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The abstracts of the Preliminary Reports that were issued from the Analytical Chemistry Division, Department 4270, during May, 1956, are presented in this report. A subject index is also included in this compilation.

Any opinions or conclusions, which are expressed in these abstracts, are tentative and subject to revision or correction before they are issued in more formal reports. Comments, suggestions, and criticisms of this material are solicited.

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MAY, 1956

R. G. BallElectrodeposition Method for the Determination of Copper by Internal Electrolysis, RGB-33 and 34

RGB-33: In a continuation of the study to improve the method for the deposition of copper by internal electrolysis, an alundum thimble was inserted in the electrolysis cell to separate the analyte and catholyte compartments. The use of this thimble was found to increase the current flow through the cell and to lessen the time that is required for the deposition of copper. A period of 30 minutes was found to be sufficient for the quantitative recovery of up to 15 mg of copper.

RGB-34: The majority of samples, which will be analyzed by this electrodeposition method for copper, will be solutions of uranyl sulfate. It was essential, therefore, to determine if any diffusion of uranium and nickel through the alundum diaphragm into the anode compartment occurs during the period of electrolysis. When 50-ml samples of uranyl sulfate were electrolyzed, a quantitative recovery of 1.5 to 36 mg of copper was achieved in a 30-minute period. Subsequent determination of the uranium, 100 mg, and the nickel, 0.1 to 10 mg, in these samples after the electrodeposition of copper revealed that no trend toward loss of these constituents is evident during electrolysis. This indicates that any diffusion of uranium and nickel into the alundum thimble is negligible over the period that is required for the deposition of copper.

C. M. BoydStability of the Light Source and Photo-Electric Cell of the Wagner Turbidimeter, CMB-44

The stability of the light source and the response of the photo-electric cell of a Wagner turbidimeter was determined since it is believed that any fluctuations of the light intensity will noticeably affect the determination of the particle-size distribution of thorium oxide, which is made by means of this instrument. A slight instability in the measured light intensity is noted when the turbidimeter is operated under the same conditions as those which are used in the actual determination of the particle-size distribution of thorium oxide. This instability appears to be due to variation in the

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photo-electric cell fatigue, and is sufficient to cause an error of 5 to 10 per cent in estimating the amount of thorium oxide that is present as particles with diameters less than values between 0.5 and 10 microns.

Determination of Particle-Size Distribution of Thorium Oxide, CMB-45

In order to determine any changes which occur as a result of circulating thorium oxide, as an aqueous slurry, in a dynamic test loop, the particle-size distribution of the oxide was determined by the Andreasen sedimentation method. Xylene that contained 0.06 per cent oleic acid was used as the suspending medium. The test results indicate that a gradual decrease in the particle-size of thorium oxide occurs during the 20 days of circulation with the major part of this degradation occurring within the first day. The degradation that occurred during the circulation was such that the amount of oxide that was composed of particles between one and five microns in diameter was diminished from 90 to 20 per cent, while the amount which was less than 0.5 microns in diameter increased from 3 to 43 per cent. In a comparison of these results with those which were obtained by the Wagner turbidimetric method, complete agreement of the results was not realized for all of the samples; however, in general, the same trend in the changes of particle-size distribution was indicated by the two methods.

R. E. Feathers, Jr.

Determination of Water in Gases by Measurement of the Dew Point, REF-1

In an effort to determine the amount of water in gases, a correlation between the concentration of water in helium and the dew point, as measured with an Alnor Dew Point Meter, was established. Values for the concentration of water in ppm by volume and ppm by weight in helium were plotted as a function of dew point temperature. It was found that the concentration of water in gases can be determined in this manner with a precision of approximately two per cent.

G. Goldberg

Determination of the Moisture Content of Gases by Measurement of the Dew Point, GG-70

Since it was established that the use of Alnor Dew Pointer is applicable to the determination of water in gases by measurement of the dew point, tests were made to compare the advantage of using either Tygon or copper tubing as gas transfer lines to the instrument. It was found that it is necessary to purge Tygon tubing at least 15 minutes before reliable results can be obtained. Reproducible measurements are achieved immediately when a copper transfer line is used.

