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HW-54125
October 1, 1958

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CHEMICAL PROCESSING DEPARTMENT
ESSENTIAL MATERIALS MANUAL

Specifications and Acceptance, Sampling,
and Analytical Procedures

Issued by

Research and Engineering Operation

CHEMICAL PROCESSING DEPARTMENT

October 1, 1958

HANFORD ATOMIC PRODUCTS OPERATION
Richland, Washington

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SECTION I

INTRODUCTION

SECTION I

INTRODUCTION

This manual has been prepared for the purpose of consolidating the specifications for essential materials used by the Chemical Processing Department of the Hanford Atomic Products Operation. These specifications serve a dual function. First, when duly authorized, they detail purchase specifications and tolerances by which these materials may be routinely purchased. Second, they detail sampling and analytical procedures which are required to:

1. Insure that the buyer has received his money's worth of material.
2. Protect the user against faulty material which may cause malfunction of a process or damage to equipment.

Such specifications may originate in any interested group within CPD. They are to be directed to the Manager, Separations Chemistry Laboratory, who is responsible for maintenance of the manual and who will secure the necessary technical review, obtain the proper approvals, and arrange for inclusion in the manual. Upon approval by the Manager, Research and Engineering Operation, these specifications are binding. For materials delivered directly to the operating plants, it is the responsibility of each plant manager to verify acceptablility of the material received in his plant or obtain a temporary waiver of specifications. For materials delivered to a central CPD warehouse, this responsibility rests with the Specialist, Production Planning and Scheduling, Production Operation. Waivers may be authorized by one of the following:

Manager, Research and Engineering Operation
Manager, Plutonium Process Engineering
Manager, Weapons Process Engineering
Manager, Purex Process Engineering
Manager, Redox Process Engineering

Section II comprises purchase and acceptance specifications. An index is found in the first part of Section II. Here materials are listed by CPD cost account number and by name. The using plants are indicated.

The specification page or pages for a material contain both purchase specifications and acceptance specifications. The latter define the manner and extent of testing for acceptance.

Section III gives sampling procedures. They are not intended as specifications. Samples are to be submitted to an appropriate laboratory for analysis. The Separations Chemistry Laboratory arranges for special non-routine testing on request.

Section IV gives analytical methods to be used. Many of these are of long standing and the original letter-number code system for designating methods is followed. The methods are assembled on the basis of element, ion, or property measured, and are arranged alphabetically according to the assigned code.

The code is based on the following principles. The first letter or pair of letters is the chemical symbol of the element determined. Complex ions are coded according to the most representative element in the compound. Organic compounds are assigned the code letters Zx; physical properties, Zy; and miscellaneous instrumental methods such as spectrographic and gas techniques are coded Zz.

APPROVAL

The signatures below signify approval of all specifications dated October 1, 1958, or before:

Issued by K. M. Harmon Mgr., Process Chemistry Operation 10-30-58
Date

Approved by V. R. Cooper Mgr., Research & Engineering 10/31/58
Date

ALPHABETICAL INDEX

MATERIAL	CPD MATERIAL NUMBER	USING OPERATION
Acetic Acid	064	Purex Fission Products
Aluminum Nitrate	060	Redox Purex Weapons
Aluminum Sulfate	100	Power
Ammonium Fluoride - Ammonium Nitrate	063	Redox
Argon	161	Weapons
Bromobenzene	220	Weapons
Calcium (U-grade)	311	Weapons
Carbon Dioxide	270	Weapons
Carbon Tetrachloride (c.p.)	280	Weapons
Carbon Tetrachloride	281	Weapons
Chemical 70-58	313	Weapons
Chemical 70-58 Oxide	315	Weapons
Chlorine	321	Power
Citric Acid	285	Fission Products
Dibutyl Butyl Phosphonate	381	Weapons
Di-sodium Phosphate	380	Power
Di(2-ethylhexyl) Phosphoric Acid (D2EHPA)	384	Fission Products
DTPA	705	Fission Products

MATERIAL	CPD MATERIAL NUMBER	USING OPERATION
Ferrous Ammonium Sulfate	430	Purex Redox Weapons
Ferrous Sulfamate	432	Purex
Filter Aids	530	Redox
Formaldehyde Solution (37%)	436	Purex
Graphite	452	Weapons
Hydrazine Solution (35%)	890	Purex
Hydrofluoric Acid (48%)	502	Weapons
Hydrogen Fluoride	510	Weapons
Hydrogen Peroxide	526	Weapons Fission Products
Hydroxy Acetic Acid	524	Purex
Hydroxylamine Nitrate Solution	529	Weapons
Iodine	550	Weapons
Lead Nitrate	576	Purex
Lime, Hydrated	590	Power
Limestone	600	Purex Weapons
Magnesium Oxide	312	Weapons
Magnesium Oxide Crucibles	361	Weapons
Magnesium Oxide Crucibles - Slip Cast	373	Weapons
Mercuric Nitrate	626	Purex Redox

MATERIAL	CPD MATERIAL NUMBER	USING OPERATION
Methyl Isobutyl Ketone	640	Redox
Nitric Acid (60%)	670	Purex Redox Weapons Fission Products
Nitric Acid (72%)	671	Weapons
Oxalic Acid	690	Purex Weapons Fission Products
Oxygen	700	Weapons
P-1 Vessel	931	Weapons
Permutit SK Anion Exchange Resin	140	Purex Weapons
Petroleum Diluent	720	Purex
Potassium Bicarbonate	743	Purex Fission Products
Potassium Permanganate	750	Purex Redox Weapons Fission Products
Propane	760	Redox
Silicon Nitride Ceramic	774	Weapons
Silver Nitrate	775	Purex Redox Fission Products
Sodium Acetate	780	Purex
Sodium Bismuthate	782	Redox

MATERIAL	CPD MATERIAL NUMBER	USING OPERATION
Sodium Carbonate	785	Purex Fission Products Weapons Power
Sodium Chloride (Rock Salt)	770	Power
Sodium Dichromate	795	Redox Purex Weapons
Sodium Dichromate Solution	796	Redox
Sodium Fluoride	799	Purex Weapons
Sodium Hydroxide (50%)	800	Purex Redox Weapons
Sodium Hydroxide (Reagent grade)	802	Purex
Sodium Nitrate	805	Purex
Sodium Nitrite	810	Purex Weapons Fission Products
Sodium Sulfate, Anhydrous	820	Purex Fission Products
Sodium Sulfite	825	Power
Sugar	842	Purex
Sulfamic Acid	870	Purex Redox
Sulfuric Acid (93%)	880	Purex Weapons

MATERIAL	CPD MATERIAL NUMBER	USING OPERATION
Sulfuric Acid (c.p.)	881	Purex Weapons
Tantalum Metal	912	Weapons
Tartaric Acid	910	Purex
Tributyl Phosphate (TBP)	920	Purex Weapons
Trichloroethylene (Tech.)	930	Weapons

NUMERICAL INDEX

CPD MATERIAL NUMBER	MATERIAL
060	Aluminum Nitrate
063	Ammonium Fluoride - Nitrate
064	Acetic Acid
100	Aluminum Sulfate
140	Permutit-SK Anion Exchange Resin
161	Argon
220	Bromobenzene
270	Carbon Dioxide
280	Carbon Tetrachloride (C.P.)
281	Carbon Tetrachloride
285	Citric Acid
311	Calcium (U grade)
312	Magnesium Oxide
313	Chemical 70-58
315	Chemical 70-58 Oxide
321	Chlorine
361	Magnesium Oxide Crucibles
373	Magnesium Oxide Crucibles - Slip Cast

CPD MATERIAL Number	MATERIAL
380	Disodium Phosphate
381	Dibutyl Butyl Phosphonate
384	Di(2-ethylhexyl) Phosphoric Acid (D2EHPA)
430	Ferrous Ammonium Sulfate
432	Ferrous Sulfamate
436	Formaldehyde Solution (37%)
452	Graphite
502	Hydrofluoric Acid (48%)
510	Hydrogen Fluoride
524	Hydroxy Acetic Acid
526	Hydrogen Peroxide
529	Hydroxylamine Nitrate Solution
530	Filter Aids
550	Iodine
576	Lead Nitrate
590	Lime, Hydrated
600	Limestone
626	Mercuric Nitrate
640	Methyl Isobutyl Ketone
670	Nitric Acid (60%)
671	Nitric Acid (72%)

CPD MATERIAL NUMBER	MATERIAL
690	Oxalic Acid
700	Oxygen
705	DTPA
720	Petroleum Diluent
743	Potassium Bicarbonate
750	Potassium Permanganate
760	Propane
770	Sodium Chloride
774	Silicon Nitride Ceramic
775	Silver Nitrate
780	Sodium Acetate
782	Sodium Bismuthate
785	Sodium Carbonate
795	Sodium Dichromate
796	Sodium Dichromate Solution
799	Sodium Fluoride
800	Sodium Hydroxide - 50%
802	Sodium Hydroxide (Reagent grade)
805	Sodium Nitrate
810	Sodium Nitrite
820	Sodium Sulfate, Anhydrous

CPD MATERIAL NUMBER	MATERIAL
825	Sodium Sulfite
842	Sugar
870	Sulfamic Acid
880	Sulfuric Acid (93%)
881	Sulfuric Acid (C.P.)
890	Hydrazine Solution
910	Tartaric Acid
912	Tantalum Metal
920	Tirbutyl Phosphate (TBP)
930	Trichloroethylene (Tech.)
931	P-1 Vessel

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Cl-2	Determination of Free Chlorine - visual
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ALPHABETICAL INDEX

MATERIAL	CPD MATERIAL NUMBER	USING OPERATION
Acetic Acid	064	Purex
Aluminum Nitrate	060	Weapons Manufacturing Redox
Aluminum Sulfate	100	Power
Argon	161	Weapons Manufacturing
Bromobenzene	220	Weapons Manufacturing
Calcium (U-grade)	311	Weapons Manufacturing
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Hydrogen Fluoride	510	Weapons Manufacturing
Hydrogen Peroxide	526	Weapons Manufacturing
Hydroxy Acetic Acid	524	Purex
Iodine	550	Weapons Manufacturing
Lead Nitrate	576	Purex
Lime, Hydrated	590	Power
Limestone	600	Weapons Manufacturing Purex
Magnesium Oxide	312	Weapons Manufacturing
Magnesium Oxide Crucibles	361	Weapons Manufacturing
Magnesium Oxide Crucibles - Slip Cast	373	Weapons Manufacturing
Mercuric Nitrate	626	Purex and Redox
Methyl Isobutyl Ketone	640	Redox

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Oxygen	700	Weapons Manufacturing
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Permutit SK Anion Exchange Resin	140	Redox Purex
Petroleum Diluent	720	Purex
Potassium Bicarbonate	743	Purex
Potassium Permanganate	750	Weapons Manufacturing Purex and Redox
Propane	760	Redox
Silicon Nitride Ceramic	774	Weapons Manufacturing
Silver Nitrate	775	Purex and Redox
Sodium Acetate	780	Purex
Sodium Carbonate	785	Weapons Manufacturing Power
Sodium Chloride (Rock Salt)	770	Power
Sodium Dichromate	795	Redox
Sodium Dichromate Solution	796	Redox
Sodium Fluoride	799	Weapons Manufacturing

MATERIAL	CPD MATERIAL NUMBER	USING OPERATION
Sodium Hydroxide (50%)	800	Weapons Manufacturing Purex Redox
Sodium Hydroxide (Reagent grade)	802	Purex
Sodium Nitrate	805	Purex Redox
Sodium Nitrite	810	Purex
Sodium Sulfate, Anhydrous	820	Purex
Sodium Sulfite	825	Power
Sugar	842	Purex
Sulfamic Acid	870	Purex
Sulfuric Acid (93%)	880	Weapons Manufacturing Purex
Sulfuric Acid (C.P.)	881	Weapons Manufacturing Purex
Tantalum Metal	912	Weapons Manufacturing
Tartaric Acid	910	Purex
Tributyl Phosphate (TBP)	920	Weapons Manufacturing Purex
Trichloroethylene (Tech.)	930	Weapons Manufacturing

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881	Sulfuric Acid (C.P.)
890	Hydrazine Solution
910	Tartaric Acid
912	Tantalum Metal
920	Tributyl Phosphate (TBP)
930	Trichloroethylene (Tech.)
931	P-1 Vessel

SECTION II

MATERIALS SPECIFICATIONS

FEB 2 8 1964

ALUMINUM NITRATE SOLUTION

CPD Material Number 0060

Purchase Specifications

a.	Aluminum nitrate solution assay (as aluminum)	Min. 5.18 Wt.%
	(as $Al(NO_3)_3 \cdot 9H_2O$)	Min. 72.0 Wt.%
b.	Specific gravity at 100 deg. F.	Min. 1.390
c.	Acidity (acid deficient), HNO_3	-1.1 to -1.6 Wt.%
	HNO_3	-0.25M to -0.35M
d.	Insolubles	Max. 0.20 Wt.%
e.	Ammonia (NH_4^+)	Max. 0.02 Wt.%
f.	Chloride (Cl^-)	Max. 0.005 Wt.%
g.	Sulfate ($SO_4^=$)	Max. 0.02 Wt.%
h.	Alkali and alkaline earth metals: as sodium	Max. 0.20 Wt.%
i.	Heavy metals (including SN) as Pb	Max. 0.01 Wt.%
j.	Silica (Si)	Max. 0.01 Wt.%
k.	Iron (Fe)	Max. 0.03 Wt.%
l.	Nitrite (NO_2	Max. 0.001 Wt.%
m.	Calcium	Max. 0.0072 Wt. %
n.	Strontium	Max. 0.0014 Wt.%

Acceptance Specifications

An 8 oz. sample from each delivery should be analyzed as follows, (sampling Method C.).

		Test Requirements		
		Value	Method	Frequency
a.	Aluminum as $Al(NO_3)_3 \cdot 9H_2O$	Min. 72.0 Wt.%	Al-1	On request of Proc. Engineers
b.	Specific Gravity at 100 F	Min. 1.390	Zy-10A	Each delivery
c.	Acidity as HNO_3 (Redox only)	-1.1 to -1.6 Wt.%	H-3	" "
d.	Insolubles	Max. 0.20 Wt.%	Zy-14	On request of Proc. Engineers
e.	Ammonia (NH_4^+)	Max. 0.02 Wt.%	N-5	" " " "
f.	Chloride (Cl^-)	Max. 0.005 Wt.%	Cl-5	Each delivery
g.	Sulfate ($SO_4^=$)	Max. 0.02 Wt.%	S-25	On request of Proc. Engineers
h.	Alkali and alkaline earths as Na	Max. 0.20 Wt.%	Na-2	" " "
i.	Heavy metals (incl. Sn) as Pb	Max. 0.01 Wt.%	Pb-2	" " "
j.	Silica (Si)	Max. 0.01 Wt.%	Si-1	" " "
k.	Iron (Fe)	Max. 0.03 Wt.%	Fe-5	" " "
l.	Nitrite (NO_2^-)	Max. 0.001 Wt.%	N-13	" " "
m.	Calcium (Ca)(Purex only)	Max. 0.0072 Wt. %	Zy-25	" " "
n.	Strontium (Sr)(Purex only)	Max. 0.0014 Wt.%	Zy-25	" " "

Issued by

O F Beaulieu

Manager, Separations Chemistry Laboratory

Approved by

H. S. Frank

Manager, Research and Engineering

AMMONIUM FLUORIDE - AMMONIUM NITRATE SOLUTION

CPD Material Number 0063

Purchase Specifications

a.	Ammonium Fluoride-Ammonium Nitrate, NH_4F-NH_4NO_3 (Aqueous Solution)	NH_4F NH_4NO_3	36.0-37.6 Wt.% 7.0- 7.4 Wt.%
b.	Insolubles	Max.	0.02 Wt.%
c.	pH at 23-25 C		7.5 ± 0.3
d.	Chloride (Cl^-)	Max.	0.02 Wt.%

Acceptance Specifications

An 8-ounce sample of each shipment (Method C) should be taken.

Test Requirements

		Value	Method	Frequency
a.	Ammonium Fluoride, NH_4F	36.0-37.6 Wt.%	F-2	Each shipment
b.	Ammonium Nitrate, NH_4NO_3	7.0-7.4 Wt.%	N-21	Each shipment
c.	pH at 23-25 C	7.5 ± 0.3	pH-1	Each shipment
d.	Chloride (Cl^-)	Max. 0.02 Wt.%	Cl-5	On request

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Manager, Separations Chemistry Laboratory

Approved by

R. E. Tomlinson

Manager, Research and Engineering

ACETIC ACID

CPD Material Number 064

Purchase Specifications

a.	Acetic acid (CH_3COOH) aqueous solution	Minimum 80 Wt.%
b.	Residue on evaporation	Maximum 0.01 Wt.%
c.	Chloride	Maximum 0.01 Wt.%

Acceptance Specifications

		Value	Test Method	Testing Frequency
a.	Acidity as CH_3COOH	To meet minimum specified on requisition	H-1	Each shipment
b.	Residue on ignition	Maximum 0.01 Wt.%	Zy-22	Each shipment
c.	Chloride	Maximum 0.01 Wt.%	Cl-5	Each shipment

An 8 oz. sample of each shipment should be taken.

Issued by

O F Beaulieu

Manager
Separations Chemistry Laboratory

Approved by

H. S. Frank

Manager
Research and Engineering

ALUMINUM SULFATE ("ALUM")

CPD Material Number 100

Purchase Specification

Dry Crystals

a. Aluminum sulfate, $Al_2(SO_4)_3.xH_2O$
Commercial grade

Al_2O_3 content — Minimum 17 Wt.%

Acceptance Specifications

On request of Power Department supervision, a 16 oz. sample from a shipment may be taken (Method B) and analyzed as follows:

	Test Requirements		
	Value	Method	Frequency
$Al_2(SO_4)_3.xH_2O$			On request of Power Dept. supervision
Al_2O_3 content	Min. 17 Wt.%	Al-1	

Issued by

Approved by

H. S. Frank

PERMUTIT SK ANION EXCHANGE RESIN

CPD Material Number 0140

Purchase Specifications

a.	Permutit SK Anion Exchange Resin	Salt form, i.e., Cl^{-} $SO_4^{=}$
b.	Particle Size	85% Min. - 20 + 40
c.	Moisture Content	45 - 55 per cent

Acceptance Specifications

A 16 oz. sample (Method B) of each shipment should be analyzed as follows. If more than one lot is represented, secure and analyze a sample of each lot.

		Test Requirements		
		Value	Method	Frequency
a.	Salt form	Positive*	Cl-5	Each shipment
b.	Particle Size	85% Min. - 20+40	Zy-24a	Each shipment
c.	Moisture Content	45-55%	O-4	Each shipment

* Qualitative test for Cl^{-} only.

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

ARGON

CPD Material Number 160

PROPERTIES		Testing Methods
*a. Argon, A Assay, min.,	99.8%	Zz-1 or Mass Spectrometer
b. Inert gas.		

* Property a required by specification.

SAMPLE

Sample each gas shipment by Method J.

COMMENTS ON STORAGE AND HANDLING

This material is purchased as a gas under high pressure (up to 2200 psi gauge).

Cylinder color marking: green-yellow.

ARGON

CPD Material 0161

Purchase Specifications

a. Argon, compressed gas	Min. 99.8 Vol. % Avg.

Note: A certified analysis should be requested from the vendor on the purchase requisition.

Acceptance Specifications

A certified vendor's analysis showing conformance to specifications is adequate for acceptance. Copy coverage to the consumer should be supplied by the Purchasing Department on request of Process Engineering.

On request of Process Engineering a sample may be taken by Method J and analyzed as follows:

		Test Requirements	
	Value	Method	Frequency
a. Argon	Min. 99.8 Vol %	Zz-1 or Mass Spec	On request of process engineers

Issued by

Manager, Separations Chemistry Laboratory

Approved by

H. S. Frank

Manager, Research and Engineering

BROMOBENZENE

CPD Material Number 0220

Purchase Specifications

a. Bromobenzene, commercial grade	
b. Specific gravity	Max. 1.495 at 20°/4°
c. Color	Water clear

Acceptance Specification

On request of Process Engineering a 10 oz. sample of each shipment should be taken by method D-1 and analyzed as follows:

	Test Requirements		
	Value	Method	Frequency
b. Specific gravity 20°/4°	Max. 1.495	Zy-10	On request of process engineers
c. Color	Water white	Visual	" "

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Approved by

Manager, Research and Engineering

CARBON DIOXIDE

CPD Material Number 0270

Purchase Specifications

Carbon dioxide, compressed gas, commercial grade CO_2	Min. 99.5 Vol. %

Note: A certified analysis should be requested from the vendor on the purchase requisition.

Acceptance Specifications

A vendor's analysis of each shipment should be forwarded by the vendor to the Purchasing Department. Copy coverage to the consumer should be supplied by the Purchasing Department on request.

Conformance to specification in the vendor's analysis is satisfactory for acceptance.

On request of process engineers, the Separations Chemistry Laboratory will arrange for sampling and analysis of this material as a spot check.

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Manager, Separations Chemistry Laboratory

Approved by

H. S. Frank
Manager, Research and Engineering

CARBON TETRACHLORIDE (C.P.)

CPD Material Number 0280

Purchase Specifications

Carbon Tetrachloride, CCl_4, C.P. grade

Acceptance Specifications

This material is procured from Stores in bottles containing eight pounds. The caption 51-1100-300 conforms to the specifications of Baker & Adamson, Catalog #1556.

This material is acceptable from Stores without testing.

Issued by

O F Beaulieu

Manager, Separations Chemistry Laboratory

Approved by

F. S. Frank

Manager, Research and Engineering

CARBON TETRACHLORIDE

CPD Material Number 0281

Purchase Specifications

a.	Carbon tetrachloride, technical grade	
b.	Boiling range at 760 mm	73.0 - 77.5° C
c.	Acidity	Neutral to methyl red
d.	Non-volatile residue	Max. 0.002 Wt.%
e.	Free chlorine	To pass test
f.	Appearance, colorless, clear liquid	

Note: A certified analysis should be requested from the vendor on the purchase requisition.

Acceptance Specifications

A certified vendor's analysis showing conformance to specifications is adequate for acceptance. Copy coverage to the consumer should be supplied by the Purchasing Department on request of Process Engineering.

On request of Process Engineering at 16 oz. sample (Method D) of a shipment should be taken and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
b.	Boiling range	73-77.5° C/760	Zy-3	On request of process engineers
c.	Acidity	Neutral to methyl red		" "
d.	Non-volatile	Max. 0.002%	Zy-22	" "
e.	Free chlorine	To pass test	Cl-2	" "
e.	Appearance	Clear colorless	Visual	" "

Issued by

Manager, Separations Chemistry Laboratory

Approved by

F. S. Frank

Manager, Research and Engineering

CITRIC ACID

CPD Material Number 285

Purchase Specifications

a.	Citric Acid ($H_3C_6H_5O_7$)	Minimum 99.5 Wt.%
b.	Chloride (Cl^-)	Maximum 0.01 Wt.%
c.	Residue on ignition	Maximum 0.01 Wt.%
d.	Phosphate ($PO_4^{\equiv}$)	Maximum 0.001 Wt.%

Acceptance Specifications

		Value	Testing Method	Testing Frequency
a.	Acidity as $H_3C_6H_5O_7$	Minimum 99.5 Wt.%	H-1	Each shipment
b.	Chloride (Cl^-)	Maximum 0.01 Wt.%	Cl-5	Each shipment
c.	Residue on ignition	Maximum 0.01 Wt.%	Zy-23	Each shipment
d.	Phosphate ($PO_4^{\equiv}$)	Maximum 0.001 Wt.%	P-2	Each shipment

An 8 oz. sample of each shipment should be taken.

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Manager
Separations Chemistry Laboratory

Approved by

H. S. Frank

Manager
Research and Engineering

CALCIUM (U-GRADE)

CPD Material Number 311

PROPERTIES

			Testing Methods
*a.	Calcium metal: min.,	96%	Ca-1
*b.	Carbon (C): max.,	350 ppm	C-2
*c.	Chlorine (Cl): max.,	0.1%	Cl-5
*d.	Nitrogen (N): max.,	400 ppm	N-3
*e.	Metallic impurities: max.,		Zz-21
	Lithium (Li)	1 ppm	
	Beryllium (Be)	1 ppm	
	Boron (B)	1 ppm	
	Sodium (Na)	10 ppm	
	Magnesium (Mg)	2000 ppm	
	Aluminum (Al)	10 ppm	
	Silicon (Si)	10 ppm	
	Manganese (Mg)	100 ppm	
	Iron (Fe)	100 ppm	
	Copper (Cu)	10 ppm	
	Chromium (Cr)	10 ppm	
	Cobalt (Co)	10 ppm	
	Nickel (Ni)	10 ppm	

f. Lustrous, silver-white surface when freshly cut, but gradually becomes gray due to coating of oxide.

* Properties a through e required by specification.

SAMPLE

50 gram sample from each shipment - Method B-1.

HAZARDOUS PROPERTIES

a. Metallic calcium reacts with moisture or acids to liberate hydrogen and can develop enough pressure in sealed container to explode. Dangerous fire hazard.

b. Calcium can ignite spontaneously in the presence of oxygen and moisture. The fumes evolved are irritating to the skin.

MAGNESIUM OXIDE

CPD Material Number 0312

Purchase Specifications

a. Magnesium oxide, MgO, electrically fused.	Min. 98 Wt.%
b. Silicon dioxide (SiO_2)	Max. 0.15 Wt.%
c. R_2O_3	Max. 0.50 Wt.%
d. Calcium oxide (CaO)	Max. 1.0 Wt.%
e. Boron (B)	Max. 0.01 Wt.%
f. Beryllium (Be)	Max. 0.01 Wt. %
g. Grain size range as specified on individual purchase requisitions	

Acceptance Specifications

A 10 oz. sample of each shipment should be analyzed as follows, (sampling Method B III).

	Test Requirements		
	Value	Method	Frequency
a. Magnesium oxide (MgO)	Min. 98 Wt.%	Mg-2	On request of Proc. Engrs.
b. Silicon dioxide	Max. 0.15 Wt.%	Si-1	" " "
c. R_2O_3	Max. 0.5 Wt.%	Fe-1	" " "
d. Calcium oxide	Max. 1.0 Wt.%	Ca-1	" " "
e. Boron (B)	Max. 0.01 Wt.%	Zz-21	" " "
f. Beryllium (Be)	Max. 0.01 Wt.%	Zz-21	" " "
g. Grain size		Zy-24 Sieve analysis	" " "

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

CHEMICAL 70-58

CPD Material Number 313

PROPERTIES

The properties, specifications, and testing methods are contained in Document HW-26242.

CHEMICAL 70-58 OXIDE

CPD Material Number 315

PROPERTIES

The properties, specifications, and testing methods are contained in Document HW-30586.

CHLORINE

CPD Material 0321

Purchase Specifications

a. Chlorine, compressed gas, Cl_2 commercial grade — Min. 99.9 Vol.%

Note: A certified analysis should be requested from the vendor on the purchase requisition.

Acceptance Specifications

A vendor's certified analysis showing conformance to specifications is adequate for acceptance. Copy coverage to the consumer should be supplied by the Purchasing Department on request of Power Department supervision.

On request of Power Department supervision the Separations Chemistry Laboratory will arrange for special testing when needed.

Issued by

Manager, Separations Chemistry Laboratory

Approved by

H. S. Frank
Manager, Research and Engineering

CHROMIC NITRATE

CPD Material Number 340

PROPERTIES		Weight Percent	Testing Methods
*a.	Chromic Nitrate, $Cr(NO_3)_3 \cdot 9H_2O$ Assay (as Cr): min.,	12.5%	Cr-1
*b.	Chloride (Cl^-): max.,	0.005%	Cl-5
*c.	Iron (Fe): max.,	0.01%	Fe-5
*d.	Substances not precipitated by NH_4OH: max., as sodium	0.10%	Na-2
*e.	Sulfate ($SO_4^{=}$): max.,	0.01%	S-25
*f.	Ammonia (NH_4^+): max.,	0.002%	N-5
g.	Purple-red crystals. Melts at 36 C		

*Properties a through f required by specification.

SAMPLE

10 oz. sample of each shipment - Method B.

HAZARDOUS PROPERTIES

Chromium nitrate is a powerful oxidizing agent in the solid form, and hence is a dangerous fire and explosion hazard.

Issued by:

KM Harman

Manager, Process Chemistry

Approved by:

Manager, Research and Engineering

MAGNESIUM OXIDE CRUCIBLES

CPD Material Number 361

Purchase Specifications

a. Magnesium oxide crucibles designated by type number and drawing number.

Type H- 7, drawing number H-2-19945, Rev. 1.
Type H- 11, drawing number H-2-24652.

b. The crucibles shall be made of electrically fused, ground, and screened magnesium oxide. The mix shall contain a minimum of 40% of grain size -10, +20 (40% of the material passes through a 10 mesh and is retained on a 20 mesh screen).

c. The magnesium oxide used for the crucibles shall conform to the following composition requirements.

MgO	Minimum	96.5 Wt.%
SiO_2	Maximum	1.5 Wt.%
	Minimum	1.0 Wt.%
R_2O_3(Al_2O_3, Fe_2O_3)	Maximum	0.5 Wt.%
CaO	Maximum	1.0 Wt.%
Be	Maximum	0.01 Wt.%
B	Maximum	0.01 Wt.%

Acceptance Specifications

Acceptability of the material will be determined by Weapons Process Engineering Group, CPD, based on performance (particularly life of crucibles in actual use).

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research & Engineering

MAGNESIUM OXIDE CRUCIBLES

CPD Material Numbers 362-369

PROPERTIES

The magnesium oxide used in these crucibles shall meet the specifications established for CPD Material Number 312, with the exception that a silica content of not more than 0.5 per cent is allowable.

CRUCIBLE SPECIFICATIONS

Crucible Number	CPD Material Number	See Drawing Number
H-5	362	H-2-20928
CD-6507	363	H-2-20954
CD-6506	364	H-2-18401
CDS-6506	365	H-2-18400
RCDS-1102	369	H-2-16724

SAMPLE

These materials are procured through the AEC. Conformance to specifications will be determined by the Finished Products Operation, C.P.D.

MAGNESIUM OXIDE CRUCIBLES - SLIP CAST

CPD Material Number 373

Purchase Specifications

Crucible Number: RS-6 Drawing Number: H-2-19756 (Secret)

The finished product must meet the dimensions specified in the reference drawing. The finished crucibles must have well sintered bodies with no appreciable surface dust, and should exhibit a high degree of strength and thermal shock resistance such that the crucibles will maintain their integrity during the reduction process.

A suggested procedure for slip casting, which has been used at Hanford, is a modification of the Los Alamos method described in the American Ceramic Society Bulletin, 37(9):409-413, September, 1958, "Casting of Magnesium Oxide in Aqueous Slips."

a.	Magnesium oxide, MgO, electrically fused	Min. 96.5 Wt.%
b.	Silicon dioxide (SiO_2) Note: Preferred SiO_2 content is 0.5% to 1.0%	Max. 1.5 Wt.%
c.	R_2O_3	Max. 0.5 Wt.%
d.	Calcium oxide (CaO)	Max. 1.0 Wt.%
e.	Beryllium (Be)	0.01 Wt.%
f.	Boron (B)	0.01 Wt.%
g.	Other elements as low as commercially feasible, subject to individual approval on presentation of typical complete analysis and process testing.	

Acceptance Specifications

Sampling practice for received material will be specified as needed by Finished Products Operation, CPD.

		Test Requirements		
		Value	Method	Frequency
a.	Magnesium oxide, MgO, electrically fused.	Min. 96.5 Wt.%	Mg-2	On request of Proc. Engrs.
b.	Silicon dioxide, SiO_2	Max. 1.5 Wt.%	Si-1	" " "
c.	R_2O_3	Max. 0.50 Wt.%	Fe-1	" " "
d.	Calcium oxide, CaO	Max. 1.0 Wt.%	Ca-1	" " "
e.	Beryllium (Be)	Max. 0.01 Wt.%	Zz-21	" " "
f.	Boron (B)	Max. 0.01 Wt.%	Zz-21	" " "

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Manager, Separations Chemistry Laboratory

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Manager, Research and Engineering

DI-SODIUM PHOSPHATE

CPD Material Number 0380

Purchase Specifications

a. Sodium Phosphate, Dibasic, Na_2HPO_4, commercial grade

Acceptance Specifications

Acceptance testing is not mandatory.

On request of Power Department supervision, the Separations Chemistry Laboratory will arrange for any necessary tests.

Issued by	Approved by
OF Beaulieu	W. S. Frank
Manager, Separations Chemistry Laboratory	Manager, Research and Engineering

DI(2 ETHYLHEXYL) PHOSPHORIC ACID

CPD Material Number 0384

Purchase Specifications

a.	Di(2-ethylhexyl) phosphoric acid. (Also called D2EHPA) $[CH_3 \cdot CH_2 \cdot CH_2 \cdot CH_2CH(CH_2CH_3)CH_2]_2O_2POOH$		97.6-100.0 Wt.%
b.	Specific gravity		0.970-0.984 20/20 C 0.965-0.979 25/4 C
c.	Refractive index		$N^{20}D$ 1.443
d.	Solubility		
	(1) In water	Max.	0.01 Wt.% at 20 C
	(2) Water in	Max.	2.4 Wt.% at 20 C
e.	Flash point: Cleveland Open Cup		385 F

Acceptance Specifications

A 16 oz. sample of each shipment (Method C or D) should be taken and analyzed as follows:

	Test Requirements		
	Value	Method	Frequency
(a) Di(2-ethylhexyl)phosphoric acid	> 97.6% < 100%	H-1	Each shipment
(b) Specific gravity	0.970-0.984 20/20	Zy-10	Each shipment

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Manager, Separations Chemistry Laboratory

Approved by

R. E. Tomlinson

Manager, Research and Engineering

FERROUS AMMONIUM SULFATE

CPD Material Number 0430

Purchase Specifications

a.	Ferrous ammonium sulfate, $Fe(NH_4)_2(SO_4)_2.6H_2O$ Ferrous iron content, Fe^{++}	Min. 13.9 Wt.%
b.	Chloride (Cl^-)	Max. 0.01 Wt.%
c.	Insolubles	Max. 0.01 Wt.%

Acceptance Specifications

On request of Process Engineering a 16 oz. sample should be taken from a shipment (Method B) and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Ferrous iron	13.9 Wt.%	Fe-3	On request of process engineers
b.	Chloride	Max. 0.01 Wt.%	Cl-5	
c.	Insolubles	Max. 0.01 Wt.%	Zy-14	

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Manager, Separations Chemistry Laboratory

Approved by

R.E. Tomlinson
Manager, Research and Engineering

FERROUS SULFAMATE

CPD Material Number 0432

Purchase Specifications

a.	Ferrous sulfamate solution $Fe(NH_2SO_3)_2$ Content:	48 to 52 Wt.%
b.	Reducing normality	2.9 to 3.2 Normal
c.	Sulfamate concentration as $Fe(NH_2SO_3)_2$	6.3 to 7.00 Molar
d.	Ferric ion	Max. 0.05 Wt.%
e.	Sulfate ($SO_4^=$)	Max. 1.0 Wt.%
f.	Chloride (Cl^-)	Max. 0.02 Wt.%
g.	pH	Max. 1.35
h.	SpGr	1.52 ± 0.02 at 25/25
i.	Calcium (Ca)	Max. 0.004 Wt.%
j.	Strontium (Sr)	Max. 0.0005 Wt.%

Acceptance Specifications

A 16 oz. sample of each shipment should be analyzed as follows (sampling Method D).

		Test Requirements		
		Value	Method	Frequency
a.*	Reducing normality	2.9 - 3.2 N	Fe-3	Each shipment
b.	Sulfamate	6.3 - 7.00M	Sa-2	On request of Proc. Engrs.
c.	Ferric ion	Max. 0.05 Wt.%	Fe-6	" " "
d.*	Sulfate ($SO_4^=$)	Max. 1.0 Wt.%	S-25	" " "
e.	Chloride (Cl^-)	Max. 0.02 Wt.%	Cl-5	Each shipment
f.	pH	Max. 1.35	pH-1	On request of Proc. Engrs.
g.*	SpGr	1.52±0.02 at 25/25	Zy-10	Each shipment
h.	Calcium (Ca)(Purex only)	Max. 0.004 Wt.%	Zy-25	Each shipment
i.	Strontium (Sr)(Purex only)	Max. 0.0005 Wt.%	Zy-25	Each shipment

* Acceptance specifications a, d, and g should be measured within 24 hours of receipt for satisfactory results.

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Manager, Research and Engineering

FORMALDEHYDE SOLUTION (37%)

CPD Material Number 0436

Purchase Specifications

a. Formaldehyde content, HCHO	Min. 37 Wt.%
b. Methanol content	Max. 7.5 Wt.%
c. Acidity as formic acid	Max. 0.05 Wt.%
d. Calcium (Ca)	Max. 0.000075 Wt.% (2 ppm)
e. Strontium (Sr)	Max. 0.0000037 Wt.% (.1 ppm)

Acceptance Specifications

A 16 oz. sample from a shipment should be analyzed as follows, (sampling Method C).

	Test Requirements		
	Value	Method	Frequency
a. Formaldehyde, HCHO	Min. 37 Wt.%	Zx-2	Each shipment
b. Acidity as formic acid	Max. 0.05 Wt.%	H-2	On request of Proc. Engineers
c. Calcium (Ca)	Max. 0.000075 Wt.%	Zy-25	Each shipment
d. Strontium (Sr)	Max. 0.0000037 Wt.%	Zy-25	Each shipment

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GRAPHITE
METALLURGICAL GRADE, UNIMPREGNATED

CPD Material Number 0452

Purchase Specifications

a. Particle size	Maximum 0.032
b. Flexural strength	Minimum 2000 psi
c. Density	Minimum 1.63 g/cc

Acceptance Specifications

Separations Chemistry Laboratory will arrange for special sampling and testing on request.

Issued by	Approved by
O F Beaulieu	W. S. Frank
Manager Separations Chemistry Laboratory	Manager Research and Engineering

HYDROXY ACETIC ACID

CPD Material Number 524

Purchase Specifications

a. Hydroxy acetic acid, $HOCH_2 \cdot COOH$. An aqueous solution (minimum of 70%) or a crystalline solid may be purchased. Calcium and strontium values to be computed as per cent of $HOCH_2 \cdot COOH$.

b. Chloride (Cl^-)	Maximum 0.2 Wt.%
c. Residue on ignition	Maximum 0.1 Wt.%
d. Strontium	Maximum 0.0001 Wt.%
e. Calcium	Maximum 0.0002 Wt.%
f. Magnesium	Maximum 0.001 Wt.%

Acceptance Specifications

	Value	Testing Method	Testing Frequency
a. Acidity as $HOCH_2COOH$	Note 1	H-1	Each shipment
b. Chloride (Cl^-)	Max. 0.1 Wt.%	Cl-5	On request
c. Residue on ignition	Max. 0.1 Wt.%	Zy-23	Each shipment
d. Strontium	Max. 0.0001 Wt.%	Zy-25	Each shipment
e. Calcium	Max. 0.0002 Wt.%	Zy-25	Each shipment
f. Magnesium	Max. 0.001 Wt.%	Mg-1	On request

Note 1: The observed acidity as $HOCH_2COOH$ should not be less than the value the vendor has agreed to supply.

An 8 oz. sample of each shipment should be taken.

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Mgr. Separations Chemistry Laboratory

Approved by

Mgr. Research and Engineering

HYDROFLUORIC ACID (48%)

CPD Material Number 502

Purchase Specifications

a. Hydrofluoric acid, HF Assay (as HF)	Min. 46%
b. Chloride (Cl^-)	Max. 0.1%

Acceptance Specifications

This material is purchased from Stores under the caption number 51-0100-260, which specifies 48% Reagent, A.C.S. material.

Assurance of conformity with A.C.S. specifications by the vendor's label is adequate for acceptance.

This material will be sampled and analyzed only on special request of Process Engineers.

Testing methods are specified in "A.C.S. Reagent Chemicals."

Issued by

O F Beaulieu

Manager, Separations Chemistry Laboratory

Approved by

H. S. Frank

Manager, Research and Engineering

HYDROGEN FLUORIDE

CPD Material Number 0510

Purchase Specifications

a. Anhydrous hydrogen fluoride (HF)	Min. 99.7 Wt.%
b. Water (H_2O)	Max. 0.25 Wt.%
c. Fluosilicic Acid (H_2SiF_6)	Max. 0.04 Wt.%
d. Sulfuric acid (H_2SO_4)	Max. 0.04 Wt.%
e. Sulfur dioxide (SO_2)	Max. 0.072 Wt.%

Acceptance Specifications

A vendor's analysis of each lot of material supplied should be forwarded by the vendor to the purchasing department. Copy coverage to the consumer should be supplied by the purchasing department on request.

Issued by

Manager, Separations Chemistry Laboratory

Approved by

W. S. Frank

Manager, Research and Engineering

HYDROXY ACETIC ACID

CPD Material Number 524

Purchase Specifications

a. Hydroxy acetic acid, $HOCH_2 \cdot COOH$. An aqueous solution (minimum of 70%) or a crystalline solid may be purchased. Calcium and strontium values to be computed as per cent of $HOCH_2 \cdot COOH$.

b.	Chloride (Cl^-)	Max. 0.2 Wt.%
c.	Residue on ignition	Max. 0.1 Wt.%
d.	Strontium	Max. 0.0001 Wt.%
e.	Calcium	Max. 0.0002 Wt.%
f.	Magnesium	Max. 0.001 Wt.%

Acceptance Specifications

An 8 oz. sample of each shipment should be taken. (Sample Method D)

		Test Requirements		
		Value	Method	Frequency
a.	Acidity as $HOCH_2COOH$	Note 1	H-1	Each shipment
b.	Chloride (Cl^-)	Max. 0.1 Wt.%	Cl-5	On request of Proc. Engrs.
c.	Residue on ignition	Max. 0.1 Wt.%	Zy-23	Each shipment
d.	Strontium	Max. 0.0001 Wt.%	Zy-25	Each shipment
e.	Calcium	Max. 0.0002 Wt.%	Zy-25	Each shipment
f.	Magnesium	Max. 0.001 Wt.%	Mg-1	On request of Proc. Engrs.

Note 1: The observed acidity as $HOCH_2COOH$ should not be less than the value the vendor has agreed to supply.

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Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

HYDROGEN PEROXIDE (12%)

CPD Material Number 526

Purchase Specifications

a.	Hydrogen peroxide, H_2O_2	Minimum 12 Wt.%
b.	Iron (Fe)	Maximum 0.5 ppm
c.	Phosphate ($PO_4^{\equiv}$)	Maximum 0.001 Wt.%
d.	Residue on evaporation	Maximum 0.01 Wt.%

Acceptance Specifications

		Value	Testing Method	Testing Frequency
a.	Hydrogen peroxide (H_2O_2)	Min. 12 Wt.%	O-1	Each shipment
b.	Iron (Fe)	Max. 0.5 ppm	Fe-5	Each shipment
c.	Phosphate ($PO_4^{\equiv}$)	Max. 0.001 Wt.%	P-2	Each shipment
d.	Residue on evaporation	Max. 0.01 Wt.%	Zy-22	Each shipment

An 8 oz. sample of each shipment should be taken.

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Manager,
Separations Chemistry Laboratory

Approved by

H. S. Frank

Manager,
Research and Engineering

HYDROGEN PEROXIDE (12%)

CPD Material Number 526

PROPERTIES		Testing Methods
*a. Hydrogen peroxide, H_2O_2 Assay: min.,	12%	0-1
*b. Iron (Fe): max.,	0.5 ppm	Fe-5

* Properties a and b required by specification.

SAMPLE

16 oz. sample of each shipment - Method C or D.

HAZARDOUS PROPERTIES

Hydrogen peroxide and its solutions are dangerous to the eyes, and in higher concentrations, are corrosive to the skin. It is a powerful oxidizing agent and can produce violent combustion if brought into contact with combustible materials. It can decompose violently if brought into contact with catalytic materials, such as iron, chromium, or copper metal or their salts.

