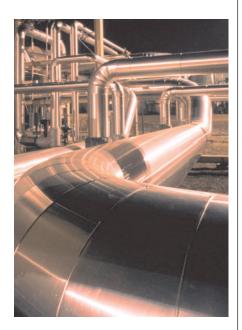


A Dulany Industries Company





Chemical **Safety** Handbook





CONTENTS

Introduction
Nomenclature
Unit Diagram-typical Sulfuric Acid Plant
Sulfuric Acid . <
Methods of analysis of Sulfuric Acid and Oleum
Safety, Health, and Environmental Information
Unloading Sulfuric Acid
Materials of Construction

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INTRODUCTION

This handbook is offered as a service to users of sulfuric acid. It provides, in easy-to-follow form, comprehensive data on the properties and handling of this important chemical. Its uses as a raw material or a processing agent are so numerous today in all branches of industry that it has been called "the universal chemical" and "the chemical that makes chemicals."

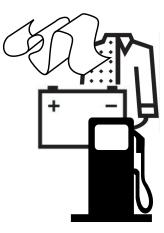
Our broad experience in sulfuric acid technology and our years of assistance to diversified customers have guided the selection and presentation of the information included in these pages. Sulfuric acid is one of the most widely used industrial chemicals. Its broad usage is due to several important factors:

- 1. Low cost
- 2. Low volatility
- 3. Ability to act as a strong oxidizing agent
- 4. Ability to act as a strong dehydrating agent
- 5. Strong acidic properties

Here are some of the growing number of end-uses and applications using sulfuric acid:

Agricultural chemicals	Lubricating oils
Aluminum sulfate	Medicinal processes
Batteries	Nitric acid concentration
Carbolic acid	Oil additives
Cellophane	Paper
Chlorine drying	Qualitative and quantitative analysis
Detergents	Rayon and rubber
Ethanol	Sugar
Explosives	Sulfonated hydrocarbons
Fertilizers	Synthetic fibers
Gasoline	Textiles
Herbicides	Titanium dioxide production
Hydrochloric acid	Uranium processing
Hydrofluoric acid	Veterinary drugs
Iron and steel pickling	Water softener regeneration
Jet fuel	Water treatment
Kerosene	Yellow pigments
Leather	







NOMENCLATURE

SULFURIC ACID

The term sulfuric acid covers both H₂SO₄ and water solutions of this compound.

For many applications, users of sulfuric acid consider the strength, or percent sulfuric acid, to be a very important quality parameter.

Specific gravity is most commonly determined by a hydrometer graduated in degrees Baumè (or Bè), an arbitrary scale.

The approximate strength of most sulfuric acid solutions is readily estimated by the use of a hydrometer. Two basic types of hydrometers, each with a different scale, are traditionally used in the sulfuric acid and related industries.

Specific Gravity Hydrometer – measures the ratio of the weight of a volume of sulfuric acid at a defined temperature to an equal volume of water at the defined temperature. Hydrometers standardized at a temperature of $60^{\circ}/60^{\circ}$ F are most commonly used for specific gravity measurements of sulfuric acid. The important use of specific gravity measurements in industrial control has caused the development of special scales. Sulfuric acid strength is often measured using one of these.

Baume Hydrometer – There are two Baumè hydrometer scales; one for use with liquids heavier than water, the other for liquids lighter than water. As originally established, the specific gravity of a 15% by weight aqueous salt (sodium chloride) solution at 15° C was designated as 15° Baumè (Bè) and pure water was designated at 0° Bè. These points were marked on a hydrometer stem and made the basis of a linear scale. For liquids lighter than water, a 16% solution of salt fixed the zero point and pure water was designated at 10° Bè; linear extrapolation was made from these points.

Sulfuric acid solutions are heavier than water, therefore, a "heavy" Baumè hydrometer is used.

The Baumè gravity of an acid sample can be calculated if the specific gravity $@~60^{\circ}$ F is known. For example, a sulfuric acid sample with a specific gravity of 1.8354 has a Baumè gravity of 66.0°.

The relationship between degrees Bè and specific gravity for sulfuric acid, at $60^{\circ}/60^{\circ}$ F is given by the following formula:

$$^{2}\text{Be} = 145 - \frac{145}{Specific Gravity}$$

With this formula, it is possible to construct a table giving the percentage composition of sulfuric acid solutions for various Baumè readings.

