RAPID ANALYSIS of NONFERROUS METALS AND ALLOYS

by George Norwitz

1958

CHEMICAL PUBLISHING CO., INC.
212 FIFTH AVENUE NEW YORK, N. Y.

Rapid Analysis of Nonferrous Metals and Alloys

ISBN: 978-0-8206-0100-7

Chemical Publishing Company:
www.chemical-publishing.com
www.chemicalpublishing.net

First Edition:

© **Chemical Publishing Company, Inc. -** New York 1958

Second Impression:

Chemical Publishing Company, Inc. - 2011

Printed in the United States of America

PREFACE

This book describes rapid methods for the analysis of nonferrous metals and alloys. Speed, however, has not been overemphasized. In addition to speed the methods chosen have the following characteristics: accuracy, simplicity, and reliability in the hands of routine operators.

The book differs from previously published books on metal analysis in the following respects: (1) There is an increased reliance on sequence procedures, (2) mathematical correction factors are advantageously used to eliminate lengthy separations, (3) colorimetric procedures are stressed, (4) increased use is made of perchloric acid, especially in destroying organic matter.

A sequence procedure as the name implies, is a method of analysis in which several determinations are made on the same sample. Such a technique has several advantages. It is obviously rapid since it eliminates the necessity of weighing and dissolving several samples. It saves glassware, chemicals, and laboratory space. Laboratories using sequence procedures are invariably neater than those which do not use them. According to statistical studies in laboratories running large numbers of samples, the results obtained by routine operators will be more accurate when sequence procedures are used. The explanation of this is that the use of sequence procedures encourages careful manipulation and reduces tendencies toward carelessness.

The use of mathematical correction factors accurately compensates for interferences. For instance, in the colorimetric determination of manganese, 10% chromium will give a reading equivalent to 1.2% manganese. Therefore, when a sample containing chromium is analyzed for manganese it is a simple matter to make a proportionate correction.

The use of colorimetric methods is almost essential in rapid metal analysis. For a combination of speed and accuracy, colorimetric methods have much to recommend them.

Perchloric acid has become an almost indispensable reagent in metal analysis. It is especially useful in destroying filter paper, cupferron, and tartaric acid. The destruction of such organic substances by the use of a mixture of perchloric and nitric acids is safe. The author has been present on hundreds of occasions when a mixture of these two acids was used to destroy organic matter, but not once did he observe a violent reaction, let alone an explosion.

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Chapter 1. *SPECIAL REAGENTS*

Reagent No. 1 — Acetate Buffer Solution (for Colorimetric Copper Determination)

Dissolve 500 g. of ammonium acetate in a mixture of 200 ml. of glacial acetic acid and 400 ml. of water. Dilute to 1 liter.

Reagent No. 2 — Aluminon Solution

A 10% benzoic acid solution, a buffer solution, and a 1% gelatin solution are required for the preparation of this reagent. To prepare the 10% benzoic acid solution, dissolve 50 g. of benzoic acid in 500 ml. of methanol. For the buffer solution, mix 470 ml. of ammonium hydroxide with 430 ml. of glacial acetic acid. Cool to room temperature and add more acid or base as necessary to adjust the *p*H at 5.25 to 5.35 when diluted 1 to 20. Dilute to 1 liter. To prepare the 1% gelatin solution, dissolve 3 g. of U.S.P. gelatin by adding hot water and stirring. Cool and dilute to 300 ml.

To prepare the aluminon reagent dissolve 0.3 g. of pure aluminon in water and add 60 ml. of the benzoic acid solution. Dilute to 300 ml. Disregard the precipitate since it will later redissolve. Add 300 ml. of the buffer solution and 300 ml. of the gelatin solution and shake. This solution should stand 3 days before using. The reagent will keep 3 months if stored in the dark.

Reagent No. 3 — Ammonium Citrate Solution

Dissolve 250 g. of citric acid in water and add 250 ml. of ammonium hydroxide. Dilute to 2.5 liters.

Reagent No. 4 — Ammonium Molybdate Solution

Dissolve 100 g. of $(NH_4)_6MoO_7O_{24}\cdot 4H_2O$ in about 900 ml. of water by warming on a steam bath at 50°C Dilute to 1 liter. If the solution is not clear, filter.

Reagent No. 5 — Ammonium Vanadate Solution

Dissolve 2.3 g. of NH_4VO_3 in hot water, add 10 ml. of nitric acid and dilute to 1 liter.

Reagent No. 6 — Antimony Trichloride Solution

Dissolve 10 g. of $SbCl_3$ in 250 ml. of hydrochloric acid and dilute to 500 ml. with water.