HYDROXYLAMINE NITRATE SOLUTION

CPD Material Number 0529

Purchase Specifications

a.	Hydroxylamine Nitrate ($NH_2OH \cdot HNO_3$) Solution	Minimum	1.2 molar
b.	Sulfate ($SO_4^=$)	Maximum	250 ppm
c.	Purity (anhydrous basis)	Minimum	99.7%

Note: A vendor's certified analysis should be requested on the purchase requisition.

Acceptance Specifications

A vendor's certified analysis showing conformance to specifications "a" and "b" is adequate for acceptance. Copy coverage to the consumer should be supplied by the Purchasing Department or CPD Production and Scheduling Operation.

In the absence of a vendor's analysis, an 8 oz. sample (Method D) should be taken and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Hydroxylamine Nitrate	Min. 1.2M	N-12	Each shipment
b.	Sulfate	Max. 250 ppm	S-25	Each shipment

Issued by

Manager, Separations Chemistry Laboratory

Approved by

R. E. Tomlinson
Manager, Research and Engineering

FILTER AIDS

CPD Material Number 0530

Purchase Specifications

These materials are purchased by trade names.

Acceptance Specifications

On request of Process Engineering a 16 oz. sample (Method B) may be taken.

The Separations Chemistry Laboratory will arrange for examination of the sample.

Issued by	Approved by
Manager, Separations Chemistry Laboratory	Manager, Research and Engineering

IODINE

CPD Material Number 0550

Purchase Specifications

a.	Sublimed Iodine	
b.	Non-volatile matter	Max. 0.020 Wt.%
c.	Chlorine and bromine (As Cl)	Max. 0.005 Wt.%

Acceptance Specifications

A 4 oz. sample (Method B-II) should be taken and analyzed as follows.

		Test Requirements		
		Value	Method	Frequency
b.	Non-volatile matter	Max. 0.02 Wt.%	Zy-22	One shipment/mo.
c.	Chlorine and bromine (As Cl) to pass test in Method Cl-6	Max. 0.005 Wt.%	Cl-6	One shipment/mo.

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

LEAD NITRATE

CPD Material Number 0576

Purchase Specifications

a. Lead Nitrate, tech. crystal, $Pb(NO_3)_2$· Lead content	Min. 62 Wt.%
b. Chloride (Cl^-)	Max. 0.01 Wt.%
c. Calcium (Ca)	Max. 0.005 Wt.% (50 ppm)
d. Strontium (Sr)	Max. 0.006 Wt.% (60 ppm)

Acceptance Specifications

An 8 oz. sample of a shipment should be analyzed as follows. (Sample Method B)

	Test Requirements		
	Value	Method	Frequency
a. Lead content (Pb)	Min. 62 Wt.%	Pb-1	On request of Proc. Engineers
b. Chloride (Cl^-)	Max. 0.01 Wt.%	Cl-5	" " "
c. Calcium (Ca)	Max. 0.005 Wt.%	Zy-25	Each shipment
d. Strontium (Sr)	Max. 0.006 Wt.%	Zy-25	Each shipment

Issued by

Manager, Separations Chemistry Laboratory

Approved by

F. S. Frank

Manager, Research and Engineering

LIME, HYDRATED

CPD Material Number 0590

Purchase Specifications

a. Hydrated lime, $Ca(OH)_2$, commercial grade, CaO content, as total alkalinity — Min. 70 Wt.%

Acceptance Specifications

On request of Power Department Supervision a 16 oz. sample (Method A or B) of a shipment should be taken and analyzed as follows:

	Test Requirements		
	Value	Method	Frequency
Total alkalinity as CaO	Min. 70 Wt.%	H-11	On request of Power supervision

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LIMESTONE

CPD Material Number 0600

Purchase Specifications

a.	Limestone, $CaCO_3$, aragonite type, total alkalinity as $CaCO_3$	Min. 96 Wt.%
b.	Nominal particle size 2-4 mesh	
c.	Insoluble materials after dissolution in 0.5M HCl and neutralization to pH 7.	Max. 1.0 Wt.%

Acceptance Specifications

On request of Process Engineering a 16 oz. sample (Method A or B) should be taken and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Alkalinity as $CaCO_3$	Min. 96 Wt.%	H-11	On request of process engineers
b.	Insoluble materials after dissolution in 0.5M HCl and neutralization to pH 7	1.0 Wt.%	Zy-18	" "

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MERCURIC NITRATE

CPD Material Number 0626

Purchase Specifications

a. Mercuric nitrate, C.P. grade, $Hg(NO_3)_2.H_2O$ content Min. 99.0 Wt.%

Acceptance Specifications

This material is purchased in small reagent bottles. Routine sampling and testing is not considered necessary. The Separations Chemistry Laboratory will arrange for sampling and testing on request of Process Engineering.

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METHYL ISOBUTYL KETONE (HEXONE)

CPD Material Number 0640

Purchase Specifications

a.	Methyl isobutyl ketone, $CH_3COC_4H_9$	99.0 Vol. %
b.	Distillation Range	114 - 117 C
c.	Refractive Index	1.3953 ± 0.004 n_D 20 C
d.	Color	15 max. (Hazen Pt-Co Std)
e.	Acidity (as acetic acid):	Max. 0.05%
f.	Methyl isobutyl carbinol:	Max. 0.6 Vol.%
g.	Mesityl oxide:	Max. 0.1 Vol.%
h.	Oxidizing impurities	Max. 0.01 N

Acceptance Specifications

A 16 oz. sample from each car should be analyzed as follows:*

		Test Requirements		
		Value	Method	Frequency
b.	Distillation Range	114-117	Zy-3	On request of proc. engrs.
c.	Refractive Index	1.3953 ± 0.004	Zy-5	Each car*
d.	Color	15 max. platinum cobalt std. (Hazen)	Zy-1	On request of proc. engrs.
e.	Acidity (as acetic acid)	Max. 0.05%	H-2	" "
f.	Methyl isobutyl carbinol	Max. 0.6 Vol.%	Zy-26	Each car*
g.	Mesityl oxide	Max. 0.1 Vol.%	Zx-7	Each car*
h.	Oxidizing impurities	Max. 0.01 N	Zy-20	On request of proc. engrs.

* Analyses must be complete and results reported to Process Engineering before car may be unloaded.

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HEDTA (Trisodium Salt)

CPD Material Number 0668

Purchase Specifications

a.	Aqueous solution of trisodium-N-hydroxyethylethylenediamine-triacetate. $C_{10}H_{15}N_2O_7Na_3$	Min. 40 Wt.%
b.	Sp.Gr. 25/25	Min. 1.28
c.	pH	11 to 14
d.	$CaCO_3$ value	120 ± 5

Acceptance Specifications

A 16 oz. sample (Method C) of each shipment should be taken and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Sp. Gr. 25/25	Min. 1.28	Zy-10	Each shipment
b.	pH	11-14	pH-1	" "
c.	$CaCO_3$ value	120 ± 5	V-1	" "
d.	Sr	2 ppm (100% basis), (0.00008%)	Zy-25	On request
e.	Ca, Mg	20 ppm (100% basis), (0.0008%)	Zy-25	On request

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Manager, Research and Engineering

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NPH (NORMAL PARAFFIN HYDROCARBON)

CPD Material Number 0669

Purchase Specifications

a.	N-paraffin hydrocarbon	Min. 97 Wt.%
b.	B.P. (C_{10} to C_{14} components)	Min. 99 Wt.%
c.	Flash point	155 to 160 F
d.	Sp.Gr. 60/60	0.74 to 0.76
e.	Viscosity	Max. 2.5 centipoises 25 C
f.	Aromatics	Max. 0.2 Wt.%
g.	Olefines	Max. 0.5 Bromine No.
h.	South Hampton Co. is only acceptable source*	

Acceptance Specification

A 16 oz. sample of each tank car (Method C) should be taken and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Boiling Range or Gas chromatograph	Min. 99% C_{10}-C_{14}	Zy-3 Zy-4	On request " "
b.	Sp.Gr. 60/60 F	0.74 - 0.76	Zy-10A	Each shipment
c.	Viscosity	Max. 2.5 centipoises 25 C	Zy-9	" "
d.	Aromatics	Max. 0.2 Wt.%	Zx-9	" "
e.	Olefines	Max. 0.5 Br. No.	Zx-21	" "

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Manager, Research and Engineering

* South Hampton Co.
Times Center Bldg.
2444 Times Blvd.
P. O. Box 6966
Houston, Texas 77005

NITRIC ACID (60%) NOMINAL

CPD Material Number 0670

Purchase Specifications

a. Nitric acid, HNO_3	Min. 57.0 Wt.%
b. Nitric Oxides	Max. 0.05 Wt.%
c. Chloride, (Cl^-)	Max. 0.05 Wt.%
d. Residue on evaporation	Max. 0.01 Wt.%
e. Calcium (Ca)	Max. 0.00012 Wt.%
f. Strontium (Sr)	Max. 0.000012 Wt.%
g. Phosphate ($PO_4^{\equiv}$)	Max. 0.003 Wt.%

Acceptance Specifications

A 4 oz. sample of each truck delivery should be taken. A composite of ten truck samples should be analyzed as follows. A 4 oz. sample of each tank car should be taken. Each tank car should be analyzed as follows:

	Test Requirements		
	Value	Method	Frequency
a. Nitric acid, HNO_3	Min. 57.0 Wt.%	H-1	Each composite or car
b. Nitric oxides*	Max. 0.05 Wt.%	N-11	" " " "
c. Chloride (Cl^-)	Max. 0.05 Wt.%	Cl-5	" " " "
d. Residue on evaporation	Max. 0.01 Wt.%	Zy-22	" " " "
e. Calcium(Ca)(Purex only)	Max. 0.00012 Wt.%	Zy-25	One composite per mo.
f. Strontium(Sr)(Purex only)	Max. 0.000012 Wt.%	Zy-25	" " " "
g. Phosphate ($PO_4^{\equiv}$)(Purex only)	Max. 0.003 Wt.%	P-2	" " " "

* Tested at Redox only on request of Process Engineers.

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NITRIC ACID (72%)

CPD Material 0671

Purchase Specifications

a.	Nitric acid, HNO_3	Min. 72.0 Wt.%
b.	Nitric oxides	Max. 0.05 Wt.%
c.	Chloride, (Cl^-)	Max. 0.05 Wt.%
d.	Residue on evaporation	Max. 0.02 Wt.%
e.	Specific gravity 20/4	Min. 1.422

Note: A vendor's certified analysis should be requested on the purchase requisition.

Acceptance Specifications

A vendor's certified analysis showing conformance to specifications is adequate for acceptance. Copy coverage to the consumer should be supplied by the Purchasing Department on request of Process Engineering.

Routine determination of Specific gravity should be carried out as well.

In the absence of vendor's analysis, a 16 oz. sample of each shipment (Method C) should be analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Nitric acid, HNO_3	Min. 72.0 Wt.%	H-1	On request of process engineers
b.	Nitric oxides	Max. 0.05 Wt.%	N-11	
c.	Chloride (Cl^-)	Max. 0.05 Wt.%	Cl-5	" "
d.	Residue on evaporation	Max. 0.02 Wt.%	Zy-22	" "
e.	Specific gravity 20°/4	Min. 1.422	Zy-10	Each shipment

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O. F. Beaulieu

Manager, Separations Chemistry Laboratory

Approved by

W. S. Frank

Manager, Research and Engineering

OXALIC ACID

CPD Material Number 0690

Purchase Specifications

a.	Oxalic acid, technical dihydrate $(COOH)_2 \cdot 2H_2O$	Min. 99.5 Wt.%
b.	Water insolubles	Max. 0.05 Wt.%
c.	R_2O_3	Max. 0.05 Wt.%
d.	Chloride (Cl^-)	Max. 0.01 Wt.%
e.	Calcium (Ca)	Max. 0.007 Wt.%
f.	Strontium (Sr)	Max. 0.0005 Wt.%

Acceptance Specifications

An 8 oz. sample of each shipment should be taken. (Sample Method B)

		Test Requirements		
		Value	Method	Frequency
a.	Oxalic acid dihydrate $(COOH)_2 \cdot 2H_2O$	Min. 99.5 Wt.%	Zx-3	Each shipment
b.	Water insolubles (Z plant only)	Max. 0.05 Wt.%	Zy-14	Each shipment
c.	R_2O_3	Max. 0.05 Wt.%	Fe-1	On request of Process Engineers
d.	Chloride (Cl^-)	Max. 0.01 Wt.%	Cl-5	Each shipment
e.	Calcium (Ca)(Purex only)	Max. 0.007 Wt.%	Zy-25	Each shipment
f.	Strontium(Sr)(Purex only)	Max. 0.0005 Wt.%	Zy-25	Each shipment

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OXYGEN

CPD Material Number 0700

Purchase Specifications

a.	Oxygen, O_2, commercial grade Assay	 Min. 99.5 Vol. %

Acceptance Specifications

A representative sample by sampling method J may be taken on request of Process Engineers.

A vendor's certified analysis of purity is adequate for acceptance.

		Test Requirements		
		Value	Method	Frequency
a.	Oxygen, O_2	Min. 99.5 Vol.%	Zz-1	On request of Process Engineers

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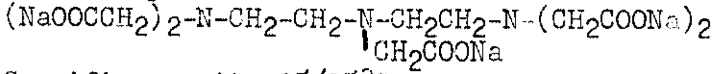
JUN 26 1964

DTPA(Sodium Salt)

CPD Material Number 0705

Purchase Specifications

a. Aqueous solution of pentasodium diethylene triamine pentacetate — Min. 40 Wt.%



$(NaOOCCH_2)_2\text{-}N\text{-}CH_2\text{-}CH_2\text{-}\underset{|\,CH_2COONa}{N}\text{-}CH_2CH_2\text{-}N\text{-}(CH_2COONa)_2$

b. Specific gravity 25/25°C — Min. 1.30

c. pH: — 11.5 - 12.0

d. $CaCO_3$ value: Mgm $CaCO_3$/gram — 78 to 80

Acceptance Specifications

A 16 oz. sample (Method D) of each shipment should be taken and analyzed as follows:

	Test Requirements		
	Value	Method	Frequency
a. Specific gravity 25/25°C	Min. 1.30	Zy-10	Each shipment
b. pH	~~Approx.~~ 11.5-12.0	pH meter	" "
c. $CaCO_3$ value, Mgm $CaCO_3$/g	78-80	V-1	" "

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OF Beaulieu

Manager, Separations Chemistry Laboratory

Approved by

H. S. Frank

Manager, Research and Engineering

POTASSIUM FLUORIDE

CPD Material Number 0713

Purchase Specifications

a.	Potassium Fluoride (KF)	Minimum	95 Wt.%
b.	Chloride (Cl^-)	Maximum	0.1 Wt.%

Acceptance Specifications

A vendor's certified analysis showing conformance to specifications "a" and "b" is adequate for acceptance. Copy coverage to the consumer should be supplied by the Purchasing Department or CPD Production and Scheduling Operation.

In the absence of a vendor's analysis, a 4 oz. sample (Method B) should be taken and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Potassium Fluoride	Min. 95 Wt.%	F-2	Each shipment
b.	Chloride	Max. 0.1 Wt.%	Cl-5	Each shipment

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Manager, Research and Engineering

PETROLEUM DILUENT

CPD Material Number 720

Purchase Specifications

a.	Soltrol-170 A proprietary material of the Phillips Petroleum Co.	
b.	Flash point	175-190 F
c.	Specific gravity	0.77-0.78 60/60 F
d.	Viscosity	Max. 2.7 centipoises/25 C
e.	Bromine Number	Max. 1.5
f.	Aromatics	Max. 1.0 Vol. %

Acceptance Specifications

A 16 oz. sample of each tank car (Method C) should be taken and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Specific Gravity	0.77-0.78 60/60 F	Zy-10A	Each shipment
b.	Viscosity	Max. 2.7 Centipoises/25 C	Zy-9	Each shipment
c.	Olefines	Bromine No. ≤1.5	Zx-21	Each shipment
d.	Aromatics	Max. 1.0 Vol.%	Zx-9	Each shipment

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Manager, Research and Engineering

POTASSIUM BICARBONATE

CPD Material Number 743

Purchase Specifications

a.	Potassium bicarbonate ($KHCO_3$)	Minimum 99.5 Wt.% Maximum 100.5 Wt.%
b.	Chloride	Maximum 0.01 Wt.%
c.	Calcium	Maximum 0.01 Wt.%
d.	Phosphate	Maximum 0.003 Wt.%
e.	Water insolubles	Maximum 0.01 Wt.%

Acceptance Specifications

		Value	Test Method	Testing Frequency
a.	Alkalinity as $KHCO_3$	Min. 99.5 Wt.% Max. 100.5 Wt.%	H-11	Each shipment
b.	Chloride	Max. 0.01 Wt.%	Cl-5	Each shipment
c.	Calcium	Max. 0.01 Wt.%	Ca-2	Each shipment
d.	Phosphate	Max. 0.003 Wt.%	P-2	Each shipment
e.	Water insolubles	Max. 0.01 Wt.%	Zy-14	Each shipment

An 8 oz. sample of each shipment should be taken.

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PHOSPHORIC ACID (85%)

CPD Material Number 0744

Purchase Specifications

a.	Phosphoric Acid (H_3PO_4)	Minimum	85 Wt. %
b.	Chloride (Cl^-)	Maximum	0.1 Wt.%

Acceptance Specifications

A vendor's certified analysis showing conformance to specifications "a" and "b" is adequate for acceptance. Copy coverage to the consumer should be supplied by the Purchasing Department or CPD Production and Scheduling Operation.

In the absence of a vendor's analysis, an 8 oz. sample (Method D) should be taken and analyzed as follows:

			Test Requirements	
		Value	Method	Frequency
a. Phosphoric Acid	Min.	85 Wt.%	H-1	Each shipment
b. Chloride	Max.	0.1 Wt.%	Cl-5	Each shipment

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POTASSIUM PERMANGANATE

CPD Material Number 0750

Purchase Specifications

a.	Potassium permanganate, $KMnO_4$ Assay; $KMnO_4$	Min. 99.0 Wt.%
b.	Water insolubles	Max. 0.20 Wt.%
c.	Strontium	Max. 20 ppm
d.	Calcium and/or magnesium	Max. 100 ppm

Acceptance Specifications

A 4 oz. sample from each shipment should be analyzed as follows (Sampling Method B).

			Test Requirements	
		Value	Method	Frequency
a.	Potassium permanganate, $KMnO_4$	Min. 99.0 Wt.%	Mn-1	Each shipment
b.	Water insolubles	Max. 0.20 Wt.%	Zy-14	" "
c.	Strontium	Max. 0.002 Wt.% (20 ppm)	Zy-25	" "
d.	Calcium and/or magnesium	Max. 0.01 Wt.% (100 ppm)	Zy-25	" "

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SODIUM (OR POTASSIUM) PERSULFATE

CPD Material Number 0752

Purchase Specifications

a.	Sodium (or potassium) persulfate $Na_2S_2O_8$ or $K_2S_2O_8$	Min. 97 Wt.%
b.	Chloride	Max. 0.01 Wt.%
c.	Insolubles	Max. 0.01 Wt.%

Acceptance Specifications

A 4 oz. sample (Method B) should be taken and analyzed as follows:

		Test Requirements		
		Value	Method	Frequency
a.	Persulfate	Min. 97% formula Wt.	O-6	Each shipment
b.	Chloride	Max. 0.01 Wt.%	Cl-5	" "
c.	Insolubles	Max. 0.01 Wt.%	Zy-14	" "

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Manager, Separations Chemistry Laboratory

Approved by

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Manager, Research and Engineering

PROPANE

CPD Material Number 0760

Purchase Specifications

a. Commercial propane gas.

Acceptance Specifications

This material is not routinely sampled.

a. On request of Power Department supervision, Separations Chemistry Laboratory will arrange for sampling and testing of this material.

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Manager, Separations Chemistry Laboratory	Manager, Research and Engineering

SODIUM CHLORIDE (ROCK SALT)

CPD Material Number 0770

Purchase Specifications

a. Rock salt, NaCl, commercial grade.

Acceptance Specifications

An 8 oz. sample (Method A) may be taken and analyzed as follows.

		Test Requirements	
	Value	Method	Frequency
a. NaCl content	-	Cl-1	On request of Power Dept. supervision

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SILICON NITRIDE CERAMIC

CPD Material Number 774

Purchase Specifications

a. Bulk density	Minimum 2.2 g/cc
b. Hardness	Minimum Rockwell A55
c. Modulus of rupture (Cross bending strength)	Minimum 9000 psi

Acceptance Specifications

Separations Chemistry Laboratory will arrange for special sampling and testing on request.

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SILVER NITRATE

CPD Material Number 0775

Purchase Specifications

a.	Silver Nitrate, $AgNO_3$	Min. 99.8 Wt.%
b.	Chloride (Cl^-)	Max. 0.002 Wt.%
c.	Substances not precipitated by HCl	Max. 0.05 Wt.%

Acceptance Specifications

An 8 oz. sample from each shipment should be analyzed as follows. (Sample Method B)

		Test Requirements		
		Value	Method	Frequency
a.	Silver Nitrate	Min. 99.8 Wt.%	Ag-1	On request of Proc. Engrs.
b.	Chloride (Cl^-)	Max. 0.002 Wt.%	Cl-5	" " "
c.	Substances not precipitated by HCl	Max. 0.05 Wt.%	Zy-17	" " "

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SODIUM ACETATE

CPD Material 0780

Purchase Specifications

a.	Sodium acetate trihydrate $NaC_2H_3O_2.3H_2O$	Min. 99 Wt.%
b.	Residue on drying	Min. 58.9 Wt.%
c.	Chloride	Max. 0.01 Wt.%
d.	Calcium-Magnesium - as calcium	Max. 0.01 Wt.%

Acceptance Specifications

A 16 oz. sample of each shipment (Method B) should be taken and analyzed as follows.

		Test Requirements		
		Value	Method	Frequency
a.	Residue on drying	Min. 58.9 Wt.%	Zy-22	Each shipment
b.	Chloride	Max. 0.01 Wt.%	Cl-5	" "
c.	Calcium-magnesium as calcium	Max. 0.01 Wt.%	Ca-2	" "

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SODIUM BISMUTHATE

CPD Material #0782

Purchase Specifications

a.	Sodium Bismuthate, $NaBiO_3$	Min.	80 Wt. %
b.	Chloride	Max.	0.01 Wt.%
c.	Insolubles	Max.	0.10 Wt.%
d.	Fineness - to pass 200 mesh	Min.	95.0 Wt.%

Acceptance Specifications

An 8 oz. sample of each shipment should be taken (Method B).

		Test Requirements		
		Value	Method	Frequency
a.	Sodium Bismuthate	Min. 80 Wt. %	0-5	Each shipment
b.	Chloride	Max. 0.01 Wt.%	Cl-5	" "
c.	Insolubles	Max. 0.10 Wt.%	Zy-16	On request of process engineers
d.	Fineness	Min. 95% through 200 mesh	Zy-24	" " "

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SODIUM CARBONATE

CPD Material Number 0785

Purchase Specifications

a. Sodium Carbonate, technical, Na_2CO_3 Na_2O content	Min. 57.3 Wt.%
b. Water insolubles	Max. 0.10 Wt.%
c. Calcium (Ca)	Max. 0.004 Wt.% (40 ppm)
d. Strontium (Sr)	Max. 0.001 Wt.% (10 ppm)

Acceptance Specifications

An 8 oz. sample of each shipment should be analyzed as follows. (Sample Method B)

	Test Requirements		
	Value	Method	Frequency
a. Sodium Carbonate, Na_2CO_3 Na_2O content	Min. 57.3 Wt.%	H-11	On request of Proc. Engineers
b. Water insolubles	Max. 0.10 Wt.%	Zy-14	Each shipment
c. Calcium (Ca)	Max. 0.004 Wt.%	Zy-25	Each shipment
d. Strontium (Sr)	Max. 0.001 Wt.%	Zy-25	Each shipment

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SODIUM DICHROMATE

CPD Material Number 795

PROPERTIES

			Testing Methods
*a.	Sodium dichromate, $Na_2Cr_2O_7 \cdot 2H_2O$ Assay: min.,	98.0%	Cr-1
*b.	Sulfate ($SO_4^{=}$): max.,	0.5%	S-25
*c.	Water insolubles: max.,	0.1%	Zy-14
d.	Red, somewhat deliquescent crystals. Loses water of crystallization at 100 C.		

* Properties a through c required by specification.

SAMPLE

16 oz. sample of each shipment - Method B.

HAZARDOUS PROPERTIES

This material is toxic and dangerous to the eyes.

SODIUM DICHROMATE (SOLUTION)

CPD Material Number 0796

Purchase Specifications

a.	Sodium dichromate solution $Na_2Cr_2O_7.2H_2O$ content	Min. 65 Wt.%
b.	Sulfate ($SO_4^{=}$)	Max. 0.4 Wt.%
c.	Water insolubles	Max. 0.1 Wt.%

Acceptance Specifications

An 8 oz. sample from each shipment should be taken (Sampling Method C) and analyzed as follows.

			Test Requirements	
		Value	Method	Frequency
a.	Sodium dichromate $Na_2Cr_2O_7.2H_2O$	Min. 65 Wt.%	Cr-1	Each shipment
b.	Sulfate ($SO_4^{=}$)	Max. 0.4 Wt.%	S-25	" "
c.	Water insolubles	Max. 0.1 Wt.%	Zy-14	" "

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SODIUM FLUORIDE

CPD Material Number 0799

Purchase Specifications

a.	Sodium Fluoride, NaF	Min. 95 Wt.%
b.	Water insolubles	Max. 0.1 Wt.%
c.	Iron, Fe	Max. 0.07 Wt.%
d.	Sodium fluosilicate (Na_2SiF_6)	Max. 0.35 Wt.%
e.	Free alkali as Na_2CO_3	Max. 0.3 Wt.%
f.	Chloride	Max. 0.1 Wt.%
g.	Calcium	Max. 0.02 Wt.% (200 ppm)
h.	Strontium	Max. 0.01 Wt.% (100 ppm)

Acceptance Specifications

A 4 oz. sample of each shipment should be analyzed as follows (Sampling Method B).

		Test Requirements		
		Value	Method	Frequency
a.	Sodium Fluoride, NaF	Min. 95 Wt.%	F-1	On request of Proc. Engineers
b.	Water insolubles	Max. 0.1 Wt.%	*	" " " "
c.	Iron, Fe	Max. 0.07 Wt.%	*	" " " "
d.	Sodium Fluosilicate	Max. 0.35 Wt.%	*	" " " "
e.	Free alkali as Na_2CO_3	Max. 0.3 Wt.%	*	" " " "
f.	Chloride	Max. 0.1 Wt. %	Cl-5	Each shipment
g.	Calcium (Purex only)	Max. 0.02 Wt.%	Zy-25	" "
h.	Strontium (Purex only)	Max. 0.01 Wt.%	Zy-25	" "

* Separations Chemistry will arrange for special testing on request.

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SODIUM FLUORIDE

CPD Material Number 799

PROPERTIES

			Testing Methods
*a.	Sodium Fluoride, NaF, Commercial grade, Assay: min.,	95%	F-1
*b.	Water insolubles: max.,	0.1%	--
*c.	Iron as Fe_2O_3: max.,	0.1%	--
*d.	Sodium fluosilicate (Na_2SiF_6): max.,	0.35%	--
*e.	Free alkali as Na_2Co_3: max.,	0.3%	--
*f.	Chloride (Cl): max.,	0.1%	Cl-5
g.	Clear, lustrous crystals or white powder		

* Properties a through f required by specification. On-plant analysis required for properties a and f only.

SAMPLE

8 oz. shipment of each sample - Method B

HAZARDOUS PROPERTIES

Sodium fluoride is poisonous. Damage may be caused either by contact with the skin or by ingestion.

SODIUM HYDROXIDE (CAUSTIC SODA) (50%)

CPD Material Number 0800

Purchase Specifications

a.	Sodium hydroxide, NaOH (Aq. solution) commercial grade	Na_2O content, 38.9 ± 1 Wt.%
b.	Chloride (Cl^-)	Max. 0.9 Wt.%
	or as NaCl	Max. 1.5 Wt.%
c.	Calcium (Ca)	Max. 0.002 Wt.%
d.	Strontium (Sr)	Max. 0.001 Wt.%

Acceptance Specifications

An 8 oz. sample of each delivery should be taken and analyzed as follows (Sampling Method C).

		Test Requirements		
		Value	Method	Frequency
a.	Sodium hydroxide, Na_2O content	38.9 ± 1 Wt.%	H-11	Each shipment
b.	Chloride (Cl^-)	Max. 0.9 Wt.%	Cl-5	Each shipment
c.	Calcium (Ca)(Purex only)	Max. 0.002 Wt.%	Zy-25	" "
d.	Strontium (Purex only)	Max. 0.001 Wt.%	Zy-25	" "

Material transferred from Redox to Z-plant should not exceed 0.1 Wt.% water insolubles. Testing for this to be as requested by Z-plant process engineers using method Zy-14.

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

SODIUM HYDROXIDE (REAGENT GRADE)

CPD Material Number 0802

Purchase Specifications

a. Solid sodium hydroxide, NaOH — Minimum 97.0 Wt.%

b. Must meet A.C.S. Specifications for reagent chemicals.

Acceptance Specifications

a. Sodium hydroxide*

b. Must conform to "A.C.S. Specifications for Reagent Chemicals."

* Separations Chemistry Laboratory will arrange for special sampling and testing on request.

Issued by

Manager
Separations Chemistry Laboratory

Approved by

Manager
Research and Engineering

JUN 19 1964

SODIUM NITRATE

CPD Material Number 0805

Purchase Specifications

a. Sodium Nitrate, $NaNO_3$	Min. 99.0 Wt.%
b. Water insolubles	Max. 0.10 Wt.%
c. Chloride (Cl^-)	Max. 0.50 Wt.%
d. Total iodine (I)	Max. 0.0005 Wt.%

Acceptance Specifications

A 16 oz. sample of each shipment (Method B) should be taken and analyzed as follows:

	Test Requirements		
	Value	Method	Frequency
a. Sodium Nitrate, $NaNO_3$	Min. 99.0 Wt.%	N-21	Each shipment
b. Water insolubles	Max. 0.10 Wt.%	Zy-14	Each shipment
c. Chloride (Cl^-)	Max. 0.50 Wt.%	Cl-5	Each shipment
d. Total Iodine (I)	Max. 0.0005 Wt.%	I-1	Each shipment

Issued by

Manager, Separations Chemistry Laboratory

Approved by

W. S. Frank
Manager, Research and Engineering

SODIUM NITRITE

TPD Material Number 0810

Purchase Specifications

a.	Sodium Nitrite, $NaNO_2$	Min. 99 Wt.%
b.	Moisture Content	Max. 0.30 Wt.%
c.	Water insolubles	Max. 0.05 Wt.%
d.	Calcium (Ca)	Max. 0.002 Wt.%
e.	Strontium (Sr)	Max. 0.0002 Wt.%

Acceptance Specifications

An 8 oz. sample of each shipment should be analyzed as follows. (Sample Method B.)

			Test Requirements	
		Value	Method	Frequency
a.	Sodium Nitrite, $NaNO_2$	Min. 99 Wt.%	N-10	Each shipment
b.	Moisture Content	Max. 0.30 Wt.%	0-2	On request of Process Engineers
c.	Water insolubles	Max. 0.05 Wt.%	Zy-14	" " "
d.	Calcium (Ca)(Purex only)	Max. 0.002 Wt.%	Zy-25	Each shipment
e.	Strontium (SR)(Purex only)	Max. 0.0002 Wt.%	Zy-25	Each shipment

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

SODIUM SULFATE, ANHYDROUS

CPD Material Number 0820

Purchase Specifications

a.	Anhydrous Sodium Sulfate, Na_2SO_4	Min. 99.0 Wt.%
b.	Chloride (Cl^-)	Max. 0.2 Wt.%
c.	Calcium (Ca)	Max. 0.001 Wt.%
d.	Strontium (Sr)	Max. 0.0002 Wt. %

Acceptance Specifications

An 8 oz. sample of each shipment should be analyzed as follows. (Sample Method B.)

		Test Requirements		
		Value	Method	Frequency
a.	Chloride	Max. 0.2 Wt.%	Cl-5	On request of Process Engineers
b.	Calcium	Max. 0.001 Wt.%	Zy-25	Each shipment
c.	Strontium	Max. 0.0002 Wt.%	Zy-25	Each shipment

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

SODIUM NITRITE

CPD Material Number 0810

Purchase Specifications

a.	Sodium Nitrite, $NaNO_2$	Min. 99 Wt.%
b.	Moisture Content	Max. 0.30 Wt.%
c.	Water insolubles	Max. 0.05 Wt.%
d.	Calcium (Ca)	Max. 0.002 Wt.%
e.	Strontium (Sr)	Max. 0.0002 Wt.%

Acceptance Specifications

		Value	Testing Method	Testing Frequency
a.	Sodium Nitrite, $NaNO_2$	Min. 99 Wt.%	N-10	Each shipment
b.	Moisture Content	Max. 0.30 Wt.%	0-2	On request
c.	Water insolubles	Max. 0.05 Wt.%	Zy-14	On request
d.	Calcium (Ca)	Max. 0.002 Wt.%	Zy-25	Each shipment
e.	Strontium (Sr)	Max. 0.0002 Wt.%	Zy-25	Each shipment

An 8 oz. sample of each shipment should be taken.

Issued by	Approved by
Mgr. Separations Chemistry Laboratory	Mgr. Research and Engineering

SODIUM SULFATE, ANHYDROUS

CPD Material Number 0820

Purchase Specifications

a.	Anhydrous Sodium Sulfate, Na_2SO_4	Min. 99.0 Wt.%
b.	Chloride (Cl^-)	Max. 0.20 Wt.%
c.	Calcium (Ca)	Max. 0.003 Wt.%
d.	Strontium (Sr)	Max. 0.0006 Wt.%

Acceptance Specifications

An 8 oz. sample of each shipment should be analyzed as follows: (Sample Method B)

		Test Requirements		
		Value	Method	Frequency
a.	Chloride	Max. 0.20 Wt. %	Cl-5	On request of Process Engineers
b.	Calcium	Max. 0.003 Wt.%	Zy-25	Each shipment
c.	Strontium	Max. 0.0006 Wt.%	Zy-25	Each shipment

Issued by

Manager, Separations Chemistry Laboratory

Approved by

BF Judson

for Manager, Research and Engineering

SODIUM SULFITE

CPD Material Number 0825

Purchase Specifications

a.	Sodium sulfite, Na_2SO_3 (anhydrous) commerical grade	Min. 85 Wt.%
b.	Water insolubles	Max. 0.5 Wt.%

Acceptance Specifications

On request of Power Department supervision, a 16 oz. sample of a shipment should be taken (Method B) and analyzed as follows.

		Test Requirements		
		Value	Method	Frequency
a.	Sodium sulfite (Na_2SO_3)	Min. 85 Wt.%	S-11	On request of Power Dept. supervision
b.	Water insolubles	Max. 0.5 Wt.%	Zy-14	" "

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

SUGAR (White, granulated)

CPD Material Number 0842

Purchase Specifications

a.	Sugar content	Min. 99.5 Wt.%
b.	Calcium	Max. 16 ppm
c.	Strontium	Max. 3 ppm

Acceptance Specifications

An 8 oz. sample of each shipment should be taken using sampling Method B.

			Test Requirements	
		Value	Method	Frequency
a.	Sugar content	Min. 99.5 Wt.%	SpG of 50% Sol*	On request of Process Engineers
b.	Calcium	Max. 16 ppm	Zy-25	Each shipment
c.	Strontium	Max. 3 ppm	Zy-25	Each shipment

* Use 20/20° tables from Handbooks.

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

SULFAMIC ACID

CPD Material Number 0870

Purchase Specifications

a.	Sulfamic acid, NH_2SO_3H - Assay	Min. 97 Wt.%
b.	Chloride (Cl^-)	Max. 0.02 Wt.%
c.	Sulfate ($SO_4^=$)	Max. 1.0 Wt.%
d.	Nitrate (NO_3^-)	Max. 0.1 Wt.%
e.	Water insolubles	Max. 0.1 Wt.%
f.	Tin (Sn)	Max. 0.01 Wt.%
g.	Heavy metals (as Pb)	Max. 0.04 Wt.%
h.	Calcium (Ca)	Max. 0.004 Wt.% (40 ppm)
i.	Strontium	Max. 0.001 Wt.% (10 ppm)

Acceptance Specifications

A 16 oz. sample of each shipment should be analyzed as follows: (Sample Method B)

			Test Requirements	
		Value	Method	Frequency
a.	Sulfamic acid, NH_2SO_3H	Min. 97 Wt.%	Sa-1	Each shipment
b.	Chloride (Cl^-)	Max. 0.02 Wt.%	Cl-5	On request of Proc. Engineers
c.	Sulfate ($SO_4^=$)	Max. 1.0 Wt.%	S-25	" " "
d.	Nitrate (NO_3^-)	Max. 0.1 Wt.%	N-23	" " "
e.	Water insolubles	Max. 0.1 Wt.%	Zy-14	" " "
f.	Tin (Sn)	Max. 0.01 Wt.%	Sn-1	" " "
g.	Heavy metals (as Pb)	Max. 0.04 Wt.%	Pb-2	" " "
h.	Calcium (Ca)(Purex only)	Max. 0.004 Wt.%	Zy-25	Each shipment
i.	Strontium(Sr)(Purex only)	Max. 0.001 Wt.%	Zy-25	Each shipment

Issued by

Manager, Separations Chemistry Laboratory

Approved by

W. S. Frank

Manager, Research and Engineering

SULFURIC ACID (93%)

CPD Material Number 0880

Purchase Specifications

a. Sulfuric acid, H_2SO_4 commercial grade H_2SO_4 content	Min. 93 Wt.%

Acceptance Specifications

On request of Process Engineering, a 16 oz. sample of each shipment should be taken (Method C) and analyzed as follows.

	Test Requirements		
	Value	Method	Frequency
a. H_2SO_4 content	Min. 93 Wt. %	H-1	On request of process engineers

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

SULFURIC ACID (C.P.)

CPD Material Number 0881

Purchase Specifications

a.	Sulfuric acid, H_2SO_4(aq.) C.P.	
	H_2SO_4 content	Min. 92 Wt.%

Acceptance Specifications

This material is obtained from Stores in small quantities: Sampling and testing will be arranged by the Separations Chemistry Laboratory on request of Process Engineering.

Issued by

Manager, Separations Chemistry Laboratory

Approved by

Manager, Research and Engineering

HYDRAZINE SOLUTION

CPD Material Number 0890

Purchase Specifications

a.	Hydrazine content, $NH_2 \cdot NH_2$	Min. 35 Wt.%
b.	Chloride (Cl^-)	Max. 0.05 Wt.%
c.	Calcium (Ca)	Max. 0.0007 Wt.% (20 ppm)
d.	Strontium (Sr)	Max. 0.00007 Wt.% (2 ppm)

Acceptance Specifications

An 8 oz. sample of a shipment should be analyzed as follows. (Sample Method D)

		Test Requirements		
		Value	Method	Frequency
a.	Hydrazine content, NH_2NH_2	Min. 35 Wt.% 357 g/l	N-25	Each shipment
b.	Chloride (Cl^-)	Max. 0.05 Wt.%	Cl-5	Each shipment
c.	Calcium	Max. 0.0007 Wt.%	Zy-25	Each shipment
d.	Strontium	Max. 0.00007 Wt.%	Zy-25	" "

Issued by

C. F. Beaulieu

Manager, Separations Chemistry Laboratory

Approved by

W. S. Frank

Manager, Research and Engineering

HYDRAZINE SOLUTION (35%)

CPD Material Number 890

PROPERTIES			Wt. %	Testing Method
a.	Hydrazine	$H2N-NH_2$		
	Assay:	minimum	35	N-25
b.	Sp.Gr.	Minimum	$1.021\frac{25^{\circ}}{4^{\circ}}$	
c.	Chlorides:	Maximum	0.05	Cl-5
d.	Appearance:	Colorless liquid		

Properties a, c required by specification. Properties a and c on each shipment. Property b on request.

SAMPLE

Eight ounces of each shipment. Method D may be used.

HAZARDOUS PROPERTIES

Hydrazine is toxic. Inhalation of vapors and contact with skin should be avoided.

Issued by:

O. F. Beaulieu
Manager, Process Chemistry

Approved by:

R. E. Tomlinson
Acting Manager, Research and Engineering

TARTARIC ACID

CPD Material Number 0910

Purchase Specifications

a.	Tartaric Acid $HOOC(CHOH)_2COOH$	Min. 99.4 Wt.%
b.	Chloride (Cl^-)	Max. 0.01 Wt.%
c.	Residue on ignition	Max. 0.02 Wt.%

Acceptance Specifications

A 16 oz. sample of each shipment (Method B) should be taken and analyzed as follows.

		Test Requirements		
		Value	Method	Frequency
a.	Tartaric Acid $HOOC(CHOH)_2COOH$	Min. 99.4 Wt.%	H-1	Each shipment
b.	Chloride	Max. 0.01 Wt.%	Cl-5	" "
c.	Residue on ignition	Max. 0.02 Wt.%	Zy-23	On request of process engineers

Issued by

Beaulieu

Manager, Separations Chemistry Laboratory

Approved by

W. S. Frank

Manager, Research and Engineering

TANTALUM METAL

CPD Material Number 912

Purchase Specifications

a. Tantalum metal, various shapes. Minimum 99.8 Wt.%.

Conforming to ASTM Designation: B364-61T particularly as to the following chemical requirements and tensile properties.

b. Chemical impurities

Carbon	Maximum	0.03 Wt.%
Oxygen	Max.	0.03 Wt.%
Nitrogen	Max.	0.015 Wt.%
Hydrogen	Max.	0.01 Wt.%
Niobium	Max.	0.10 Wt.%
Iron	Max.	0.02 Wt.%
Titanium	Max.	0.01 Wt.%
Tungsten	Max.	0.03 Wt.%
Silicon	Max.	0.02 Wt.%
Nickel	Max.	0.02 Wt.%

c. Tensile properties

Conditions:	Ultimate Strength:
Cold worked	Minimum 75,000 psi
Stress relieved	Minimum 55,000 psi
Fully annealed	Minimum 35,000 psi

Acceptance Specifications

Separations Chemistry Laboratory will arrange for special sampling and testing on request.

Issued by

Manager, Sep. Chem. Lab.

Approved by

H. S. Frank

Manager, Research and Engineering

TRIBUTYL PHOSPHATE

CPD Material Number 0920

Purchase Specifications

a. Tributyl Phosphate $(C_4H_9)_3PO_4$	
b. Butanol (reducing normality)	Max. 0.05 Wt.%
c. Acidity (mono and dibutyl phosphates as normality)	Max. 0.02 N
d. Suspended solids	Max. 0.01 Wt.%
e. Specific gravity	0.973 to 0.983 at 20/20 C
f. Water: No turbidity when one volume TBP is mixed with 19 volumes of 60° Be gasoline at 20 C	

Acceptance Specifications

A 16 oz. sample of each shipment (Method C or D) should be taken and analyzed as follows.

	Test Requirements		
	Value	Method	Frequency
a. Butanol (reducing normality)	0.05 Wt.%	Zx-5	Each shipment
b. Acidity (mono and dibutyl phosphates as normality	Max. 0.02 N	H-2	" "
c. Suspended solids	Max. 0.01 Wt.%	Zy-15	" "
d. Specific gravity	0.973-0.983 20/20	Zy-10	" "
e. Water: No turbidity when one volume TBP is mixed with 19 volumes of 60° Be gasoline at 20 C	---	Zy-1	" "

Issued by

Manager, Separations Chemistry Laboratory

Approved by

H. S. Frank

Manager, Research and Engineering

TRICHLOROETHYLENE (TECH.)