UNCLASSIFIEDExamination of Cold Trap from Heat Exchanger, GG-69 and 71

GG-69: An examination was made of the contents of a cold trap which was removed from Intermediate Heat Exchanger Test Stand B. Residual NaK was drained from the trap, following which the trap was cut in half and washed thoroughly with water. The drainings from the trap were analyzed for sodium, potassium, oxygen, uranium, zirconium, nickel, iron, and chromium.

GG-71: A cold trap from Small Heat Exchanger Test Stand B was drained to remove the residual NaK. After the trap was washed, the drainings were analyzed for sodium, potassium, oxygen, iron, nickel, and chromium.

G. GoldsteinDetermination of Free Acid in Solutions of Uranyl Sulfate, GGs-1 and 2

GGs-1: In an attempt to develop a method for the determination of the free acid in solutions of uranyl sulfate, the applicability of a Sargeant-Malmstadt Automatic Titrator for this purpose was evaluated. Attempts to titrate 50 to 70 ml of 0.002 to 0.01 N sulfuric acid in solutions of uranyl sulfate which contained 50 to 100 mg of uranium with this instrument were not successful, since the termination of the titration did not occur at the end point which corresponds to the neutralization of free acid (pH 3.5 to 4.0). The titration, however, was automatically terminated at the end point which corresponds to the neutralization of the free acid plus the complete hydrolysis of the uranium (pH 9).

GGs-2: In a further effort to adapt the Sargent-Malmstadt Automatic Titrator to the determination of free acid in solutions of uranyl sulfate, a sensitivity control was installed on the instrument. At an increased sensitivity setting, it was found that 0.1 to 0.5 meq of sulfuric acid can be automatically titrated in the presence of as much as 50 mg of uranium. The increased sensitivity adjustment is not necessary when sulfuric acid alone is titrated with sodium hydroxide.

C. A. HortonDetermination of Trace Amounts of Molybdenum, CAH-4 and 5

CAH-4: In order to determine the effect of certain substances on the determination of trace amounts of molybdenum by the thiocyanate, colorimetric method, different concentrations of molybdenum, iron, uranium, aluminum, and vanadium were extracted with n-butyl acetate. It was found that vanadium, in amounts equal to and greater than molybdenum, interferes seriously with the thiocyanate method for the determination of molybdenum, while iron, uranium, and aluminum do not interfere when present in

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concentrations up to 5 mg per ml of the test solution. The presence of either citrate or oxalate does not prevent the extraction of vanadium with n-butyl acetate.

CAH-5: Since the presence of vanadium leads to interference in the determination of molybdenum by the thiocyanate method, attempts were made to separate vanadium from molybdenum prior to the estimation of the latter element. The proposed method of separation depends on the precipitation of the interferences and iron as hydroxides after ferrous iron is added to reduce vanadium. Apparently, vanadium cannot be separated in this manner, inasmuch as considerable interference was still noticed in the determination of molybdenum.

J. R. Lund

Determination of Fluoride by a Flame Photometric Method, JRL-28 and 29

JRL-28: The effect of mercurous chloride as a carrier in the precipitation of calcium fluoride and the subsequent indirect determination of fluoride was evaluated. Under certain conditions, it was found that, in the absence of fluoride, calcium does not co-precipitate with mercurous chloride. When the same experimental conditions were applied, however, in the presence of fluoride, no improvement was achieved in the estimation of fluoride by a flame photometric method for the determination of calcium. It was concluded that mercurous chloride is not suitable as a carrier for the precipitation of calcium fluoride under the conditions of this experimental work.

JRL-29: Since all efforts to estimate fluoride by methods based on the evaluation of excess calcium that remains in the filtrate after a precipitation of calcium fluoride were unsuccessful, an attempt was made to substitute barium for calcium in subsequent experiments with isopropyl alcohol as the solvent. The calculated recovery of fluoride was neither constant nor quantitative in a series of tests in which a standard alcoholic solution of fluoride was taken, or in another series in which an aqueous solution of fluoride was used for the standard; however, after a correction factor is applied, the recovery values are either constant, for the alcoholic standards of fluoride, or both constant and quantitative, for the aqueous standards.