Reagent No. 7 — Barium Chloride-Hydrochloric Acid Solution

Add 10 ml. of 10% barium chloride solution and 10 ml. of hydrochloric acid to 1 liter of water.

Reagent No. 8 — Dimethylglyoxime Solution

Dissolve 0.8 g. of the sodium salt of dimethylglyoxime in about 200 ml. of water and add 150 ml. of ammonium hydroxide. Dilute to 2.5 liters. This solution will keep about 2 weeks.

Reagent No. 9 — Ferrous Ammonium Sulfate Solution

Dissolve 23 g. of $(NH_4)_2SO_4\cdot FeSO_4\cdot 6H_2O$ in 1 liter of sulfuric acid (1 to 10).

Reagent No. 10 — Formic Acid Mixture

Add 200 ml. of formic acid and 30 ml. of ammonium hydroxide to about 500 ml. of water. Dilute to 1 liter.

Reagent No. 11 — Formic Acid Wash Solution

Add 25 ml. of formic acid mixture (Reagent No. 10) to 1 liter of water and saturate with hydrogen sulfide.

Reagent No. 12 — Gum Arabic Solution

Add 10 g. of gum arabic (acacia) to 500 ml. of hot water while stirring. Keep on a hot plate under the boiling point for 1 hour in a covered beaker. Do not boil. Keep well stoppered and refrigerated.

Reagent No. 13 — Iodine Solution (0.1 N; for Colorimetric Nickel Determination)

Dissolve 12.7 g. of iodine and 25 g. of potassium iodide in 25 ml. of water, and dilute to 1 liter.

Reagent No. 14 — Iodine Solution (0.1 N; for Tin Determination)

Dissolve 12.7 g. of iodine and 25 g. of potassium iodide in 25 ml. of water and dilute to 1 liter. Standardize against pure metallic tin (0.3 g.). Store this solution in a glass-stoppered brown bottle in a dark place.

Reagent No. 15 — Iodine Solution (0.02 N; for Tin Determination)

Pipet 100 ml. of the standardized 0.1N iodine solution (Reagent No. 14) into a 500-ml. volumetric flask and dilute to the mark. Transfer to a dry glass-stoppered brown bottle and store in a dark place.

Reagent No. 16 — Periodate-Phosphoric Acid Solution

Dissolve 17 g. of potassium periodate in a mixture of 100 ml. of warm water and 335 ml. of 85% phosphoric acid by heating on a steam bath with occasional stirring. Dilute to 2.5 liters.

Reagent No. 17 — Potassium Bromate Solution (0.02 N)

Dry pure potassium bromate (low bromide content) at 180°C. for 1 hour. Dissolve 0.5567 g. in water and dilute to 1 liter in a volumetric flask. This is a primary standard.

Reagent No. 18 — Potassium Dichromate Solution

Dissolve 2.8284 g. of pure potassium dichromate in water and dilute to 1 liter. One milliliter of this solution contains 1.0 mg. of chromium.

Reagent No. 19 — Rubeanic Acid Solution

Dissolve 0.5 g. of rubeanic acid (dithiooxamide) in 500 ml. of 95% ethyl alcohol. The solution is stable for 2 or 3 months.

Reagent No. 20 — Stannous Chloride Solution (35%)

Dissolve 350 g. of $SnCl_2 \cdot 2H_2O$ in 500 ml. of hydrochloric acid by warming on a steam bath at 50°C. Cool, and dilute to 1 liter with hydrochloric acid.

Reagent No. 21 — Starch Solution

Make a paste of 2 g. of soluble starch in 10 ml. of water. Add this paste to 200 ml. of boiling water. Cool to room temperature. This solution should be prepared fresh weekly.

Reagent No. 22 — Succinate Solution

Dissolve 60 g. of succinic acid, 100 g. of ammonium chloride, and 100 g. of urea in about 1.5 liters of water. Dilute to 2 liters.

Reagent No. 23 — Thiourea Solution

Dissolve 5 g. of thiourea in water and dilute to 100 ml. Filter if not clear. This solution should be prepared fresh daily.

Throughout this book, when ammonium hydroxide or acids are specified, it should be understood that the following concentrations are intended:

	%
Ammonium Hydroxide	28
Hydrobromic Acid	48
Hydrochloric Acid	37.0–38.5
Hydrofluoric Acid	48
Nitric Acid	70
Perchloric Acid	70
Phosphoric Acid	85
Sulfuric Acid	97

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