CPD Material Number 0930

Purchase Specifications

a.	Trichloroethylene Technical Grade, Type I Regular	
b.	Boiling range at 760 mm	86.0 C to 90 C
c.	Non-volatile residue	Max. 0.004 Wt.%
d.	Moisture content	Max. 100 ppm
e.	Free chlorine	Max. 0.01%
f.	Specific gravity	1.450 to 1.475 $\frac{20}{20}$ C
g.	Color	Clear
h.	Appearance	Free from suspended matter or sediment

Acceptance Specifications

A 16 oz. sample from each drum shall be analyzed as follows: (Method D)

		Test Requirements		
		Value	Method	Frequency
b.	Boiling range at 760 mm	86.0 to 90.0 C	Zy-3	Each drum
c.	Non-volatile residue	Max. 0.004%	Zy-22	Each drum
d.	Moisture content	Max. 100 ppm	No turbidity at 0 C for 10 min.	Each drum
e.	Free chlorine	Max. 0.01%	Cl-2	Each drum
f.	Specific gravity	1.450 to 1.475 $\frac{20}{20}$ C	Zy-10	Each drum

Issued by

Manager, Separations Chemistry Laboratory

Approved by

R. E. Tomlinson
Manager, Research and Engineering

P-1 VESSEL

CPD Material Number 0931

Purchase Specifications

a. Vessels must meet specifications present on drawing H-2-23835.

b. Material is to be silicon nitride ceramic (CPD Material #0774) with the following specifications:

1.	Bulk density	Min. 2.2 g/cc
2.	Hardness	Min. Rockwell A55
3.	Modulus of Rupture	Min. 9000 psi cross bending strength

c. Vessels must be free of loose oxides and residue.

Notes:

1. A vendor's certification, including test results, should be procured for item (b) with copy coverage to Weapons Fabrication Operation.

2. A mercury leak test of the plug seat is not required. However, the customer will lend the vendor a tantalum plug for seat testing if desired.

3. New vendors must submit a minimum of five vessels for process evaluation before their product can be given consideration.

Acceptance Specifications

a. Five per cent of the vessels in a lot or shipment should be taken as a sample and delivered to Weapons Fabrication Operation.

b. Weapons Fabrication Operation will perform leak tests and line testing on samples prior to acceptance.

c. The vendor's certificate will normally suffice for acceptance under item (b).

Issued by

Manager, Separations Chemistry Laboratory

Approved by

W. S. Frank

Manager, Research and Engineering

SECTION III

STANDARD SAMPLING PROCEDURES
FOR
ESSENTIAL MATERIALS

II. STANDARD METHODS FOR SAMPLING ESSENTIAL MATERIALS

Method A - Standard Procedure for Sampling Solids in Bulk (other than coal)

1. Safety Precautions

   a. Gloves should be worn to protect the hands and coverall goggles to protect the eyes.

   b. When sampling "Lime" a respirator should be worn to protect the membranes of the nose and throat from irritation.

   c. In general, plant safety rules applicable to the handling of materials also apply to this sampling method.

2. Equipment Required

   a. Clean, dry, wide mouth bottles as specified.

   b. Shovel.

   c. Sampling thief as per attached sketch.

3. Sampling procedure

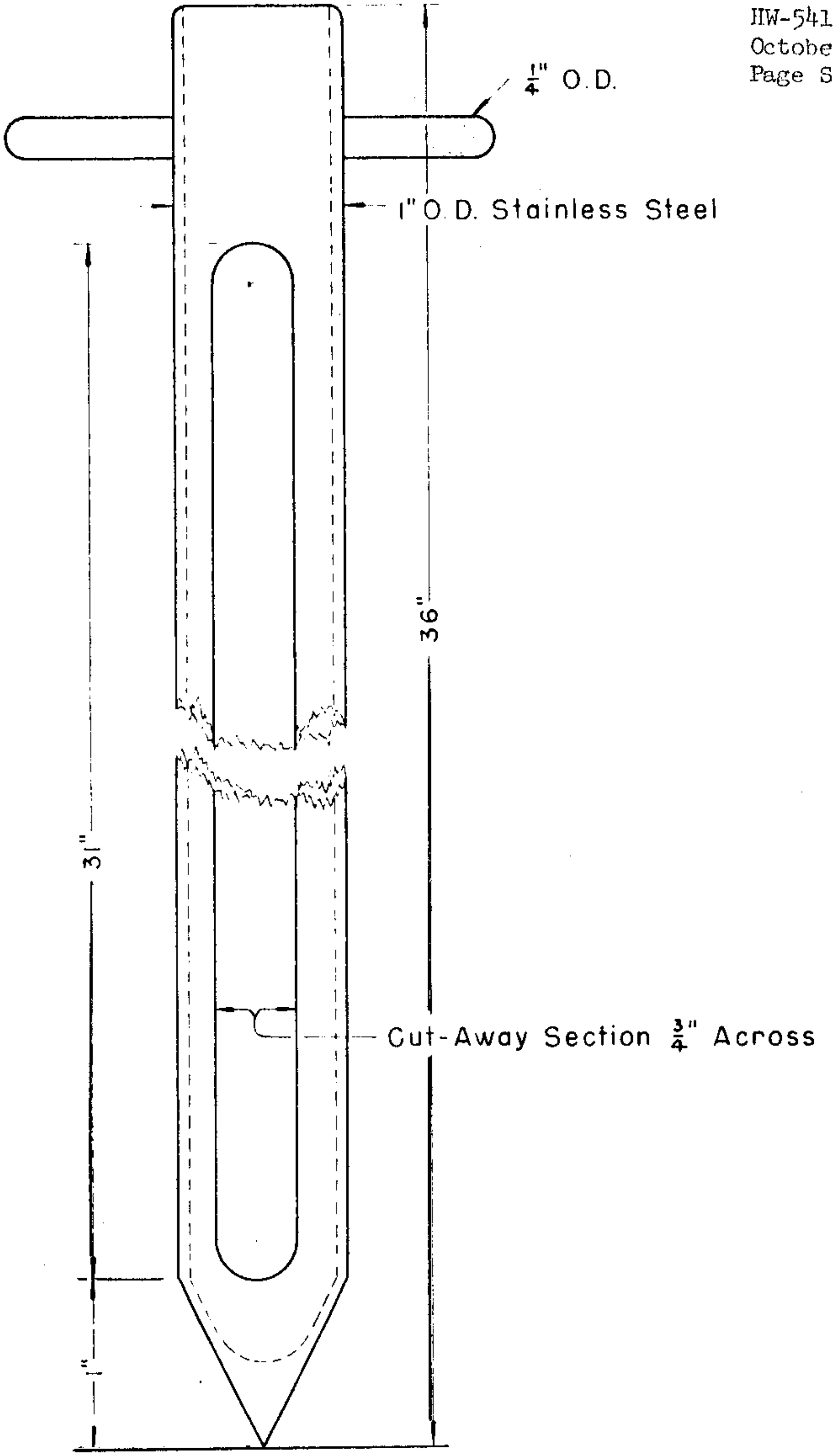
   a. Every sample submitted to the laboratory for analysis should be representative of the lot of material in question.

   b. A gross sample of fifty to one hundred pounds for a unit of thirty tons or less of material shall be taken as follows:

   By removing increments at regular intervals, i.e., one shovelfull after every ten thousand pounds of material has been removed from the car, or by removing increments with a sampling thief from various sections of the car in a geometric pattern so that equal amounts will be taken from all sections of the car, including bottom as well as top material.

   c. The gross sample shall then be crushed to 3/16" size if necessary, thoroughly mixed on a clean, dry working surface, then cored, flattened, quartered, and opposite quarters discarded.

   d. The remaining sample is then mixed, cored, flattened and quartered as above until a quantity of sample equal to the amount specified is obtained. The sample is then placed in a clean, dry, wide mouth, screw cap bottle, labeled in the specified manner and delivered to the appropriate laboratory.



GENERAL ELECTRIC CO.
HANFORD WORKS

SCALE NONE
APPROVED

[illegible] 11-2-48

SAMPLING THIEF FOR SOLIDS

DATE 10-25-48 DRAWN BY BGD
CHECKED CJ PROJ. NO.
E. R. NO. 2448 BLDG. NO.
DWG. NO. SK-7-106

G. E. CO. NO. 350 5-48

Method B - Standard Procedure for Sampling Solids in Containers

1. Safety Precautions

   a. Gloves must be worn to protect the hands.

   b. Safety glasses or goggles should be worn to protect the eyes.

   c. Plant Safety Rules covering the handling of materials in this category also apply to sampling.

2. Equipment Required

   a. Clean, dry, screw cap, wide mouth bottles as specified.

   b. Scoop, medium size.

   c. Sampling Thief as used in Method "A".

   d. Bottle, wide mouth, clean, dry, large.

3. Sampling Procedure

   a. Each sample submitted to the laboratory for analysis shall represent a composite of samples taken from at least ten (10) per cent of the containers of any one shipment, selecting the containers for sampling at random. In case a shipment involves less than twenty (20) but more than one (1) containers, a minimum of two (2) containers shall be opened for sampling.

   b. Individual samples shall be taken in such a manner that an accurate cross-section of the material in the container shall be obtained as follows:

      1. Powdered or crystalline material shall be sampled with a sampling thief, inserting the "thief" at one side of the container so that it travels diagonally across and through the entire depth of the material.

      2. Lump or coarse material shall be sampled by removing increments from the selected containers with a sampling thief wherever possible; otherwise a sample shall be removed by first moving the top material to one side and then taking a sample from the newly exposed material.

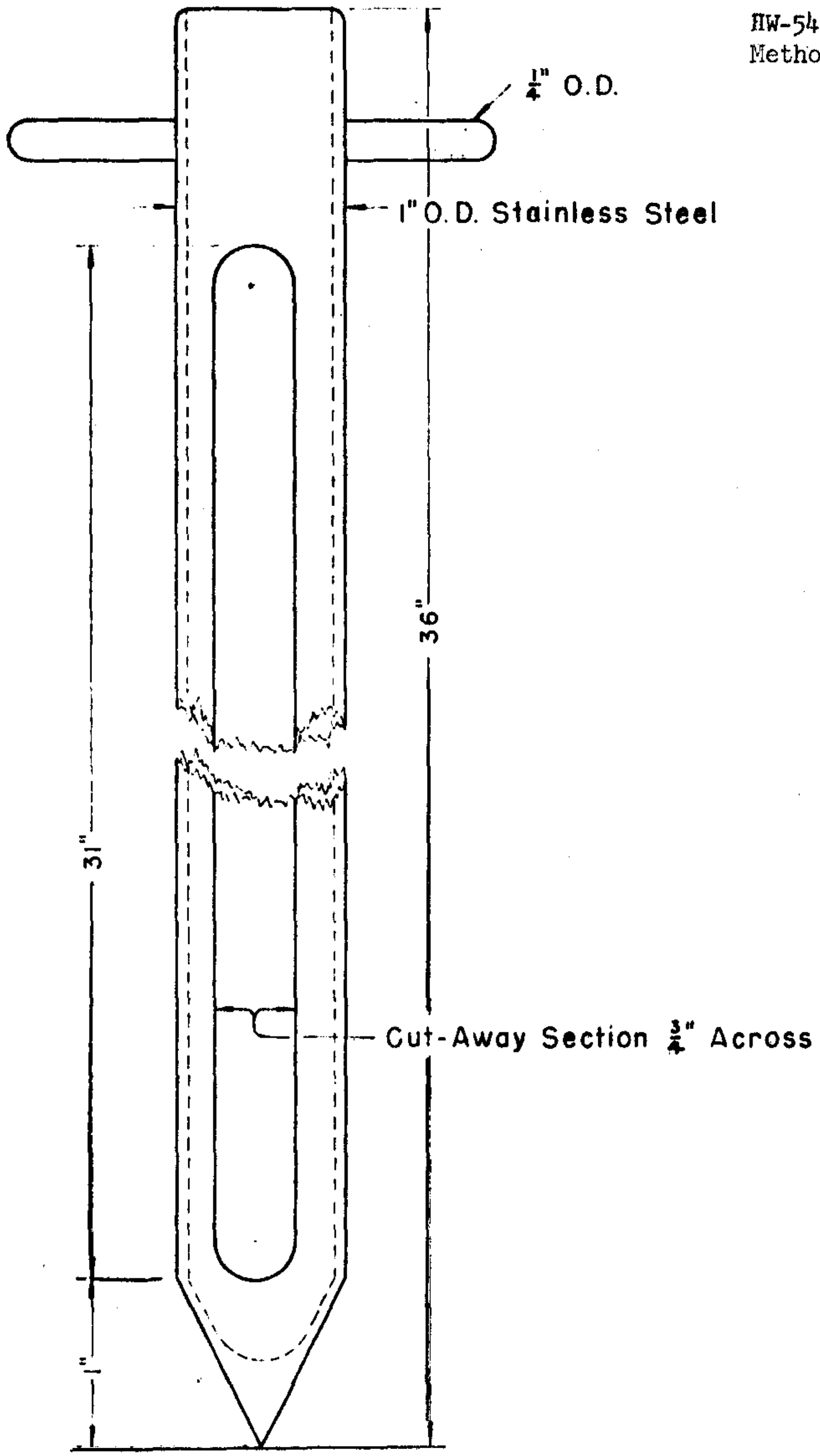
   c. From each container selected for sampling, remove approximately 1/4 lb. of material and place in a large, clean, dry, wide mouth bottle.

   d. The composite thus obtained is then thoroughly mixed by rolling and shaking the bottle for at least a five minute period.

e. Remove a portion of the composite sample to the size and type of bottle specified on the summary sheet, seal with the appropriate closure, identify the sample by affixing a label on which the necessary information is recorded and deliver the sample to the appropriate laboratory.

f. Upon receipt of the sample, the laboratory group will again mix the sample before analyzing.

HW-54125
Method A



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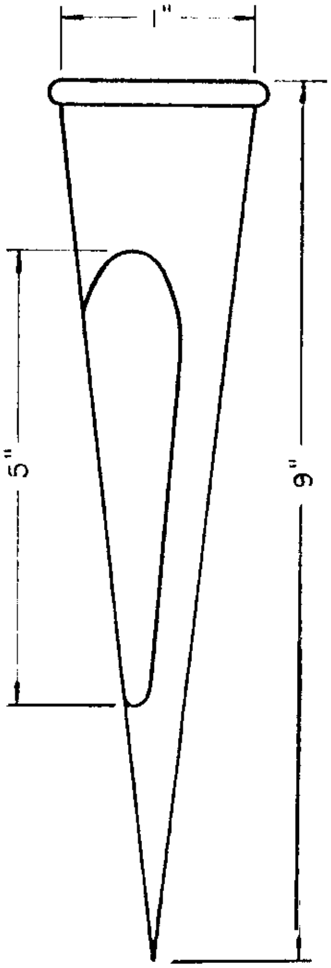
GENERAL ELECTRIC CO.
HANFORD WORKS

SAMPLING THIEF FOR SOLIDS

SCALE NONE
APPROVED
[illegible]
[illegible] 11-3-48
[illegible]

DATE 10-25-48 DRAWN BY BGD
CHECKED [illegible] PROJ. NO.
E. R. NO. 2448 BLDG. NO.
DWG. NO. SK-7-106



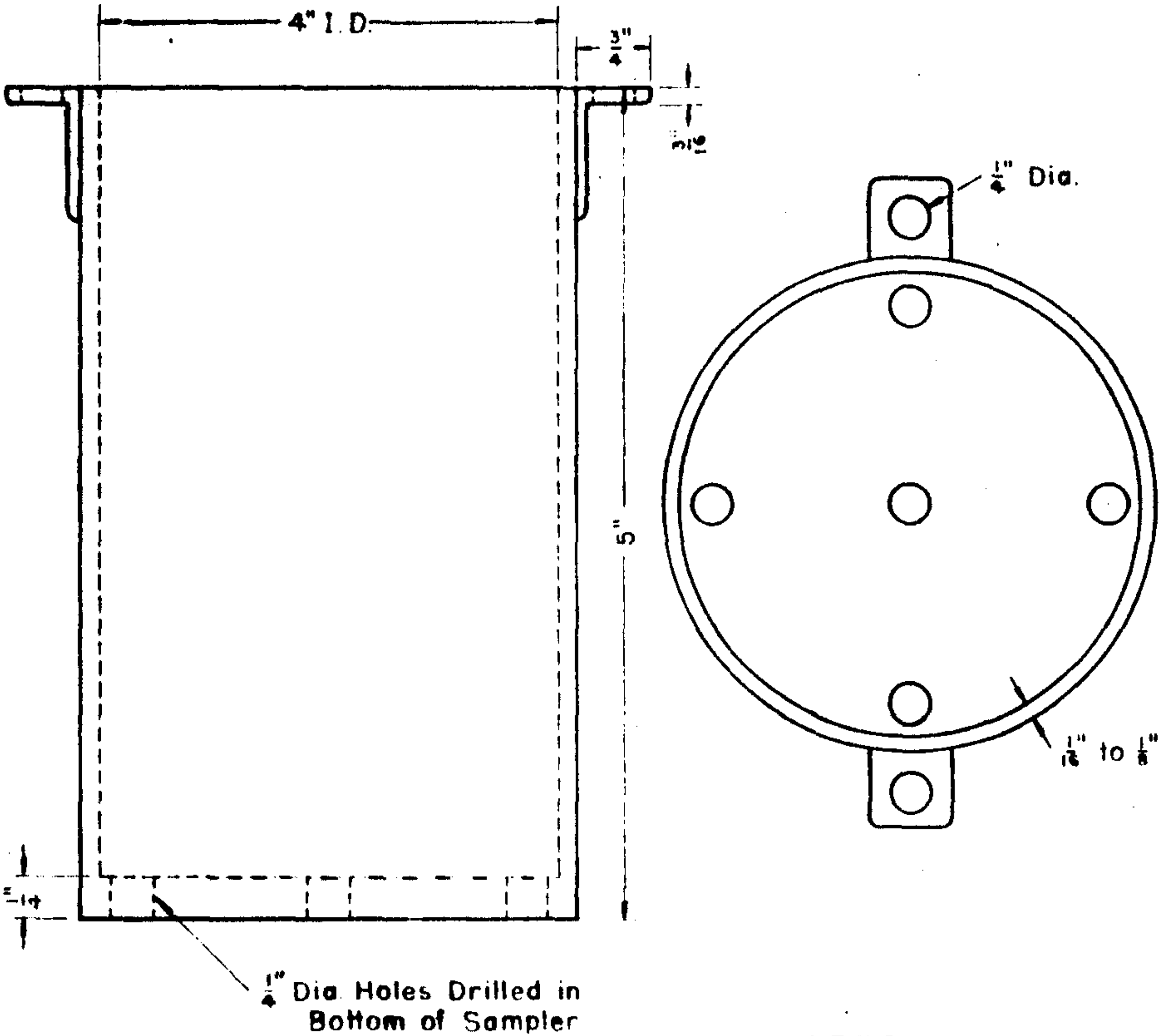
GENERAL ELECTRIC CO.
HANFORD WORKS

SCALE NONE
APPROVED

THIEF FOR SAMPLING MATERIAL RECEIVED IN BAGS

DATE 10-25-48 DRAWN BY BGD
CHECKED
PROJ. NO.
E R NO. 2448
BLDG. NO.

DWG. NO. SK-7-107



NOTES

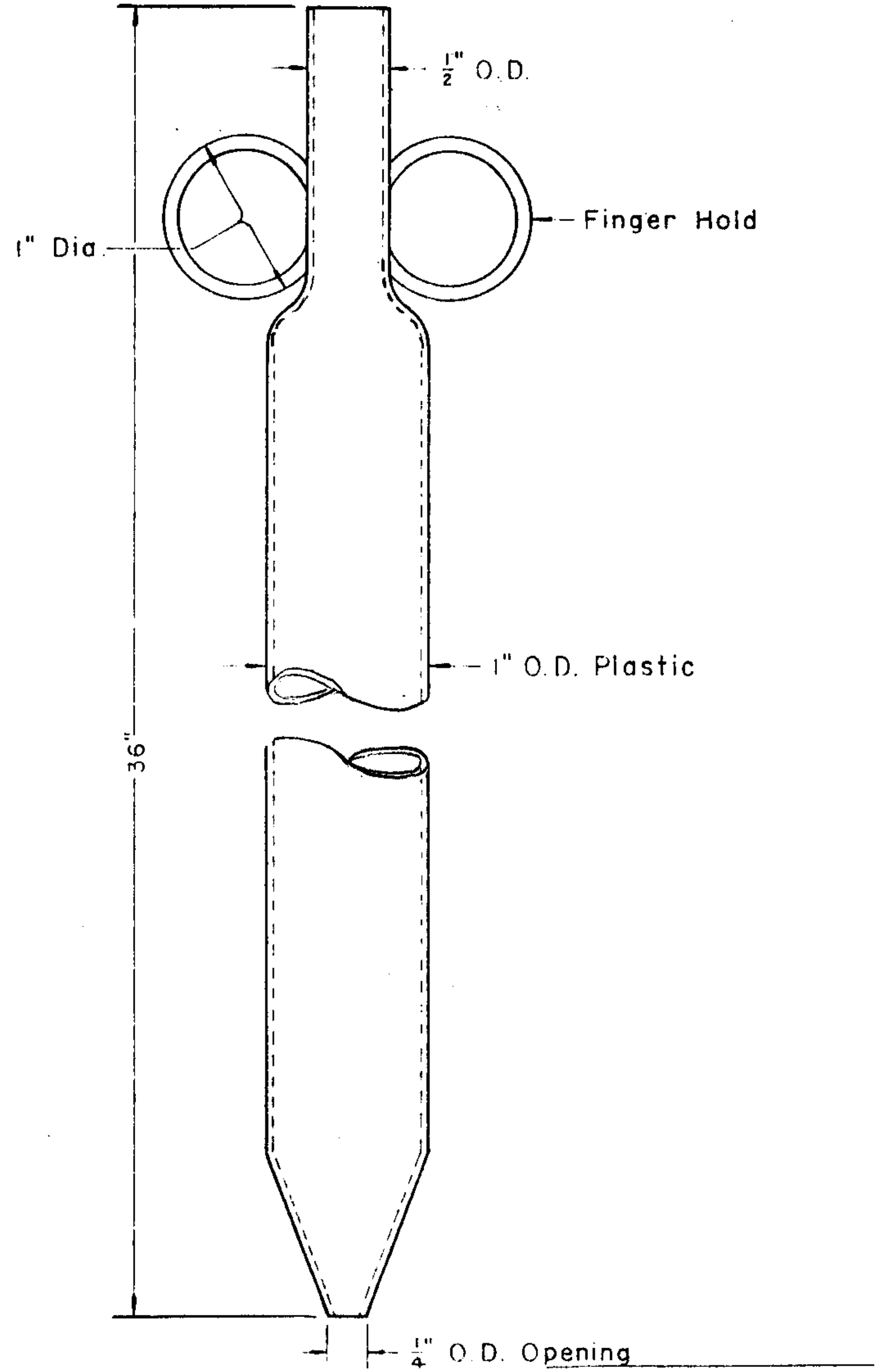
1. Stainless steel construction.
2. 20' Of flexible stainless steel chain to be provided with each sampler.

GENERAL ELECTRIC CO.
HANFORD WORKS

SCALE NONE
APPROVED

SAMPLING THIEF FOR LIQUIDS RECEIVED IN TANK CAR LOTS

DATE 10-25-48 DRAWN BY BGK
CHECKED CK PROJ. NO.
E. R. NO. 2448 BLDG. NO.
DWG. NO. SK-7-108

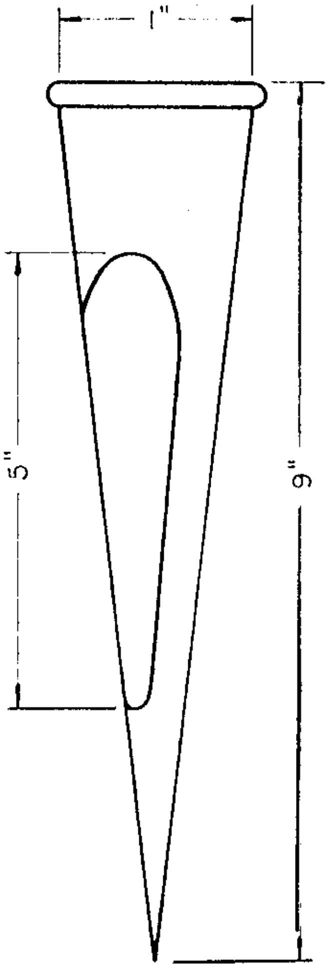


GENERAL ELECTRIC CO.
HANFORD WORKS

SCALE NONE
APPROVED

SAMPLING THIEF FOR LIQUIDS RECEIVED IN CONTAINERS

DATE 10-25-48
DRAWN BY
CHECKED
PROJ. NO.
E. R NO. 2448
BLDG. NO.
DWG. NO. SK-7-109



GENERAL ELECTRIC CO.
HANFORD WORKS

SCALE NONE
APPROVED

THIEF FOR SAMPLING MATERIAL RECEIVED IN BAGS

DATE 10-25-48 DRAWN BY
CHECKED PROJ. NO.
E R NO. 2448 BLDG. NO.
DWG. NO. SK-7-107

G E CO NO 350 5-48

Method B-I - Procedure for Sampling Granular Calcium

1. Safety Precautions

a. Wear leather gloves and safety glasses.

b. Make sure that all equipment used in the sampling is dry.

c. Avoid spilling any of the Calcium.

2. Equipment Required

a. Pint Mason jars with jar rings.

b. Storage barrel, fitted to vacuum and Argon lines, and equipped with a filter to remove water in the lines (See Remark 1).

c. Small Crescent wrench and screw driver.

d. Small scoop or sample thief(to be used for Calcium only.)

3. Sampling Procedure

a. One sample shall be submitted for each of the containers sampled. Ten (10) % of the containers shall be sampled or two (2) containers if there are less than twenty (20).

b. Carefully clean the sample jar and scoop, rinse well with distilled water, and dry on a steam plate.

c. Break the seal on the container being sampled and loosen, but do not remove the lid.

d. Place the container and an opened sample jar into a storage barrel and close the barrel.

e. Evacuate the barrel and refill with Argon.

f. Repeat step e.

g. Open the storage barrel and remove the lid of the container being sampled.

h. Using scoop or sample thief, transfer the required amount of sample to the jar, scraping away the surface layer to obtain portions from below.

i. Close the storage barrel and repeat step e twice.

j. Open the storage barrel and close the container and sample jar. Remove the sample jar and tighten the jar lid.

k. Repeat steps c through j for each container sampled.

l. Leaving the containers sampled in the storage barrels, repeat step e four (4) times.

m. Label each sample with the required information and deliver to the appropriate laboratory.

Remarks

1. An assault mask cannister may be attached on the end of the gas line within the storage barrel as a temporary expedient.

Method B-II - Procedure for Sampling Iodine

1. Safety Precautions

   a. Rubber gloves and safety glasses must be worn.

   b. Containers to be sampled shall be removed to a well-ventilated hood for sampling.

2. Equipment Required

   a. Glass, Bakelite scoop or sampling thief.

   b. Wide-mouth glass-stoppered bottle, 16 oz.

3. Sampling Procedure

   a. Each sample submitted to the laboratory shall be a composite of samples taken from at least ten (10)% of the containers represented by the sample, or from at least two (2) of the containers if there are less than twenty (20).

   b. The portions taken from each container shall be of equal size, and when combined in the sample bottle, shall fill it no more than half full.

   c. Carefully clean the sampling equipment, rinse with distilled water, and dry on a steam plate.

   d. Remove the lid from the container to be sampled, being careful not to allow paraffin or other material used as a seal to fall into the container.

   e. Insert a sampling thief, if available, diagonally to the full depth of the container, and withdraw the desired portion; otherwise, scoop from the top. Place in the sample bottle. Repeat with the other containers to be sampled.

   f. Replace the stopper on the sample bottle, affix a label filled out with the required information, and deliver to the appropriate laboratory.

Method B-III - Procedure for Sampling Magnesium Oxide

1. Safety Precautions

None

2. Equipment Required

a. Sampling thief or scoop, to be used for mangesium oxide only.

b. Wide-mouth screw-cap bottle, 16 oz.

c. One gallon wide-mouth jar, to be used for sampling Magnesium Oxide only.

3. Sampling Procedure

a. Each sample submitted to the laboratory shall be a composite of samples taken from at least ten (10) % of the containers represented by the sample, or from at least two (2) of the containers if there are less than twenty (20).

b. Make sure that the sampling equipment is clean and dry.

c. Insert a sampling thief, if available, diagonally to the full depth of the container being sampled, withdraw the sample, and discharge it into the large jar. If a scoop is used, first move the top material to one side, and then scoop an equivalent amount from the newly-exposed material.

d. Sample the remaining containers in the same manner, removing at least one lb. from each container, and composite the individual samples in the large jar.

e. Thoroughly mix the contents of the jar by shaking for five minutes.

f. Remove a portion of the composite to the sample bottle, affix a label filled out with the required information, and deliver to the appropriate laboratory.

g. Do not return the excess composite to the original container, but discard it.

Method C - Standard Procedure for Sampling Liquids in Bulk

1. Safety Precautions

   a. Rubber gloves and coverall goggles shall be worn to protect the hands and eyes, respectively.

   b. A gas mask, designed for protection against acid vapors, shall be worn in any case where plant Safety Rules require use of same.

   c. All plant Safety Rules covering the handling of corrosive materials also apply to this sampling procedure.

2. Equipment Required

   a. Clean, dry, glass stoppered bottle as specified.

   b. Tank car liquid thief as per attached sketch.

   c. Safety carriers for bottles.

3. Sampling Procedure

   One sample representative of the lot shall be removed from each tank car shipment as follows:

   a. Rapidly lower a weighted sample thief, containing an unstoppered bottle of the desired size, to the bottom of the tank car.

   b. Allow the sample thief to stay in this position for approximately three minutes and then raise the sampling thief slowly to the top of the car.

   c. Pour part of the contents of the sample bottle into the car, so that an air-space is left above the liquid, and replace the stopper.

   d. Remove the bottle from the sampling thief and wash the outside of the bottle to remove any adhering liquid; however, extreme care should be exercised to prevent water from flowing around the stopper of the bottle and thus diluting or contaminating the sample.

   e. Repeat step d for all other equipment used in the sampling operation.

   f. The sample should then be labeled in the specified manner and delivered to the appropriate laboratory.

Remarks

Clean rubber stoppers should be used as bottle closures in the case of sodium hydroxide and sodium silicate.

Procedure - Analyis of Hydrazine Solution (< 0.05% Cl) (#890)

Place 40 ml of water in each of two glass-stoppered cylinders. To each add 1 ml of 1 + 3 nitric acid and 1 ml of acid silver nitrate. To one add further 1 ml of concentrated nitric acid and 1.0 ml of the hydrazine solution sample. To the other add 5 ml of 0.1 mgm/ml chloride standard. Mix. Compare. The turbidity of the sample tube should be less than that of the standard.

Procedure - Analysis of Potassium Persulfate #0752 - 0.01% Cl^-

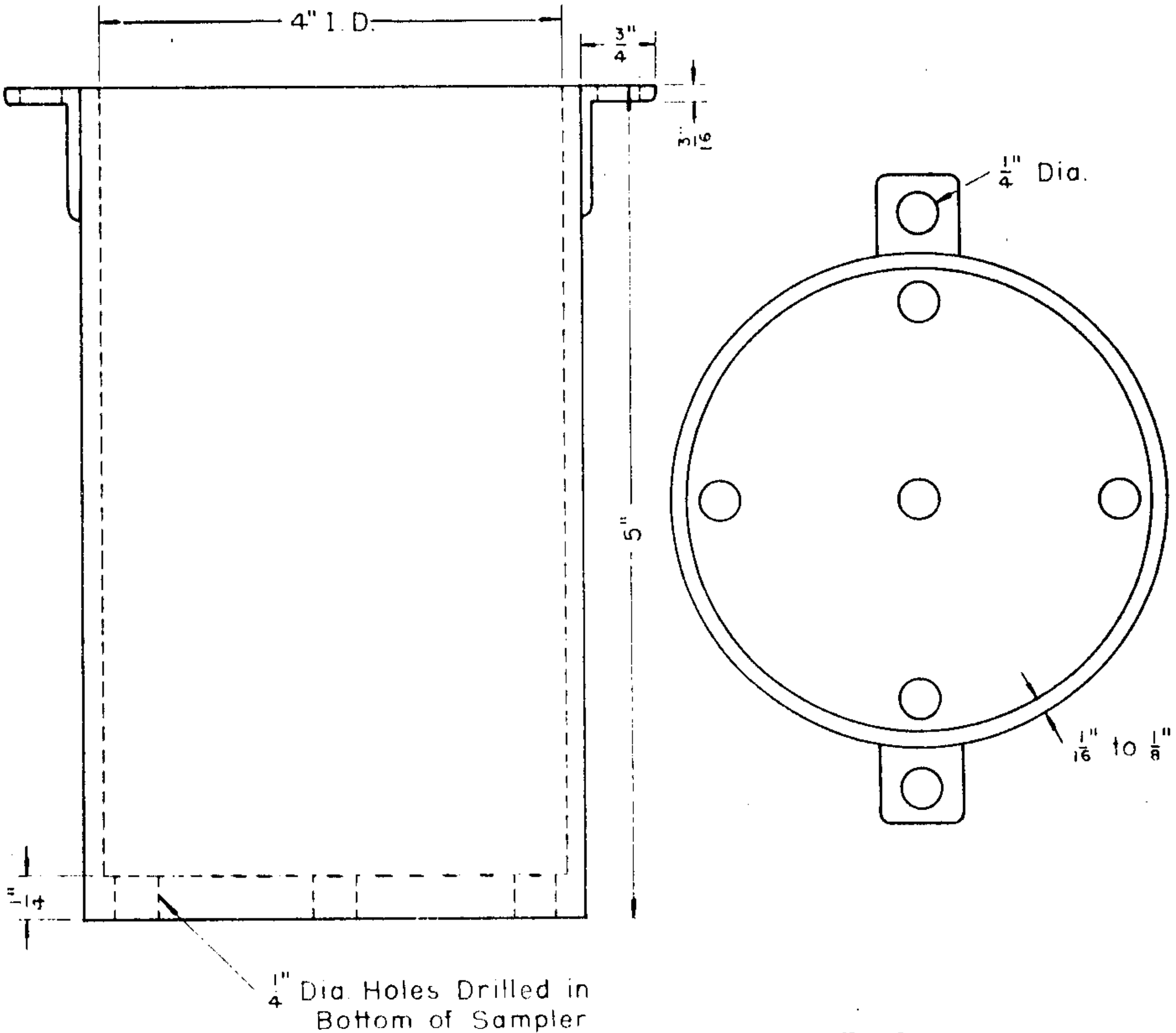
Dissolve 1 g in 30 ml of hot water and transfer to a 50 ml glass-stoppered cylinder. Add 1 ml of 1 + 3 nitric acid and 1.0 ml of acid silver nitrate.

Compare with a freshly prepared standard made with 1.0 ml of standard 0.1 mg per ml chloride.

Procedure - Analysis of Sodium Bismuthate #0782 - 0.01%

Add 2 g to 25 ml of water, heat to boiling and keep at the boiling temperature for 10 minutes. Dilute to 50 ml and filter through a filter that has been washed free of chlorides. To 25 ml of the filtrate, add 0.15 ml of 30% hydrogen peroxide to clear the solution.

Add 1 ml of nitric acid and 1 ml of silver nitrate reagent. The turbidity should not exceed that produced by 1 ml of standard 0.1 mg per ml chloride.



NOTES

1. Stainless steel construction.
2. 20' Of flexible stainless steel chain to be provided with each sampler.

GENERAL ELECTRIC CO.
HANFORD WORKS

SCALE NONE
APPROVED

SAMPLING THIEF FOR LIQUIDS
RECEIVED IN TANK CAR LOTS

DATE 10-25-48 DRAWN BY
CHECKED PROJ. NO.
E. R. NO. 2448 BLDG. NO.

DWG. NO. SK-7-108

Method D - Standard Procedure for Sampling Liquids in Containers

1. Safety Precautions

   a. Rubber gloves and cover-all goggles are worn to protect the hands and eyes, respectively.

   b. Samples should only be taken in a well ventilated room, using a gas mask of the proper type whenever necessary.

   c. All plant Safety Rules covering handling of materials of this category also apply to this sampling procedure.

2. Equipment Required

   a. Clean, dry, glass stoppered bottles of the size specified.

   b. Sampling thief as per attached sketch.

   c. Funnel (glass).

   d. Safety carriers for bottles.

3. Sampling Procedure

   Samples should be obtained by selecting at random ten per cent of the containers in any one shipment and removing equal portions of material from each container selected as follows:

   a. Immerse a clean sampling thief in the liquid and allow liquid to flow into the tube.

   b. Place the thumb over the top opening of the sample thief and remove the thief from the container.

   Use this portion of the material to rinse the tube and then discard.

   c. Now lower the sampling thief into the container until the bottom opening of the thief is approximately one-half inch above the bottom of the container.

   d. When sampling thief has filled again replace the thumb over the top opening and remove from the container. Allow this liquid to drain into the sample bottle.

   e. Repeat steps a through d for each container to be sampled, compositing all individual samples to obtain one sample representative of the shipment.

Method D-I - Procedure for Sampling Bromobenzene

1. Safety Precautions

    a. Coveralls, safety glasses, and rubber gloves shall be worn.

    b. All sampling shall be done in a well-ventilated hood, away from open flames.

    c. Open bottles behind a hood window as a precaution against escaping spray.

2. Equipment Required

    a. Short-stemmed glass funnel.

    b. Safety carriers for bottles.

    c. 50 ml. Mohr pipette.

    d. Brown glass-stoppered bottle, 16 oz.

    e. Paraffin, small brush, masking tape.

3. Sampling Procedure

    a. Carefully clean the sample bottle, funnel, and pipette, rinsing with concentrated nitric acid and distilled water, and drying in an oven or on a steam plate.

    b. Select at random at least one bottle for each ten cases in the shipment, but at least two bottles from different cases if there are less than twenty.

    c. Immerse a Mohr pipette slowly into the liquid to its full depth, allowing it to fill.

    d. Cover the top of the pipette with the thumb and remove it from the bottle being sampled.

    e. Discard this portion into the sink, using a large volume of water to flush it down.

    f. Repeat c and d, draining this portion into the sample bottle. Use a funnel to avoid spilling any liquid.

    g. Repeat steps c and d until the amount desired from one bottle is obtained. Sample the remaining bottles in the same manner, compositing all individual samples in the same sample bottle.

h. Replace the stopper on the sample bottle and seal by brushing with melted paraffin.

i. Secure the stopper to the sample bottle with masking tape and rinse or wipe off any liquid spilled on the outside of the bottle.

j. Affix a label to the bottle, filled out with the required information and deliver to the appropriate laboratory.

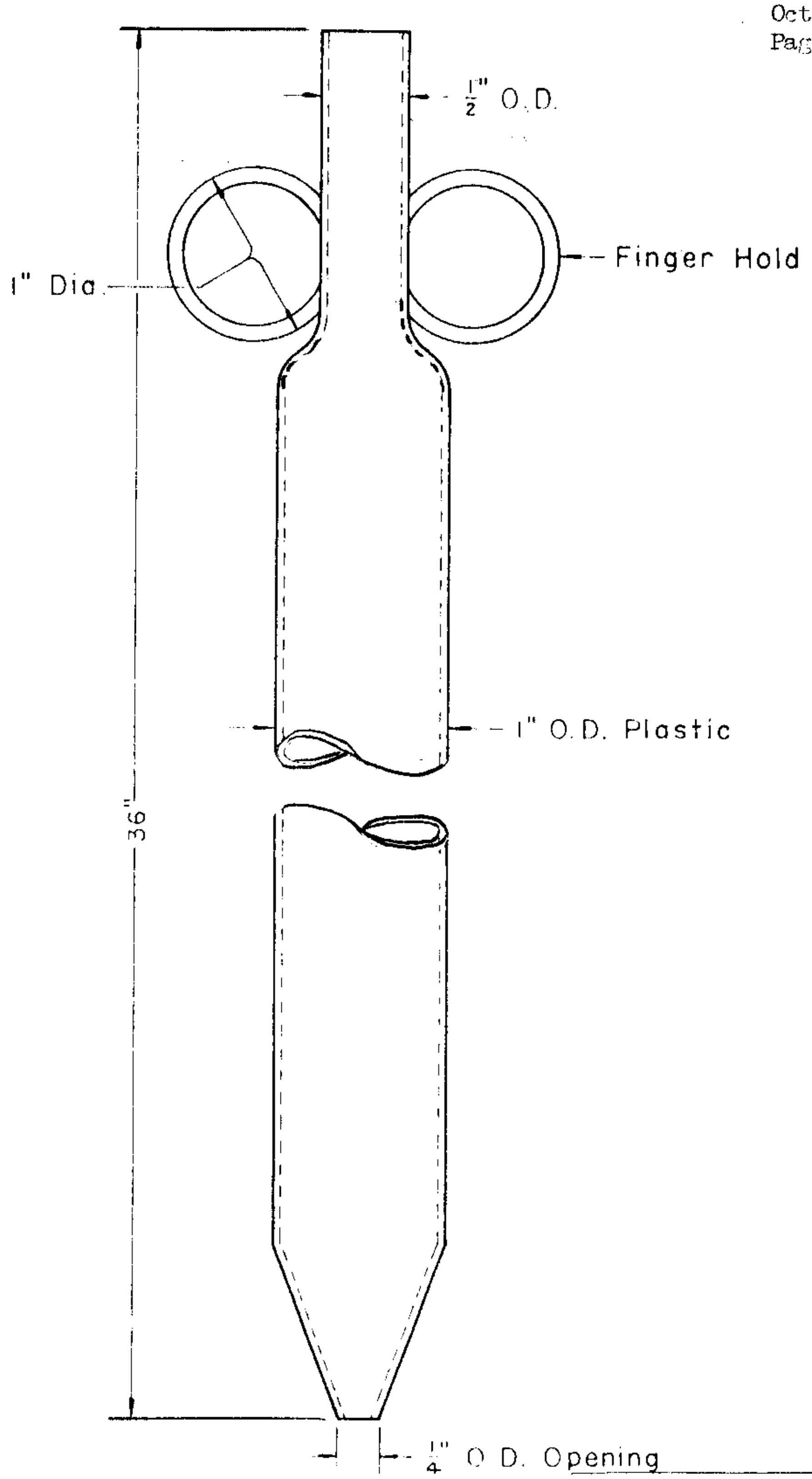
f. Replace the stopper in the bottle containing the composite sample and mix the contents thoroughly.

g. Transfer a portion of the composite sample to a bottle of the type and size specified and remove any material which might have been spilled on the outside of the bottle by washing with water.

h. The sample should then be labeled in the specified manner and delivered to the appropriate laboratory.

Remarks

In any case where the number of containers received in any one shipment is less than twenty, but more than one, two containers should be selected at random and sampled as above.



GENERAL ELECTRIC CO.
HANFORD WORKS

SCALE NONE
APPROVED

SAMPLING THIEF FOR LIQUIDS RECEIVED IN CONTAINERS

DATE 10-25-48 DRAWN BY
CHECKED PROJ. NO.
E. R. NO. 2448 BLDG. NO.

DWG. NO. SK-7-109

Method J - Sampling Procedure for Gases (Non-Corrosive), Argon, Helium, Oxygen, CO_2

1. Safety Precautions

a. Gloves and goggles should be worn to protect the hands and eyes, respectively.

b. All equipment used in the sampling operation should be tested to withstand 3000 lbs. per square inch internal pressure.

c. Observe all plant Safety Rules which cover handling of gas cylinders.

2. Equipment Required

a. Gas Cylinders (Size B - Ohio Chemical Company).

b. Necessary couplings and wrenches for making connections to tanks.

c. Vacuum Pump (Hyvac or Megavac).