E. C. Lynn

Analysis of Tap Water, Building 9213, ECL-6 and CDS-7

Partial analyses were made on two samples of water, both of which were reported to have been taken from different faucets in Building 9213. Both samples were analyzed for total solid matter, iron, calcium, magnesium, sodium, and potassium. The methods of analysis were in essential compliance with those given in ASTM D1256-53T.

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R. L. McCutchen

Fluorometric Determination of Uranium, RLM-26 and 27

RLM-26: A series of experiments was made to determine the validity of the hypothesis that the reproducibility of the calibration data for a newly prepared batch of flux improves with time. It was found that the reproducibility of the calibration data is not dependent on the age of the flux.

RLM-27: In order to determine if the calibration factors for the 10- and 100-scales of the Photovolt fluorophotometer are significantly different, a series of tests was made to compare the factors. Initially, a significant difference in the factors for the two scales was observed. After the instrument was re-adjusted and the scales were re-aligned, however, the factors for the two scales were found to be essentially the same.

B. L. McDowell

Separation of Rare Earths from Stainless Steel and Inconel, BLM-53

A procedure was developed for the concentration and separation of trace amounts of rare earths in several types of stainless steel and inconel. After separation, the individual rare earths were determined by spectrographic procedures. The recovery of these elements by this method of separation and preparation for analysis is quantitative within the precision of the spectrographic procedure.

Separation of Cerium from Samples of Cast Iron, BLM-54

The procedure for the concentration of rare earths with a yttrium oxide carrier prior to the spectrographic determination of the individual elements was applied to the determination of cerium in cast iron. The results for the cerium content of several samples of cast iron agree essentially with the results from a wet analysis of cerium which were furnished with the samples.

W. J. Ross

Determination of Boron in Fluoride Salts, WJR-92

A further investigation of the effect of acid concentration on the extraction of boron with ether from solutions of $\text{HCl-AlCl}_3\text{-C}_2\text{H}_5\text{OH}$ was made. It was established that the value for the partition coefficient of boron diminishes as the concentration of HCl is increased from 0 to 4 M. The value remains essentially constant when 4 M up to 6 M HCl is the medium of extraction.

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I. B. Rubin

Dissolution of the Acid-Insoluble Residue of Mixtures of Thorium Oxide-Aluminum Silicate, IBR-47

Several methods were studied for the dissolution of thorium oxide which contains aluminum silicate as mullite. It was found that essentially complete dissolution is achieved by dissolving the sample in nitric acid with a small amount of hydrofluoric acid, following which the acid-insoluble residue is fused with a sodium carbonate-sodium tetraborate flux. This fused mixture can be dissolved in hydrochloric acid. After dissolution, the aluminum content of the samples can be determined by the 8-quinolinol, spectrophotometric method. Results by this method were in agreement with those found by an ASTM gravimetric method.

The Effect of Tiron as a Complexing Agent on the Determination of Aluminum, IBR-48 and 49

IBR-48: The use of disodium-1,2-dihydroxybenzene-3, 5-disulfonate (Tiron) was studied as a complexing agent for iron in the determination of aluminum by the 8-quinolinol method. It was established that Tiron apparently prevents the formation and extraction of the aluminum-8-quinolinol complex into chloroform; but, it does not affect the formation and subsequent extraction of the iron-8-quinolinol complex under the same conditions.

IBR-49: Since the presence of Tiron appeared to prohibit the formation and extraction of aluminum as the 8-quinolinol complex, further tests were made to establish the conditions under which this extraction is prevented. The tests that were made revealed that less than one per cent of aluminum is recovered from solutions which contain an excess of Tiron when the solutions are equilibrated for three minutes, although 95 per cent of the aluminum is extracted under the same conditions if Tiron is absent. The extraction time was found to be critical, since 25 per cent of aluminum is extracted, in the presence of Tiron, if the samples are extracted for a period of nine minutes.

C. D. Susano, See E. C. Lynn

Analysis of Tap Water, Building 9213, ECL-6 and CDS-7

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