aluminum in silicon can not be determined by means of thermal neutron activation. Aluminum-28 activity comes predominantly from the (n,p) reaction on silicon and masks any Al^{28} formed by the thermal neutron capture on stable Al^{27} . The gamma energy of Al^{28} was found to be 1.75 ± 0.03 Mev.

Nuclear Analyses Methods: Determination of Boron in Aqueous and Organic Solutions (WAB-23)

Boron can be determined by nuclear analyses methods that utilize neutron density measurements. It can be determined directly in the concentration range 0.05 to 3.00 mg/ml.

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Activation Analysis: Determination of Impurities in "Fabrifax"
(WAB-24)

Neutron activation analysis was used to identify impurities in a sample of aluminum silicate fiber ("Fabrifax"). The radioisotopes identified indicated the presence of manganese, iron, copper, molybdenum, chromium, hafnium, tantalum, phosphorus, and probably sodium.

Activation Analysis: The Use of Discriminatory Gamma Counting for the Determination of Fe⁵⁹ Separated from Zirconium Metal Samples (WAB-25)

A method is given for the counting of Fe⁵⁹ in the presence of as much as 10⁵ c/m of Zr⁹⁵-Nb⁹⁵.

Activation Analysis: Use of Discriminatory Gamma Counting for the Measurement of Cobalt-60 in the Presence of Cobalt-58 (WAB-26)

A method is given for the measurement of Co-60 in the presence of as much as 40000 c/m of Co-58. Counter calibration and conditions are listed.

Activation Analysis: Determination of Uranium in Inconel Flakes (WAB-27)

Enriched U²³⁵ was determined in a small sample of Inconel flakes. The method, calculations, and results are given briefly.

Activation Analysis: Determination of Microgram Quantities of Scandium in Beryllium (WAB-28)

Scandium was determined in beryllium by a direct gamma spectrometric measurement after a neutron irradiation. The scandium content was 9.7 ± 0.5 µg/g..

Activation Analysis: Determination of Tin in Solutions of Zirconium-Tin Alloy (WAB-29)

Tin was determined by neutron activation analysis in four solutions of Zr-Sn alloys. Counting conditions are given in detail. The limit of measurement for the method was 0.001 mg/ml.

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Activation Analysis: Detection of Impurities of Platinized Petroleum Catalysts (WAB-30)

Neutron activation analysis was used to identify impurities in six samples of platinized petroleum catalysts. The radioisotopes identified indicated the presence of manganese, zinc, copper, sodium, phosphorus, tungsten, hafnium, strontium, gold, mercury, calcium, molybdenum, chromium, and probably antimony, arsenic, indium, protactinium, zirconium, iron, silver, and bromine.

Activation Analysis: Detection of Trace Elements in Malignant Tissue (WAB-31)

Neutron activation analysis was used to identify trace elements in a sample of malignant tissue. The radioisotopes identified indicated the presence of phosphorus, sodium, copper, bromine, manganese, iron, zinc, cobalt, and probably nickel and chromium.

Activation Analysis: Detection of Trace Elements in Transistor-Grade Silicon (WAB-32)

Neutron activation analysis showed that the trace elements present in a particular transistor-grade silicon were phosphorus, sodium, arsenic, chromium, copper, manganese, and antimony.

Activation Analysis: Detection and Quantitative Determination of Trace Elements in Tin (WAB-33)

Qualitative analysis of a sample of tin by neutron activation analysis disclosed arsenic, antimony, copper, phosphorus, and potassium as the major contaminants. Iron and sodium were probably present. Quantitative analysis gave 0.0479 ± 0.0009 mg of antimony and 0.061 ± 0.002 mg of arsenic, per gram of tin.

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Activation Analysis: Determination of Samarium in Cerium (WAB-34)

Samarium was determined in a sample of cerium by neutron activation analysis. The results of spectrochemical and activation analyses were in agreement. Lanthanum and dysprosium were identified as being present in the sample.

Activation Analysis: Determination of Sulfur and Phosphorus in Transistor-Grade Silicon and in Chromium (WAB-35)

Sulfur and phosphorus have been determined in transistor-grade silicon and in several specimens of chromium. Phosphorus can be determined without difficulty. The sulfur determination is subject to counting errors because of the presence of the weak beta (0.17-Mev) radiation.

Activation Analysis: Detection of Impurities in $Y(NO_3)_3$ and Determination of Yttrium (WAB-36)

The activation analysis of a sample of yttrium nitrate indicated that it contained copper, manganese, dysprosium, sodium, and probably lanthanum and bromine. The yttrium content was found to be 0.311 ± 0.005 g per gram of sample, corresponding to between 0 to 1 mole of water of hydration.

R. E. Druschel

Activation Analysis: Determination of Aluminum in High-Purity Silicon (WAB-22:RED-1)

See under W. A. Brooksbank above.

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W. T. Mullins

Activation Analysis: Determination of Silicon in Niobium (WIM-3b)

The determination of silicon in niobium by activation analysis was undertaken using the slow neutron-capture reaction, $\text{Si}^{30}(\text{n}, \gamma)\text{Si}^{31}$, $t_{1/2} = 2.7$ hours. A radiochemical separation by means of silicon carrier was utilized, and a silicon comparator sample was irradiated along with the unknown sample. The limit of measurement of silicon under the irradiation and processing conditions used was 5 μg per gram of niobium.

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ANALYTICAL CHEMISTRY DIVISION

X-10 SITE

JANUARY - JUNE, 1956

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