3. Sampling Procedure

a. Cylinder gases shall be sampled to the extent of 10% of the number of cylinders received in a single shipment, with samples being taken at random.

b. All tank car shipments shall be sampled.

c. Evacuate the sample cylinder and then close the valve (Remark 1).

d. Connect the coupling to the tank or cylinder to be sampled and to the sample cylinder. Attach the vent valve or coupling to a small line leading outside the building, or if the sampling is done in the open, the small line should extend to a point 20 feet away from the operator.

e. Flush the coupling with the sample by first opening the vent valve on the coupling and then opening the tank valve. Let gas flow for a minute to insure removal of air.

f. Close vent valve.

g. Open valve on sample cylinder slowly, maintaining a positive gas pressure in the coupling.

h. Slowly open the valve on the tank being sampled and permit tank and sample cylinder pressures to equalize.

i. Close the valve on the tank or cylinder being sampled.

j. Open vent valve and bleed-off gas in sample cylinder until atmospheric pressure is attained.

k. Close vent valve as soon as pressure in sample cylinder reaches atmospheric.

l. Repeat steps 7 through 11 for three successive times, to be certain that the sample cylinder has been completely flushed.

m. Retain the fourth sample.

n. Close the valves on the sample cylinder and tank or cylinder being sampled.

o. Open vent valve momentarily to relieve gas pressure within the coupling.

p. Disconnect and remove coupling.

q. Clearly identify the sample by affixing a label on which the necessary information is recorded.

r. Deliver the sample cylinder to the appropriate laboratory.

Remarks

1. Care should be exercised in turning the valve on the Type "B" cylinder since the seat of the needle valve is easily damaged.

STANDARD METHODS FOR SAMPLING ESSENTIAL MATERIALS

Foreword

Department and plant safety rules applicable to the handling of a material apply to the sampling. The plant Chemical Hazards Review supplies information toward safe handling.

Note: A general discussion of sampling practices is given in "Scott's Standard Methods of Chemical Analysis." N. H. Furman, Editor, 5th Edition, Vol. 2, D. Van Nostrand Co., Inc. Princeton, N. J., 1939.

Practical sampling procedures based on use experience and on the degree of control required are outlined for the following items:

Method A	Solids in bulk
B	Solids in containers
B-I	Granular calcium
B-II	Iodine
B-III	Magnesium Oxide
C	Liquids in bulk
C-1	Liquids in tank trucks
D	Liquids in containers
D-1	Bromobenzene
J	Non-corrosive gases

Method A - Standard Procedure for Sampling Solids in Bulk (Car or Truck) (Other than coal)

1. Equipment Required
   a. Clean, dry, wide mouth bottles or jars.
   b. Shovel
   c. Sampling thief as per attached sketch.
   d. Labels for bottles or jars.

2. Sampling Procedure

a. Every sample submitted to the laboratory for analysis should be representative of the material in question.

b. A gross sample of 50 to 100 pounds for a unit of 30 tons or less of material shall be taken as follows:

By removing increments at regular intervals, i.e., one shovelful after every 10,000 pounds of material has been removed from the car, or by removing increments with a sampling thief from various sections of the car in a geometric pattern so that equal amounts will be taken from all sections of the car, including bottom as well as top material.

c. The gross sample shall then be crushed to 3/16" size, if necessary, thoroughly mixed on clean, dry working surface, then coned, flattened, quartered, and opposite quarters discarded.

d. The remaining sample is then mixed, coned, flattened, and quartered as above until a quantity of sample equal to the amount specified on the specification page of this manual is obtained. The sample is then placed in a clean, dry, wide mouth, screw cap bottle. Send the labeled sample in the sealed screw cap container to the laboratory of the plant receiving or using the material.

Information on the label should include the name of the material, purchase order number, vendor's name, date, and name of sampler. The number of a rail car may also be noted.

Method B - Standard Procedure for Sampling Solids in Containers

1. Equipment Required

   a. Clean, dry, screw cap, wide mouth bottles or jars.

   b. Scoop, medium size.

   c. Sampling thief as used in Method A.

   d. Labels for bottles or jars.

2. Sampling Procedure

   a. Each sample submitted to the laboratory for analysis shall represent a composite from the containers according to the following schedule:

Number of containers in shipment	Number of containers sampled for composite
25 or more	5
Less than 25	2
Less than 5	1 only

   b. Individual samples shall be taken in such a manner than an accurate cross-section of the material in the container shall be obtained as follows:

      1. Powdered or crystalline material shall be sampled with a sampling thief, inserting the "thief" at one side of the container so that it travels diagonally across and through the entire depth of the material.

      2. Lump or coarse material shall be sampled by removing increments from the selected containers with a sampling thief wherever possible; otherwise a sample shall be removed by first moving the top material to one side and then taking a sample from the newly exposed material.

   c. From each container selected for sampling, remove approximately 1/4 lb. of material and place in a large, clean, dry, wide mouth bottle or jar.

   d. The composite thus obtained is then thoroughly mixed by rolling and shaking the bottle for at least a five-minute period.

e. Send a labeled sample from the composite, and of the amount noted on the specification page of this manual, in a sealed jar or bottle to the laboratory of the plant receiving or using the material.

Information on the label should include the name of the material, purchase order number, vendor's name, date, and name of the sampler.

Method B-1 - Procedure for Sampling Granular Calcium

1. Equipment Required

   a. Clean, dry, wide mouth bottles or jars with screw caps.

   b. 15 ml sample bottles with screw caps.

   c. Argon supply and purge lines (dry).

   d. Crescent wrench and screw driver.

   e. Clean, dry, polyethylene scoop (to be used for calcium only).

   f. Labels for bottles or jars.

2. Sampling Procedure

   a. One sample shall be submitted from one container per lot. A lot consists of five 100-pound barrels.

   b. Remove the lid from the barrel being sampled.

   c. Scrape the surface of the calcium away and insert the polyethylene scoop deep into the material.

   d. Remove a good cross section of the calcium and place in a 4-ounce, wide mouth bottle. Purge the sample with argon.

   e. Screw the cap on the bottle and shake to thoroughly mix the contents.

   f. Remove the cap and transfer approximately 10 grams to a clean, dry, 15 ml sample bottle. (The bottle should be about 2/3 full).

   g. Purge the sample with argon; screw the cap on.

   h. Label the sample giving the name of the material, purchase order number, identifying lot number, if available, vendor's name, date, and sampler's name. Deliver the sample to the laboratory.

Method B-II - Procedure for Sampling Iodine

1. Equipment Required

a. Glass, Bakelite scoop, or sampling thief.
b. Wide mouth, glass-stoppered bottle or jar, 16 oz.
c. Labels for bottles or jars.

2. Sampling Procedure

a. Each sample submitted to the laboratory shall be a composite of samples taken from at least ten (10) per cent of the containers represented by the sample, or from at least two (2) of the containers if there are less than twenty (20).

b. The portions taken from each container shall be of equal size, and when combined in the sample bottle, shall fill it no more than half full.

c. Carefully clean the sampling equipment, rinse with distilled water, and dry on a steam plate.

d. Remove the lid from the container to be sampled, being careful not to allow paraffin or other material used as a seal to fall into the container.

e. Insert a sampling thief, if available, diagonally to the full depth of the container, and withdraw the desired portion; otherwise, scoop from the top. Place in the sample bottle. Repeat with the other containers to be sampled.

f. Replace the stopper on the sample bottle. Label the bottle, giving the name of the material, purchase order number, vendor's name, identifying lot number, if available, date, and sampler's name. Deliver the sample to the laboratory.

Method B-III - Procedure for Sampling Magnesium Oxide

1. Equipment Required

   a. Sampling thief or scoop, to be used for magnesium oxide only.

   b. Wide mouth, screw cap bottle or jar, 16 oz.

   c. One-gallon, wide mouth jar, to be used for sampling magnesium oxide only.

   d. Labels for bottles and jars.

2. Sampling Procedure

   a. Each sample submitted to the laboratory shall be a composite of samples taken from at least ten (10) per cent of the containers represented by the sample, or from at least two (2) of the containers if there are less than twenty (20).

   b. Make sure that the sampling equipment is clean and dry.

   c. Preferably insert a sampling thief, if available, diagonally to the full depth of the container being sampled, withdraw the sample, and discharge it into the large jar. If a scoop is used, first move the top material to one side, and then scoop a clean sample from the newly exposed material.

   d. Sample the remaining containers in the same manner, removing at least one pound from each container, and composite the individual samples in the large jar.

   e. Thoroughly mix the contents of the jar by shaking for five minutes.

   f. Remove a portion of the composite to the sample bottle. Label the sample bottle, giving the name of the material, purchase order number, identifying lot number, if available, vendor's name, date, and sampler's name. Deliver the sample to the laboratory.

Method C - Standard Procedure for Sampling Liquids in Bulk

1. Equipment Required

a. Clean, dry, glass-stoppered bottle or jar, as specified.

b. Tank car liquid thief as per attached sketch.

c. Safety carriers for bottles.

d. Labels for bottles or jars.

2. Sampling Procedure

One sample representative of the lot shall be removed from each tank car shipment as follows:

a. Rapidly lower a weighted sample thief, containing an unstoppered bottle of the desired size, to the bottom of the tank car.

b. Allow the sample thief to stay in this position for approximately three minutes and then raise the sampling thief slowly to the top of the car.

c. Pour part of the contents of the sample bottle into the car, so that an air-space is left above the liquid, and replace the stopper.

d. Remove the bottle from the sampling thief and wash the outside of the bottle to remove any adhering liquid; however, extreme care should be exercised to prevent water from flowing around the stopper of the bottle and thus diluting or contaminating the sample.

e. Carefully rinse all other equipment used in the sampling operation.

f. Label the sample giving the name of the material, the purchase order number, the identifying lot number, if available, the load number from a truck, or the rail car number, the name of the vendor, date, and sampler's name.

Method C-1 - Standard Procedure for Sampling Aluminum Nitrate Solution and Nitric Acid Received in Tank Trucks.

Note. Chemical tank trucks are connected up, emptied into their respective receiving tanks, and disconnected by the truck drivers.

1. Equipment Required

   a. A clean, dry bottle for the sample size noted under acceptance specifications, and a polyethylene-lined screw cap should be used.

   b. A label for the bottle.

2. Sampling Procedure

   a. Observe that the in-line trap sampler is drained and that the drain valve is closed before the transfer line from a truck is connected for unloading.

   b. After the truck contents have been unloaded and the transfer line from the truck has been disconnected, take a sample via the sampler drain valve. Cap the bottle. Wash any spilled material from the bottle.

   c. Label the sample, giving the name of the material, the identifying lot number or truck number, the name of the vendor, date and sampler's name.

   d. Deliver the sample to the plant laboratory.

   e. Drain the sample trap and leave the sampler drain valve open.

Method D - Standard Procedure for Sampling Liquids in Containers

1. Equipment Required

a. Clean, dry, glass bottles or jars with polyethylene-lined bakelite caps for the sample size specified in the specification sheet.

b. Sampling thief as per attached sketch.

c. Funnel (glass).

d. Safety carriers for bottles.

e. Labels for bottles or jars.

2. Sampling Procedure

Each sample submitted to the laboratory for analysis shall represent a composite from the containers according to the following schedule:

Number of containers in shipment	Number of containers sampled for composite
25 or more	5
Less than 25	2
Less than 5	1 only

a. Immerse a clean sampling thief in the liquid and allow liquid to flow into the tube.

b. Place the thumb over the top opening of the sample thief and remove the thief from the container. Use this portion of the material to rinse the tube and then discard.

c. Now lower the sampling thief into the container until the bottom opening of the thief is approximately one-half inch above the bottom of the container.

d. When sampling thief has filled again, replace the thumb over the top opening and remove from the container. Allow this liquid to drain into the sample bottle.

e. Repeat steps a through d for each container to be sampled compositing all individual samples to obtain one sample representative of the shipment.

f. Replace the stopper in the bottle containing the composite sample and mix the contents thoroughly.

g. Transfer a portion of the composite sample to a bottle of the type and size specified and remove any material which might have been spilled on the outside of the bottle.

h. Label the sample giving the name of the material, the purchase order number, and, if available, an identifying lot number, the name of the vendor, date, and sampler's name. Deliver the sample to the appropriate plant laboratory.

Method D-1 - Procedure for Sampling Bromobenzene

1. Equipment Required

a. A clean dry, 50 ml pipet.

b. A rubber squeeze bulb to fit the pipet.

c. A clean, dry, 8 oz. amber bottle with a polythene lined cap.

d. Labels

2. Sampling Procedure

a. Select at random at least one bottle for each ten cases, but at least two bottles if there are less than 20 cases in a shipment.

b. Prepare a composite consisting of nearly equal amounts from the bottles selected and in the following manner.

c. Insert the pipet well into the bottle being sampled and by means of a rubber bulb draw up about 50 ml of the liquid. In the usual way replace the bulb by the forefinger and, so holding the pipet full, transfer the liquid to an 8 oz. amber bottle.

Without cleaning the pipet, transfer 50 ml portions from the remaining selected bottles to the 8 oz. bottle.

Note: Where a shipment of more than 40 cases is sampled, a 16 oz. bottle will be needed.

d. Tighten the cap of the sample bottle and wipe off any spilled liquid from the outside of the bottle.

e. Label the bottle giving the date, name of material, sampler's name, and, if possible, the vendor's name and shipment or lot number.

f. Deliver the sample to the 234-5 laboratory.

Note: The pipet may be cleaned with water and acetone and then should be dried by drawing air through it or by allowing it to stand in a vertical position until dry.

Method J - Sampling Procedure for Gases (Non-Corrosive) - Argon, Helium, Oxygen, CO_2, Nitrogen

This procedure is for sampling the gas phase if there are two phases. Accordingly, the outlet valve of the vessel being sampled should be at the top of the vessel.

1. Equipment Required

a. Sample cylinders (Size B-Ohio Chemical Co.) - 3 3/4" O.D. x 16" high)

Note: All equipment used in the sampling operation should be tested to withstand 3000 pounds per square inch internal pressure.

b. A "T" shaped arrangement of a pipe, a valve, and couplings suitable for the sample cylinder to the tank being sampled with the valves arranged so that pressure can be released at the "T".

c. Vacuum pump. (Hyvac or Megavac) (Pump should be capable of a 5 mm vacuum)

d. Labels or tags.

2. Sampling Procedure

a. In sampling cylinder gases samples shall be taken at random from ten per cent of cylinders received in a shipment.

b. In sampling tank cars a sample shall be·taken for every car.

c. Evacuate the sample cylinder and close the valve (Remark 1)

d. Connect the sampler coupling to the sample cylinder and to the tank to be sampled. Arrange the vent line so that gas may be vented outside the building or, if in the open, to a distance of 20 ft. from the operator.

e. Flush the coupling with ghe gas in order to remove air by first opening the vent valve and then slowly opening the tank valve far enough to give a moderate flow of gas for 1 to 2 minutes.

f. Close vent valve. The pressure in the coupling will build to tank pressure at this point as the tank valve is slightly open.

g. Open the valve on the sample cylinder slowly so that a positive pressure is maintained in the coupling.

h. Slowly increase the opening of the valve on the tank being sampled, permitting tank and sample cylinder pressures to equalize.

i. Close the valve on the tank being sampled.

j. Open the vent valve and bleed off the gas in the sample cylinder until atmospheric pressure is reached.

k. Close the vent valve as soon as the pressure in the sample cylinder reaches atmospheric.

l. In order to flush the sample cylinder thoroughly, go through the above operation three times; i.e.:

   1. Close vent.
   2. Slightly open tank valve until pressure in the two cylinders is equalized.
   3. Close tank valve.
   4. Open vent and allow pressure to bleed off.
   5. Go back to step 1, close vent, etc.

m. Finally, when the two cylinders are at equal pressure as in h or l (2) above, close the sample cylinder valve.

n. Close tank valve.

o. Open vent.

p. Disconnect the cylinders.

q. Label the sample, giving an identifying number such as shipment or order number, the name of the gas, the date, the sampler's name, and the vendor.

r. Tanke the sample to the plant laboratory for analysis.

Remarks

1. Care should be exercised in turning the valve on the Type "B" cylinder since the seat of the needle valve is easily damaged.

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Hg-1	Mercury - volumetric
I-1	Iodine - extraction
I-2	Iodine - colorimetric
Mg-1	Magnesium - colorimetric
Mn-1	Manganese - volumetric
N-3	Nitrogen - volumetric
N-5	Nitrogen - Nessler
N-10	Nitrite - volumetric
N-11	Nitrogen oxides - volumetric
N-13	Nitrite - colorimetric
N-21	Nitrate - volumetric
N-23	Nitrate - colorimetric
N-25	Hydrazine - volumetric
Na-2	Alkali and alkaline earth - gravimetric
O-1	Determination of hydrogen peroxide - volumetric
O-2	Determination of water - distillation
O-3	Determination of water - qualitative
O-4	Determination of Moisture Content
P-1	Determination of Phosphate - gravimetric
P-2	Determination of Phosphate - colorimetric
Pb-1	Lead - volumetric
Pb-2	Heavy metals - turbidimetric
S-11	Sulfite - volumetric
S-25	Sulfate - turbidimetric
Sa-1	Sulfamic acid - gravimetric
Sa-2	Sulfamate - gravimetric
Si-1	Silicon - gravimetric
Sn-1	Tin - colorimetric

V-1	Versene - volumetric
Zx-3	Oxalic acid - volumetric
Zx-5	Butanol - volumetric
Zx-7	Mesityl Oxide - spectrophotometric
Zx-9	Aromatics - extraction
Zx-21	Olefins - volumetric
Zy-1H	Color - visual
Zy-3	Distillation Range - special
Zy-5	Refractive Index - special
Zy-6	Flash Point - closed cup
Zy-9	Viscosity - kinematic
Zy-10A	Specific Gravity - hydrometer
Zy-14	Water Insoluble - gravimetric
Zy-15	Suspended Solids - gravimetric
Zy-17	Not Precipitated by hydrochloric
Zy-20	Oxidizing Impurities - spectrophotometric
Zy-22	Loss on Drying - Residue - gravimetric
Zy-23	Residue on ignition - gravimetric
Zy-24	Determination of Particle Size - sieve
Zy-24a	Determination of Particle Size
Zy-25	Flame Photometry
Zy-26	Methyl Isobutyl Carbinol
Zz-1	Gases - absorption
Zz-21	Impurities - spectrographic

SECTION IV

ANALYTICAL PROCEDURES

DETERMINATION OF SILVER

Gravimetric Method

Principle

Silver ion reacts with HCl to form insoluble silver chloride, a suitable salt for gravimetric analysis. Antimony, lead, and mercury will also be precipitated, if present.

Procedure - Analysis of Silver Nitrate

Weigh accurately 1 g. of sample, dissolve in about 100 ml. of water, and heat to boiling. Add dropwise, with stirring, 5 ml. of 2 N HCl. Let stand for a few minutes, then decant the solution into a Selas crucible, and test the filtrate with a drop of dilute HCl to make sure all the silver is precipitated. Wash three times by decantation, using hot 1% HNO_3. Transfer the precipitate to the crucible and wash until free from chlorides, about six to eight additional washings being sufficient. Wash once with alcohol and dry at 110° C to constant weight.

Calculations

$$\% \ AgNO_3 = \frac{\text{g. AgCl} \times 118.52}{\text{g. sample}}$$

Literature References

1. Furman, "Scott's Standard Methods of Chemical Analysis", D. Van Nostrand Co., Inc., New York, 821 (1939) (5th Ed.).

Written by: L. F. Kendall (July, 1952)

DETERMINATION OF ALUMINUM

8-Hydroxyquinoline Method

Principle

8-Hydroxyquinoline precipitates aluminum quantitatively from a weak acid solution. A large number of other cations are also precipitated by this reagent. Interference by some of them, such as Mg, Ca, Ba, can be eliminated by carrying out the precipitation in a buffered acetic acid solution. However, other common ions, such as iron, nickel, copper, zinc, etc., precipitate under the same conditions and can best be removed by electrolysis at a mercury cathode. For the analyses indicated below, no interferences are normally expected. The procedure is generally applicable for 0.2-50 mg. of aluminum. Larger quantities give high results because of adsorption of excess precipitant. The precipitate forms according to the following reaction:

$$Al^{+++} + 3HOC_9H_6N \longrightarrow Al(OC_9H_6N)_3\downarrow + 3H^+$$

Solutions

8-Hydroxyquinoline, 5%. Dissolve 50 g. of 8-hydroxyquinoline in 120 ml. of glacial acetic acid and dilute to 1 liter with water. Filter if necessary and store in a dark bottle.

Procedure - Analysis of Alum

Weigh 0.4-0.55 g. of the sample. Dissolve in 200 ml. of water and continue as in general procedure.

Procedure - Analysis of Aluminum Nitrate Solution

Weigh a 10 g. sample and transfer quantitatively to a 250 ml. volumetric flask, dilute to volume and mix. Pipet a 10 ml. aliquot into a beaker and adjust volume to 100 ml., then proceed as indicated below.

Procedure - General

Adjust pH of sample to 4 with 1:1 NH_4OH. Add sufficient 5% 8-hydroxyquinoline to precipitate the aluminum. One ml. of the solution will precipitate 3 mg. of aluminum; an excess of 15-25% should be used. Heat to 60-70° C, add 4 M ammonium acetate dropwise until the yellow color deepens, then add 5 ml. additional for each 100 ml. of solution. The pH should now be between 5.0 and 5.4. Stir well and allow to stand 4 hrs. at room temperature.

Filter through a tared, sintered glass or Selas crucible and wash with cold water. Dry for 2 hrs. in an oven at 135-140° C. Cool in a desiccator and weigh.

Calculations

$$\% \ Al_2O_3 \text{ in alum} = \frac{\text{g. residue} \times 0.111 \times 100}{\text{g. sample}}$$

$$\% \ ANN = \frac{\text{g. residue} \times 0.8165 \times 100}{\text{g. sample in aliquot}}$$

References

1. Furman, N. H., "Scott's Standard Methods of Chemical Analysis", D. Van Nostrand, New York (1939), p. 12.

2. Mellan, I., "Organic Reagent in Inorganic Analysis", The Blakiston Co., Philadelphia (1941), pp. 119-129 and 239-244.

3. Welcher, F. J., "Organic Analytical Reagents", D. Van Nostrand Co., Inc., New York (1947) Vol. 1, pp. 285-292.

Written by: G. B. Barton (July, 1952)

DETERMINATION OF CALCIUM

Gravimetric Method

Principle

The determination of free calcium metal is carried out by ignition of the sample in air to the oxide. The gain in weight is used to calculate free calcium.

The determination of total calcium is accomplished by precipitation as the oxalate from a slightly ammoniacal solution or oxalate-oxalic acid buffered solution of the salts. The precipitate is ignited to calcium oxide for weighing. Other alkaline earths, ferric iron and aluminum, manganese and some of the rare earths may interfere. To avoid contamination of the calcium precipitate by Mg, Sr, and Ba, a double precipitation is desirable, particularly in the presence of large amounts of Mg. The presence of NH_4Cl hinders the precipitation of Mg. The remainder of the interfering elements if present in appreciable amounts, are removed prior to analysis by hydroxide precipitation. The precipitation from an oxalate-oxalic acid buffered solution enables direct separation from small amounts of these elements.

Procedure - Analysis of Calcium for Free Calcium

Weigh a one gram sample into a covered platinum dish, and cautiously add 30 to 40 ml. of distilled water (in hood). After the sample has cooled, wash the platinum covers, and then evaporate the sample to dryness on a hot plate. Ignite to constant weight at 1200° C, cool in a desiccator, and weigh (covered).

Procedure - Analysis of Magnesium Oxide

Weigh a 1 g. sample into a platinum crucible, add about 6 times as much Na_2CO_3, and mix thoroughly. Cover the crucible and cautiously bring the contents to the melting point, continuing the fusion until the melt is clear and homogeneous.

Cool the crucible, rotating it so the melt solidifies on the sides in a thin layer, immerse in a beaker of water and heat until the solid is dissolved. Carefully add 20 ml. of 70% perchloric acid to the solution, cover with a watch glass, and evaporate to fumes of $HClO_4$. Boil for 10 or 15 minutes after the fuming point (Remark 2), cool and dilute about 1 to 5. Heat to boiling, filter and wash with 1:100 HCl solution (Remark 3).

Dilute the combined filtrate and washings to 250 ml., take a 25 ml. aliquot for analysis, dilute it to approximately 100 ml., and neutralize with NH_4OH. Acidify the sample with concentrated oxalic acid, add a 10 ml. excess, and then add 20 ml. of saturated ammonium oxalate slowly and with continuous stirring.

Digest, filter, and wash the precipitate according to the general procedure, and then instead of igniting, dissolve the precipitate in 50 ml. of HCl. Neutralize with ammonium hydroxide, acidify with concentrated oxalic acid, and carry out the precipitation again in the same manner as above. Continue according to the general procedure.

Procedure - General

After the addition of ammonium oxalate is complete, allow the solution to digest for one hour on a steam bath, then filter through an ashless type medium filter paper (Whatman #42), and wash with water containing 2 g/l of ammonium oxalate.

Transfer the precipitate to a tared platinum crucible, dry, and ignite at about 1200° C till constant weight is obtained. Cool in a desiccator and weigh with a lid on the crucible.

Calculations

$$\% \text{ free Ca} = \frac{(\text{g. residue} - \text{g. sample}) \times 2.505 \times 100}{\text{g. sample}}$$

$$\% \text{ total CaO} = \frac{\text{g. ignited precipitate} \times 100}{\text{g. sample in aliquot}}$$

Remarks

1. In the determination of CaO in MgO this pretreatment is provided to remove silica. Instead of this step, the filtrate and washings from the determination of silica in the MgO sample may be taken and used at this point. Dilute to 250 ml., take a 25 ml. aliquot and continue as outlined above.

2. The ignited CaO must be weighed quickly since it will pick up moisture from the air. The desiccator must contain a better desiccant than CaO. $CaCl_2$ is not satisfactory.

Literature References

1. Furman, "Scott's Standard Methods of Chemical Analysis", D. Van Nostrand, Co., Vol. 1, p. 210 (1939).

2. Ibid., p. 214.

3. Griffin, "Technical Methods of Analysis", 2nd Ed., p. 428.

Written by: R. J. Brouns (July, 1952)

CALCIUM BY VERSENE

Volumetric

Principle

Calcium is estimated by titrating a solution of sample with a standard solution of the disodium salt of ethylenediamine tetraacetic acid (Versene). This standard solution has 0.1 g/l magnesium chloride added to provide a pink background color necessary in the use of Eriochrome black T indicator. An appropriate buffer must be used.

Interference by ions such as zinc, manganese, cadmium, aluminum, and magnesium can occur. This would lead to discovery of these elements in an essential material and is an advantage of the method. Magnesium in the standard is accounted for in standardization.

References:

Diehl, H., Goetz, C. A., and Hack, C. C. J. Am. Water Works Assoc., 42 40 (1950).

Betz, J. D., Noll, C. A. ibid, 42 49 (1950).

Goetz, C. A., Loomis, T. C. Analyt. Chem. 22 798 (1950)

Solutions

Standard Hard Water

Dissolve 1.000 g $CaCO_3$ in a minimum of HCl. Neutralize with ammonia solution (litmus). Gently boil to remove excess ammonia (litmus). Make to 1 L volume with distilled water. One ml is equivalent to 1 mg of $CaCO_3$ or 0.4004 mg calcium.

Standard Versene Solution

Prepare a solution in water of 4.0 g/l of the monohydrated disodium salt of ethylenediamine tetraacetic acid (Versene) containing 0.1 g/l magnesium chloride. Filter if necessary. Standardize by titrating 25.00 ml portions of standard hard water following the general procedure noted below.

Buffer Solution

Dissolve 67.5 g ammonium chloride in 386 ml distilled water. Add 570 ml of concentrated ammonia solution.

Indicator Solution

Weigh 0.500 g of Eriochrome Black T (color Index Number 203). Dissolve as far as possible in 100 ml methyl alcohol. Filter through filter paper.

Procedure - General

Dissolve samples to be tested in water. Adjust to neutrality with 1 + 2 ammonia solution using litmus. Add 2.0 ml of buffer solution. Add 3 drops of indicator titrate with Versene solution using the disappearance of the violet cast as the endpoint. Record mgm of Ca for one ml of Versene solution as the standard value.

Calculation

$$\% \text{ Ca} = \frac{\text{ml of Versene} \times 0.0400}{\text{g sample}}$$

Procedure - Analysis of Sodium Sulfate (< 0.02%) (#820)

Weigh a 50 g sample. Dissolve in 100 ml water. No adjustment with ammonia is necessary. Otherwise, follow general procedure.

Procedure - Analysis of Potassium Bicarbonate (<0.02%) (#743)

Weigh a 20 gm sample and transfer to a 400 ml beaker. Dissolve in 50 ml of water and carefully acidify by addition of 1:1 hydrochloric acid. Neutralize with 1 + 3 ammonia solution. Follow the general procedure.

Procedure - Analysis of Sodium Acetate (< 0.01%) (#780)

Follow the procedure for sodium sulfate using a 20 gram sample.

DETERMINATION OF CHLORIDE

Volumetric Method

Principle

Sodium chloride reacts with silver nitrate to precipitate silver chloride. The excess silver nitrate is then titrated with potassium thiocyanate, using ferric alum as an indicator. The silver thiocyanate precipitates, and when all the silver ions are removed, ferric thiocyanate forms, as indicated by the formation of a red color. The first tinge of a permanent pink in the solution indicates the endpoint of the reaction.

If the reaction flask stands exposed to light, the silver salts darken and turn gray. This obscures the endpoint and tends to give low values.

$$NaCl + AgNO_3 \xrightarrow{HNO_3} AgCl + NaNO_3$$

$$AgNO_3 + KSCN \longrightarrow AgSCN + KNO_3$$

$$6KSCN + 2(NH_4)Fe(SO_4)_2 \longrightarrow 2Fe(SCN)_3 + 3K_2SO_4 + (NH_4)_2SO_4$$

Solutions

Standardized potassium thiocyanate, 0.1 N.

Ferric alum indicator, saturated. Dissolve 140 g. of ferric ammonium sulfate ($Fe(NH_4)(SO_4)_2 \cdot 12H_2O$) in 400 ml. hot water. Cool and filter into a 500 ml. volumetric flask. Add 40 ml. concentrated nitric acid and dilute to the mark.

Procedure - Analysis of Sodium Chloride (Rock Salt)

Dry several grams of rock salt sample at 105-110° C for several hours, preferably overnight. Weigh 0.20-0.25 g. into a 300 ml. Erlenmeyer flask and dissolve in 20 ml. of distilled water. Add 5 ml. concentrated nitric acid and exactly 50 ml. of 0.1 N silver nitrate solution, mix thoroughly, and allow to stand in a dark cupboard for 30 min. (Remark 1).

Add about 3 ml. of ferric alum indicator and titrate with standardized potassium thiocyanate until the first permanent pink color is reached. Carry a reagent blank through the procedure.

Calculations

$$\% \text{ NaCl} = \frac{(A - B) \times N \text{ of KSCN} \times 5.845}{\text{g. sample}}$$

A = ml. KSCN for blank.
B = ml. KSCN for sample.

Remarks

1. The endpoint may be easier to see if the silver chloride is filtered out at this point. If this is done, the filter paper, flask, and precipitate should be thoroughly washed with several portions of 1:99 nitric acid.

References

1. Rosin, "Reagent Chemicals and Standards", D. Van Nostrand Co., 392 (1937).

Written by: M. C. Lambert (July, 1952)

DETERMINATION OF CHLORIDE

Turbidimetric

Principle

Silver ion forms a sufficiently reproducible turbidity with chloride ion to provide a measure of chloride ion. The sample must be dissolved in water and consideration given to such interferences as lead, monovalent mercury, monovalent copper, thallium, iodides, bromides, cyanides, thiocyanates, and thiosulfates. Specific procedures are noted below. The final solution should contain only a slight excess of silver nitrate and nitric acid. An excessive amount of either of these materials causes dissolution of silver chloride and leads to a low result. Chloride free water must be used for all reagent and dilutions.

Solutions

Silver nitrate. Prepare a 17 g/l solution of $AgNO_3$ (approximately 0.1 N) in 1 + 3 nitric acid. (Dilute conc. nitric acid 1 + 3). (Approx. 4 N).

Standardized chloride, 0.1 mg. Cl per ml. Prepare a 0.1648 g/l solution of sodium chloride in water.

Procedure - General

Weight or measure, preferably in a clean beaker, an amount of sample to yield 0.1 mgm chloride on the basis of the permissible maximum. Dissolve the sample in 20-25 ml of water. Add 1.0 ml of 1 + 3 nitric acid and mix. If the solution is turbid, filter preferably into a 50 ml glass stoppered cylinder. Rinse filter with four 5 ml portions of water bringing the volume in the cylinder to 40-45 ml. and proceed as below.

If the solution is clear, transfer to a 50 ml glass stoppered cylinder and add water to 40 ml. volume. Add 1.0 ml. of acid silver nitrate solution. Make to 50 ml. volume with water. Stopper and mix.

Prepare a standard. Place 40 ml of water, 1.0 ml. of 1 + 3 nitric acid, 1.0 ml. of standard chloride, and 1.0 ml. of acid silver nitrate in a 50 ml glass stoppered cylinder. Make to 50 ml. volume with water. Stopper, and mix.

Compare sample cylinder and standard. Turbidity in the sample must be less than in standard.

Variations on the general procedure are necessary with certain samples and are noted below.

Analysis of ANN Solution SpG 1.39 (< 0.005% Cl) (#060)

Measure 10 ml. (14 gm) of material by means of a volumetric flask or pipet. Transfer to a 100 ml. volumetric flask. Rinse pipet or 10 ml. volumetric flask with four 5-10 ml. portions of water and add to the 100 ml. flask. Make to volume, stopper, and mix. Filter a portion if not clear.

Transfer 14 ml. of clear solution representing 2.0 gm of original sample to a 50 ml. glass stoppered cylinder. Add 1.0 ml. of 1 + 3 nitric acid. Add water to 40 ml. volume. Add 1.0 ml. of acid silver nitrate. Make to 50 ml. volume with water. Stopper and mix.

Compare with a standard freshly made with 1.0 ml. of standard chloride.

Procedure - Analysis of Sodium Sulfate (< 0.01%) (#082)

Dissolve 1.0 gm in 20 ml. of water. Transfer to a glass-stoppered cylinder. Add 1 ml. of 1 + 3 nitric acid. Add 20 ml. of water and 1.0 ml. of acid silver nitrate. Stopper and mix. Compare turbidity with that of a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Citric Acid (< 0.01% Cl) (#285)

Dissolve 1.0 g in 20 ml of water. Add 1.0 ml of 1 + 3 nitric acid. Transfer to a 50 ml glass-stoppered cylinder. Add 1.0 ml of acid silver nitrate. Make to 50 ml volume and mix. Compare with a fresh standard made with 1.0 ml of standard chloride representing 0.1 mg of chloride ion.

Procedure - Analysis of Calcium (< 0.1% Cl) (#311)

Place a 1.0 g sample in a clean 250 ml beaker. Cover with a watch glass. Add 1 + 3 nitric acid slowly until the sample is dissolved and an acid reaction to litmus is obtained. Add 10 ml excess 1 + 3 nitric acid. Transfer the clear solution to a 100 ml volumetric flask and make to volume. Transfer 10 ml of this solution to a 50 ml glass-stoppered cylinder. Add water to 40 volume. Add 1.0 ml of acid silver nitrate. Make to 50 ml volume in water. Stopper and mix. Compare with fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Chromium Nitrate (< 0.005% Cl) (#340)

Dissolve a 2.0 g sample in 20-25 ml of water. Add 1.0 ml of 1 + 3 nitric acid, or equivalent. If solution is not clear, follow filtration step. If solution is clear, transfer to a 50 ml glass-stoppered cylinder. Add water to 40 ml volume. Add 1.0 ml of acid silver nitrate. Make to 50 ml volume with water. Stopper and mix. Compare turbidity with that of a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Ferrous Ammonium Sulfate (< 0.01% Cl) (#430)

Dissolve a 1.0 g sample in 20-25 ml of water. Add 1.0 ml of 1 + 3 nitric acid or equivalent. If solution is not clear, follow filtration step. If clear, transfer to a 50 ml glass-stoppered cylinder, add water to 40 ml volume, add 1.0 ml of acid silver nitrate. Make to 50 ml volume with water. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Ferrous Sulfamate (< 0.02% Cl) (Sp.G 1.5) (#432)

Transfer 5.0 ml (7.5 g) of sample to a 200 ml volumetric flask. Make to volume with water and mix.

Transfer 13 ml of the clear solution to a 50 ml glass-stoppered cylinder. Add 1.0 ml of 1 + 3 nitric acid. Dilute to 40 ml. Add 1.0 ml of acid silver nitrate. Make to 50 ml volume with water. Stopper, mix, and compare with a standard made with 1 ml of standard chloride.

Procedure - Analysis of Lead Nitrate (< 0.01% Cl) #576)

Dissolve 1.0 g in 20 ml of water. Transfer to a glass-stoppered cylinder. Add 1 ml of 1 + 3 nitric acid. Add 20 ml of water and 1.0 ml of acid silver nitrate. Stopper and mix. Compare turbidity with that of a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Nitric Acid (SpG ca. 1.4) (< 0.05% Cl) (#670) (671)

Transfer 3.6 ml (5 g) of nitric acid to a 100 ml volumetric flask. Make to volume with water. Transfer 4 ml of this solution representing 0.2 g of sample to 50 ml glass-stoppered cylinder. Add water to 40 ml volume. Add 1.0 ml of acid silver nitrate solution. Make to 50 ml volume with water. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Oxalic Acid (< 0.01% Cl) (#690)

Dissolve a 1.0 g sample in 40 ml of water. Add 1.0 ml of 1 + 3 nitric acid. Transfer to a 50 ml glass-stoppered cylinder. Add 1.0 ml of acid silver nitrate. Make to 50 ml volume with water. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Potassium Bicarbonate (< 0.01% Cl) (#743)

Dissolve 1.0 g in 20 ml of water. Add 1 + 3 nitric acid to acidify and 1.0 ml excess. Add 20 ml of water. Transfer to a glass-stoppered cylinder and add 1.0 ml of acid silver nitrate. Compare with a freshly prepared standard made with 1.0 ml of standard chloride solution representing 0.1 mg of chloride.

Procedure - Analysis of Silver Nitrate (<0.002% Cl) (#775)

Dissolve a 5.0 g sample in 40 ml of water. Add 1.0 ml of 1 + 3 nitric acid. Transfer to a 50 ml glass-stoppered cylinder. Make to 50 ml volume with water. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Sodium Acetate (<0.01% Cl) (#780)

Follow procedure for sodium sulfate.

Procedure - Analysis of Sodium Fluoride #799 (<0.1% Cl), Potassium Fluoride #713 (<0.1% Cl)

Dissolve 1.0 g in water. Transfer to 100 ml volumetric flask and make to volume with water. Transfer 10 ml representing 0.1 g to a glass-stoppered cylinder. Add 1.0 ml of 1 + 3 nitric acid. Add water to 40 ml volume. Add 1.0 ml of acid silver nitrate and 0.5 g boric acid. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Sodium Hydroxide (<0.9% Cl) (#800)

Mix 5.0 g sample with water and transfer to a 500 ml volumetric flask. Transfer 1.1 ml of this solution to a 50 ml glass-stoppered cylinder. Add water to 40 ml volume. Add 2.0 ml of 1 + 3 nitric acid. Add 1.0 ml of acid silver nitrate. Make to 50 ml volume with water. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Sodium Nitrate (<0.50% Cl) (#805)

Dissolve 1.0 g of sample in distilled water. Transfer to a 100 ml volumetric flask and make to volume. Transfer 2.0 ml representing 0.020 g of sample to a glass-stoppered cylinder. Add 1.0 ml of 1 + 3 nitric acid. Add 1.0 ml of acid silver nitrate solution. Make to 50 ml volume with water. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Sulfamic Acid (<0.02% Cl) (#870)

Dissolve a 0.5 g sample in 40 ml of water. Add 1.0 ml of 1 + 3 nitric acid. Transfer to a glass-stoppered cylinder. Add 1.0 ml of acid silver nitrate. Make to volume, stopper, and mix. Compare with a fresh standard made with 1.0 ml of standard chloride.

Procedure - Analysis of Hydrazine Solution (<0.05% Cl) (#890)

Place 40 ml of water in each of two glass-stoppered cylinders. To each add 1 ml of 1 + 3 nitric acid and 1 ml of acid silver nitrate. To one add further 1 ml of concentrated nitric acid and 1.0 ml of the hydrazine solution sample. To the other add 5 ml of 0.1 mgm/ml chloride standard. Mix. Compare. The turbidity of the sample tube should be less than that of the standard.

Procedure - Analysis of Tartaric Acid ($< 0.01\%\ Cl^-$) (#910)

Dissolve 1.0 g in 20 ml of water. Add 1.0 ml of 1 + 3 nitric acid. Transfer to a 50 ml glass-stoppered cylinder. Add 1.0 ml of acid silver nitrate. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride representing 0.1 mg of chloride ion.

Procedure - Analysis of Acetic Acid ($< 0.01\%\ Cl^-$) (#064)

Dissolve 1.0 ml in 20 ml of water. Add 1.0 ml of 1 + 3 nitric acid. Transfer to a 50 ml glass-stoppered cylinder. Add 1.0 ml of acid silver nitrate. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard chloride of 0.1 mg/ml concentration.

Procedure - Analysis of Permutit Anion Resin (#140)

To about 1 gram of 1 ml of the wet resin, add 10 ml of 1 + 1 HNO_3. Mix. Decant the supernate. Add 1 ml of 0.1 N silver nitrate. A distinct silver chloride precipitate should be observed.

Procedure - Analysis of Hydroxy Acetic Acid - #524 (< 0.1 Wt.% Cl^-)

Place a sample representing 1 gram of hydroxy acetic acid in a 100 ml volumetric flask. Make to volume with chloride free water. Transfer 10 ml to a glass-stoppered cylinder. Add 1.0 ml of 1 + 3 nitric acid. Add water to 40 ml volume and 1 ml of acid silver nitrate. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard 0.1 mg per ml chloride solution.

Procedure - Analysis of Ammonium Fluoride - Ammonium Nitrate - #0063 (< 0.1 Wt.% Cl^-)

Weigh a 2.0 g sample. Mix with 40 ml of water and transfer to a 50 ml g.s. cylinder. Add 1 ml of acid silver nitrate and 0.5 g boric acid. Mix.

Compare with a standard made with 4.0 ml of 0.1 mg/ml standard chloride solution.

Procedure - Analysis of Dibutyl Butyl Phosphonate - #0381

Place a 1.0 g (or ml) sample in a 100 ml volumetric flask. Add 50 ml of chloride-free water and 1 ml of concentrated nitric acid. Shake vigorously and make to volume with water. Decant through a filter paper that has been wetted with chloride-free water.

Transfer 10 ml of the clear filtrate to a glass-stoppered cylinder. Add 1.0 ml of 1 + 3 nitric acid. Add water to 50 ml volume and 1 ml of acid silver nitrate. Stopper and mix. Compare with a fresh standard made with 1.0 ml of standard 0.1 mg per ml chloride solution.

Procedure - Analysis of Sodium Bismuthate #0782 (.01%)

Add 2 g to 25 ml of water, heat to boiling and keep at the boiling temperature for 10 minutes. Dilute to 50 ml and filter through a filter that has been washed free of chlorides. To 25 ml of the filtrate, add 0.15 ml of 30% hydrogen peroxide to clear the solution.

Add 1 ml of nitric acid and 1 ml of silver nitrate reagent. The turbidity should not exceed that produced by 1 ml of standard 0.1 mg per ml chloride.

DETERMINATION OF COMBINED CHLORIDE AND BROMIDE

Turbidimetric Method

Principle

Silver chloride and silver bromide are soluble in ammonium hydroxide; silver iodide is insoluble and may be separated from a mixture of the three halides by filtration. Acidification of the filtrate destroys the soluble silver-ammonium complexes and reprecipitates the halides. The combined chloride and bromide may then be estimated turbidimetrically. If free halogen is present in the original sample, it must first be reduced to halide by some agent, such as sulfurous acid. Cyanides, thiosulfates, thiocyanates, and strongly colored ions interfere.