Calculations: $^{\circ}Be \ gravity @ \ 60^{\circ}F = 145 - \frac{145}{Specific \ Gravity}$ $Be \ gravity @ \ 60^{\circ}F = 145 - \frac{145}{1.8354}$ $Be \ gravity @ \ 60^{\circ}F = 145 - 79.0$ $Be \ gravity @ \ 60^{\circ}F = 66.0$







Specific gravity of a sulfuric acid solution can be calculated if the degrees baumè gravity (@ 60° F is known. For example, an acid solution with a Baumè gravity of 65.5 degrees has a specific gravity (@ 60°/60° F of 1.8239

Calculations:

$$Sp. Gr. @ 60^{\circ}/60^{\circ}F = \frac{145}{145 - {}^{\circ}\text{Be} @ 60^{\circ}\text{F}}$$

$$Sp. Gr. @ 60^{\circ}/60^{\circ}F = \frac{145}{145 - 65.5^{\circ}^{\circ}\text{Be}}$$

$$Sp. Gr. @ 60^{\circ}/60^{\circ}F = \frac{145}{79.5}$$

$$St. Gr. @ 60^{\circ}/60^{\circ}F = 1.8239$$

Sulfuric Acid solutions more concentrated than about 93.19 percent H_2SO_4 (66° Bè) change density very little with an increase in concentration. Therefore solution strength is usually defined in terms of percent H_2SO_4 rather than density or degrees Baumè.

OLEUM

Sulfur trioxide dissolved in 100 percent H_2SO_4 is known as oleum or fuming sulfuric acid. Usually the strength of oleum is expressed in terms of percent by weight of free sulfur trioxide. Strength is also expressed as percent equivalent H_2SO_4 .

VIRGIN ACID

Virgin acid is sulfuric acid that has never been used in a process. It is commonly called commercial grade sulfuric acid. Two grades of virgin or commercial grade acid are industrially important.

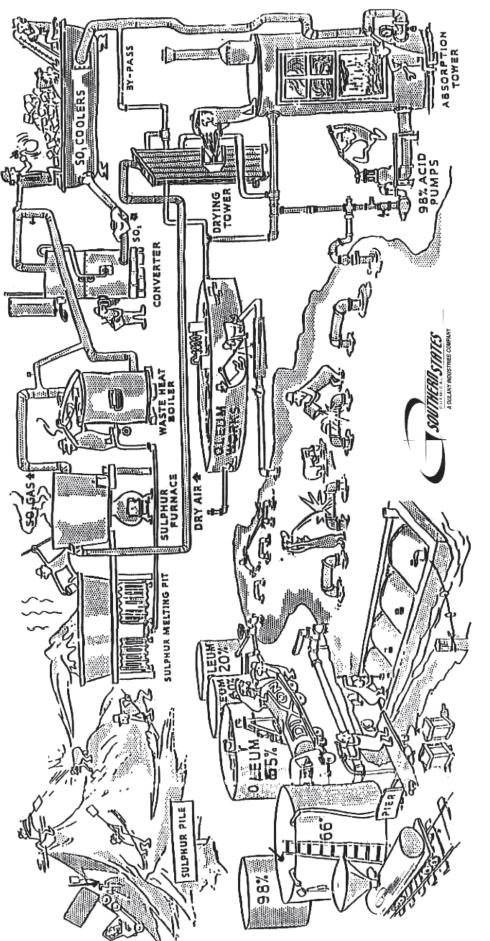
They are:

66° Bè acid (93.19% H₂SO₄) 98-99% H₂SO₄

The 98-99% sulfuric acid has a high freezing point. For this reason, it is usually shipped in insulated containers equipped with steam coils. The 66° Bè sulfuric acid has a freezing point far below zero and is sometimes called "winter acid."

SPENT ACID

Sulfuric acid that has been used in a process is called "spent acid." This term is misleading because spent acid may retain full acid value. However, spent acid may contain impurities that vary widely in nature and content, depending on the generator of the spent acid.





THE PRODUCTION PROCESS

UNIT DIAGRAM – TYPICAL ACID PLANT



SULFURIC ACID

PHYSICAL PROPERTIES OF SULFURIC ACID

INDEX

Typical characteristics and properties of sulfuric acid	5
	5
Viscosity of sulfuric acid solutions	6
Boiling point/freezing point of sulfuric acid solutions	7
Total vapor pressure of sulfuric acid solutions and oleum	7
Heat of infinite dilution of sulfuric acid	8
Specific heat of sulfuric acid solutions	9
Enthalpy of sulfuric acid solutions	9



TYPICAL CHARACTERISITCS & PROPERTIES OF SULFURIC ACID

Physical state	:	Liquid
Color	:	Clear, colorless to cloudy
Flash point	:	None
Corrosivity	•	Highly corrosive to most metals; particu- larly at concentrations below 60° Bè with evolution of hydrogen gas.
Reactivity	:	In addition to attacking many metals, the acid in its concentrated form is a strong oxidizing agent and may cause ignition on contact with organic materi- als and such material as nitrates, car- bides, chlorates, etc. It also reacts exothermally with water.