Solutions

Standardized chloride, 0.1 mg. Cl per ml. Dissolve 4.120 g. NaCl in 1 l. water.

Procedure - Analysis of Iodine

Grind 5 g. sample with 25 ml. water, let stand for 30 min. with occasional stirring and filter. To 20 ml. of the filtrate add sulfurous acid until the amber color disappears. Avoid an excess. Carry through general procedure, using 2 ml. of standardized chloride as comparison standard (aliquot = 0.80).

Procedure - General

Dilute the sample aliquot and the comparison standard to 25 ml. Add 1 ml. of ammonium hydroxide (28%) to each solution, then add with stirring 2 ml. of 3.5% silver nitrate. Boil gently 5 min., cool thoroughly, and filter (Remark 1). Wash filter with 5 ml. water. Add 1:1 nitric acid dropwise to the filtrate until cloudiness appears; then, add 10 drops in excess. Visually compare turbidity of sample with that of the aliquot (Remark 2).

Calculations

$$\% \text{ Cl(Br)} = \frac{\text{ml. std. chloride} \times 0.01}{\text{g. sample} \times \text{aliquot}}$$

Remarks

1. Caution: Silver-ammonium complexes have been known to form dangerously explosive mixtures on standing. Therefore, do not interrupt the procedure after the silver nitrate has been added to the ammonia solution. Also do not allow the solution to boil dry.

2. For more accurate work dilute the solutions to a final volume of 50 ml. and compare their transmittancies on a spectrophotometer. A standard curve may be prepared for this purpose, using the standardized chloride solution.

Literature References

1. A.C.S., "Specifications for Analytical Reagents", (1942).

2. Rosin, "Reagent Chemicals and Standards", D. Van Nostrand Co., (1939).

Written by: J. E. Meinhard (July, 1952)

CARBON IN CALCIUM

Principle

Carbon in calcium metal is oxidized to carbon dioxide in a stream of purified oxygen at 900 C. The carbon dioxide is separated from water and other absorbable combustion products by means of traps. The carbon dioxide is collected by freezing in a trap cooled with liquid nitrogen. Excess oxygen is pumped off. The trap and manometer system are isolated. The carbon dioxide is vaporized and its volume measured.

Apparatus

The carbon apparatus is shown in Figure 1.

Reagents

1. Silica gel
2. Dri-Rite
3. Ascarite
4. Copper turnings, amalgamated
5. Magnesium perchlorate (Anhydrone)
6. Liquid nitrogen
7. Reagent grade oxygen.

Procedure

Weigh 100 ± 5 mg of calcium into a capsule. Verify the preheater temperature of 900 C and combustion furnace temperature at 850 C. Verify proper operation of vacuum pumps. Make sure valves V2 and V3 are closed. Slowly open valve V3. Adjust mercury to zero.

Open V1 and the other valves in the oxygen line. Adjust the oxygen pressure to 2-4 pounds. Keep oxygen pressure low. The arrow on V4 should point up. Open V2 slowly until mercury column reads about 13 cm. Do <u>NOT</u> try to over-control V2.

Place the sample in the dumper. Arrange liquid nitrogen at the cold trap.

Close V1, turn arrow on V4 to the right momentarily, then turn arrow back to the upward position.

Dump sample.

When the buzzer sounds (after approximately 10 minutes) open V1, and slowly close V2.

Adjust the mercury to zero and close V3. Remove the liquid nitrogen and warm the cold trap. Tap manometer gently and record reading.

To run the blank, omit the dumping of the sample.

Calculations:

$$\text{ppm C} = \frac{\text{W sample} - \text{W blank}}{\text{grams sample}}$$

where $W = \mu g\ C = (A + BX)X$

A and B are calibration constants. X is manometer reading.

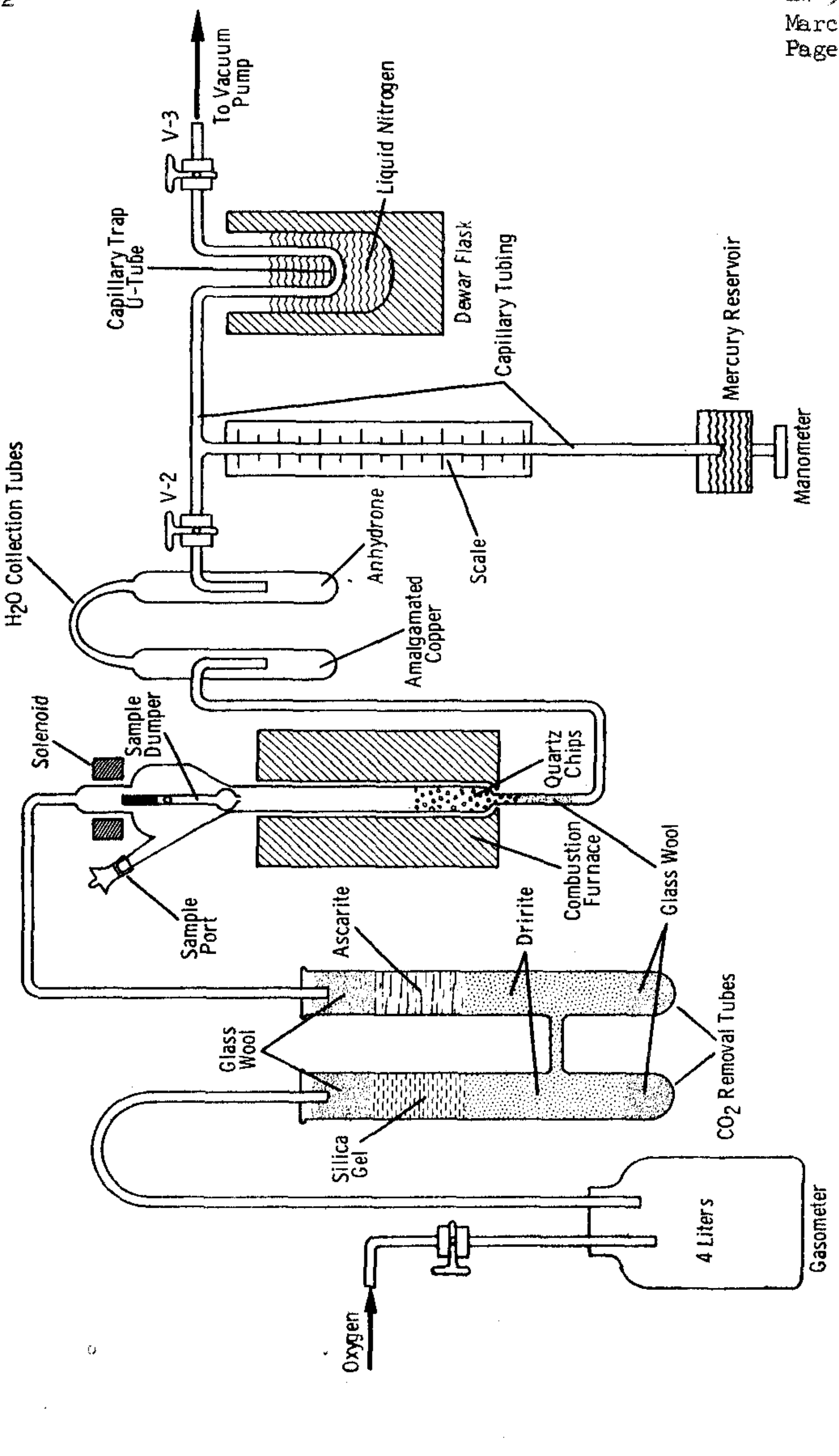


FIGURE - I
CARBON APPARATUS

DETERMINATION OF FLUORIDE

Volumetric

Principle

Fluoride ion is titrated with standard thorium nitrate using sodium alizarin sulfonate as indicator.

Reagents

0.05% sodium alizarin sulfonate in water.
0.02 N sodium hydroxide (not standardized).
0.02 N hydrochloric acid (1.6 ml conc. HCl per liter).
1.0 N acetic acid (6.0 ml glacial acetic per 100 ml).

Standard 0.1 N thorium nitrate, standardized against reagent grade sodium fluoride dried at 150 C for two hours.

Procedure - General

Weigh a sample containing between 0.06 and 0.08 grams of fluoride ion in a plastic beaker. Add 25 ml of water and 0.5 ml of indicator. Adjust the acidity by dropwise addition of 0.02 N base or acid until upon addition of acid the pink color is just discharged. Add 0.4 ml of 1 N acetic acid. While stirring, titrate slowly with 0.1 N thorium solution to a permanent pink color.

Calculations

1.00 ml of 1 N thorium is equivalent to 0.018998 grams fluoride ion.

$$\text{ml of thorium } \frac{\text{sol'n x normality}}{\text{g sample}} \times .018998 \times 100 = \%\ F$$

Reference: Rosin, J. Reagent Chemicals and Standards. G. Van Nostrand Co., Inc. 4th Ed. (1961) p. 335.

Procedure - Analysis of Ammonium Fluoride, Ammonium Nitrate Mixture (#0063)

Place about 0.2 ml of sample in a tared 125 ml Erlenmeyer flask. Weigh accurately. Add 25 ml of water and 0.5 ml of indicator, add 5 ml of 0.02 N HCl and 1.0 ml of 1 N acetic acid. Titrate slowly with 0.1 N thorium solution to a permanent pink color.

Note 1. With this sample size of this material the titration may be taken rapidly to 20 ml and then in one-drop increments to the end point. Observe the end point against a white background.

Note 2. As this material is strongly buffered, a comparatively large amount of acid is needed to adjust the pH.

1 ml of 1 N thorium solution is equivalent to 0.03704 g NH_4F

$$\frac{\text{ml of thorium sol'n} \times \text{normality}}{\text{g sample}} \times 0.03704 \times 100 = \%\ NH_4F$$

Procedure - Analysis of Potassium Fluoride (#0713)

Place about 1.4 g of sample in a tared 125 ml Erlenmeyer flask. Weigh accurately. Add 25 ml of water, add 1 ml of indicator. Add 5 ml of 0.02 N HCl and 1.0 ml of 1 N acetic acid. Titrate slowly with 0.1 N thorium nitrate to a permanent pink color. One ml of 0.1 N thorium nitrate is equivalent to 0.005810 g KF.

DETERMINATION OF R_2O_3

Gravimetric Method

Principle

The sample is converted to a soluble form and the insoluble hydroxides of the R metal ions are ignited to R_2O_3.

Anions, such as phosphate, arsenate, vanadate, and silicate, form insoluble compounds and must be absent. Organic materials, as tartaric acid or sugar, form soluble complexes with iron and aluminum, and must therefore be removed. Oxidizing agents, such as bromine, may oxidize divalent manganese and cause its precipitation. In the absence of phosphate or carbonate, the alkali and alkaline earth metals present no difficulties when sufficient ammonium salt is present.

$$R^{+++} + 3NH_4OH \longrightarrow R(OH)_3 + 3NH_4^+$$

$$2R(OH)_3 \xrightarrow{\Delta} R_2O_3 + 3H_2O$$

Procedure - Analysis of Magnesium Oxide

Weigh accurately a 1 g. sample into a platinum crucible, add 5 g. sodium carbonate, mix, and cover. Heat cautiously until the mixture becomes a clear and homogeneous melt.

After the molten mass has solidified on the sides of the crucible, immerse the crucible and cover in a beaker of water and heat to effect solution of the contents. After the crucible has been rinsed and removed, add 20 ml. of perchloric acid, and boil the solution for 15 min. after copious fumes of the perchloric acid evolve. Do not permit the contents of the beaker to become solid or the silica separation will be incomplete.

Cool the contents of the beaker, dilute 1:5 with distilled water, heat to boiling, and quantitatively remove the silica by filtration through Whatman No. 42 filter paper. Wash with 1:99 hydrochloric acid and then with water until washings are acid free. Dilute the filtrate and washings to 250 ml. Take a 50 ml. aliquot and add 15 ml. of concentrated hydrochloric acid. Continue according to the general procedure.

Procedure - Analysis of Oxalic Acid

Weigh accurately 20 g. of sample and transfer to a platinum dish. Heat slowly and carefully with a Bunsen flame until the acid melts; ignite the fumes given off in order to obviate the danger of explosive air-fume mixtures. After the residue has been ignited, add 5 g. of potassium acid sulfate or potassium pyrosulfate, and fuse until the melt is clear. Extract the melt with 100 ml. of distilled water to which has been added 15 ml. of concentrated hydrochloric acid and continue as indicated in the general procedure.

Procedure - General

Add 1 ml. of concentrated nitric acid to the solution and boil for 5 min. Filter through a No. 40 Whatman paper and wash with water.

Add a few drops of methyl red (0.2% alcoholic solution) and heat just to boiling. Carefully add dilute ammonium hydroxide drop by drop until the color of the solution changes to a distinct yellow. Boil gently for 1-2 min. and filter immediately through a No. 41 Whatman paper or OK Swedish paper. Wash the precipitate thoroughly with hot, 2% ammonium chloride and then suck dry (Remark 1).

Place the paper and precipitate in a tared platinum crucible and cautiously smoke off the paper. Ignite the crucible in a muffle at 900° C. and accurately weigh the residue.

Calculations

$$\% \; R_2O_3 = \frac{\text{g. residue} \times 100}{\text{g. sample in aliquot}}$$

Remarks

1. If the nature or amounts of other elements present call for it, dissolve the precipitate in 25 ml. of 1:4 hydrochloric acid and reprecipitate as before.

References

1. Treadwell and Hall, "Analytical Chemistry", John Wiley and Sons, Inc., Ed. 9, Vol. II, 148-171 (1937).

2. Griffin, "Technical Methods of Analysis", McGraw-Hill Book Co., Inc., 148 (1927).

3. Kolthoff and Sandell, "Testbook of Inorganic Quantitative Analysis", MacMillan Co., 294-307 (1936).

4. Hillebrand and Lundell, "Applied Inorganic Analysis", John Wiley and Sons, Inc., 341, 397 (1929).

Written by: T. K. Bierlein (July, 1952)

IRON DETERMINATION

Colorimetric Method
Range 20 to 120 micrograms

Principle

O-phenanthroline reacts with ferrous iron in slightly acid solution (pH 3-6) to form a red-colored complex. In order to determine total iron, it is necessary to reduce any ferric iron present by means of hydroxylamine hydrochloride.

Substances precipitated by ammonium hydroxide (group III Al, V, etc.) interfere by precipitating at the pH used. The addition of 1 ml. of 10% ammonium or sodium citrate prevents such interference. The addition should be made just before neutralizing. No other interference has been encountered.

Solutions

Ortho-phenanthroline solution, 0.1%. Dissolve 0.5 g. ortho-phenanthroline in 50 ml. of distilled water in a 500 ml. volumetric flask and dilute to the mark with distilled water.

Standard ferrous sulfate solution, 1 ml. = 10 micrograms of iron. Carefully add 50 ml. concentrated sulfuric acid to 250 ml. of distilled water in a 500 ml. volumetric flask. Dissolve 0.0351 g. of ferrous ammonium sulfate in this solution and dilute to volume with distilled water.

Procedure - Analysis of Aluminum Nitrate (ANN)

Weigh accurately a 10.0 g. sample of aluminum nitrate solution into a beaker and add 25 ml. of concentrated sulfuric acid and 5 ml. of water. Add formic acid and heat if necessary to remove the nitrate; continue the formic acid addition until no brown fumes are expelled. Heat the solution until white fumes are observed, cool, and add 100 ml. water. Heat to dissolve crystals and filter into a 250 ml. volumetric flask. Dilute to volume with water. Using a 1 ml. aliquot, proceed as described in the general procedure.

Procedure - Analysis of Chromium Nitrate Salt

Weigh accurately 2.50 g. sample and dissolve with 50 ml. of water, 5 ml. of 50% sodium hydroxide, and 5 ml. of 30% hydrogen peroxide. Boil to expel the excess peroxide. Acidify with HCl, neutralize with NH_4OH, and boil to expel excess ammonia. Filter and wash the precipitate with cold water, discarding filtrate. Dissolve the precipitate with 10 ml. of hot 1:1 HCl. Transfer the solution through a filter into a 100 ml. volumetric flask and wash filter with hot water. Using a 10 ml. aliquot, proceed as described in the general procedure.

Procedure - Analysis of Hydrogen Peroxide

Weigh accurately 100 ml. of sample into a platinum dish. After evaporating to dryness, rinse down the sides with 2 ml. of hydrochloric acid and 5 ml. water. Re-evaporate to 1-2 ml. volume and transfer to the 50 ml. volumetric flask. Using the entire sample, proceed as described in the general procedure.

Procedure - General

Deliver the specified aliquot into a 50 ml. volumetric flask. Add 1 ml. of 10% hydroxylamine hydrochloride and mix solution. Add 1 ml. of ortho-phenanthroline and 5 ml. of 10% ammonium citrate. Adjust the pH of the solution 3-6 with 1 M hydrochloric acid or 1 M ammonium hydroxide as required. Dilute to mark with water and mix solution. Compare in spectrophotometer at 515 mu against sample blank prepared in the same manner and containing all reagents except ortho-phenanthroline, using 2 cm. cuvettes.

Compare the transmittancy obtained against the transmittancy obtained by carrying 1, 3, 5, and 7 ml. of the standard iron solution through the same procedure.

Calculations

$$\% \text{ Fe} = \frac{\text{g. Fe in aliquot} \times 100}{\text{g. sample in aliquot}}$$

$$\text{ppm Fe} = \% \text{ Fe} \times 10^4$$

References

1. Smith, G. F., "Phenanthroline and Substituted Phenanthroline Indicators," The G. Fredrich Smith Chemical Co., Columbus, O., 103 pp. (1944).

Written by: C. A. Goodall (July, 1952)

Procedure - Analysis of Phosphoric Acid (#0744)

Weigh accurately a 0.3 to 0.4 g sample in a 150 ml beaker. Add 20 ml of distilled water and two drops of 1% thymolphthalein indicator. Titrate with 0.2 N NaOH to a faint blue end point.

1 ml of N NaOH corresponds to 0.04900 g of H_3PO_4

HYDROGEN ION, ACID ASSAY

Volumetric Method

Principle

The amount of standard sodium hydroxide solution needed to neutralize the acid present in a sample of the essential material is determined. An indicator that changes color at the equivalence point is used.

Solutions

Standardized sodium hydroxide solution of low carbonate content.

0.1% alcoholic solution of methyl red.

0.1% alcoholic solution of phenolphthalein (preferably in isopropyl alcohol).

General Procedure

Transfer a weighed quantity of the sample to a suitable vessel such as a 200 ml Erlenmeyer flask. Add 25 ml of water, or, in special cases, isopropyl alcohol. Add 3 drops of indicator solution and titrate with standard base.

Calculations

$$\%\text{ of the acid} = \frac{(\text{ml of NaOH})(\text{Normality of NaOH})(F)(100)}{\text{g of sample}}$$

If a measured volume of sample was used, then the g of sample must be obtained by multiplying the volume of sample by an observed (pycnometer) specific gravity of the sample.

The factor F is supplied under each of the following procedures.

Procedure - Analysis of Nitric Acid

Weigh accurately 2.0 - 2.5 g of sample into an Erlenmeyer flask containing 25 ml of distilled water. Titrate the sample with normal sodium hydroxide to the endpoint using 1 drop of 0.1% alcoholic solution of methyl red as the indicator.

F = 0.06320 = g HNO_3 per ml of 1.0 N sodium hydroxide.

Procedure - Analysis of Sulfuric Acid

Weigh accurately 0.1-0.14 g of sample into an Erlenmeyer flask containing 25 ml of water. Titrate the sample with standardized sodium hydroxide to the endpoint, using 1 drop of 0.1% alcoholic solution of methyl red as the indicator.

F = 0.04904 = g H_2SO_4 per ml of 1.0 N sodium hydroxide.

Procedure - Analysis of Citric Acid #285

Accurately weigh a 2.5 to 3.0 g sample of the solid. Transfer to a flask. Dissolve in 25 ml of water. Add 3 drops of phenolphthalein. Titrate with standard normal sodium hydroxide.

F = 0.06404 = g citric acid per ml of 1.0 N sodium hydroxide.

Procedure - Analysis of Di(2-ethylhexyl) Phosphoric Acid #384

Weigh accurately about 1.6 - 2.4 g of sample into an Erlenmeyer flask. Add about 50 ml of isopropyl alcohol and about 3 drops of neutral phenolphthalein indicator solution. Titrate to pink color with standard fifth normal sodium hydroxide.

F = 0.3224 = g D2EHPA per ml of 1 N sodium hydroxide

Procedure - Analysis of Tartaric Acid #910

Weigh accurately about 4.0 g of the sample. Transfer to an Erlenmeyer flask. Add 25 ml of water to dissolve sample. Titrate with standard 1.0 N NaOH, using 3 drops of phenolphthalein indicator. One ml N NaOH is equivalent to 0.07504 grams of tartaric acid.

Procedure - Analysis of Acetic Acid (#064)

Place about 10 ml of water in a glass-stoppered flask. Stopper and weigh accurately. Carefully add about 0.5 ml of sample to the flask. Stopper and weigh.

Titrate with standard 0.2 N or normal sodium hydroxide of low carbonate content using phenolphthalein indicator.

1 ml of normal sodium hydroxide is equivalent to 0.06005 grams of CH_3COOH.

Procedure - Analysis of Hydroxy Acetic Acid #0524 (M. Wt. 76.03)

Tare a 250 ml g S Erlenmeyer flask, add from 3.3 to 3.5 g of 70% hydroxyl acetic acid (2.3 to 2.5 g of 100%) and weigh accurately. Record the net weight. Add 50 ml of water to the flask and 5 drops of neutral phenolphthalein indicator solution. Titrate to a phenolphthalein endpoint with standard normal sodium hydroxide. At this point all the free hydroxy acetic acid has been neutralized. This volume may be noted, if of interest. Continue the addition of the standard normal sodium hydroxide until a total of about 40 ml have been added. Record the buret reading. Place a small funnel in the neck of the flask and boil gently (low hot plate) for 30 minutes. Replace any water lost on boiling. Back-titrate with standard normal sulfuric acid. Record the volume.

Calculation

Compute wt.% of hydroxy acetic acid:

$$\text{Wt.\%} = \frac{(\text{Ml base x normality - ml sulfuric acid x normality})}{\text{g sample}} \times .07603 \times 100$$

Note: The difference in the two end points is attributed to an anhydride.

DETERMINATION OF FREE ACID

Volumetric Method

Principle

Free acid in a solution of a hydrolyzable material such as aluminum nitrate is determined by direct titration, after the addition of potassium oxalate. The oxalate is used to repress the hydrolytic effect (equation 1) through the formation of a complex ion (equation 2).

1) $Al^{+3} + H_2O = Al(OH)^{+2} + H^+$

2) $Al^{+3} + 3\ C_2O_4^{=} = Al(C_2O_4)_3^{-3}$

Solutions

Standardized sodium hydroxide, 0.3 N.

Standardized nitric acid 0.1 N.

Procedure - Analysis of Aluminum Nitrate

Weigh accurately 10 g of sample into a beaker. Add 10 ml distilled water to dissolve the sample.

Adjust a pH meter, checking the meter and electrodes with standard buffer mixtures. Immerse the electrodes in the beaker containing the sample, so that the electrodes are not touching the beaker bottom or walls.

Add potassium oxalate crystals in small portions until further addition causes little change in the pH. (The pH should be about 4.7). If the pH rises to 7, spike the samples with a known amount of standard nitric acid and add oxalate crystals again.

Titrate the solution with 0.1 N sodium hydroxide, making the additions in small increments. Measure the pH after the addition of each increment and record the volume of reagent and pH. Continue the titration until a large change in pH, which indicates the end point, is passed.

Calculations

Ml NaOH at end point = total ml NaOH added from all increments preceding

$$A \text{ plus } \frac{A-B}{2A-C} \times D$$

A = maximum change in pH in one increment
B = change in pH for increment preceding A
C = change in pH for increment following A
D = increment used in titration

$$\text{Free acid, as } HNO_3\% = \frac{\text{ml NaOH}^{*} \times \underline{N} \text{ NaOH} \times 6.302}{\text{wt. sample}}$$

* An appropriate correction to the volume of NaOH used must be made if an acid spike is added.

Procedure - Analysis of **A**luminum Nitrate Solution #060 (1.1 to 1.6 wt. per cent) acid deficient.

Accurately transfer a sample of about 4.0 g to a 100 ml beaker. (If the specific gravity is known, a 3.0 ml micro pipet may be used and rinsed). Add 15 ml of water to the sample.

Provide a pH meter with glass and calomel electrodes checked with a standard buffer. Provide a magnetic stirrer.

Immerse the electrodes in the beaker containing the sample. Start stirring. Add accurately from a burette or micro pipet 2.0 ml of 1 N nitric acid. Add potassium oxalate crystals in small portions until further addition causes little change in the pH. The pH should be between 4.7 and 4.9.

Titrate with standard sodium hydroxide preferably 0.3 N using 0.2 ml increments. Record the pH at each increment and the volume of titrant. Continue the titration until the large change in pH, which indicates the end point is passed.

Calculations

Compute the end point.

Compute the per cent acid deficiency as negative free acid.

$$(\text{minus})\%\ HNO_3 = \frac{(\text{ml NaOH} \times \text{norm} - \text{ml } HNO_3 \times \text{norm}) \times 6.320}{\text{Wt. sample}}$$

DETERMINATION OF ACIDITY

Volumetric Method

Principle

The sample is titrated with sodium hydroxide to the phenolphthalein endpoint to determine the acid content.

Solutions

Phenolphthalein, 1%. Dissolve 5 g phenolphthalein, $C_{20}H_{14}O_4$, commercial dye, in 250 ml 95% ethyl alcohol in a 500 ml volumetric flask. Dilute to the mark with the alcohol. Mix well and transfer to a glass-stoppered bottle.

Standardized sodium hydroxide, 0.1 N.

Procedure - Analysis of Hexone #0640, Tributyl Phosphate #0920, Dibutyl Butyl Phosphonate #0381

Pipet a 50 ml sample into 50 ml of distilled water in an Erlenmeyer flask. Add three drops of phenolphthalein and titrate with 0.1 N sodium hydroxide to a faint pink endpoint which persists for 30 seconds. Shake the flask vigorously after each addition of sodium hydroxide.

For a blank, titrate 50 ml of the distilled water, to which three drops of phenolphthalein have been added, with standard sodium hydroxide.

Calculations

$$\text{Acidity, as normality} = \frac{\text{ml of std. NaOH (sample - blank)} \times \underline{N} \text{ of NaOH}}{50}$$

$$\text{Acidity, as acetic acid, wt.\%} = \frac{\text{ml of std. NaOH (sample-blank)} \times \underline{N} \text{ of NaOH} \times 6.003}{50 \times \text{Sp. G of sample}}$$

$$\text{Acidity, as acetic acid, Vol. \%} = \frac{\text{ml of std. NaOH(sample-blank)} \times \underline{N} \text{ of NaOH} \times 5.724}{50}$$

Original by: E. M. Kinderman (July, 1952)

DETERMINATION OF MERCURY

Volumetric

PRINCIPLE

A direct titration with thiocyanate with ferric nitrate as indicator serves.

SOLUTIONS

Standard 0.1 N potassium or ammonium thiocyanate.

Ferric Nitrate Indicator solution (10 g ferric nitrate and 2 ml concentrated nitric acid in 100 ml water.)

PROCEDURE - GENERAL

Accurately transfer an appropriate sample to an Erlenmeyer flask. Dissolve in 5 ml nitric acid and 100 ml water. Add 2 ml of ferric nitrate indicator and titrate with standard 0.1 N thiocyanate.

$$\% \text{ Mercury} = \frac{\text{ml thiocyanate} \times \text{normality} \times .1713}{\text{g sample}} \times 100$$

PROCEDURE - Analysis of Mercuric Nitrate (#0626)(99%)

Accurately weigh about 0.5 g. Follow the general procedure. Multiply per cent mercury by 1.708 to give per cent $Hg(NO_3)_2.H_2O$.

Code H-11

Procedure - Analysis of Limestone

Weigh accurately about 2.0 g of sample into a 250 ml flask. Proceed as in Lime, hydrated above.

Procedure - Analysis of Potassium Bicarbonate #743 (99.5 - 100.5%)

Accurately weigh about 3 grams of sample. Dissolve in 50 ml of water and titrate with 1 N acid using Methyl orange indicator.

One milliliter of 1 N acid is equivalent to 0.1001 grams of $KHCO_3$

Calculations

$$\%Na_2O = \frac{\text{ml. acid} \times N \times 0.0310 \times 100}{\text{g sample in aliquot}}$$

$$\%CaO = \frac{(\text{ml. acid} \times N - \text{ml. base} \times N) \times 0.02804 \times 100}{\text{g sample}}$$

$$\%KHCO_3 = \frac{\text{ml acid} \times N \times 0.1001}{\text{g sample}}$$

DETERMINATION OF IODINE

Colorimetric Method

Principle

Iodide and iodine are oxidized to iodate by hot bromine water in dilute acid solution. Iodine is liberated by potassium iodide after the bromine is expelled thus giving six times as much iodine as was originally present. The color developed with starch indicator is measured with the Beckman Quartz Spectrophotometer.

$$5HI + HIO_3 \longrightarrow 3I_2 + 3H_2O$$

Solutions

Potassium iodide	.001 mg I/ml
Starch indicator	
Sulfuric acid	2N
Bromine water	Saturated
Salicylic acid	1.0%
Potassium iodide	5%

Procedure - Sodium Nitrate

Weigh accurately 10.0 gram sample. Dissolve in 30 ml. water and filter into 100 ml. volumetric flask. Dilute to volume and mix. Pipet 5 ml. into a 50 ml. Erlenmeyer flask. Add 2 drops 2N sulfuric acid and 5 drops of saturated bromine water. Heat to boiling until yellow color disappears. Add 2 drops 1 percent salicylic acid while hot. Cool to room temperature, add 3 drops starch solution and 3 drops 5 percent potassium iodide.

Transfer to 10 ml. volumetric flask and dilute to volume.

Transfer a portion of the sample into an absorption cell having a path length of 1 cm. Measure the optical density against a reference cell containing all reagents.

Determine the mg. of iodine from a standard curve prepared by running 0, 1, 2, 3, 4, 5, 6, 7, and 8 ml. of the 0.001 mg/ml iodide standard through the above procedure.

Procedure - Sodium Nitrate (Solution)

Use a 20 gram sample. Dilute to 30 ml. and follow above procedure.

Calculations

$$\text{Percent Iodine} = \frac{\text{mg. Iodine}}{\text{sample wt. x aliquot x 10}}$$

Literature Reference

R. G. Turner, J. Am. Chem. Soc. 52, 2768-73 (1930)

Written by: F. P. Roberts (May, 1956)

DETERMINATION OF IODINE

Colorimetric Method

Principle

Iodide and iodine are oxidized to iodate by hot bromine water in dilute acid solution. Iodine is liberated by potassium iodide after the bromine is expelled thus giving six times as much iodine as was originally present. The color developed with starch indicator is measured with the Beckman Quartz Spectrophotometer.

$$5HI + HIO_3 \longrightarrow 3I_2 + 3H_2O$$

Solutions

Potassium iodide	.001 mg I/ml
Starch indicator	
Sulfuric acid	2N
Bromine water	Saturated
Salicylic acid	1.0%
Potassium iodide	5%

Procedure - Sodium Nitrate

Weigh accurately 10.0 gram sample. Dissolve in 30 ml. water and filter into 100 ml. volumetric flask. Dilute to volume and mix. Pipet 5 ml. into a 50 ml. Erlenmeyer flask. Add 2 drops 2N sulfuric acid and 5 drops of saturated bromine water. Heat to boiling until yellow color disappears. Add 2 drops 1 percent salicylic acid while hot. Cool to room temperature, add 3 drops starch solution and 3 drops 5 percent potassium iodide.

Transfer to 10 ml. volumetric flask and dilute to volume.

Transfer a portion of the sample into an absorption cell having a path length of 1 cm. Measure the optical density against a reference cell containing all reagents.

Determine the mg. of iodine from a standard curve prepared by running 0, 1, 2, 3, 4, 5, 6, 7, and 8 ml. of the 0.001 mg/ml iodide standard through the above procedure.

Procedure - Sodium Nitrate (Solution)

Use a 20 gram sample. Dilute to 30 ml. and follow above procedure.

Calculations

$$\text{Percent Iodine} = \frac{\text{mg. Iodine}}{\text{sample wt. x aliquot x 10}}$$

Literature Reference

R. G. Turner, J. Am. Chem. Soc. 52, 2768-73 (1930)

Written by: F. P. Roberts (May, 1956)

MAGNESIUM

Colorimetric

Principle

The pink color shown by Titan Yellow (Clayton Yellow) dye with magnesium ion in alkaline solution affords a measure of magnesium concentration.

Solutions

(1) Standard magnesium solution containing 0.01 mg of magnesium in 1 ml. Dissolve 1.014 grams of clear crystals of magnesium sulfate heptahydrate, $MgSO_4.7H_2O$, in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 liter.

(2) Titan Yellow Solution.

Dissolve 0.1 gm of Titan Yellow in 100 ml of water.

(3) Sodium Hydroxide Solution, 1 N.

Dissolve 4.0 grams of reagent sodium hydroxide in 100 ml of water.

Procedure - General

Prepare an aqueous neutral solution from the sample or from the residue on ignition (Zy-23). This solution should be practically colorless.

Transfer an aliquot that should contain, as a limit, less than 0.10 milligrams of magnesium to a 50 ml glass stoppered cylinder. From the cylinder transfer one-fourth of the aliquot to a second comparison cylinder leaving an aliquot representing less than a limit of 0.075 milligrams in the first cylinder.

To the comparison cylinder add 5 ml of standard magnesium solution (0.05 milligrams).

Adjust to equal volumes in both cylinders with water. Add 0.15 ml of Titan yellow solution to each cylinder. Mix. Add 1.0 ml 1 N sodium hydroxide to each cylinder. Mix. The sample cylinder should show less color than the comparison cylinder. One third the color due to sample in the sample cylinder is compensated by sample in the comparison cylinder. The other two thirds of the aliquot in the sample cylinder must thus show less than 0.05 milligrams of magnesium to pass the test.

ASSAY OF MAGNESIUM OXIDE

Volumetric

Principle

An ignited sample is treated with a measured excess of standard sulfuric acid. Excess acid is then titrated with standard sodium hydroxide using methyl orange indicator.

A correction for calcium oxide should be made.

Procedure, General

Ignite about 500 mg of sample to constant weight in a small tared platinum crucible. Weigh the residue accurately.

Place the crucible and residue in a 250 ml beaker. Add accurately about 30 ml of standard normal sulfuric acid.

When reaction is complete titrate the residual acid with standard normal sodium hydroxide using methyl orange indicator.

One ml of normal sulfuric acid is equivalent to 0.02016 g of MgO.

Procedure, Analysis of Magnesium Oxide #312, or of Crucibles, 361, 373.

Follow the general procedure.

Reference: Rosin, J., "Reagent Chemicals and Standards". D. Van Nostrand Co., Inc. Princeton, N. J., 1961.

Procedure - Analysis of Hydroxy Acetic Acid

Dissolve the residue from the ignition (Zy-23) of a 10 gram sample (8 ml of 70% solution) in water. The solution should be nearly colorless. (If not, evaporate and ignite further at 600 C.) Transfer the solution to a graduated cylinder and adjust to a volume of 20 ml.

Transfer 5 ml to a second comparison cylinder. To this cylinder add 5 ml of standard magnesium solution. Add 5 ml of water.

To each cylinder add 0.15 ml of Titan Yellow solution. Mix. To each cylinder add 2 ml of 1 N sodium hydroxide. Mix.

The sample cylinder should show less color than the comparison. This is to say that 5 grams of original sample contains less than 0.05 milligrams of magnesium or 10 ppm.

Reference: See Calcium Carbonate "Reagent Chemicals" A. C. S. Specifications.

DETERMINATION OF MANGANESE

Volumetric Method

Principle

The permanganate ion is quantitatively reduced to divalent manganese in acid solution by a reducing agent, such as divalent iron. The amount of permanganate can be determined by adding an excess of divalent iron and back titration with a standardized permanganate solution to a permanent faint pink color at the endpoint.

Divalent manganese can be determined by oxidizing the manganese to permanganate with bismuthate in a cold acid solution. The permanganate formed can be determined after filtering off the excess bismuthate.

$$2KMnO_4 + 10FeSO_4 + 8H_2SO_4 \longrightarrow 5Fe_2(SO_4)_3 + 2MnSO_4 + K_2SO_4 + 8\ H_2O$$

$$4MnSO_4 + 10NaBiO_3 + 14H_2SO_4 \longrightarrow 4NaMnO_4 + 5Bi_2(SO_4)_3 + 3Na_2SO_4 + 14H_2O$$

Solutions

Standardized potassium permanganate, 0.1 N.

Procedure - Analysis of Potassium Permanganate

Dissolve 2.5 - 3.0 g. of accurately weighed sample in water. Transfer the solution to a 1 l. volumetric flask, dilute to the mark with water, and mix. Pipet a 10 ml. aliquot into an Erlenmeyer flask containing 100 ml. of water, and proceed as described under "General".

Procedure - General

Add 20 ml. of 0.1 N ferrous sulfate and 3 ml. of concentrated phosphoric acid to the permanganate solution and stir. Titrate the excess ferrous sulfate with standardized 0.1 N potassium permanganate solution to the appearance of a permanent faint pink color.

Carry a reagent blank through the general procedure, substituting 100 ml. of water for the sample.

Calculations

$$\%\ KMnO_4 = \frac{(B-A) \times N \times 3.161}{\text{g. of sample in aliquot}}$$

A = ml. of permanganate used to titrate sample
B = ml. of permanganate used to titrate blank
N = normality of permanganate solution.

Code Mn-1

References

1. Furman, "Scott's Standard Methods of Chemical Analysis", I, 5th Ed., D. Van Nostrand Co. (1939) pp. 474-571.

2. Hillebrank & Lundell, "Applied Inorganic Analysis", John Wiley & Sons, New York, (1929) pp. 343-6.

Written by: R. E. Ewing (July, 1952)

DETERMINATION OF NITROGEN

Ammonia Distillation - Volumetric Method

Principle

Nitrogen is reduced by strong reductants to the negative trivalent form. Nitride nitrogen in metallic calcium is converted to ammonia in an alkaline medium. The ammonia is distilled into a boric acid solution, which is subsequently back-titrated to the original pH with standard acid to determine the ammonia.

$$Ca_3N_2 + 6H_2O \longrightarrow 3Ca(OH)_2 + 2NH_3$$

$$H_2NH_4BO_3 + HCl \longrightarrow H_3BO_3 + NH_4Cl$$

Solutions

Standardized hydrochloric acid, 0.01 N.

Methyl red, 0.1%. 50 mg. per 50 ml. of absolute ethyl alcohol.

Bromcresol green, 0.1%. 50 mg. in 50 ml. of absolute ethyl alcohol.

Boric acid - indicator reagent; 20 g. of boric acid, 7.5 ml. of 0.1% bromcresol green solution, and 1.5 ml. of 0.1% methyl red solution in 1 l.

Procedure - Analysis of Calcium

Weigh 0.5 g. of calcium by difference and transfer it to a 100 ml. Kjeldahl flask. Connect the flask to a micro Kjeldahl distillation apparatus.

Place 5 ml. of the boric acid - indicator reagent in a 50 ml. Erlenmeyer flask and insert it under the end of the condenser tube, so that the exit is about 1/4" below the surface of the liquid.

Admit steam slowly to the distillation flask until the calcium is dissolved. Then, add 5 ml. of 50% sodium hydroxide solution and continue the steam distillation for 10 min., or until 20-25 ml. of distillate has been collected.

Remove the receiving flask and titrate with the standardized hydrochloric acid to the first tinge of red. Carry a blank through the entire procedure and titrate to an exactly matching color.

Calculations

$$\text{ppm N} = \frac{(A - B)\ C \times 0.014 \times 10^6}{\text{g. sample}}$$

A = ml. HCl to titrate sample
B = ml. HCl to titrate blank
C = normality of HCl

Literature References

1. Niederl and Niederl, "Micromethods of Quantitative Organic Analysis", John Wiley and Sons, Inc., 69 (1947).

Written by: A. H. Bushey (July, 1952)

DETERMINATION OF NITROGEN COMPOUNDS

Nessler Method

Principle

Nessler's reagent, K_2HgI_4, reacts in alkaline solution to give a pale yellow solution to a brown precipitate, depending on the concentration of ammonia. The test is delicate but is subject to interference from aldehydes, cyanides, sulfides, and bicarbonates.

The test may be applied to higher valent inorganic nitrogen compounds by making prior reduction with aluminum in alkaline medium.

$$NH_3 + 2K_2HgI_4 + 3KOH \longrightarrow NH_2IHg_2O + 7KI + 2H_2O$$

Solutions

Nessler's Reagent: weigh about 50 g of KI into a 1 liter volumetric flask and dissolve in 35 ml of water (Remark 1). Add saturated mercuric chloride ($HgCl_2$) solution slowly, and with stirring, till a slight red precipitate remains. Add about 400 ml of 50% KOH solution, mix, and dilute to the mark with water. Allow to stand 24 hours, then decant the clear liquid to a Pyrex container.

Standardized Ammonium Chloride, 0.01 mg. NH_4^+ per ml. Prepare a more concentrated solution by dissolving 2.965 g of NH_4Cl with water to 1 liter in a volumetric flask. Dilute 10 ml of this solution to 1 liter.

Procedure - Analysis of Aluminum Nitrate Solution #060, $<0.02\%$ NH_4^+

Prepare a 1:10 weight volume dilution of the sample. Transfer a 1.0 ml portion of the dilution to a 100 ml Nessler tube or graduated cylinder. Add 5 ml of 10% reagent sodium hydroxide solution and 2 ml of Nessler's solution. Make to 100 ml volume with ammonia free water and mix.

From a graduated pipet transfer 2.0 ml of standard ammonium chloride solution (0.01 mg NH_4^+ per ml) to a 100 ml Nessler tube or graduated cylinder. Add 5 ml of 10% sodium hydroxide solution and 2 ml of Nessler's reagent. Make to volume and mix.

The color of the sample should be no darker than that of the reference.

Procedure - Analysis of Chromium Nitrate #340, $<0.002\%$ NH_4^+

Dissolve 2.0 g of sample in 20 ml of water in a 100 ml Kjeldahl flask. Assemble apparatus and add 5 ml of 50% sodium hydroxide solution. Steam distill 50 ml into 10 ml of water containing 1 drop of concentrated hydrochloric acid.

Dilute the distillate to 100 ml in a volumetric flask. To a 50 ml aliquot, add 5 ml of 10% NaOH, 2 ml of Nessler solution, and make up to the mark with water in a 100 ml Nessler tube.

From a graduated pipet, transfer 2.0 ml of standardized ammonium chloride (0.01 mg NH_4^+ per ml) to a 100 ml Nessler tube. Add 5 ml of 10% NaOH solution and 2 ml of Nessler's reagent. Make to volume.

The color of the sample should be no darker than that of the reference.

Remarks

1. It is essential that ammonia-free water be employed and that the test not be carried out in an atmosphere containing ammonia fumes.

DETERMINATION OF NITRITE

Oxidation Method

Principle

Nitrite is determined by oxidation in acid solution to nitrate. Since nitrites are unstable in acid, an excess of oxidant is added immediately to the sample and the excess determined. Reducing agents other than nitrite in the sample interfere.

$$5NaNO_2 + 2KMnO_4 + 3H_2SO_4 \longrightarrow 5NaNO_3 + K_2SO_4 + 2MnSO_4 + 3H_2O$$

$$5(NH_4)_2Fe_2(SO_4)_3 + 2KMnO_4 + 8H_2SO_4 \longrightarrow 5(NH_4)_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + K_2SO_4 + 8H_2O$$

The range is from 0 to 100%.

Solutions

Standardized potassium permanganate, 0.1 N.

Standardized ferrous ammonium sulfate, 0.1 N.

Preoxidized acidulated water. Add 20 ml. conc. H_2SO_4 to 1 l. distilled water and $KMnO_4$ to a persistent pink color (2 sec.) (about 10-15 drops 0.1 N).

Procedure - Analysis of Sodium Nitrite

Weigh accurately 1 g. of sample into a 100 ml. volumetric flask, dissolve in 20 ml. water, and dilute to the mark. Add from a buret 30 ml. of standard permanganate to 300 ml. of oxidized acidulated water and mix. Add slowly with rapid stirring and with the tip of the buret held under the surface of the liquid a 10 ml. aliquot of sample to the permanganate solution.

Allow to stand 5 min., then add 15 ml. of standardized ferrous ammonium sulfate, and allow to stand for 5 min. more. Titrate the excess reductant to a permanent pink color (2 sec.) with the $KMnO_4$ solution.

Calculations

$$\% \ NaNO_2 = \frac{(V_1 \times N_1) - (V_2 \times N_2) \times 0.0345 \times 100}{\frac{V_3}{V_4} \times W}$$

where V_1 = ml. of permanganate used
V_2 = ml. of ferrous ammonium sulfate
V_3 = ml. of aliquot
V_4 = ml. of sample dilution
N_1 = normality of permanganate
N_2 = normality of ferrous ammonium sulfate
W_3 = weight of sample in grams.

Literature References

1. Rosin, "Reagent Chemicals and Standards", p. 48, D. Van Nostrand Co., (1937).

Written by: W. N. Carson (July, 1952)

DETERMINATION OF LOWER OXIDES OF NITROGEN

Permanganate Method

Principle

For practical purposes the lower oxides of nitrogen in nitric acid may be determined volumetrically by titrating with standardized potassium permanganate. The oxides are caluclated and expressed as nitrogen dioxide (NO_2).

$$5\ NO_2 + MnO_4^- + H_2O \longrightarrow 5\ NO_3^- + Mn^{++} + 2H^+$$

Solutions

Standardize potassium permanganate, 0.1 N.

Procedure - Analysis of Nitric Acid

Pipet 10 ml. of sample (Remark 1) into 150 ml. of water in a 250 ml. Erlenmeyer flask. Hold the pipet tip below the liquid surface during the addition and remove it just as the pipet empties.

Swirl the flask and titrate immediately with standardized 0.1 N potassium permanganate to a faint pink endpoint which persists for 30 seconds (Remark 2).

Calculations

$$\%\ NO_2 = \frac{V_1 \times N \times 4.6}{V_2 \times G}$$

N = normality of potassium permanganate
V_1= ml. of potassium permanganate
V_2= ml. of sample
G = specific gravity of sample.

Remarks

1. The sample weight is found from the volume and specific gravity of the sample.

2. The reaction may be slow near the endpoint, hence adequate time must be allowed for complete oxidation.

Literature References

1. Furman, "Scott's Standard Methods of Chemical Analysis", II, 5th Ed., D. Van Nostrand Co., Inc., New York, 2212 (1935).

Written by: A. H. Bushey (July, 1952)

ASSAY OF HYDROXYLAMINE NITRATE

Volumetric Method

Principle

Ferric iron is reduced by hydroxylamine and the resultant ferrous iron is titrated with standard permanganate solution.

$$(NH_2OH)_2\ H_2SO_4 + 4\ FeNH_4(SO_4)_2 \rightarrow 4\ FeSO_4 + 4(NH_4)HSO_4 + H_2SO_4 + H_2O + N_2O$$

Reagents

a. Ferric ammonium sulfate solution.

Dissolve 200 grams of reagent of ferric ammonium sulfate in 600 ml of water in a beaker. Add 20 ml of concentrated sulfuric acid. Decant from sediment to a graduated vessel and make to one liter volume with water.

b. Solid sulfamic acid.

c. Standard 0.1 N permanganate.

Procedure: Analysis of Hydroxylamine Nitrate Solution. #0529

1. With a micropipet accurately transfer a 1.000 ml sample to a 300 ml Erlenmeyer flask.
2. Add solid sulfamic acid (0.2 to 0.5 g), estimated, on the tip of a spatula.
3. From a graduated cylinder, add 20 ml of water and 25 ml of ferric ammonium sulfate solution.
4. Place a short-stemmed funnel in the neck of the flask.
5. Boil gently for 5 to 6 minutes on a hot plate.
6. Promptly cool, add 125 - 150 ml of water and titrate with standard permanganate using a 50 ml buret.

Calculations

Ml of titrant x normality x 0.5 = molarity of hydroxylamine in sample.

References:

American Chemical Society, "Reagent Chemicals" assay for hydroxylamine hydrochloride, Letter C. W. Pollock to O. F. Beaulieu, August 14, 1964.

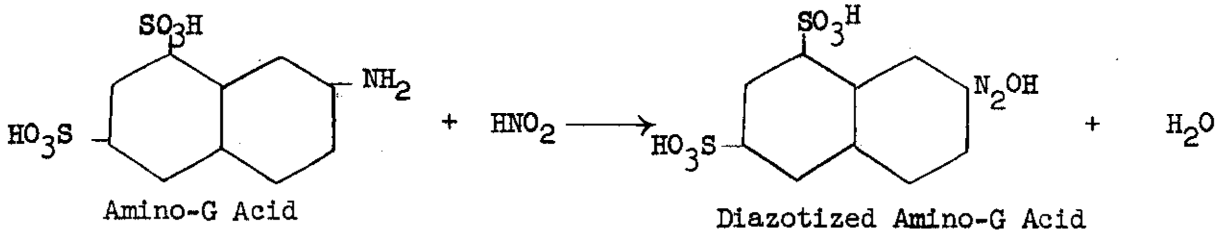
Note: with triplicate assays a standard deviation of the mean of ± 0.1% has been observed.

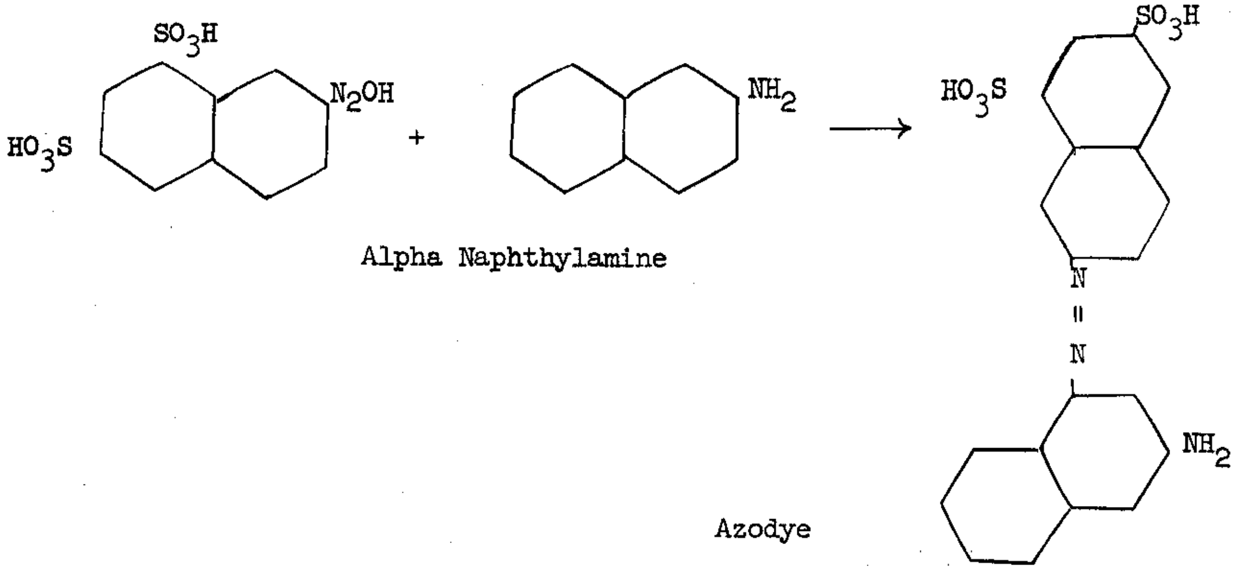
DETERMINATION OF NITRITE

Diazotization Method

Principle

In the presence of aromatic amines, nitrite in acid solution is converted to diazonium salts, which can be coupled with aromatic amines to give intensely colored azodyes. By using an amine that does not couple to give dyes to make the diazonium salt, and then coupling with another aromatic amine, the amount of dye formed becomes stoichiometric with respect to nitrite.





Solutions

Standardized nitrite, 1.5M. Dissolve approx. 103.5 g of C.P. sodium nitrite in 1 liter of distilled water. Standardize against 0.1 N permanganate by adding an aliquot of nitrite solution to an excess of the standard permanganate in dilute acid, using care to prevent the decomposition of nitrite by the acid or air. Add standard ferrous ammonium sulfate solution in excess of remaining permanganate and back titrate excess ferrous with additional standard permanganate to permanent pink (2 sec.) color.

$$\underline{M}\ NO_2^- = \frac{(V_{MnO_4} \times N_{MnO_4}) - (V_{Fe} \times N_{Fe})}{2 \times \text{ml. of aliquot}}$$

$$1 \text{ ml N } KMnO_4 = 0.02300 \text{ g } NO_2^-$$

0.3% Amino-G acid solution. Dissolve 3 g of amino-G acid (7 naphthylamine 1, 3 disulfonic acid) in 1 liter distilled water.

Alpha naphthylamine solution. Approximately 0.3%. Add 0.5 g alpha naphthylamine to 10 ml of water in a small beaker. Carefully bring to a gentle boil. Continue gentle boiling for 2 - 5 minutes. A milky aqueous phase is usually observed. Allow the mixture to cool. A clear colorless aqueous phase should result and amount to about 10 ml. Decant this carefully into a 100 ml volumetric flask and make to volume with water. This solution should be prepared fresh weekly.

Dilute nitrite standard. Add 100 µl of 1.5 ± 0.01 molar nitrite to 690 ml of distilled water. Mix. 1 ml provides 0.00001 g NO_2^-. This solution is good for about 48 hours.

Procedure - Analysis of Aluminum Nitrate Solution

1. Weigh 10 grams of sample. Transfer to a 100 ml volumetric flask and make to volume with distilled water. Mix.
2. Transfer 10 ml of this stock solution to a 50 ml Nessler tube. Add 10 ml of distilled water.
3. Place 20 ml of water in a second Nessler tube. Add to this tube 1 ml of the dilute nitrite standard amounting to 0.00001 g NO_2^-.
4. To each tube add 1 ml glacial acetic acid and mix.
5. To each, add 1 ml of amino-G-acid. Mix.
6. To each, add 3 ml of alpha naphthylamine solution. Mix.
7. Allow to stand 5 minutes.
8. Compare pink colors visually. The color of sample should be less than that of the standard. If so, report "less than 0.001% NO_2^-. If not, verify and report "out of specification."

DETERMINATION OF NITRATE

Ferrous Sulfate Method

Principle

Ferrous sulfate reduces nitrate to nitric oxide in sulfuric acid medium; excess ferrous sulfate combines with the liberated nitric oxide to form a brown addition complex. The reaction is employed qualitatively as the basis of the familiar brown ring test. It may be employed for a colorimetric determination by making comparison with standards, or volumetrically by titrating with standard ferrous solution to the brown colored endpoint.

$$2NaNO_3 + 6FeSO_4 + 4H_2SO_4 \longrightarrow 3Fe_2(SO_4)_3 + Na_2SO_4 + 2NO + 4H_2O$$

$$FeSO_4 + NO \longrightarrow FeSO_4 \cdot NO$$

Solutions

Standardized ferrous sulfate, 0.5 N. Dissolve about 139 g. of $FeSO_4 \cdot 7H_2O$ in about 400 ml. of water and add 50 ml. of concentrated sulfuric acid. Cool and dilute to 1 l.

To standardize, dry "nitrometer standard", or recrystalized C.P. KNO_3 to constant weight at 135° C. Weigh and titrate a portion as described under "Procedure - Analysis of Sodium Nitrate".

$$N = \frac{W}{0.337 \times V}$$

W = g. of potassium nitrate
N = normality of ferrous sulfate
V = ml. of ferrous sulfate

Standardized nitrate solution. 0.5 mg. HNO_3/ml. Measure 495 ml. of concentrated sulfuric acid with a graduate and transfer to a 600 ml. beaker in an ice bath. Add with a pipet exactly 5 ml. of a solution prepared by dissolving 8.022 g. of dried KNO_3 in exactly 100 ml. of aqueous solution; keep the pipet tip in constant motion during the addition.

Ferrous Sulfate, 0.2 M. Dissolve 27.8 g. of $FeSO_4 \cdot 7H_2O$ in 300 ml. of water. Add 150 ml. of sulfuric acid, cool, and make to 500 ml. Add a 1" piece of pure iron wire, hold the solution in an amber bottle, and do not keep for longer than one month.

Procedure - Analysis of Sodium Nitrate

Weigh exactly 0.12-0.14 g. of sample, dissolve it in 3-4 ml. of water, and add about 30 ml. of concentrated phosphoric acid.

Titrate with standardized 0.5 N ferrous sulfate solution (Remark 1) to the appearance of the first permanent yellowish-brown color.

$$\% \text{NaNO}_3 = \frac{\text{ml ferrous x normality}}{\text{g sample}} \times 0.02833 \times 100$$

$$\% \text{NaNO}_3^- = \underline{\text{ml ferrous x normality}} \times 0.02067 \times 100$$

Procedure - Analysis of ammonium nitrate, ammonium fluoride solution (0063)

a. Standardization of ferrous sulfate solution.

Arrange for titration with a 50 ml buret. (As NO gas is evolved it is desirable to do the titration near or in a hood, rather than on a desk.) A magnetic stirrer and a large stirring bar are desirable.

Place the ferrous sulfate solution in the buret.

Place an accurately weighed 0.4 g portion of dried reagent potassium nitrate in a 250 ml beaker. Dissolve with 5 ml of water. Add 50 ml of 85% phosphoric acid. Arrange for titration.

Add 10 drops of ferrous sulfate from the buret and allow to stir until a dark color develops and fades. Continue the titration slowly. A fast dropping rate is suitable until near the endpoint. The color at the endpoint should persist for more than 2 minutes.

To assure technique, run duplicates. Use the average of results agreeing within 0.1 ml.

Compute the normality of the ferrous solution. This solution deteriorates and should be used on the same day it is standardized.

b. Assay of sample

Tare a 250 ml beaker. Add about 5 ml of the sample and weigh. Add 50 ml of 85% phosphoric acid and titrate as in the above standardization.

$$\frac{\text{ml ferrous x normality}}{\text{g sample}} \times 0.02668 \times 100 = \% \text{NH}_4\text{NO}_3$$

Remark

1. The reaction of ferrous sulfate and nitrate undergoes an induction period that is retarded by an initial excess of ferrous ion. Therefore, add the first 0.5 - 1.0 ml of reagent and stir until the reaction starts, as indicated by the gradual appearance of a dark brown color that fades suddently.

Literature Reference

1. Furman, "Scott's Standard Methods of Chemical Analysis," D. Van Nostrand Co., 2214-19 (1939).

DETERMINATION OF NITRATE

Phenoldisulfonic Method

Principle

Phenoldisulfonic acid, $C_6H_3OH(SO_3H)_2$, reacts with nitrate to form a yellow, water soluble compound that can be employed as a basis for nitrate determination. Carbonate, chloride, nitrates, and organic matter constitute interferences.

Solutions

1, 3, 5 phenoldisulfonic acid reagent, as purchased.

Standardized nitrate solution, 0.5 mg. NO_3/l. Dissolve 0.815 g. of KNO_3 in water and make to exactly 1 l.

Procedure - Analysis of Sulfamic Acid

Dissolve a 0.5 g. sample in a small volume of water and make basic to pH paper with 1:1 ammonium hydroxide. Boil to a small volume and evaporate to dryness on a steam bath (Remark 1).

Add 2 ml. of phenoldisulfonic acid reagent to the dry sample and rub with a stirring rod to assure that the reagent contacts all the dried sample. Add 15 ml. of water and 1:1 ammonium hydroxide until the solution is strongly basic to pH paper and the yellow color develops.

Transfer the solution to a 50 ml. Nessler tube, filtering if necessary, and make to volume. Compare the sample color with that of a control containing 1 ml. of standard 0.5 mg/ml NO_3 solution.

Calculation

$$\% \; NO_3 = \frac{A \times B \times 100}{C}$$

A = ml. of nitrate standard
B = g/ml. of NO_3 in standard
C = g. of sample.

Remarks

1. Avoid overheating the sample to prevent volatilization of ammonium nitrate.

Literature References

1. Millan, "Organic Reagents in Inorganic Analyses", p. 480 (1941).

Written by: A. H. Bushey (July, 1952)

DETERMINATION OF HYDRAZINE

Principle

Hydrazine in hydrochloric acid solution is titrated with standard iodate. Ammonia does not interfere. A hydrochloric acid concentration of 3 to 5 molar is desirable.

$$N_2H_4 + KIO_3 + 2HCl \longrightarrow KCl + ICl + N_2 + H_2O$$

Solutions

Standard 0.1 N potassium iodate (0.01666 M) prepared by weighing reagent potassium iodate.

Procedure: Analysis of Hydrazine Solution, 35%.

Place 20 ml of water in a glass stoppered Erlenmeyer flask. Using a 50 ul pipet, accurately measure a sample at 25 degrees and transfer to the flask. Rinse the pipet with water.

Add 20 ml of conc. (12 N) hydrochloric acid. Titrate with iodate until 25 ml have been added. Add about 2 ml of carbon tetrachloride. Continue titrating to 31 ml. Stopper and shake the flask. Continue titrating using small increments and with shaking until the pink color in the carbon tetrachloride layer just disappears.

One ml of N iodate is equivalent to 0.005341 gm N_2H_4.

Compute g/l N_2H_4.

Compute per cent as g/l N_2H_4 x 0.0980, when sample is measured at $25^\circ \pm 1^\circ$. Otherwise, measure the Sp.Gr. and compute.

If less than 35.0% is observed, report as out of specification.

DETERMINATION OF ALKALI AND ALKALINE EARTHS

Gravimetric Method

Principle

After the interfering elements are removed, the sample is evaporated to dryness with excess sulfuric acid to yield a residue of the sulfates of the alkali and alkaline earth metals. All heavy metals interfere and must be removed.

Procedure - Analysis of Aluminum Nitrate Solution #060

Evaporate a 3 g sample twice with 7 ml of HCl. Take up the residue in 50 ml of hot water and pour the solution into a mixture of 10 ml of NH_4OH and 70 ml of water. Boil until nearly free from NH_3 odor, filter, and wash to 150 ml. Take a 50 ml aliquot and follow the general procedure. (Remark 1).

Procedure - Analysis of Chromium Nitrate #340

Dissolve 4 g of chromium nitrate in 150 ml of water. Add 15 ml of concentrated ammonium hydroxide to precipitate the chromium. Boil to expel the excess ammonia, cool, filter, and dilute to 200 ml. Take a 50 ml aliquot and follow the general procedure.

Procedure - General

Transfer the 50 ml aliquot to a tared platinum dish, add 5 drops of H_2SO_4, and evaporate carefully to dryness. Ignite at 850 - 900 C, cool, and weigh. (Remark 2).

Calculations

$$\% \text{ Alkaline earths as sodium} = \frac{\text{g residue} \times 100 \times 0.324}{\text{g sample in aliquot}}$$

Remarks

1. The solution obtained at this point can be used for the sulfate determination as well as the alkaline earth determination.

2. Not more than 0.003 g of residue should remain.

References

1. Rosin, J., "Reagent Chemicals and Standards," D. Van Nostrand Co., Inc., p. 25, (1937).

2. Ibid., p. 142.

DETERMINATION OF HYDROGEN PEROXIDE

Ceric Oxidation - Volumetric Method

Principle

Hydrogen peroxide is oxidized quantitatively by ceric sulfate in a volumetric titration:

$$4Ce(HSO_4)_4 + 2H_2O_2 \longrightarrow 2Ce_2(SO_4)_3 + 2O_2 + 10H_2SO_4$$

Reducing agents interfere, if present, causing high results.

Solutions

Ceric sulfate, 0.1 N.

Ferroin indicator.

Procedure - Analysis of Hydrogen Peroxide

Weigh accurately 1-2.5 g. sample into a 500 ml. volumetric flask containing about 300 ml. distilled water and dilute to the mark, mixing well. Transfer a 25 ml. aliquot to an Erlenmeyer flask, acidify, and add 5 ml. excess 6 N sulfuric acid. Add one drop of ferroin indicator. Titrate with standardized 0.1 N ceric sulfate solution to a pale blue endpoint.

Calculations

$$\% \ H_2O_2 = \frac{\text{ml. standard ceric x normality x 1.701}}{\text{g. sample in aliquot}}$$

References

1. Oesper, "Newer Methods of Volumetric Analysis", D. Van Nostrand, 44 (1938).

Written by: U. L. Upson (July, 1952)

DETERMINATION OF WATER

Distillation Method

Principle

Water may be removed from a variety of materials by boiling them with solvents, such as benzene (C_6H_6), toluene (C_7H_8), and xylene (C_8H_{10}), all of which are immiscible with water. If the distillate is collected, the water and solvent separate into two phases. If the collector is graduated, the amount of water may be read directly. Toluene or xylene are generally used as the organic solvents.

Apparatus

Dean and Stark water test apparatus, A.S.T.M. Designation D95.

Procedure - Analysis of Sodium Nitrite

Weigh 100 g. of sample and place in the distilling flask. Add 100 ml. of toluene or xylene to the flask and assemble the apparatus. Heat the flask, regulating the temperature so that the distillation proceeds at a rate of 2-5 drops per second. Continue the distillation until the water level in the trap remains constant and read this level. Determine a reagent blank on each batch of solvent used.

Calculations

$$\% \text{ water} = \frac{(A - B) \times 100}{C}$$

A = g. of water collected
B = g. of water in an equal volume of blank
C = wt. of sample in g.

Remarks

1. If water content of a sample is believed to be greater than 10%, a proportionally smaller sample size should be used.

References

1. A.S.T.M. Standards, Part III, Designation D95-40, American Society for Testing Materials, Philadelphia, Pa., 262-265 (1942).

Written by: W. G. Burch (July, 1952)

DETERMINATION OF WATER

Qualitative Method

Principle

The presence of water in many materials may be detected by its producing a cloudy appearance.

Procedure - Analysis of Tributylphosphate

Measure 38 ml. of white gasoline (60° Baume' - Sp. Gr. 0.745) into a flask equipped with a stirrer and maintained at 20° C in a constant temperature bath. Add 2 ml. of sample and stir. If water is present, turbidity will result.

Procedure - Analysis of Tetrachloroethylene

Measure approximately 20 ml. of the sample and transfer to a 50 ml. test tube. To a 400 ml. beaker add 200 ml. of acetone and cool the acetone to -10° C by dropping in small pieces of dry ice. Immerse the test tube containing the sample into the cold acetone long enough to be cooled to -10° C, then remove the sample and allow to stand until room temperature is reached. Any resulting cloudiness orturbidity indicates a suspension of water.

Written by: W. G. Burch (July, 1952)

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May 18, 1964

DETERMINATION OF MOISTURE CONTENT

Oven Drying Method

Principle

The moisture content of thermally stable ion exchange resins may be determined by direct oven drying. Most thermally unstable resins may be converted to stable forms in order to apply the oven drying method.

Procedure - Permutit SK Anion Exchange Resin

A 1-5 gram sample of resin in a tared open container (oven dried to constant weight) is placed in an oven and dried to constant weight at 105 C. At least four hours drying time is preferable before first weighing of dried sample. Use of a desiccator while cooling is essential.

Handling of the original bulk sample is important. A moisture-tight container is filled to near capacity with the resin sample. Immediate weighing is necessary after transferring sample to analysis container.

Calculations

$$\text{Moisture content (per cent)} = \frac{\text{Wt. before drying - wt. after drying}}{\text{Wt. of sample before drying}} \times 100$$

References

1. R. Kunin. Ion Exchange Resins, John Wiley & Sons. Inc., New York, 1958. Second ed., pp. 320-321.

2. J. Mitchell, Jr., "Water," Analytical Chemistry of the Elements, Treatise on Analytical Chemistry, edited by I. M. Kolthoff, P. J. Elving, E. B. Sandell, Interscience Publishers, New York, 1961. Pp. 109-116.

Submitted by: C. A. Colvin (May 1964)

Approved by: O. F. Beaulieu (May 1964)

Code: 0

ASSAY OF SODIUM BISMUTHATE (#0782)

Principle

The oxidizing capacity of sodium bismuthate toward ferrous sulfate is taken as a measure of the quality of the material.

Procedure

Weigh accurately about 0.7 gram, place in a flask, add 25.0 ml of acid ferrous sulfate solution, and stopper the flask. Transfer 25 ml of the acid ferrous sulfate to another flask and stopper the flask. Allow each flask to stand for 30 minutes, shaking frequently, and titrate the ferrous sulfate in each with 0.1 N potassium permanganate. The difference in the volume of permanganate consumed in the two titrations is equivalent to the sodium bismuthate. One milliliter of 0.1 N permanganate corresponds to 0.01400 gram of $NaBiO_3$. The acid ferrous sulfate must be freshly prepared by dissolving about 7 grams of clear crystals of ferrous sulfate ($FeSO_4 \cdot 7H_2O$) in 100 ml of oxygen-free dilute sulfuric acid (1 + 9).

Reference: ACS "Reagent Chemicals" 1960, p. 442.

ASSAY OF POTASSIUM (OR SODIUM) PERSULFATE (#0752)

Principle

The oxidizing capacity toward iodide ion is used as a measure of the quality of the material.

$$K_2S_2O_8 + 2KI \longrightarrow 2K_2SO_4 + 2I$$

Procedure

Weigh accurately about 0.5 g of sample and transfer to a glass-stoppered flask or bottle. Add 30 ml of water and agitate until the sample is dissolved. Add 10 ml of 10% sulfuric acid (6 ml H_2SO_4 + 90 ml H_2O). Allow to stand in the dark for 30 minutes, then titrate the liberated iodine with standard 0.1 N sodium thiosulfate.

One ml of 0.1 N thiosulfate = 0.01352 g $K_2S_2O_8$

One ml of 0.1 N thiosulfate = .01191 $Na_2S_2O_8$

Reference - Rosin, J. "Reagent Chemicals and Standards, 4th Ed. D. Van Nostrand Co., N. Y.

PHOSPHATE, GRAVIMETRIC

Principle

Phosphate is quantitatively precipitated as magnesium ammonium phosphate. The precipitate is collected, ignited, and weighed as magnesium pyrophosphate $Mg_2P_2O_7$.

Reagents

Magnesia solution. Dissolve 5.5 gm of magnesium chloride and 7 gm of ammonium chloride in 75 ml of water, add 14 ml of concentrated ammonia solution. Make to 100 ml volume. Allow the mixture to stand for two days, tightly stoppered. Filter before using.

Procedure- General

Weigh a sample representing about 0.3 gm of $Po_4^{\equiv}$. Transfer to a 250 ml beaker and dissolve in about 100 ml of water. Add 1.0 ml of hydrochloric acid. Add 20 ml of magnesia solution and 15 ml of concentrated ammonia solution. Mix and allow to stand for four hours. Collect the precipitate on a previously ignited tared filtering crucible and wash with four 20 ml portions of a 1 + 5 ammonia solution or until free of chloride. Dry and ignite to constant weight.

$$\frac{\text{Gm } Mg_2P_2O_7}{\text{Gm sample}} \times F \times 100 = \% \text{ of specified phosphate compound}$$

Procedure - Sodium Phosphate

Follow general procedure.
Use F = 1.276 for calculation.

PHOSPHATE, COLORIMETRIC

Principle

In the presence of phosphate a solution of ammonium molybdate is reduced by p-methylaminophenol to "molybdenum blue". The color under fixed conditions is proportional to $PO_4^{\equiv}$ concentration.

High temperatures are undesirable in drying samples because of possible formation of pyrophosphate.

Solutions

Standard Phosphate Solution 0.1 mg $PO_4^{\equiv}$ per ml.

Place 1.443 gm KH_2PO_4 in a 100 ml volumetric flask. Dissolve in water, make to volume and mix. Transfer 1.0 ml of this solution to a 100 ml volumetric flask, make to volume and mix. 1ml is equal to 0.0001443 gm KH_2PO_4 or 0.000100 gm $PO_4^{\equiv}$.

p - Methylaminophenol sulfate solution

Dissolve 1 gram of p-methylaminophenol in 50 ml of water. Transfer 10 ml of this solution to a beaker. Add 90 ml of water and 20 grams of sodiumbisulfite ($NoHSO_3$). Confirm the reagent by the following test.

In each of four 50 ml g.s. cylinders place 25 ml of 0.5N sulfuric acid. Add 1 ml of the above reagent and 1 ml of 5% ammonium molybdate solution. Add 0.01 mg $PO_4^{\equiv}$ to one of the cylinders, 0.02 to a second and 0.04 to a third. Mix and allow to stand 2 hrs. Compare with the blank. Differences in color should be readily perceptible.

5% Ammonium Molybdate

Dissolve 5 grams of reagent ammonium molybdate in 95 ml of water.

0.5N Sulfuric Acid.

Add 14 ml of concentrated reagent sulfuric acid to 985 ml of water.

Procedure - General

Prepare the sample to avoid oxidizing or reducing agents or excessive concentrations of organic matter. Avoid high temperatures which might convert phosphate to pyrophosphate. In case a high temperature is used digest for more than one hour with dilute acid to hydrolyse pyrophosphate.

Dissolve an appropriate sample in 25 ml of 0.5N sulfuric acid. Transfer to a 50 ml g.s. cylinder. Add 1 ml of 5% ammonium molybdate, 1 ml of p-methylaminophenol reagent solution. Mix and allow to stand for 2 hours.

Compare with a reagent blank and with an appropriate standard made using standard phosphate solution.

This test follows the practice of "Reagent Chemicals" Applied Publications, American Chemical Society, Washington D.C. 1961.

Procedure - Analysis of Potassium Bicarbonate #743 (0.003% $PO_4^{\equiv}$)

Dissolve 4 grams in 20 ml of water. Add 5 ml of hydrochloric acid. Evaporate to dryness on a steam bath or very low hot plate. Dissolve the residue in 25 ml of 0.5N sulfuric acid. Follow the general procedure.

Compare with a reagent blank and with a standard made with 1.2 ml of 0.1mg/ml $PO_4^{\equiv}$ standard solution.

Procedure - Analysis of Nitric Acid #670 (0.003%)

Evaporate 5 ml of sample to dryness on a low hot plate. Dissolve the residue in 25 ml of 0.5 N sulfuric acid. Follow the general procedure.

Compare with a reagent blank and with a standard made with 2.1 ml of 0.1 mg/ml $PO_4^{\equiv}$ standard solution.

Procedure, Analysis of Citric Acid #285 (0.001%)

Dissolve 2 grams in a small amount of water, add 0.5 gram of magnesium nitrate, evaporate to dryness, and ignite until all carbon is removed.

Dissolve the residue in 5 ml. of water, add 5 ml. of nitric acid and evaporate to dryness at low heat. Dissolve the residue in 25 ml of 0.5N sulfuric acid and follow the general procedure.

Compare with a reagent blank and with a standard made with 0.20 ml of 0.1 mg $PO_4^{\equiv}$/ ml standard phosphate.

Procedure - Analysis of Hydrogen Peroxide #526 (0.001%)

Evaporate 3.6 ml (4 grams) to dryness at low heat. Dissolve the residue in 25 ml of 0.5N sulfuric acid. Follow the general procedure.

Compare with a reagent blank and with a standard made with 0.40ml of 0.1mg/ml $PO_4^{\equiv}$ standard.

DETERMINATION OF LEAD

Volumetric

PRINCIPLE

The sample is dissolved in water and acetic acid. A measured amount of standard dichromate is then added to precipitate lead chromate. Excess dichromate is then measured on clear supernate and the lead is so measured.

SOLUTIONS

Standard 0.1 N potassium dichromate.

Starch indicator (0.5%).

Standard 0.1 N sodium thiosulfate.

PROCEDURE - GENERAL

Accurately weigh an appropriate sample. Dissolve in 10 ml water and 1.0 ml of acetic acid. Transfer to a 200 ml volumetric flask. Add 60 ml of water. Heat to boiling. Add 50 ml of 0.1 N dichromate. Boil 2-3 minutes. Cool and make to the mark with water. Mix and allow to settle. Accurately measure 50 ml or 100 ml of the clear supernate. Transfer to a glass-stoppered flask or bottle.

Add 10 ml of 10% sulfuric acid and 1 g of potassium iodide. Allow to stand 10 minutes. Titrate liberated iodine with 0.1 N thiosulfate adding about one ml of starch indicator toward the end.

CALCULATION

If 100 ml of supernate used

$$\% \text{ Pb} = \frac{(\text{ml of dichromate} \times \text{normality}) - 2(\text{ml of thiosulfate} \times \text{norm})}{\text{g sample}} \times 6.907$$

PROCEDURE - Analysis of Lead Nitrate (> 62%) #281

Accurately weigh a sample of about 0.4 g. Follow the general procedure.

DETERMINATION OF HEAVY METALS

Hydrogen Sulfide Method

Principle

Hydrogen sulfide is passed through a slightly acidic solution of the sample and the metal sulfide color is compared with a control containing the maximum permissible amount of heavy metals as lead. The solution is made alkaline to detect iron. This evaluation will indicate whether the heavy metal content is above or below the maximum permissible limit.

$$Pb(CH_3COO)_2 + H_2S \longrightarrow PbS + 2CH_3COOH$$

Solutions

Phenolphthalein, 1%. Dissolve 1 g in 100 ml of 95% ethyl alcohol.

Standardized lead solution, 0.1 mg Pb/ml. Dissolve 0.1599 g of anhydrous lead nitrate in 1 liter of water.

Procedure - Analysis of Aluminum Nitrate Solution - #060, 0.01%

Prepare a dilution in the proportion of 10 gm of sample in 100 ml volume using distilled water. Transfer 10 ml to a Nessler tube, add 2.5 ml of a 0.1 mgm Pb per ml standard lead nitrate solution. Add 25 ml of water. Transfer 35 ml of sample dilution to a second Nessler tube. To each tube add 10 ml of a recently prepared solution of hydrogen sulfide in water, or bubble H_2S through the solution. If the color in the tube containing 35 ml of sample dilution is less than the standard, report the heavy metal content as less than the maximum permissible limit.

Procedure - Analysis of Sulfamic Acid - #870

Weigh 1 g of sample and proceed as described under "General." Maximum permissible is 0.01%.

Procedure - General

Place the weighed sample in a 100 ml Nessler tube containing 60 ml of distilled water. Add 2 drops of phenolphthalein and neutralize to a pale pink color by adding concentrated ammonium hydroxide. Add 1 ml of glacial acetic acid. Mix thoroughly.

Place 1 ml of lead solution and 1 ml of glacial acetic acid in a 100 ml Nessler tube containing 60 ml of distilled water. Mix thoroughly.

Bubble hydrogen sulfide gas through both solutions for 5 minutes or add 10 ml of a fresh solution of H_2S in water. If the sample is lighter than the standard, report the heavy metal content as less than the maximum permissible limit.

Literature References

1. Rosin, "Reagent Chemicals and Standards", 321, D. Van Nostrand Co., Inc., New York, 1939.

2. American Chemical Society, "Specifications for Analytical Reagents", American Chemical Society, Washington, 1941.

Written by: H. H. Van Tuyl (July, 1952)

DETERMINATION OF pH

Principle

A pH meter with a glass electrode and a calomel electrode is used. The meter should be calibrated against a standard buffer having a value near that of the sample.

Procedure - General

Rinse the electrodes thoroughly with water. Wipe with a paper tissue. Immerse the electrodes in standard buffer and adjust the meter to read the pH of the buffer at room temperature.

Rinse the electrodes thoroughly with water. Wipe with a paper tissue. Immerse the electrodes in the sample and read the pH at room temperature.

Note: Directions for operation of various meters and for care of the electrodes are found in manufacturer's literature.

Procedure - Analysis of Ferrous Sulfamate Solution #0432

Buffer the meter at pH 4.00 and follow the general procedure.

DETERMINATION OF SULFITE

Volumetric Method

Principle

A weighed sample reacts with a known excess of iodine in acid solution. The excess iodine is titrated with sodium thiosulfate, using starch indicator. The amount of iodine which reacted with the sample is used to calculate the amount of sulfite present.

Any substances, other than sulfite, which can reduce iodine to iodide will interfere.

$$SO_3^{=} + I_2 + H_2O \longrightarrow SO_4^{=} + 2HI$$

$$I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$$

Solutions

Standardized iodine, 0.1 N.

Standardized sodium thiosulfate, 0.1 N.

Procedure - Analysis of Sodium Sulfite

Weigh accurately 0.25 g. of sample into a glass stoppered flask. Proceed as described under general procedure.

Procedure - General

Add about 10 ml. of distilled water, 50 ml. of standardized 0.1 N iodine solution, and 5 ml. of conc. hydrochloric acid. Titrate with standardized 0.1 N sodium thiosulfate solution until the brown color is a light yellow. Add 1 ml. of 1% starch indicator solution and titrate to disappearance of the blue color.

Run a blank on the iodine solution, using the same amounts of all reagents but omitting sample.

Calculations

$$\% \; Na_2SO_3 = \frac{(\text{ml. blank - ml. sample titration}) \times \text{N. of thiosulfate} \times 6.303}{\text{g. sample}}$$

References

1. Furman, "Scott's Standard Methods of Chemical Analysis", I, 5th ed., D. Van Nostrand, N.Y., 926 (1936).

Written by: M. B. Leboeuf (July, 1952)

DETERMINATION OF SULFATE
Turbidimetric

Principal

The turbidity produced by the formation of insoluble $BaSO_4$ on addition of barium chloride to a solution containing sulfate ion is visually compared with a standard. Reagent blanks should be run and results corrected when blanks are significant.

Solutions

Standard sulfate solution, 0.1 mg sulfate per ml. Weigh accurately 0.1814 g of potassium sulfate into a 1 liter volumetric flask. Dissolve with 100 ml of water. Make to the mark and mix. (Note: A stronger solution is used for dichromate samples).

6 N HCl: Mix equal volumes of concentrated hydrochloric acid and water.

10% $BaCl_2$: Dissolve 10 g reagent barium chloride in 90 ml of distilled water. A clear solution is necessary.

Procedure - General

Add 1 ml of 6 N HCl and 5 ml of 10% barium chloride solution to a solution of sample in a 50 ml Nessler tube. Dilute to 50 ml with distilled water. To second Nessler tube add 30 ml of distilled water, 1 ml of 6 N HCl, 5 ml of barium chloride. Add an appropriate amount of standard sulfate to the second tube and dilute to the mark. Mix and compare with sample.

Procedure - Analysis of Aluminum Nitrate Solution (#060) 0.02%

Weigh accurately 4 g of sample into a beaker. Add 7 ml of concentrated hydrochloric acid and evaporate to dryness on a steam bath. Repeat evaporation to dryness with 7 ml of concentrated hydrochloric acid. Dissolve the residue in 50 ml of hot water and pour into 80 ml of 2 M ammonium hydroxide. Boil off excess ammonia. Filter and wash precipitate with water until total volume of filtrate and washings is 200 ml. Transfer a 25 ml aliquot of the solution to a Nessler tube and neutralize with 1 N HCl and methyl orange. Proceed as described under "General", comparing turbidity with that formed by 1 ml of standard sulfate.

Procedure - Analysis of Ferrous Sulfamate Solution (#432) 1.0%

Transfer 0.66 ml of solution (SpG 1.52) to a 100 ml volumetric flask. Dilute to the mark and mix. Transfer 1.0 ml to a Nessler tube. Compare with a standard made by using 1.0 ml of standard sulfate following the general procedure.

Procedure - Analysis of Sodium Dichromate (#795, 0.5% sulfate)

One gram of solid sample is placed in a Nessler tube and dissolved in about 30 ml of distilled water. One ml of 6 N hydrochloric acid and 10 ml of 10% barium chloride solutions are added. The solution is brought to 50 ml volume, mixed, and the turbidity visually compared with a standard. The standard is prepared using one gram of reagent sulfate free sodium dichromate and the above amounts of acid and barium but with the addition of 5 ml of a 0.001 g/ml standard sulfate solution.

Note: This standard sulfate is ten times the concentration of that described under "Solutions."

Procedure: Analysis of Chromic Nitrate (#340, < 0.01%)

Dissolve 1.0 g of sample in 40 ml of water in a Nessler tube. Add 1.0 ml of 6 N HCl and 5 ml of 10% barium chloride. Mix.

To a second Nessler tube, add 40 ml of water, 1.0 ml of 6 N HCl, 5 ml of 10% barium chloride, and 1.0 ml of standard 0.1 mg per ml sulfate solution. Mix and compare the two tubes.

Procedure - Analysis of Hydroxylamine Nitrate #0529, < 250 ppm or 0.025%.

With a graduated pipet, transfer 3.48 ml of sample (0.4 g) to a Nessler tube. Add 1 ml of 6 N HCl and 5 ml of 10% barium chloride. Dilute to 50 ml and mix. Compare with a standard made with 1 ml standard sulfate.

Procedure - Analysis of Sodium Dichromate Solution (#796, 0.4% sulfate)

1.0 g of liquid sample is mixed with about 30 ml of distilled water in a Nessler tube. One ml of 6 N hydrochloric acid and 10 ml of 10% barium chloride solutions are added. The solution is made to 50 ml volume, mixed, and the turbidity visually compared with that of a standard.

The standard is similarly prepared using 0.65 g of reagent sulfate free sodium dichromate, 30 ml of distilled water, and the above amounts of acid and barium, but with the addition of 4 ml of a 0.001 g/ml standard sulfate solution.

Procedure - Analysis of Sodium Nitrate (#805) < 0.50%

Weigh 2.00 g and dissolve in 20 ml of water. Transfer to a 500 ml volumetric flask and make to volume. Transfer 5.0 ml to a 50 ml Nessler tube. Follow standard procedure, comparing with a standard made with 1 ml of standard sulfate.

Procedure - Analysis of Sulfamic Acid (#870) < 1.0%

Dissolve 1.0 g of sample water in a 100 ml volumetric flask. Transfer 1.0 ml to a Nessler tube. Add 1 ml of 6 N HCl and 5 ml of 10% barium chloride. Dilute to 50 ml and mix. Compare with a standard made with 1 ml of standard sulfate.

DETERMINATION OF SILICON

Perchloric Acid Dehydration Method

Principle

Silicon and its compounds are converted by proper treatment of the sample to silicic acid, which is then dehydrated to impure silica by fuming with perchloric acid. The amount of silica in the impure solid is determined by volatization of the silica with hydrofluoric acid and weighing the impurities.

$$Na_2SiO_3 + 2HCl \longrightarrow H_2SiO_3\downarrow + 2NaCl$$

$$H_2SiO_3 \xrightarrow{HClO_4} H_2O + SiO_2\downarrow$$

$$SiO_2 + 4HF \longrightarrow SiF_4\uparrow + 2H_2O$$

For the interferences consult the literature references.

Procedure - Analysis of Magnesium Oxide

Weigh 1 g. of sample into a platinum crucible. Add approximately 6 times as much C.P. sodium carbonate and mix well. Cover crucible and apply heat cautiously (Fisher burner) until mixture melts, increasing the heat during the reaction. Continue until the melt is clear and homogeneous. Remove the crucible from the heat and rotate the crucible so that a thin layer of melt forms on the side of the crucible. When the melt solidifies, drop crucible and lid into 1" of water, contained in a 400 ml. beaker. Add water to cover the crucible and heat gently until material is in solution. Remove crucible and lid, rinsing well, and proceed as under "General".

Procedure - Analysis of Aluminum Nitrate Solution

Take 10 ml. sample and add to 400 ml. beaker. Add 10 ml. concentrated sulfuric acid, heat gently, and add formic acid dropwise until NO_2 fumes are all evolved. Proceed as under "General".