SULFURIC ACID

Sulfuric Acid			Reference
Degrees Baumè	66	66.3	
% H ₂ SO ₄	93.19	99.00*	8, 10
Specific Gravity @ 60°/60° F	1.8354	1.8424	8
Freezing Point ℃ °F	-32 -26	7 44	9
(Approximate) Boiling Point °F (1 Atmos.)	529	589	1, 2, 3, 4, 5
Total Vapor Pressure @ 250°F mm Hg.	1.7	1	6
Viscosity in Centipoise 70° F 110° F 150° F	22.2 11.3 7.1	26.5 12.8 7.8	2, 6, 7
Density, pounds per gallon (60° F)	15.286	15.363	в
Specific Heat, BTU/(pound) (°F) @ 68°F	0.38	0.34	6, 9

REFERENCES

1. R. H. Perry and C. H. Chilton. "Chemical Engineers Handbook," 5th Ed. McGraw-Hill (1973).

 E. W. Washburn, et al "International Critical Tables of Numerical Daya, Physics, Chemistry and Technology," Vol. 3., McGraw-Hill (1928).
 A. M. Fairle. "Sulfuric Acid Manufacture" Reinhold Publishing Corp. (1936).

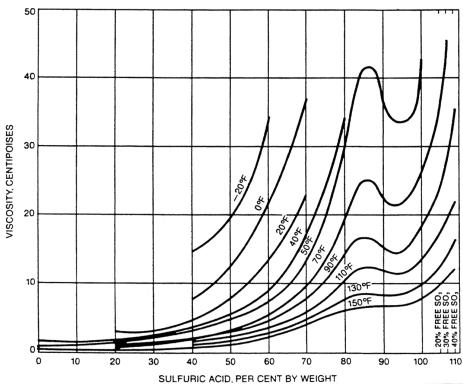
4. W. W. Duecker and J. R. West "The Manufacture of Sulfuric Acid, "American Chemical Society Monograph Series, Facsimile of 1959 Edition, Robert E. Kringer Publishing Co., Inc. Huntington, NY (1977).

 S. "Sulfur Manual, "Texas Gulf Sulfur (1959).
 T. R. Bump and W. L. Sibbitt. "Industrial and Engineering Chemistry, " 47,1665 (1956).
 N. F. H. Bright H. Hutchinson and D. Smith "Journal of the Society of Chemical Industry" 65, 385 (1946).

 "Chemical Plant Control Data Handbook,"
 9th Ed., Chemical Construction Corp. (1969).
 A. Stauden, Editor, "Kirk-Othmer Encyclopedia of Chemical Technology," 2nd Ed., Vol. 19 Interscience Publishers (1969).

10. F. D. Snell and L. S Ettre. "Encyclopedia of Industrial Chemical Analysis," 1B, 415, Interscience Publishers (1973).

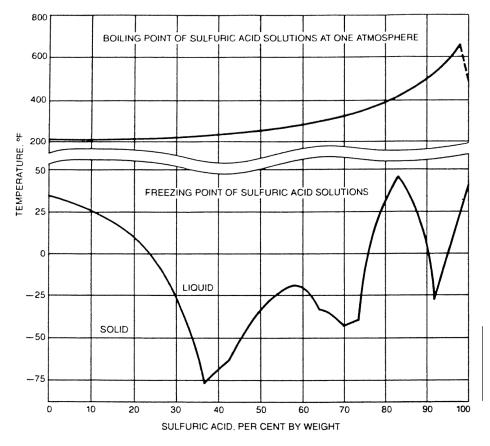
* THIS AND OTHER STRENGTHS AVAILABLE UPON REQUEST.



VISCOSTIY OF SULFURIC ACID SOLUTIONS AT VARIUOS TEMPERATURES

DATA OBTAINED FROM N.F.H. BRIGHT, H. HUTCHINSON AND D. SMITH, "THE VISCOS-ITY AND DENSITY OF SULFURIC ACID AND OLEUM," JOURNAL OF THE SOCIETY OF CHEMICAL INDUSTRY, DECEMBER, 1946, PP. 385-388.