Procedure - Analysis of Sulfamic Acid

Take 10 g. sample and dissolve in 20 ml. of water in a 400 ml. beaker. Proceed as under "General".

Procedure - General

Add concentrated hydrochloric acid to the sample until it is strongly acid, if it is not already acid. Add 20 ml. of perchloric acid and evaporate to fumes of perchloric acid. Fume at least 15 min. (Remark 1), cool, add 100 ml. hot, distilled water, stir well, and heat until salts are dissolved. Filter through Whatman #42 filter paper and wash thoroughly with 10% (by volume) sulfuric acid.

Evaporate the filtrate to small volume, add 10 ml. perchloric acid, and evaporate to fumes as above. Filter as above, using the same filter paper. Wash filter well with hot, distilled water to remove free acids (Remark 2).

Dry precipitate and filter in a platinum crucible, burn paper off slowly, and then ignite at 1000° C for 1/2 hr. Cool in desiccator and weigh.

Moisten residue with concentrated sulfuric acid and add 10 ml. concentrated hydrofluoric acid carefully. Evaporate (steam bath) to dryness and repeat hydrofluoric acid treatment. Ignite residue at 1000° C for 1/2 hr., cool in desiccator, and weigh.

Calculations

Grams SiO_2 in sample = weight before HF treatment minus weight of the treatment = W.

$$\% \ SiO_2 = \frac{W}{S} \times 100 = \frac{W}{V \times Sp.G.}$$

S = g. sample
V = ml. sample
Sp.G = specific gravity

$$\% \ Si = \% \ SiO_2 \times 0.4672$$

$$ppm \ Si = \% \ Si \times 10^4$$

Remarks

1. The residue must be thoroughly dehydrated to reduce the solubility of SiO_2 in the subsequent filtration and washing.

2. All the perchloric acid must be removed by thorough washing to prevent a violent reaction when the filter paper is dried and ignited.

Literature References

1. Hildebrand and Lundell, "Applied Inorganic Analysis", John Wiley & Sons, Inc., 722 (1929).

2. Furman, "Scott's Standard Methods of Chemical Analysis", D. Van Nostrand Co., 804 (1939).

3. A.S.T.M., Chemical Methods of Metals, 145 (1946).

Written by: W. N. Carson (July, 1952)

DETERMINATION OF TIN

Silico-Molybdate Colorimetric Method

Principle

Stannous chloride reduces silico-molybdate to molybdenum blue, which is measured spectrophotometrically. Nitrates and hydrogen peroxide interfere.

Solutions

Silicate stock solution. Fuse 1 g. of pure silica with 5 g. of sodium carbonate and dissolve in 1 l. of water.

Molybdate stock solution. Dissolve 5.3 g. of ammonium molybdate in 190 ml. of water and 10 ml. of concentrated sulfuric acid.

Silico-molybdate reagent. Dilute 10 ml. of molybdate stock solution to about 800 ml. with water. Add 2.5 ml. of silicate stock solution and dilute to 1 l. with water. Prepare fresh daily. Let stand at least 30 min. before using.

Standard tin solution, 0.04 mg. Sn/ml. Dissolve 0.08 g. of pure tin in 100 ml. of 6 $\underline{N}$ hydrochloric acid and dilute to 2 l. with 6 $\underline{N}$ hydrochloric acid.

Procedure - Analysis of Aluminum Nitrate Solution

Weigh 0.4 g. of solution into a beaker, dissolve in a small amount of water, add 2.5 ml. of concentrated sulfuric acid, and heat gently. Add concentrated formic acid dropwise until all brown fumes of nitrogen dioxide are gone. Heat till white fumes appear and fume for 1 hr. Add 15 ml. of water and proceed as described under "General". Maximum permissible limit, 0.005%.

Procedure - Analysis of Sulfamic Acid

Weigh 0.2 g. of sample into a beaker, add 15 ml. of water, and neutralize to the litmus paper endpoint with concentrated ammonium hydroxide. Proceed as described under "General". Maximum permissible limit, 0.01%.

Procedure - General

To the solution of the sample add 1 ml. of concentrated hydrochloric acid and about 0.5 g. (a small scoop) of test lead. Cover, boil for 10 min., and pour immediately into a 25 ml. graduated cylinder containing 5 ml. of the silico-molybdate reagent. Dilute to 25 ml. with water. Let stand 5 min. before measuring on the spectrophotometer.

Perform a standard in the same manner, substituting 500 ul. of standard tin solution for the sample. Read the sample and the blank against a water reference at 700 mu, using the red sensitive phototube and 5 cm. cells. If the optical density of the sample is less than that of the standard, report as less than the maximum permissible limit.

Literature References

1. Ind. and Eng. Chem., Anal. Ed., 16, 269 (1944).

Written by: H. H. Van Tuyl (July, 1952)

Code: V

CALCIUM CARBONATE VALUE OF VERSENE AND ANALOGS

Volumetric

PRINCIPLE

The weight of sample required to react with a known amount of standard calcium solution is determined.

Note: The principle is the converse of Method Ca-2.

SOLUTIONS

Standard Hard Water

1 ml is equivalent to 1 mg $CaCO_3$. See under Method Ca-2.

Buffer Solution

See under Method Ca-2.

Indicator Solution

See under Method Ca-2.

PROCEDURE - GENERAL

Accurately weigh an appropriate portion of the sample. Transfer to a 200 ml volumetric flask. Make to volume with water and mix.

Fill a 50 ml burette with this solution.

Accurately pipet an appropriate amount of standard hard water to a flask or bottle. Add 2.0 ml of buffer and 3 drops of indicator. Titrate with the sample dilution.

CALCULATION

$$\text{mgm } CaCO_3 \text{ per gram of sample} = \frac{\text{ml hard water} \times 200}{\text{ml of sample dilution} \times \text{g sample}}$$

PROCEDURE - Analysis of DTPA (#705)(78-80 mg $CaCO_3$/g)

Accurately weigh about 3 g of the liquid sample. Quantitatively transfer to a 200 ml volumetric flask and make to volume with water. Mix and fill a 50 ml burette with this solution.

Place 50 ml of the standard hard water in a flask. Add buffer and indicator. Titrate with the sample dilution.

Note: 500 ul of a 1.203 g/l solution of $MgSO_4$ may be added to improve the end point. If this is done, 500 ul must be added to the hard water volume before calculation.

Procedure - Analysis of HEDTA (Trisodium Salt) #0668
($CaCO_3$ value 120 ± 5)

Accurately weigh about 6 grams (5 ml) in a tared 100 ml beaker. Quantitatively transfer to a 200 ml volumetric flask and make to volume. Mix thoroughly. Fill a 50 ml buret with this solution.

Accurately pipet 50 ml of standard hard water to a flask. Add 3 ml of buffer and 3 drops of indicator. Titrate with the sample solution. Compute as under "General."

FORMALDEHYDE ASSAY

Volumetric

Principle

Formaldehyde is oxidized by peroxide to formic acid in the presence of a known amount of standard sodium hydroxide. The sodium hydroxide consumed is a measure of the formaldehyde content of the sample.

$$CH_2O + NaOH + H_2O_2 \longrightarrow NaCHO_2 + 2H_2O$$

Solutions

Standard 1 N sodium hydroxide
Standard 1 N sulfuric acid
3% hydrogen peroxide
Bromothymol blue (0.1% in 1+1 ethanol) indicator.

Procedure, General

Tare a small glass-stoppered flask or weighing bottle containing 15 ml of water. Quickly add about 3 ml of the formaldehyde solution, stopper, and weigh. Add this solution carefully to 50.0 ml of 1 N sodium hydroxide. Carefully add 50 ml of 3 per cent hydrogen peroxide, neutralized to bromothymol blue. Cover with a small funnel and heat on the steam bath for 15 minutes, shaking occasionally with a gentle rotary motion. Cool to room temperature in cold water and rinse the funnel and walls of the flask with water. Add 0.2 ml of bromothymol blue indicator solution and titrate the excess sodium hydroxide with 1 N sulfuric acid to a bluish green end point. Make a blank test with the same quantities of water, sodium hydroxide, and hydrogen peroxide, and titrate to the same end point. The difference in quantities of sulfuric acid used is equivalent to the sodium hydroxide neutralized by the formic acid corresponding to the formaldehyde in the sample, correction being made for any significant amount of acid in the 3 ml sample of formaldehyde. One milliliter of 1 N sulfuric acid corresponds to 0.03003 gram of HCHO.

Procedure, Analysis of 37% Formaldehyde Solution

Follow the general procedure.

Reference: Reagent Chemicals, A.C.S. Specifications (1960)

OXALIC ACID DETERMINATION

Volumetric Method

Principle

Oxalic acid, like many unsaturated organic compounds, acts as a reducing agent for strong oxidizing agents. The reaction is stoichiometric and can be used for the quantitative determination of the compound. The presence of other reducing agents interferes.

$$5(COOH)_2 + 2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O$$

Solutions

Standardized potassium permanganate, 0.1 N.

Procedure

Weigh accurately 3 g. of sample into a 500 ml. volumetric flask containing about 100 ml. of water. Dissolve the solid completely and dilute to the mark. Mix the solution and pipet a 10 ml. aliquot to an Erlenmeyer flask. Add 3 ml. each of concentrated sulfuric and phosphoric acids. Dilute the solution with 25 ml. of water. Add from a buret 8 ml. of standardized potassium permanganate. Heat the solution to 90°C and complete the titration of the hot solution to the endpoint, which is indicated by a pink coloration that is stable for about one min.

Calculations

$$\% \ (COOH)_2 \cdot 2H_2O = \frac{(\text{ml. KMnO4})(\text{normality})(.06303)(100)}{\text{g. of sample in aliquot}}$$

Literature References

1. Furman, "Scott's Standard Methods of Chemical Analysis," II, 5th Ed., D. Van Nostrand Co. (1939).

2. Rosen, "Reagents, Chemicals, and Standards", D. Van Nostrand Co. (1937).

Written by: R. E. Ewing (July, 1952)

DETERMINATION OF BUTANOL

Volumetric Method

Principle

Alcohols are oxidized by dichromate in acid solution. Since the reaction is slow, an excess of standard dichromate is added, the sample is heated, and the excess is then determined by titration with standard ferrous ammonium sulfate. Any reducing substance will be reported as butanol.

Solutions

Standardized potassium dichromate, 0.1 N.

Standardized ferrous ammonium sulfate, 0.1 N.

Ferroin indicator, 0.025M.

Procedure - Analysis of Tributyl Phosphate #0920, Dibutyl Butyl Phosphonate #0381

Pipet 10 ml of the sample into an Erlenmeyer flask, add 10 ml of 6 N sulfuric acid and 0.1 N standard potassium dichromate solution until a stable yellow color appears. Heat on the hot plate, with mixing, for 3 minutes. Cool under running water. Add one drop of ferroin indicator and titrate with standard ferrous ammonium sulfate to the first permanent orange color.

Calculations

$$\text{Reducing normality} = \frac{(V_D \times N_D) - V_F \times N_F}{\text{ml sample}}$$

V_D = ml of potassium dichromate
N_D = normality of potassium dichromate
V_F = ml of ferrous ammonium sulfate
N_F = normality of ferrous ammonium sulfate

Original by: G. B. Barton (July, 1952)

DETERMINATION OF MESITYL OXIDE

Spectrophotometric Method

Principle

The mesityl oxide concentration is determined spectrochemically at a wave length of 231 millimicrons against pure iso-octane as a blank. It is necessary to obtain a spectrochemical factor from known amounts of pure mesityl oxide. This factor must be redetermined when a new batch of solvent is used, or if the mesityl oxide is redistilled.

Hexone in concentrations greater than 2 ug. per ml. will give high results for which a correction must be applied. The accuracy of the method is dependent upon the purity of the mesityl oxide used as a standard, the care with which dilutions are made, and the proper use of the Beckman quartz spectrophotometer.

Solutions

Iso-octane, highest purity.

Hexone, Shell purified.

Mesityl oxide, freshly distilled. Add 35 ml. of mesityl oxide into a 50 ml. distilling flask with 4 or 5 bumping stones. Use a water condenser to condense the vapor and a glass-col mantle to heat the sample.

Collect approximately 2 ml. of distillate in a 10 ml. graduated cylinder, using this to rinse the graduate. Discard this rinse and continue to collect the distillate at the rate of 10 drops per minute until the temperature reaches 130° C, or until the flask is practically dry.

Store in a glass stoppered Pyrex bottle. The boiling point of mesityl oxide is 128-130° C.

Procedure - Analysis of Hexone

Pipet three 50 ul. portions of the sample (Remark 1) into separate 10 ml. volumetric flasks. Rinse the pipet three times with iso-octane, adding the rinsings to the flask. Pipet 25 ul. of mesityl oxide stock solution into one of the flasks as a spike. Dilute to mark with iso-octane and shake well.

Pipet 50 ul. (or size of sample) of Shell purified hexone into another 10 ml. volumetric flask. Dilute to mark with iso-octane.

Read the optical densities of the samples, spike, and hexone dilution against pure iso-octane at 231 mu and a slit width of 0.9 mm. in 1 cm. silica cells.

To calibrate the instrument, prepare a stock solution consisting of 50 ul. of mesityl oxide (Remark 2), diluted to 50 ml. with iso-octane. Pipet 25, 50, 75, and 100 ul. of stock solution into separate 10 ml. volumetric flasks. Rinse the pipet three times with iso-octane, adding the rinsings to the flask. Dilute to mark with iso-octane. Mix well by shaking.

Read the optical density of the solutions against pure iso-octane at 231 mu with a slit width of 0.9 mm. in 1 cm. silica cells.

Determine the spectrochemical factor for the mesityl oxide stock solution by averaging the individual factors found for each dilution.

$$\text{Factor} = \frac{V \times 10^{-6}}{W}$$

V = ul. of mesityl oxide stock solution
W = optical density.

Calculations

$$\% \text{ by volume mesityl oxide} = \frac{(A-B)C \times 100}{D}$$

A = average optical density of duplicate samples
B = optical density of hexone dilution
C = spectrochemical factor
D = ml. of sample.

$$\% \text{ recovery} = \frac{(E-A)C \times 100}{F}$$

E = optical density of spike
F = ml. of mesityl oxide in spike

Remarks

1. The sample should contain 0.03-0.10 ul. of mesityl oxide. This will normally be contained in 50 ul. of hexone.

2. Mesityl oxide is hygroscopic and must be redistilled once every month.

References

1. Barton, G. B., Hanford Works Secret Notebook, HW-2469-T.

2. Knott, D. M., Hanford Works Secret Notebook, HW-2197-T.

3. Temple, M. K., Hanford Works Secret Notebook, HW-2715-T.

Written by: D. W. Brite (July, 1952)

DETERMINATION OF AROMATIC HYDROCARBONS

Semi-Quantitative Method

Principle

Aromatic hydrocarbon compounds, when contacted with anhydrous sulfuric acid, are converted to sulfonates. The sulfonates are soluble in the sulfuric acid phase.

Phosphorus pentoxide may be employed to insure that the sulfuric acid remains anhydrous.

$$P_2O_5 + 3H_2O \longrightarrow 2H_3PO_4$$

Olefinic hydrocarbons, if present in the sample, also react with sulfuric acid.

$$R - CH = CH \quad R + H_2SO_4 \longrightarrow R - \underset{H}{CH} - \underset{HSO_4}{CH} - R$$

The resulting alkylsulfuric acid, like the sulfonates, is soluble in the sulfuric acid phase. An independent olefin determination must therefore be performed and a correction for olefins must be applied.

Solutions

Sulfuric acid phosphorus pentoxide mixture, 70% sulfuric acid-30% phosphorus pentoxide. Add 454 g (1 lb.) of phosphorus pentoxide to 578 ml of concentrated sulfuric acid in a 2 liter beaker. Stir until the phosphorus pentoxide is dissolved. Pour the solution into 500 ml glass-stoppered bottles. Use a glass cap to keep moisture from collecting on the lip of the bottle.

Procedure - Analysis of Petroleum Diluent #0720 or NPH #0669

Add 25 ml of the sulfuric acid - phosphorus pentoxide reagent to a sulfonation flask, stopper the flask, and place in an ice bath in a hood for 5 minutes.

Record the temperature of the sample. Pipet 10 ml into the flask, allowing the sample to run down the sides of the flask so that mixing of the two layers does not occur. Place the flask in the ice bath for another 5 minutes.

Use face mask (Remark 2). Shake the flask cautiously in the ice bath for 2 minutes so that the layers mix. Raise the flask above the ice bath and shake for 20 seconds with a vertical wrist motion at a rate of 150-200 3 to 4-inch strokes per minute. Replace immediately in the ice bath and shake for 40 seconds. Continue shaking the flask alternately out of the bath 20 seconds and in the bath 40 seconds, until a total shaking time of 10 minutes has elapsed.

Remove the flask from the ice bath and vent the flask momentarily. Centrifuge for 3 minutes at 1000 rpm. Pour concentrated sulfuric acid down the side of the flask until the hydrocarbon layer is entirely within the calibrated portion of the flask. Centrifuge for 3 minutes more.

Adjust the temperature of the sample to within 1° C of the temperature at which the sample was measured. Determine the volume of the unreacted portion of the sample.

Calculations

Percent by volume aromatics = 100 - 10R - C - U

R = ml of unreacted sample
C = correction for solubility of the unreacted portion in the acid layer, which is taken as 1
U = % olefins as found in olefin determination

Remarks

1. There is a large initial heat of reaction for samples rich in olefins. For such samples it is desirable to vent the small amount of pressure which builds up with the first shaking.

2. A face mask should be worn by the analyst from this point on.

References

1. A.S.T.M. Standards 1949, Part 5, pp. 1040-1044 (D875-46T).
2. A.S.T.M. Standards 1964, Part 17, p 373 (D1019-62)
3. A.S.T.M. Standards 1964, Part 17, p 399 (D1158-59T)

Written by: D. W. Brite (July, 1952)

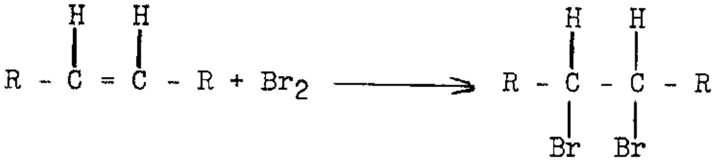
DETERMINATION OF OLEFINIC HYDROCARBONS

Volumetric Method

Principle

Olefinic hydrocarbons enter into an addition reaction with free bromine. If an excess amount of bromine is present, the excess can be determined by the addition of iodide and the titration of the resulting iodine with thiosulfate. The bromine number of the sample (grams of bromine that react with 100 grams of the sample) is determined in this manner. The olefin concentration may be calculated from the bromine number if the average molecular weight of the olefins is known (Remark 1).

$$6HAc + KBrO_3 + 5KBr \longrightarrow 6KAc + 3Br_2 + 3H_2O$$



$$R-CH=CH-R + Br_2 \longrightarrow R-CHBr-CHBr-R$$

$$Br_2 + 2KI \longrightarrow I_2 + 2KBr$$

$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$$

Solutions

Potassium bromide-bromate solution, 0.5N. Dissolve 49.6 g of potassium bromide and 13.9 g of potassium bromate in distilled water and dilute to 1 liter. Pipet exactly 5 ml of this solution, drop by drop, at a rate of 1 to 2 drops per second, while the flask is shaken with a swirling motion, into 50 ml of acetic acid and 5 ml of carbon tetrachloride in a 500 ml iodine-number flask. Shake the solution well and allow to stand 5 minutes.

Proceed as in paragraph 3 of the procedure for determining olefins.

$$\text{Normality of bromide-bromate} = \frac{V_1 N_1}{5}$$

V_1 = ml of thiosulfate
N_1 = normality of thiosulfate

Standardized sodium thiosulfate solution, 0.1 N.

Procedure - Analysis of Petroleum Diluent #0720 or NPH #0669

Weigh or pipet 3-5 g of the sample into a volumetric flask and dilute to exactly 50 ml with carbon tetrachloride. Pipet immediately a 5 ml aliquot into a 500 ml iodine-number flask containing 50 ml of glacial acetic acid.

Titrate with the bromide-bromate reagent at the rate of 1-2 drops per second while swirling the contents of the flask until a distinct yellow color persists for 5 seconds. Do not expose the flask to direct sunlight. Add an additional 1 ml of reagent, stopper the flask, and continue shaking for 40 seconds. Record the volume of reagent used.

Add immediately 5 ml of potassium iodide solution (150 g per liter), by placing in the lip of the flask and removing the stopper momentarily. Shake vigorously, add 100 ml of water, and shake vigorously again for 1 minute. Titrate with 0.1N sodium thiosulfate. When the solution becomes light in color, add 1 ml of starch solution (5 g per liter) and continue the titration to a colorless endpoint.

Calculations

$$\text{Bromine number} = \frac{V_b N_b - V_t N_t}{A} \times 7.992$$

V_b = ml of bromide-bromate
N_b = normality of bromide-bromate
V_t = ml of thiosulfate
N_t = normality of thiosulfate
A = g of sample in aliquot

$$\text{Percent olefins by volume} = \frac{B \times M}{160}$$

B = bromine number
M = molecular weight of olefins

Remarks

1. Relation of olefin molecular weight to A.S.T.M. 50% boiling point.

A.S.T.M. 50% Boiling Point	Molecular Weight of Olefin
300 F	127
350 F	145
400 F	164
450 F	185
500 F	208

References

1. A.S.T.M. Standards 1949, Part 5, pp. 1037-1040 (D875-46T)
2. A.S.T.M. Standards 1964, Part 17, p 299 (D875-62T)

Written by: D. W. Brite (July 1952)

DISTILLATION RANGE

Principle

The distillation range is a measure of the purity of a liquid. It is defined as the difference between the temperature at which distillation begins and the temperature at which all of the material has been vaporized. The temperatures themselves, as well as the range, are specifications.

The procedure given here derives from ASTM D1078-58 and from the USP XIII practices.

Equipment

1. Distilling flasks

   a. A 200 ml distilling flask with a neck of 18-24 mm ID, 10-12 cm long, having an outlet tube of 5-6 mm ID and 10-12 cm long, set at 70-75 deg. angle midway of the neck is used for low-boiling liquids.

   b. For higher boiling liquids, a 50 ml flask with a 10-12 cm neck of 14 to 16 mm ID and an outlet 10-12 cm long and 4 to 5 mm ID is used.

2. Heaters

   A Bunsen burner adjusted to a small flame may be used for non-flammable solvents. An electric heater may be used if its performance has been assured.

3. Thermometer

   Those listed in Stores under captions 57-3195-500 and 57-3195-600 may be used. Thermometers meeting ASTM specification E-1 may be used.

   Thermometers should be graduated for 0.2 or 0.1° C. They may be checked at the steam point and 760 mm, if needed.

4. Condenser and Receiver

   A straight glass condenser with a water jacket from 40-60 cm long is used. An adapter tube leads from the delivery end of the condenser into the graduated cylinder used as a receiver. The adapter tube may be fitted into the receiver through a two-hole stopper. A capillary vent tube in the second hole of this stopper is desirable when flammable solvents are distilled.

5. Asbestos Boards

   A soft asbestos board, about 6 1/2" square and 1/4" or more thick with a hole 50 mm in diameter in the center, is used to protect the 200 ml flask from superheating. A second board with a 4" hole placed under the first board is recommended. An asbestos board with a 25 mm hole is used for the 50 ml flask.

6. Rubber stoppers may be used, although some attack by solvent may occur. A sleeve in a standard taper joint on the distilling flask may be used for the thermometer if attack of the rubber stopper is great. Cork stoppers are permissible.

Procedure, General

1. Assemble the apparatus. Have the distilling flask positioned on the hole in the asbestos board. Adjust the thermometer so that the top of the mercury bulb is even with the center of the outlet tube.

2. Measure the amount of material specified in a graduated cylinder and transfer to the distilling flask. Place the graduated cylinder without drying it in position as the receiver. (Note 1) Cover the cylinder cylinder with foil or a pledget of glass wool.

3. Regulate the heating so that the first drop of liquid is collected in 5 to 10 minutes. Read the initial temperature when 5 drops of liquid fall into the receiver.

   Note: If the heating is slow, the thermometer bulb will have time to warm up to give a good value. The ring of condensing vapor should take about 3 minutes to rise from the distilling bulb to the outlet in the neck of the flask.

4. Distill at the rate of 4 to 5 ml per minute (two drops/second).

5. Record the temperature when the last liquid leaves the bottom of the flask.

6. Read a barometer in or near the room where the test was made. Correct the temperature readings by allowing 0.1 C for each 2.7 mm variation from 760 mm.

7. For high boiling liquids apply an emergent stem correction.

$$\text{Correction} = 0.00015 \times n(T-t)$$

Where N = number of degrees emergent counting from the bottom of the stopper, T is the observed distillation temperature, and t is the temperature at midway on the emergent stem read by an auxiliary thermometer during the distillation.

Note 1: The graduated cylinder may serve in observing the amounts distilled between various temperatures when this information is needed.

Procedure - Analysis of Carbon Tetrachloride #0281

Use a 100 ml sample in a 200 ml flask. Follow the general procedure.

Procedure - Analysis of Trichloroethylene #0930

Use a 100 ml sample in a 200 ml flask. Follow the general procedure.

Procedure - Analysis of Hexone #0640

Use a 100 ml sample in a 200 ml flask. Use an electric heater such as the 550 watt "Fisher Monotemp" heater (Scientific Supplies Catalog #33805). With the full 120 volts this gives a satisfactory distillation rate near 760 mm. A "variac" may be used to control the heater if the distillation rate is too fast.

Hexone is flammable. A closed receiver with a capillary vent is desirable.

DETERMINATION OF REFRACTIVE INDEX

Physical Method

Principle

All liquids have a characteristic refractive index value which can serve as a purity criterion. These values are by no means unique since other liquids or mixtures of liquids may yield the characteristic value of the pure substance.

Apparatus

Refractometer

Procedure - Analysis of Hexone

Read the operating instructions which pertain to the instrument and measure the refractive index of a suitable sample. Report the refractive index as the value obtained for a sample at 20° C and with reference to sodium light (589 mu). Estimate the value to the fourth decimal place.

Written by: T. K. Bierlein (July, 1952)

FLASH POINT OF PETROLEUM DILUENT

Closed Cup Method

Principle

The temperature of the oil under test is gradually raised until a flash of light is detected with the test flame. In the closed cup method, the oil vapor is confined within the enclosed walls of the tester. In the open cup method, as the name implies, the tester is open and the oil vapor is not confined but escapes freely into the atmosphere.

Apparatus

Tag closed tester.

Two A.S.T.M. P.M. and Tag thermometers, +20° to +230°F.

Procedure - Analysis of Petroleum Diluent

Place oil cup in proper position and measure 50 ml. of sample into it. Temperature of sample and of water bath must be at least 20° F below probable flash point when test is started.

Destroy air bubbles on surface of sample and attach cover with flash point thermometer to bath collar.

Light test flame and adjust it to size of white bead on cover. Light gas burner and place it centrally in base of tester. Adjust flame so that temperature of sample rises approximately 1.8° F per min., not faster than 2° F nor slower than 1.6° F per min. Record barometric pressure and initial temperature of sample.

When temperature of sample is 9° F below its probable flash point, turn knob on cover to introduce test flame into vapor space of cup and immediately turn back again. About 1 full second is consumed in turning knob down and back.

Record time and temperature of sample at first introduction of test flame. Repeat application of test flame after each 1° F rise in temperature of sample until distinct flash is observed.

Do not confuse flash with bluish halo sometimes surrounding test flame during applications immediately preceding actual flash. Record time and temperature of sample when flash point is reached. If test is to be repeated, use fresh sample. When barometric pressure is below 760 mm., add 1.6° F for each 25 mm. of barometer difference; when above 760 mm., deduct 1.6° F for each 25 mm.

References

1. A.S.T.M. Standards, 1949, Part 5 (D56-36), pp. 687-690.

Written by: R. Ko (July, 1952)

DETERMINATION OF VISCOSITY

Kinematic Method

Principle

Viscosities of petroleum diluents are measured in terms of the number of seconds required for a specified volume of diluent to flow, at a given temperature, through a capillary tube of specified length and diameter. Kinematic viscosity in centistokes is obtained by multiplying the efflux time by the viscosimeter constant.

The Series 50 modified Ostwald viscosimeter is limited to the range of 300-1500 seconds efflux time or of 0.8-3 centistokes.

Apparatus

Modified Ostwald viscosimeter, Series 50.

A.S.T.M. kinematic viscosity test thermometer, range 74.5 - 79.5° F.

Procedure - Analysis of Petroleum Diluents (#0720, #0669)

Clean the viscosimeter by rinsing twice with petroleum ether and remove the ether by suction. If the sample is not clean, filter more than 10 ml through a medium sintered glass filter or a 100 mesh screen.

Introduce 10 ml of the sample into the viscosimeter through the arm with the large bore. Immerse the viscosimeter in a water bath regulated at 77° F (25 C), so that the upper bulb is below the surface of the water. Adjust to a vertical position and allow at least five minutes for the sample to attain bath temperature.

Apply suction to the capillary arm to draw the sample up to a point about 5 mm above the mark between the bulbs. Release the suction and measure the time in seconds required for the meniscus to pass from the upper to the lower mark.

Run a check determination by again drawing the sample above the etched line between the bulbs and timing as before.

Calculations

Viscosity in centipoises = t x c x sp. gr.

t = efflux time in seconds
c = viscosimeter constant (Remark 1)
sp.gr. = specific gravity of sample.

Remarks

1. The viscosimeter constant is determined by the above procedure, using the efflux times for distilled water at 77 F. The kinematic viscosity of water shall be taken as 0.894 centistokes at 77 F.

$$\text{Viscosimeter constant} = \frac{0.894}{\text{Efflux time in seconds}}$$

References

1. A.S.T.M. Standards 1949 (D445-46T), Part 5, pp. 906-909.

Written by: M. C. Lambert (July, 1952)

DETERMINATION OF SPECIFIC GRAVITY

Hydrometer Method

Principle

The specific gravity of a liquid may be measured by observing the extent to which a float or hydrometer sinks in the liquid. Control of temperature is, of course, necessary. Specific gravity hydrometers are designed to measure the density of the liquid at 60 F (15.56 C) referred to water at 60 F.

Procedure - General

Place a sample in a clean dry glass cylinder and adjust the temperature of the sample to 60 F preferably by allowing the cylinder to stand in a 60 F water bath.

Place the clean dry hydrometer carefully in the cylinder so that the emergent stem is not unnecessarily wetted by the sample. Read the specific gravity from the scale at the liquid surface.

Wetting the emergent stem with a liquid increases the weight of the float and so can cause a low reading.

The cylinder should be of large enough diameter so that the hydrometer floats free without touching the cylinder wall.

The liquid surface is at the bottom of the meniscus.

Procedure - Analysis of NPH #0669 (Sp.G. 60/60 F 0.75-0.77)

Follow the general procedure. See ASTM Standards 1964, Part 17, (D1298 55)

Procedure - Analysis of Nitric Acid (#670)

Follow the general procedure.

The following table correlates percentage of nitric acid with various hydrometers.

% HNO_3	57%	58%	59%	60%
Sp.G (60/60 F)	1.3578	1.3635	1.3689	1.3741
Sp.G (77/68F)	1.3680	1.3737	1.3791	1.3843
SpG (25/4 C)	1.3445	1.3499	1.3552	1.3604
Density 25C	1.3441	1.3495	1.3548	1.3600

SPECIFIC GRAVITY BY PYCNOMETER

Principle

A known volume of liquid is measured in a pycnometer at a fixed temperature and weighed.

Apparatus

1. Pycnometer. A 5 ml modified Sprengel tube, drawing No. H-3-1704, is used. An identifying mark on the pycnometer should be made.

2. A constant temperature bath controlled to ± 0.1° C.

Procedure - Calibration of Pycnometer

1. Clean the pycnometer with chromic acid cleaning solution, rinse thoroughly with distilled water and then with acetone. Dry thoroughly by drawing a current of air through the pycnometer. Weigh to 0.0001 g.

2. Record this weight and the identifying mark on the pycnometer.

3. Using boiled and cooled distilled water, fill the pycnometer slightly beyond the mark. Immerse in a constant temperature bath at the specified temperature (usually 25 C) for at least 5 minutes.

4. Adjust liquid to the calibration mark by blotting the tip of the pycnometer without removing it from the bath. Remove from the bath and dry with wipes. Weigh to 0.0001 g.

Calculation

1. Gram of water contained at the specified temperature:

$$g = \text{Wt. full} - \text{Wt. empty}$$

2. Ml volume at specified temperature referred to water at 4° C.

$$\text{Ml volume} = \frac{\text{Wt. full} - \text{Wt. empty}}{\text{density of water at temperature specified}}$$

$$\text{Ml volume at } 25/4 = \frac{\text{Wt. full} - \text{Wt. empty}}{0.99707}$$

$$\text{Ml volume at } 20/4 = \frac{\text{Wt. full} - \text{Wt. empty}}{0.99823}$$

3. Record the calibration values and the tare weights.

Note 1

The pycnometer should be handled with a paper wipe or with cotton gloves. Rubber gloves can cause a static charge and so an error in weighing.

Procedure - Analysis of Sample - General

1. Fill the clean dry calibrated pycnometer with sample. Place in water bath for at least 5 minutes. Touch to the mark without removing from bath. Remove from the bath and dry with wipes. Weigh to 0.0001 g.

Calculate as specified for the particular sample.

Procedure - Specific gravity of bromobenzene #0220. Max. 1.495 20/4 C.

Follow the general procedure using a pycnometer calibrated at 20 C.

Calculation

$$\text{Sp. Gr. of sample } 20/4 \text{ C} = \frac{\text{Net Wt. of sample}}{\text{ml volume at } 20/4 \text{ C}}$$

Procedure - Specific gravity of di-2 ethylhexyl phosphoric acid. #0384

0.965 - 0.979 25/4 C

Follow the general procedure using a pycnometer calibrated at 25 C.

Calculation

$$\text{Sp. Gr. of sample } 25/4 \text{ C} = \frac{\text{Net Wt. of sample}}{\text{ml volume at } 20/4 \text{ C}}$$

Procedure - Specific gravity of ferrous sulfamate solution. #0432

1.52 ± 0.02 at 25/25 C.

Use a pycnometer on which the net grams content of water at 25 C is known. Fill the pycnometer with sample at 25 C and obtain the net weight.

Calculation

$$\text{Sp. Gr. } 25/25 = \frac{\text{Net wt. of sample}}{\text{Net wt. of water}}$$

Procedure - Specific Gravity of HEDTA (Trisodium Salt) Solution #0668
Min. 1.30 25/25 C

Use a pycnometer in which the net grams content of water at 25° C is known. Fill the pycnometer with sample at 25 C and obtain the net weight.

Procedure - Specific gravity of nitric acid. #0671 Min. 1.422 20/4 C

Follow the general procedure using a pycnometer calibrated at 20/4 C

Calculation

$$\text{Sp.Gr. } 20/4\text{ C} = \frac{\text{Net Wt. of sample}}{\text{ml volume } 20/4\text{ C}}$$

Procedure - Specific gravity of DTPA solution. #0705. Min. 1.30 25/25 C

Use a pycnometer on which the net grams content of water at 25° C is known. Fill the pycnometer with sample at 25°C and obtain the net weight.

Calculation

$$\text{Sp.Gr. } 25/25 = \frac{\text{Net Wt. of sample}}{\text{Net Wt. of water}}$$

Procedure - Specific gravity of TBP. #0920 0.973 - 0.983 20/20

Use a pycnometer on which the net grams content of water at 20°C is known. Fill the pycnometer with sample at 20 C and obtain the net weight.

Calculation

$$\text{Sp.Gr. } 20/20 = \frac{\text{Net Wt. of sample}}{\text{Net Wt. of water}}$$

Procedure - Specific gravity of trichloroethylene. #0930 1.450 - 1.475 20/20 C

Follow the procedure for TBP.

WATER INSOLUBLES DETERMINATION

Gravimetric Method

Principle

All material not dissolved by water is classified as water insoluble material.

Procedure - General

Digest the solution of the sample on the steam bath for 1 hour. Filter the hot solution through a tared Gooch or Selas crucible. Wash the beaker and residue with six 10 ml. portions of hot water.

Dry crucible and contents to constant weight at 105 C.

Calculations

$$\text{Percent insoluble} = \frac{(\text{g. insoluble})\ (100)}{\text{g. sample}}$$

Procedure - Analysis of Aluminum Nitrate Solution #0060, 0.2%

Weigh 15 g of solution into beaker. Dilute with 100 ml of hot water and proceed as described under "General."

Procedure - Analysis of Ferrous Ammonium Sulfate #0430, 0.01%

Weigh 10 g of sample into a beaker and dissolve with 0.5 ml of concentrated sulfuric acid, diluted to 100 ml with water. Proceed as described under "General."

Procedure - Analysis of Oxalic Acid #0690, .05%, Sodium Carbonate #0785, 0.1%, Sodium Nitrate #0805, 0.1%, Sodium Sulfite #0825, 0.5%, Sodium Nitrite #0810, 0.05%, and Potassium Bicarbonate #0743, .01%

Weigh 20 g of sample into a clean beaker and dissolve in 100 ml of water free of reducing materials. Proceed as described under "General."

Procedure - Analysis of Potassium Permanganate #0750, 0.2%

Weigh 10 g of sample into a clean beaker and dissolve in 100 ml of hot water free of all reducing materials. Proceed as described under "General," using a coarse sintered glass crucible instead of the Gooch crucible.

Procedure - Analysis of Potassium (or Sodium) Persulfate #0752, 0.01%

Weigh 10 g of sample and transfer to an 800 ml beaker. Add 500 ml of warm water or add cold water and heat gently. Stir. Collect any insoluble material on a tared Selas or Gooch crucible and weigh. Compute as under "General."

Procedure - Analysis of Sodium Dichromate Solution #0796, 0.1%

Weigh 20 g of sample into a beaker Dilute with 100 ml of hot water and proceed as described under "General."

Procedure - Analysis of Sulfamic Acid #0870

Weigh 10 g of sample into a 250 ml beaker. Dissolve in 100 ml of water and proceed as under "General."

Literature References

1. Rosin, "Reagent Chemicals and Standards," D. Van Nostrand Co., (1934)

DETERMINATION OF SUSPENDED SOLIDS

Gravimetric Method

Principle

Extraction of tributyl phosphate with kerosene removes the kerosene soluble suspended solids. Weighing the precipitate remaining after filtration permits calculation of the kerosene insoluble suspended solids originally present.

Procedure - Analysis of Tributyl Phosphate #0920, Dibutyl Butyl Phosphonate #0381

Weigh 100 ml. of sample into a 250 ml. stoppered graduate and add 100 ml. kerosene. Extract by inverting the graduate 25 times. Filter through a fine, tared, sintered glass crucible, using vacuum. Wash several times with kerosene and dry in overn at 110° C. Cool in desiccator and weigh.

Calculations

$$\% \text{ Solids} = \frac{\text{g of solids} \times 100}{\text{g of sample}}$$

Original by: C. E. Michelson (July, 1952)

UNCLASSIFIED

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HW-54125
March 15, 1965
Zy-16-1

INSOLUBLES - SPECIAL PROCEDURES

Principle

A tolerance for a residue not soluble in acids, alkalies, or other solvents is sometimes specified in purchasing a material. No general procedure is applicable. A special procedure for each material is used.

Procedure - Analysis of Sodium Bismuthate #0782

Weigh a 10 gram ± 0.5 gram sample and transfer to a 250 ml beaker. Add 25 ml of water and 25 ml of concentrated nitric acid. Mix and carefully add 10 ml of 30% hydrogen peroxide. Cover the beaker with a watch glass and heat on a low hot plate for 10 minutes. Collect any residue on a tared Selas or Gooch crucible. Wash twice with 3 N nitric acid and then with water. Dry at 105 C for four hours or to constant weight. Cool and weigh. Report percent insolubles.

DETERMINATION OF SUBSTANCES NOT PRECIPITATED BY HYDROCHLORIC ACID

Gravimetric Method

Principle

Silver chloride is precipitated from an aqueous solution by adding hydrochloric acid. Total solids remaining in solution are classified as substances not precipitated by hydrochloric acid.

Procedure - Analysis of Silver Nitrate

Weigh 25 g. of sample into a 600 ml. beaker, add 350 ml. of water, and heat to boiling. Slowly add 15 ml. of concentrated hydrochloric acid, dilute to about 400 ml., and let stand overnight. Filter and dilute filtrate to exactly 500 ml.

Evaporate a 100 ml. aliquot to dryness. Heat the residue with 10 ml. of water containing 2 drops of hydrochloric acid, and filter. Wash with 10 ml. of water, and dry the filtrate to constant weight in a tared evaporating dish.

Perform a blank, using the same procedure, without addition of the sample.

Calculations

$$\% A = \frac{(B-C) \times 500}{D}$$

where A = substances not precipitated by hydrochloric acid
B = g. of residue from sample
C = g. of residue from blank
D = g. of initial sample

Literature References

1. American Chemical Society, "Specifications for Analytical Reagents", p. 101 (1941).

2. Rosin, "Reagent Chemicals and Standards", D. Van Nostrand Co., p. 365 (1934).

Written by: H. H. Van Tuyl (July, 1952)

DETERMINATION OF OXIDIZING IMPURITIES

Spectrophotometric Method

Principle

Oxidizing impurities in hexone react with ferrous ammonium sulfate to form ferric iron, which is determined with the spectrophotometer after formation of the thiocyanate complex of ferric iron. The iron must be kept in the ferrous state prior to addition of the sample and is made up in sulfamic acid for this purpose. The SCN^- concentration must be high to minimize errors resulting from the formation of colorless ferric complexes with chloride, phosphate, sulfate, and other ions in acid solution. Several metal ions also form colored complexes with thiocyanate and will interfere. The concentration of these interferences is reduced by a preliminary water wash.

Solutions

Standard ferric solution, 0.1 N. Dissolve 39.214 of $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ in 100 ml. 1 N HNO_3, heat to boiling, cool, and make up to 1 liter with distilled water in a volumetric flask.

Dilute standard ferric solution, 0.001 N. Dilute 1 ml. of 0.1 N ferric solution to exactly 100 ml.

Dilute standard ferric solution, 0.005 N. Dilute 5 ml. of 0.1 N ferric solution to exactly 100 ml.

Dilute standard ferric sultuion, 0.010 N. Dilute 10 ml. of 0.1 N ferric solution to exactly 100 ml.

Dilute standard ferric solution, 0.015 N. Dilute 15 ml. of 0.1 N ferric solution to exactly 100 ml.

Standardized ferrous ammonium sulfate, 0.05 N. Make up 19 ml. concentrated HNO_3 + 0.9709 g. NH_2SO_3H + 19.6075 g. $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ to 1 liter with distilled water.

Sodium thiocyanate, 10 g. per liter. Make up 25 ml. concentrated H_2SO_4 + 10 g. NaSCN to 1 liter with distilled water.

Procedure - Analysis of Hexone

Dilute 1 ml. of each of the dilute standard ferric solutions to 100 ml. with the sodium thiocyanate solution and read in the spectrophotometer in 1 cm. cells at 475 mu against a NaSCN blank. Plot a calibration curve showing per cent transmittance against normality of the dilute standard ferric solutions added.

Shake 10 ml. of the sample with 0.5 ml. of distilled water and discard the aqueous phase. Add 2 ml. of the ferrous ammonium sulfate solution and shake for 1 hr. Dilute 1 ml. of the aqueous phase to 100 ml. with the sodium thiocyanate solution and read immediately in the spectrophotometer in 1 cm. cells at 475 mu against a NaSCN blank.

Calculations

$$\text{oxidizing normality} = \frac{\text{normality from calibration curve}}{5}$$

Literature References

1. Marsden, J. to Greager, O. H., GE-N-12983 (6/15/48).

Written by: A. Chetham-Strode (July, 1952)

DETERMINATION OF RESIDUE

Gravimetric

Principle

Impurities may be observed in a volatile material by evaporating a weighed sample in a tared dish and weighing the residue.

Also, certain materials may be characterized by loss in weight observed under controlled conditions.

Procedure - General

Heat a sample in an appropriate manner as directed below and obtain the weight of residue.

Calculations

$$\% \text{ Residue} = \frac{\text{grams residue} \times 100}{\text{grams sample}}$$

Procedure - Analysis of Iodine

Weigh to two decimal places by difference from a glass-stoppered weighing bottle about 10 grams of sample. Transfer to a tared porcelain dish of about 2½ or 3 inches diameter. Place a watch glass cooled by a stream of air or a water-cooled condenser made from a round-bottomed blask closely over the dish. Heat the dish on the low of a reliable hot plate or on a steam bath until the iodine has sublimed to the condenser. Dry the dish and residue in an oven at 105 C. Cool and weigh.

Procedure - Analysis of Nitric Acid

Pipet 10 ml of sample into a tared 100 ml platinum or porcelain dish. (10ml amounts to 13.7 grams of 60% nitric acid). Evaporate on a low hot plate. Heat the dish and residue at 105 C in an oven. Cool and weigh.

Procedure - Analysis of Sodium Acetate (min. 59.0%) (#780)

Accurately weigh a sample of 1 to 2 g in a tared weighing bottle. Dry at 80 C for 16 hours and then at 120 C for 4 hours. Cool and weigh. The weight of residue should be no less than 59% of the sample weight.

Procedure - Analysis of Hydrogen Peroxide (#526) (< 0.01%)

Place 20 ml of sample in a tared porcelain dish. Evaporate at steam bath temperature (low hot plate). Dry at 105°C, cool and weigh. Assume Sp. G of 1.0 for the 20 ml sample.

Procedure - Analysis of Acetic Acid (#064) ($<$ 0.01%)

In a dry tared porcelain evaporating dish place 95 ml (100 g) of sample. Evaporate to dryness on a steam bath or very low hot plate. Dry at 105 C in an oven for 20 minutes. Cool and weigh. The weight of residue should not exceed 0.01 g.

Procedure - Analysis of Carbon Tetrachloride #0281 (Max. 0.002 %)

In a dry tared evaporating dish or beaker place 100 ml (158 g) of sample. Evaporate to dryness on a steam bath or very low hot plate. Dry the residue at 105 C in an oven. Cool and weigh. The weight of residue should not exceed 0.0032 grams.

Procedure - Analysis of Trichloroethylene #0930, (Max. 0.004%)

In a dry tared evaporating disk or beaker place 50 ml (73 g) of sample. Evaporate to dryness on a steam bath or very low hot plate. Dry the residue at 105 C in an oven. Cool and weigh. The weight of residue should not exceed 0.0029 grams.

RESIDUE AFTER IGNITION

Gravimetric

Principle

The Method

In general a weighed sample in a tared porcelain or platinum dish of 25 or 50 ml capacity is carefully heated in a hood until charred. Organic materials may tend to foam and overflow the container if heating is rapid. A small flame is most practical for heating.

The char is cooled and moistened with 1 to 2 ml of concentrated sulfuric acid. The char is then ignited (hood) until all carbon is burned out and no further SO_3 fumes are observed. It is then further ignited at 600-800 C for 15 minutes in a muffle.

The dish and residue are cooled and weighed. The net weight is recorded.

Calculation

$$\% \text{ Residue} = \frac{\text{Net Wt. of residue} \times 100}{\text{Sample Wt.}}$$

Procedure - Analysis of tartaric acid #910 ($<0.02\%$).

Accurately weigh a sample of about 5 grams. Follow the general procedure.

Procedure - Analysis of citric acid #285 ($<0.01\%$)

Accurately weigh a sample of about 5 grams. Follow the general procedure.

Procedure - Analysis of Hydroxy Acetic Acid #524 ($<0.1\%$)

Use a 10 gram sample (8 ml of a 70% solution). Weigh on rough balance or measure with a graduated pipet. Follow the general procedure. The residue may be used in Mg-1.

DETERMINATION OF PARTICLE SIZE

Sieve Method

Principle

A sample is placed in the top of a nest of U. S. sieves and the sieves are shaken for a definite period. The residue in each sieve is weighed and the percentage passing or retained is computed.

Procedure - General

Weigh 100 g of sample to the nearest 0.1 g and place on the top sieve. Place sieves in Ro-top shaker and shake for 30 minutes. Weigh residue on each sieve.

Calculations

% passing on 1st screen = 100 (a)
% passing on 2nd screen = 100 (a + b)
% passing on 3rd screen = 100 (a + b + c)

where a = weight on first screen
b = weight on second screen
c = weight on third screen

Procedure - Assay of Magnesium Oxide (#312)

Use sieves appropriate to specifications under which a shipment is bought.

Procedure - Analysis of Sodium Bismuthate (#0782)

Use 200 mesh screen. 95 Wt.% should pass through the screen.

Literature Reference

1. Griffins, "Technical Methods of Analysis," pp. 814-815, (1927).

RESIDUE AFTER IGNITION

Gravimetric

Principle

The Method

In general a weighed sample in a tared porcelain or platinum dish of 25 or 50 ml capacity is carefully heated in a hood until charred. Organic materials may tend to foam and overflow the container if heating is rapid. A small flame is most practical for heating.

The char is cooled and moistened with 1 to 2 ml of concentrated sulfuric acid. The char is then ignited (hood) until all carbon is burned out and no further SO_3 fumes are observed and finally at 600-800 C for 15 minutes in a muffle.

The dish and residue are cooled and weighed. The net weight is recorded.

Calculation

$$\% \text{ Residue} = \frac{\text{Net wt. of residue} \times 100}{\text{Sample wt.}}$$

Procedure - Analysis of tartaric acid #910 <(0.02%).

Accurately weigh a sample of about 5 grams. Follow the general procedure.

Procedure - Analysis of citric acid #285 (<0.02%).

Accurately weigh a sample of about 5 grams. Follow the general procedure.

DETERMINATION OF PARTICLE SIZE

Wet-Sieve Method

Principle

Two techniques for screening resin are employed in this method.

The resin is first displaced by flowing water from a large graduated cylinder onto a nest of standard sieves. The rate of overflow is controlled such that the finer resin beads pass over first and thus are passed through the larger mesh screens before larger resin particles blind the top screens. Incremental increases in flow rate, over at least two hours, allow partitioning.

A dishpan technique is used for final screening. The largest size sieve is placed in a dishpan and water is added to about one-half the height of the sieve. The sieve is gently raised and lowered so as alternately to lift the particles on the screen and float them off again. The contents of the dishpan are added to the next size screen and the same procedure applied.

After all screens have been washed by the dishpan technique, the resin from each screen is washed into separate graduates. The screens are left to dry overnight on paper towels. After drying, the screens are brushed thoroughly (with a soft brush) and the resin thus obtained on the paper towels is added to the graduate containing resin from that screen.

The resin is tapped to constant volume in the graduates and the volume recorded. Per cent of resin retained on each screen is calculated by dividing the volume recorded for the sieve by the sum of the recorded volumes and multiplying by 100.

Apparatus

U. S. Standard Sieves - preferably stainless steel. Specific screens are dependent on requirements. A. U. S. No. 74 (200 mesh) is included as the bottom screen.

White enamel dishpan (12 inch). The white enamel surface makes observing the efficiency of screening easier.

500 ml graduate without pouring spout.

Smaller graduates as required for measuring final volumes. (Graduated cylinders ranging from 250 ml to 10 ml should be available.)

Sink and tap water source.

Flexible water tubing tipped with non-flexible 10 mm tubing.

Procedure

1. Place a sample of approximately 150 ml of resin in a 500 ml graduate (without a pouring spout).
2. A nest of sieves is placed (level) in a sink, a U.S. No. 74 (200 mesh) being the bottom sieve.
3. The graduate containing the sample is placed on the first screen and centered.
4. The water inlet tube is placed approximately 1/8 inch from the bottom of the graduate and centered.
5. Turn the water on and slowly increase the flow rate until the bed expands enough to cause only the finest resin particles to overflow the graduate. Incrementally increase the flow rate as needed. Do not rush. A minimum of two hours should be used for this step. The amount of resin overflowing at any time should be small. Occasionally rotate the graduate.
6. After the graduate is nearly void of resin, wash any remaining and adhering resin into the sieve.
7. Fill a dishpan with water to a depth of about one-half the height of a sieve.
8. Carefully remove the top sieve from the nest and place in the dishpan.
9. Gently raise and lower the sieve so as to lift the particles on the screen and float them off again. This operation is repeated approximately 25 times.
10. Set the sieve aside.
11. Wash the contents of the dishpan into the remaining sieve nest.
12. Replace the sieve from step 10 into the water-filled dishpan and repeat steps 9 through 11 until no more than a dozen particles seem to come through after a washing.
13. Wash contents of sieve into a graduate for measuring.
14. Place sieve on a paper towel and allow to dry overnight.
15. After drying, thoroughly brush (with a soft brush) the resin from the screen. Add the recovered resin to the graduate from step 13.
16. Tap the graduate to a constant volume of resin. Record volume.
17. Repeat steps 7 through 16 for each sieve.

Calculations

Calculate the volume per cent of resin retained on each screen as follows:

$$\text{Vol. \% retained on U.S. Std. Sieve No. A} = \frac{B \times 100}{C}$$

where A = the U.S. Std. sieve number (include mesh size in parenthesis)

B = vol. of resin retained on U.S. Std. Sieve A

C = total volume retained on all sieves.

Particle Size of Permutit SK Anion Exchange Resin (Ionac A-580)

Use U. S. No. 841 (20 mesh), U. S. No. 420 (40 mesh), and U. S. No. 74 (200 mesh) sieves.

References

1. R. Kunin. Ion Exchange Resins, John Wiley & Sons, Inc., New York, 1958. Second ed., pp. 322-323.

2. "Ion Exchange Resins: Sieve Analysis," Essential Materials Methods, SRP Laboratory Manual. February 1962.

Submitted by: C. A. Colvin (June 1964)

Approved by: O. F. Beaulieu (June 1964)

FLAME PHOTOMETRY

Principle

Calcium, potassium, sodium, and strontium occurring as impurities in certain substances may be determined best by use of a flame photometer such as the Beckman Model DU with flame attachments.

The method is essentially an extension of emission spectrography. Atoms are excited in a flame and the intensity of emitted light at appropriate wave lengths is observed by means of a photometer rather than on film.

Procedure - General

The directions given in "Reagent Chemicals" American Chemical Society Specifications 1960 page 13 are appropriate and are quoted here.

"The test is a semi-internal-standard method and requires two solutions. For Sample Solution A, a definite amount of the sample is dissolved and diluted to a definite volume. For Control Solution B, an equal amount of sample plus specified quantities of various impurities to be determined is dissolved and diluted to the same volume as Sample Solution A. Adjust the flame photometer to give as near as possible to 100 per cent transmittance at the designated characteristic wave length of the impurity to be determined with Control Solution B and record this value. Without changing the adjustment of the instrument, determine the emission of Sample Solution A at this wave length. Readjust only the monochromator by the small specified amount and determine the emission of Sample Solution A at this new wave length. This latter value is used to correct the emission of Sample Solution A for background emission produced by the salt and solvent. The salt being tested contains less than the specified amount of impurity if the difference (D_1) between the intensities observed for Sample Solution A at the characteristic wave length of the impurity and at the wave length used to correct for background is less than the difference (D_2) between the intensities observed for Sample Solution A and Control Solution B at the characteristic wave length of the impurity. This test is designed for a limiting test; the determination of the actual amount of impurity present requires a more elaborate procedure, especially as to controls and calibration curves.

The preparation of the various solutions and the adjustments required for the flame photometer are described in each particular test. However, no absolute directions that are infallible can be given. This type of analysis, like every other, depends on experimental conditions and the analytical technique of the operator.

Calcium. Use a flame photometer of the type previously described. Set the slit-width control at 0.03 mm. and the selector switch at 0.1. Adjust the instrument to give the maximum emission with Control Solution B at the 422.7-mμ calcium line and note this reading. Without changing any of the controls, determine the emission of Sample Solution A at this wave length. Change only the monochromator by the amount specified in the particular test and determine the emission of Sample Solution A at this new wave length.

The directions for preparing Sample Solution A and Control Solution B as well as the calculations are specified in each test.

Potassium. Use a flame photometer of the type previously described and use the red-sensitive phototube. Set the slit-width control at 0.3 mm. and the selector switch at 0.1. If the salt being tested is a calcium salt, one must use an oxyhydrogen burner, not an oxyacetylene burner. Adjust the instrument to give the maximum emission with Control Solution B at the 766.5-mμ potassium line and note this reading. Without changing any of the controls, determine the emission of Sample Solution A at this wave length. Change only the monochromator by the amount specified in the particular test and determine the emission of Sample Solution A at this new wave length.

The directions for preparing Sample Solution A and Control Solution B as well as the calculations are specified in each test.

Sodium. Use a flame photometer of the type previously described. Set the slit-width control at 0.01 mm. and the selector switch at 0.1. Adjust the instrument to give the maximum emission with Control Solution B at the 589-mμ sodium line and note this reading. Without changing any of the controls, determine the emission of Sample Solution A at this wave length. Change only the monochromator by the amount specified in the particular test and determine the emission of Sample Solution A at this new wave length.

The directions for preparing Sample Solution A and Control Solution B as well as the calculations are specified in each test.

Strontium. Use a flame photometer of the type previously described. Set the slit-width control at 0.03 mm. and the selector switch at 0.1. Adjust the instrument to give the maximum emission with Control Solution B at the 460.7-mμ strontium line and note this reading. Without changing any of the controls, determine the emission of Sample Solution A at this wave length. Change only the monochromator by the amount specified in the particular test and determine the emission of Sample Solution A at this new wave length.

The directions for preparing Sample Solution A and Control Solution B as well as the calculations are specified in each test.

Code: Zy-25

Procedure - Analysis of 50% Sodium Hydroxide for Calcium and Strontium

(Limits 0.002% Ca, 0.001% Sr amounting to 40 ppm and 20 ppm on 100% basis.)

A. Special Reagents and Equipment

1. Cation exchange resin of analytical grade, preferably Chelex-100, BioRad Laboratories, Richmond, Calif. (This corresponds to commercial Dowex A1.)

2. Ion exchange columns of Pyrex tubing 3 mm ID, and 3 cm in height with a 15 ml reservoir and a dropper type tip. Glass wool is used to support a resin bed 2 cm high. Glass wool is also placed on top of the resin to prevent fouling.

3. Standard 0.1 g/l calcium solution made by dissolving reagent calcium carbonate in approximately 1N HCl.

4. Standard 0.1 g/l strontium solution made by dissolving strontium chloride hexahydrate ($SrCl_2 . 6H_2O$) in approximately 1N HCl.

B. Separation from Sodium

1. Weigh exactly 4.0 g samples of 50% NaOH into two tared plastic bottles (2 oz.) labeled "A" and "B". Record weights. (Sample weight should be 4.0 ± 0.05 g.)

2. Pipet 5.0 ml of distilled water into each bottle and mix with a Teflon spin bar on a magnetic stirrer.

3. Add slowly (no agitation) 4.2 ml of 12M HCl to each bottle; cool to room temperature in ice water. Mix.

4. Add exactly 0.08 mg of Ca and 0.04 mg of Sr to bottle "B" and mix. (800 ul of 0.1 g/l Ca, 400 ul of 0.1 g/l Sr.)

5. Check the pH of both samples and adjust if necessary to 5.0 or below.

6. Adjust the sample pH to between 7.5 and 8.5 with 1.0M NH_4OH.

7. Transfer the contents of each bottle to a Chelex-100 ion exchange column. Rinse each bottle with 2.0 ml of distilled water and transfer the rinse to the ion exchange column reservoir. Discard the column effluents.

8. Wash each column with 3.0 ml of H_2O. Discard the wash.

9. Place clean, 2 oz. plastic bottles labeled "A" and "B" under the appropriate column. Then add exactly 5.0 ml of 1M HCl to each column. Collect effluent in appropriate bottle.

10. Add exactly 5 ml of distilled water to each bottle, mix, and analyze for Sr and Ca as described below in C, Flame Photometry.

C. Flame Photometry

a. Calcium

1. Check instrument set-up with 0.008 mg/ml calcium at the 422.7 mu calcium emission line, and at 430 mu for background.

2. Set the emission of control solution "B" at 422.7 mu to 100% transmission.

3. Determine the emission of sample solution "A" at 422.7 mu and record the %T (transmission).

4. Repeat (2) and (3). The transmission values for "A" must agree within ± 1%. If they do not, repeat (2) and (3) again.

5. Determine the emission of sample solution "A" at 430 mu, and record %T as background.

b. Strontium

1. Check the instrument set-up with 0.004 mg/ml strontium at the 460.7 mu strontium emission line, and at 457 mu for background.

2. Set the emission of control solution "B" at 460.7 mu to 100% transmission.

3. Determine the emission of sample solution "A" at 460.7 mu and record the %T (transmission).

4. Repeat (2) and (3). The transmission values for "A" must agree within ± 1%. If they do not, repeat (2) and (3) again.

5. Determine the emission of sample solution "A" at 457 mu, and record as background.

D. Calculation

a. Calcium

1. The sample is within the specifications if:

$$\%T_B - \%T_A > \%T_A - \%T_{bkgd}$$

2. Parts calcium per million parts 100% NaOH may be calculated:

$$\text{ppm Ca} = \frac{0.08\left(\dfrac{\%T_A - \%T_{bkgd}}{\%T_B - \%T_A}\right) \times 10^3}{\text{sample weight in grams} \times (0.5)}$$

where: 0.08 = mg of calcium added to control sample weight in grams,
0.5 = sample weight of 100% NaOH.

This calculation should not be used if either $(\%T_A - \%T_{bkgd})$ or $(\%T_B - \%T_A)$ is less than 10.

b. Strontium

1. The sample is within the specifications if:

$$\%T_B - \%T_A > \%T_A - \%T_{bkgd}$$

2. Parts strontium per million parts 100% NaOH may be calculated:

$$\text{ppm Sr} = \frac{0.04\left(\dfrac{\%T_A - \%T_{bkgd}}{\%T_B - \%T_A}\right) \times 10^3}{\text{Sample weight in grams} \times (0.5)}$$

where: 0.04 = mg Sr added to control.

This calculation should <u>not</u> be used if either $(\%T_A - T_{bkgd})$ or $(\%T_B - \%T_A)$ is less than 10.

Procedure - Analysis of Sodium Sulfate for Calcium and Strontium

(Limit 0.001% Ca, 10 ppm and 0.0002% Sr, 2 ppm)

A. Special Reagents and Equipment

(See under Sodium Hydroxide)

B. Separation from Sodium

1. Weigh two 2.0 gram samples and transfer to 2 oz. plastic bottles labeled "A" and "B". Add 10 ml of water to each bottle and mix until solution is effected. Thorough mixing for at least 10 minutes is essential to insure solution of last traces of small hard crystals.

2. Add exactly 0.02 mg of Ca and 0.004 mg Sr to bottle "B" and mix (200 ul of 0.1 g/l Ca, 40 ul of 0.1 g/l Sr).

3. Check the pH of both "A" and "B" and adjust to pH 7.5 to 8.5 with 1.0M ammonia solution.

4. Transfer the contents of each bottle to a Chelex-100 ion exchange column. Rinse each bottle with 2.0 ml of distilled water and transfer the rinse to the ion exchange column reservoir. Discard the column effluents.

5. Wash each column with 3.0 ml of H_2O. Discard the wash.

6. Place clean, 2 oz. plastic bottles labeled "A" and "B" under the appropriate column. Add exactly 5.0 ml of 1M HCl to each column. Collect effluent in appropriate bottle.

7. Add exactly 5 ml of distilled water to each bottle, mix, and analyze for Sr and Ca as described below in C, Flame Photometry.

C. Flame Photometry

a. Calcium

1. Check instrument set-up with 0.008 mg/ml calcium at the 422.7 mu calcium emission line, and at 430 mu for background.

2. Set the emission of control solution "B" at 422.7 mu to 100% transmission.

3. Determine the emission of sample solution "A" at 422.7 mu and record the %T (transmission).

4. Repeat (2) and (3). The transmission values for "A" must agree within ± 1%; if they do not, repeat (2) and (3) again.

5. Determine the emission of sample solution "A" at 430 mu and record %T as background.

b. Strontium

1. Check the instrument set-up with 0.004 mg/ml strontium at the 460.7 mu strontium emission line, and at 457 mu for background.

2. Set the emission of control solution "B" at 460.7 mu to 100% transmission.

3. Determine the emission of sample solution "A" at 460.7 mu and record the %T (transmission).

4. Repeat (2) and (3). The transmission values for "A" must agree within ± 1%; if they do not, repeat (2) and (3) again.

5. Determine the emission of sample solution "A" at 457 mu and record as background.

D. Calculation

a. Calcium

1. The sample is within specifications if:

$$\%T_B - \%T_A > \%T_A - \%T_{bkgd}$$

2. Parts calcium per million parts of sodium sulfate may be caluclated.

$$\text{ppm Ca} = \frac{\text{Control factor} \times \left(\dfrac{\%T_A - \%T_{bkgd}}{\%T_B - \%T_A}\right) \times 10^3}{\text{Sample weight in grams}}$$

This calculation should not be used if either ($\%T_A - \%T_{bkgd}$) or ($\%T_B - \%T_A$) is less than 10.

Control factor = milligrams of calcium added to control = 0.02

b. Strontium

Compute as for calcium. For ppm use control factor 0.004.

Procedure - Analysis of Sodium Carbonate for Calcium and Strontium

(Limits 0.004% Ca, 40 ppm, and 0.001% Sr, 10 ppm)

A. Special Reagents and Equipment

(See under Sodium Hydroxide)

1. Weigh two 2.0 g samples of sodium carbonate and transfer each to a 2 oz. plastic bottle. Label bottles "A" and "B".
2. Pipet 5.0 ml of distilled water into each bottle and mix with a Teflon spin bar on a magnetic stirrer.
3. Add slowly 3.2 ml of 12M hydrochloric acid to each bottle. Mix.
4. Add exactly 0.08 mg of Ca and 0.02 mg of Sr to bottle "B" and mix. (800 ul of 0.1 g/l Ca and 200 ul of 0.1 g/l Sr)
5. Check the pH of both samples and adjust if necessary to 4.0 or below.
6. Adjust the sample pH to between 7.5 and 8.5 with 1.0M ammonia solution.
7. Transfer the contents of each bottle to a Chelex-100 ion exchange column. Rinse each bottle with 2.0 ml of distilled water and transfer the rinse to the ion exchange column reservoir. Discard the column effluents to waste.
8. Wash each column with 3.0 ml of H_2O. Discard the wash.
9. Place clean, 2 oz. plastic bottles labeled "A" and "B" under the appropriate columns. Add exactly 5.0 ml of 1M HCl to each column. Collect the effluent in the appropriate bottles.
10. Add exactly 5 ml of distilled water to each bottle. Mix and analyze for Sr and Ca as described below under Flame Photometry.

C. Flame Photometry

Follow the practice described under Sodium Sulfate.

D. Calculation

Follow the practice described under Sodium Sulfate.

Control factor for calcium = 0.08
Control factor for strontium = 0.02

Procedure - Analysis of Sodium Nitrite for Calcium and Strontium

(0.002% Ca, 20 ppm and 0.0002% Sr, 2 ppm)

A. Special Reagents and Equipment

(See under Sodium Hydroxide)

B. Separation from Sodium

1. Weigh two 2.0 gram samples and transfer to 2 oz. plastic bottles labeled "A" and "B". Add 10 ml of water to each bottle and mix until solution is effected.

2. Add exactly 0.04 mg calcium and 0.004 mgm strontium.

   (400 ul of 0.1 g/l Ca, 40 ul of 0.1 g/l Sr)

3. Check the pH of both "A" and "B" and adjust to pH 7.5 to 8.5 with 1.0M ammonia solution.

4. Transfer the contents of each bottle to a Chelex-100 ion exchange column. Rinse each bottle with 2.0 ml of distilled water and transfer the rinse to the ion exchange column reservoir. Discard the column effluents.

5. Wash each column with 3.0 ml of H_2O. Discard the wash.

6. Place clean, 2 oz. plastic bottles labeled "A" and "B" under the appropriate column. Add exactly 5.0 ml of 1M HCl to each column. Collect effluent in appropriate bottle.

7. Add exactly 5 ml of distilled water to each bottle. Mix. Analyze for Sr and Ca as described below under Flame Photometry.

C. Flame Photometry

Follow the practice described under sodium sulfate.

D. Calculation

Follow the practice described under sodium sulfate.

Control factor for Ca = 0.04
Control factor for Sr = 0.004

Procedure - Analysis of Hydroxy Acetic Acid

(0.0002% Ca, 2 ppm and 0.0001% Sr, 1 ppm)

A. Special Reagents

1. Standard 0.1 g/l calcium solution made by dissolving reagent calcium carbonate in approximately 1N HCl.
2. Standard 0.1 g/l strontium solution made by dissolving strontium chloride hexahydrate ($SrCl_2 \cdot 6H_2O$) in approximately 1 N HCl.

B. Preparation of Sample

1. Weigh an amount of the essential material hydroxy acetic acid sample to give 10 grams of the acid on an anhydrous basis. Transfer to a 25 ml volumetric flask or g.s. cylinder and label "A".

   Prepare a second portion in the same way and label "B".

2. To flask "B" add exactly 0.02 milligrams of calcium and 0.01 milligrams of strontium.

   (200 ul of 0.1 g/l calcium and 100 ul of 0.1 g/l strontium)

3. Make both flasks to volume with 1N HCl.

C. Flame Photometry

a. Calcium

1. Check instrument set-up with 0.008 mg/ml calcium at the 422.7 mu calcium emission line, and at 430 mu for background.
2. Set the emission of control solution "B" at 422.7 mu to 100% transmission.
3. Determine the emission of sample solution "A" at 422.7 mu and record the %T (transmission).
4. Repeat (2) and (3). The transmission values for "A" must agree within ± 1%. If they do not, repeat (2) and (3) again.
5. Determine the emission of sample solution "A" at 430 mu and record %T as background.

b. Strontium

1. Check the instrument with 0.004 mg/ml strontium at the 460.7 mu strontium line and at 457 mu for background.
2. Set the emission of control solution "B" at 460.7 mu to 100% transmission.
3. Determine the emission of sample solution "A" at 460.7 mu and record the %T (transmission).
4. Repeat (2) and (3). The transmission values for "A" must agree within ± 1%. If they do not, repeat (2) and (3) again.
5. Determine the emission of sample solution "A" at 457 mu and record as background.

D. Calculation

a. Calcium

1. The sample is within specifications if:

$$\%T_B - \%T_A > \%T_A - \%T_{bkgd}$$

2. Parts calcium per million parts of the essential material on a bone dry or 100% basis may be calculated.

$$\text{ppm Ca} = \frac{\text{control factor} \times \left(\dfrac{\%T_A - \%T_{bkgd}}{\%T_B - \%T_A}\right) \times 10^3}{\text{Sample Wt. in grams (100\% basis)}}$$

This calculation should not be used if either ($\%T_A - \%T_{bkgd}$) or ($\%T_B - \%T_A$) is less than 10.

Control factor = milligrams of calcium added to control "B" = 0.02 mg.

b. Strontium

As for calcium using control factor 0.01.

Procedure - Analysis of Sulfamic Acid

(Limits 0.004% Ca, 0.001% Sr, 40 and 10 ppm)

A. Special Reagents

(See under Hydroxy Acetic Acid)

B. Preparation of Sample

1. Prepare two samples as follows:

   Weigh a 2-gram sample and transfer to a 25 ml volumetric flask or g.s. cylinder. Add 15 ml of water and mix to effect solution. Label the flasks "A" and "B".

2. To flask "B" add 0.08 mg of calcium and 0.02 mg of strontium. (800 ul of 0.1 g/l Ca and 200 ul of 0.1 g/l Sr.)

3. Make both flasks to volume with water. Mix.

C. Flame Photometry

Follow procedure under Hydroxy Acetic Acid.

D. Calculations

Follow procedure under Hydroxy Acetic Acid.

1. Calcium Control Factor = 0.08

2. Strontium Control Factor = 0.02

Procedure - Analysis of 60% Nitric Acid for Calcium and Strontium

(Limits 0.00012% Ca and 0.000012% Sr amounting to 2 ppm Ca and 0.2 ppm Sr on a 100% basis.)

A. Special Reagents

See under Hydroxy Acetic Acid

B. Preparation of Sample

1. Prepare two samples as follows:

   Measure a 60 ml sample amounting to 50 grams of HNO_3 and transfer to a clean evaporating dish. Carefully evaporate to dryness. Do not bake.

   Using about 15 ml of 1N HCl dissolve the residue and transfer to a 25 ml volumetric flask or g.s. cylinder. Label the flasks "A" and "B".

2. To flask "B" add 0.1 mg of calcium and 0.01 mgm strontium (1000 ul of 0.1 g/l Ca and 100 ul of 0.1 g/l Sr).

3. Make both flasks to volume with 1N HCl. Mix.

C. Flame Photometry

Follow procedure under Hydroxy Acetic Acid.

D. Calculation

Follow procedure under Hydroxy Acetic Acid.

1. Calcium Control Factor = 0.1.

2. Strontium Control Factor = 0.01.

Procedure - Analysis of Sugar for Calcium and Strontium

(Limits 0.0016% Ca and 0.0003% Sr of 16 and 3 ppm)

A. Special Reagents

(See under Hydroxy Acetic Acid)

B. Preparation of Sample

1. Prepare two samples as follows:

   Weigh a 5 gram portion of the material and transfer to a 25 ml volumetric flask or g.s. cylinder. Dissolve in 15 ml of 1N HCl. Label the flasks "A" and "B".

2. To flask "B" add 0.08 mg of calcium and 0.015 mg of strontium. (800 ul of 0.1 g/l Ca and 150 ul of 0.1 g/l Sr.)

3. Make both flasks to volume with 1N HCl. Mix.

C. Flame Photometry

Follow procedure under Hydroxy Acetic Acid.

D. Calculation

Follow procedure under Hydroxy Acetic Acid.

1. Calcium Control Factor = 0.08
2. Strontium Control Factor = 0.015

Procedure - Analysis of Sodium Fluoride for Calcium and Strontium

(Limits 0.02 Wt.% Ca, 0.01 Wt.% Sr, 200 ppm and 100 ppm)

A. Special Reagents and Equipment

(As for Sodium Sulfate)

B. Preparation of Sample

1. Weigh two 0.500 gram samples and place in clean platinum dishes. The dishes should be marked distinctly to avoid error.
2. To one dish add 5 ml of water and 1.0 ml of concentrated sulfuric acid.
3. To the second dish add 0.10 milligram of calcium and 0.05 milligram of strontium (1000 ul of 0.1 g/l Ca and 500 ul of 0.1 g/l Sr). Add 5 ml of water and 1.0 ml of concentrated sulfuric acid.
4. Heat both dishes under a lamp to drive off HF. Heat until SO_3 fumes have nearly ceased.

C. Separation from Sodium

1. Add 10 ml of water to each dish to dissolve all solids. Warm if necessary.
2. Adjust the pH to 7.5 - 8.5 with 1.0M ammonia solution.
3. Transfer the contents of each dish to a Chelex-100 ion exchange column. Rinse each dish with 2.0 ml of distilled water and transfer the rinse to the ion exchange column reservoir. Discard the column effluents.
4. Wash each column with 3.0 ml of water. Discard the wash.
5. Place clean 2 oz. plastic bottles labeled "A" and "B" under the appropriate column. Add exactly 5.0 ml of 1M HCl to each column. Collect effluent in appropriate bottle.
6. Add exactly 5 ml of distilled water to each bottle, mix, and analyze for Sr and Ca by flame photometry.

D. Flame Photometry

(See under Sodium Sulfate)

E. Calculation

(See under Sodium Sulfate)

1. Control factor for calcium = 0.10
2. Control factor for strontium = 0.05

Procedure - Analysis of Hydrazine for Calcium and Strontium. (35% Sol'n)

(Limits 0.0007% Ca and 0.00007% Sr, 20 ppm and 2 ppm on 100% basis.)

A. Special Reagents

(See under Hydroxy Acetic Acid)

B. Preparation of Sample

1. Prepare two samples as follows:

   Weigh a 14.3 gram sample of the 35% solution representing 5 grams of hydrazine in a porcelain casserole.

   Evaporate under a lamp to practical dryness. Transfer the residue by means of four successive 5 ml portions of 1N HCl to a 25 ml volumetric flask. Label the flasks "A" and "B".

2. To flask "B" add 0.1 mg of calcium and 0.01 mg of strontium. (1000 ul of 0.1 g/l Ca and 100 ul 0.1 g/l Sr).

3. Make both flasks to volume with water. Mix.

C. Flame Photometry

Follow procedure under Hydroxy Acetic Acid.

D. Calculations

Follow procedure under Hydroxy Acetic Acid.

1. Calcium control factor = 0.1

2. Strontium control factor = 0.01

Procedure - Analysis of Lead Nitrate for Calcium and Strontium

(Limits 0.005% calcium and 0.006% strontium, 50 and 60 ppm)

A. Special Reagents

(See under Hydroxy Acetic Acid)

B. Preparation of Sample

1. Weigh a 0.40 gram sample and transfer to a 25 ml volumetric flask. Dissolve in 5 ml of water. Add 5 ml of concentrated hydrochloric acid. Make to volume and mix. When the precipitate has settled, filter into a plastic bottle labeled "A".

2. Weigh a second 0.40 gram sample and transfer to a 25 ml volumetric flask. Dissolve in 5 ml of water. Add 0.02 mg of Ca and 0.03 mg Sr to the flask. (200 ul of 0.1 g/l Ca and 300 ul of 0.1 g/l Sr) Add 5 ml of concentrated hydrochloric acid. Make to volume and mix. Filter into a plastic bottle labeled "B".

C. Flame Photometry

(See under Hydroxy Acetic Acid)

D. Calculation

(See under Hydroxy Acid)

1. Control factor for Ca = 0.02.

2. Control factor for Sr = 0.03.

Procedure - Analysis of Oxalic Acid for Calcium and Strontium

(Limits 0.007% Ca and 0.0005% Sr, 70 and 5 ppm.)

A. Special Reagents

(See under Hydroxy Acetic Acid)

B. Preparation of Sample

1. Weigh a 5.0 gram sample and place in a casserole (Size 3 or 3A). Add 25 ml of dilute (1 + 24) nitric acid and 15 ml of 30% H_2O_2. Cover with a watch glass and digest on a steam bath or low hot plate until reaction ceases. Add 5 ml more of the peroxide and when reaction ceases uncover and evaporate (without boiling) to dryness. Dissolve the residue in 50 ml of dilute (1 + 99) nitric acid. Label "A".

2. Weigh a second 5.0 gram sample and place in a casserole. Add 0.35 mg of calcium and 0.025 mg of Sr. (3.5 ml of 0.1 g/1 Ca and 250 ul of 0.1 g/1 Sr.) Then treat as above. Label "B".

C. Flame Photometry

Follow procedure under Hydroxy Acetic Acid.

D. Calculation

Follow procedure under Hydroxy Acetic Acid.

1. Control factor for Ca = 0.35

2. Control factor for Sr = 0.025

Procedure - Analysis of Ferrous Sulfamate Solution (50%) for Calcium and Strontium

(Limits 0.004% Ca and 0.0005% Sr, 80 and 10 ppm)

A. Special Reagents and Equipment

1. Dowex 1 x 4, 50-100 mesh, in the chloride form is prepared by wetting in 6M HCl.
2. 8M hydrochloric acid.
3. 30% hydrogen peroxide.
4. 0.1 g/l Ca standard and 0.05 g/l Sr standard.
5. Ion exchange columns 4" long, 10 mm O.D., glass tubing with a 15 ml reservoir on top and a stopcock with buret tip on the bottom.

B. Separation from Iron

1. Prepare two ion exchange columns, each containing 4 ml of Dowex 1 x 4 in the chloride form. After preparation, wash each column with 5 ml of 8M HCl and discard the wash. Label the columns "A" and "B".
2. Label two 15 ml vials "A" and "B". Place a glass stir bar in each. Add reagents in the order noted below.

Reagent	In Vial A	In Vial B
H_2O	2000 ul	1000 ul
0.1 g/l Ca Std	None	300
0.05 g/l Sr Std	None	75
Ferrous Sulfamate 3.25 molar (50%) (Sp G 1.5)	500 ul (0.75 g)	500 ul
Stir at this point and then add		
Conc. HCl (12M)	5000 ul	5000 ul
30% H_2O_2	250 ul	250 ul

3. Stir the prepared solution 45 minutes to complete the oxidation and degas.
4. Transfer the contents of the vials to appropriate columns. Catch the effluent in 25 ml g.s. graduated cylinders. Flow rate should be less than 0.5 ml per minute.
5. Rinse the vials with 5 ml of 8M HCl and transfer the rinse to the columns. Accumulate the rinse in the graduated cylinders also.

6. Adjust the volumes in each graduate to 13.2 ml with water.

C. Flame Photometry

Follow the procedure under Hydroxy Acetic Acid.

D. Calculation

Follow the procedure under Hydroxy Acetic Acid.

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Procedure - Analysis of 72% Aluminum Nitrate for Calcium and Strontium

(Limits 0.0072% Ca, 0.0014% Sr, 100 ppm and 20 ppm)

A. Special Reagents

1. Standard 0.1 g/l calcium solution made by dissolving reagent calcium carbonate in approx. 1 N HCl.
2. Standard 0.1 g/l strontium solution made by dissolving strontium chloride hexahydrate ($SrCl_2.6H_2O$) in approx. 1 N HCl.
3. Solid ammonium chloride.
4. 10% ammonia solution (2 + 3).
5. Methyl red indicator solution.

B. Preparation of Sample

1. Preferably using a dry filmed micropipet, transfer 4.0 ml of sample to each of two 50 ml volumetric flasks. Label one flask A and the other B.
2. To both flasks add 10 ml H_2O and 1 g of NH_4Cl. Mix.
3. To flask B add exactly (dry filmed micropipet) 4 ml of 0.1 g/l calcium solution and 0.8 ml of 0.1 g/l strontium solution. Mix.
4. To both flasks add two drops of methyl red indicator. Mix. Add from a pipet 10% NH_3 solution (12-13 ml) to a one-drop excess. (Readjust with HCl and NH_3 solution, if necessary.)
5. Make the solution in the volumetric flasks to volume and mix thoroughly.

Filter each by pouring the solution on a dry filter paper and gather the filtrates in plastic bottles labeled A and B.

Flame Photometry

Proceed as under Hydroxy Acetic Acid - Zy-25-8.

1. Control factor for calcium = 0.4.
2. Control factor for strontium = 0.08.

ESTIMATION OF METHYL ISOBUTYL CARBINOL

Application and Principle

When methyl isobutyl carbinol (MIBC) is reacted with an acid dichromate solution, Cr(III) is the reduction product. The optical density of the Cr(III) may be measured with a spectrophotometer at 580 mu. Unreacted dichromate reagent is used as a blank to minimize the very slight interference which Cr(VI) causes at this wave length. With concentrations of methyl isobutyl carbinol of 0.1 volume per cent or less, there is a linear relationship between its concentration and the optical density of the Cr(III) formed. Higher concentrations can be diluted to this range with standard, low impurity, hexone. The reaction rate depends on the temperature which should be controlled to 25 ± 2 C. Under these conditions, precision of the determination is ± 0.01 volume per cent at the 99% confidence level. The lower limit for the method is 0.01 volume per cent.

Procedure

A. MIBC in Pretreated Hexone

1. Pipet 5.0 ml of dichromate reagent into separatory funnel, followed by 22.0 ml hexone sample.
2. Immediately lower stirrer so that the bottom of the stirrer is approximately 1/2 inch below the aqueous-hexone interface and stir for exactly 10 minutes (Remark 1).
3. Allow 10 seconds for separation of phases and transfer a portion of the aqueous phase to a 1.0 cm Beckman cell (Remark 2).
4. Read the optical density immediately (Remark 3) at 580 mu, 0.04 mm slit width against unreacted dichromate as a blank. Use step 1 of Calculations to find volume per cent MIBC in samples.

B. MIBC in Sample Containing More than 0.1 volume per cent MIBC

1. Pipet 5.0 ml of hexone sample into a 50.0 ml volumetric flask and fill to the mark with standard hexone.
2. Pipet 5.0 ml of dichromate reagent into funnel, followed by a 20 ml aliquot of the diluted hexone sample and proceed as in Part A. Calculate by step 2 in Calculations.

Calculations

1. Volume per cent MIBC = (optical density reading - .014)x .473 (Remark 4).

2. Volume per cent MIBC in samples diluted with standard hexone = 10 (volume per cent MIBC in diluted sample)- .9 (volume per cent MIBC in standard hexone).

3. Weight per cent MIBC = volume per cent x 1.015

Remarks

1. The reaction of methyl isobutyl carbinol with dichromate reagent is not complete after 10 minutes. It is, therefore, necessary that the stirring time be exactly 10 minutes to obtain good precision.

2. Do not fill cells any more than necessary to cover the windows. If hexone, which is inadvertently drawn off with the aqueous phase, is spilled, it tends to "creep" along the outside of a Beckman cell and may coat the outside of the windows.

3. Since a small amount of hexone is still in contact with the aqueous phase, the reaction continues after separation. The reading of optical density should be completed within 2 minutes after separation. If the value changes rapidly, the emulsion formed during stirring has broken up forming bubbles of hexone which decrease light transmission. Pour sample into small beaker and then back into cell and measure optical density again.

4. The value 0.014 optical density units is subtracted from the optical density reading obtained for the sample to correct for the reaction of hexone with the dichromate reagent. The value 0.473 is the volume per cent MIBC increment per optical density unit.

Reference: HW-24403 (6-20-52)

DETERMINATION OF IMPURITIES

Spectrographic Method

Principle

Analysis is accomplished by exciting the sample in a direct current arc, evaluating the intensity of the impurity emission lines, and comparing the latter with those of standard samples.

Apparatus

Emission spectrograph, source, and densitometer.

Procedure - Analysis of Calcium

To one g. of sample, add dropwise slightly more water than required to effect conversion to calcium hydroxide. Evaporate the excess water and ignite at 750° C for one hour. Grind the resulting oxide in a mortar. Mix a portion of the ground material with 5% of pure gallium oxide.

For the determination of B, Be, and Cu, pellet 25 mg. of the gallium oxide mixture and place the pellet in a crater electrode. For the determination of Li, Na, Mg, Al, Si, Mn, Cr, Co, Ni, and Fe, use 10 mg. of the mixture.

Excite the sample at 12 amp. (short circuit) with the direct current arc. Compare the resultant spectra with those produced with pure matrix material to which known amounts of impurities (as solutions) have been added.

Procedure - Analysis of Magnesium Oxide

To convert the fused sample to a soft form, dissolve one g. in silica-distilled nitric acid, dry, and ignite at 750° C. Grind the resulting oxide in a mortar.

Prepare mixtures indicated below for the required determinations:

Element Line	Sensitivity, ppm	Sample mixture and weight
Al 3082, 3093	1	1:20:20 Ga_2O_3, MgO, graphite powder; 20 mg.
B 2497	1	MgO; 10 mg.
Be 3131	1	MgO; 10 mg.
K 7665	10	1:20 Ga_2O_3, MgO; 10 mg.
Li 6707	1	1:20 Ga_2O_3, MgO; 10 mg.
Na 5890, 5896	1	1:20 Ga_2O_3, MgO; 10 mg.

Excite the sample at 12 amp. (short circuit) with the direct current arc. Compare the resultant spectra with those produced with pure matrix material to which known amounts of impurities (as solutions) have been added.

Remarks

Element Lines -- Li 6104, 6708

Sensitivity -- 50 ppm (0.005%).

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3. Letter A. J. Zeitz to E. P. Galbraith, Aug. 6, 1953, entitled, "Determination of Lithium in Flux Consisting of Barium Chloride, Potassium Chloride, and Sodium Chloride."

Written by: L. F. Kendall (September, 1955)