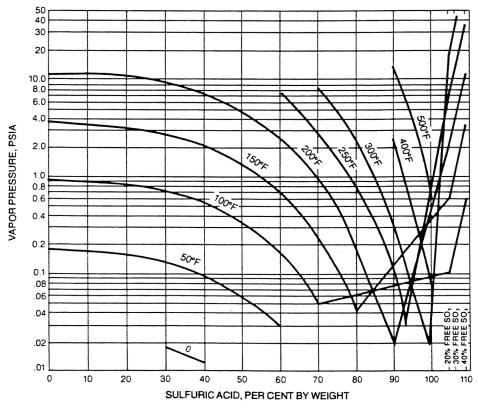




BOILING POINT/FREEZING POINT OF SULFURIC ACID SOLUTIONS

C.M. GABLE, H.F. BETZ AND S.H. MARON, "PHASE EQUILIBRIA OF THE SYSTEM SULFUR-TRIOXIDE—WATER." JOURNAL OF THE AMERI-CAN CHEMICAL SOCIETY, 72 (1950), PP. 1445-1448.

TOTAL VAPOR PRESSURE OF SULFURIC ACID SOLUTIONS AND OLEUM



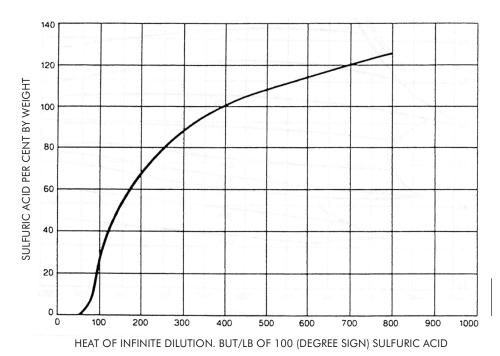
T.T. BUMP AND W.L. SIBBITT, •AQUEOUS SOLUTIONS OF NITRIC ACID AND OF SUL-FURIC ACID," INDUSTRIAL AND ENGINEERING CHEMISTRY, 47 (1955), PP. 1665-1670.



HEAT OF INFINITE DILUTION OF SULFURIC ACID

Determining Heat Evolved During Dilution of Sulfuric Acid and Oleum

This chart, showing the heat of infinite dilution for all concentrations of sulfuric acid, can be used to determine the heat evolved in diluting one strength of acid to another.



BICHOWSKY AND ROSSINI, "THERMOCHEM-ISTRY OF CHEMICAL SUBSTANCES, " (1963).

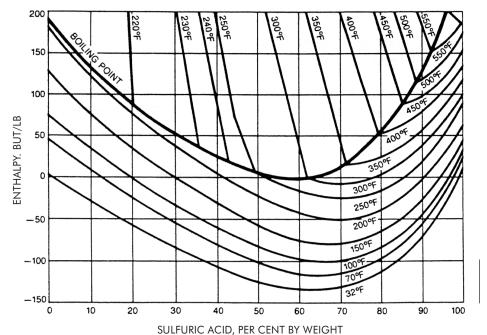
The following is an example of the use of this chart:

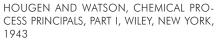
PROBLEM – Determine the heat evolved when 100 pounds of 98 percent Sulfuric Acid (98 pounds of 100 percent H_2SO_4 Basis) is diluted to 77 percent acid.

SOLUTION – Read carefully from the chart the heat of infinite dilution for 98 percent acid (390 BTU/lb. of 100 percent acid) and for 77 percent acid (245 BTU/lb of 100 percent acid). The difference (145 BTU/lb of 100 percent acid) is then multiplied by the weight of 100 percent sulfuric acid being diluted.

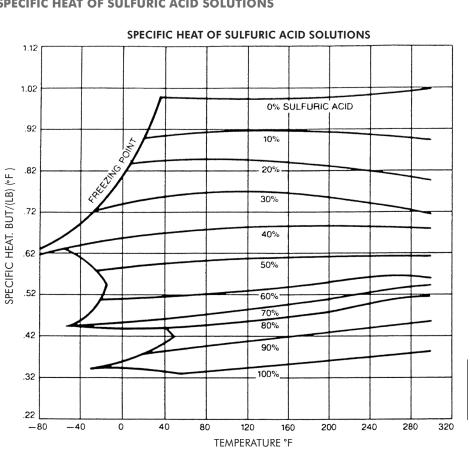
The heat of dilution for the problem given is 14,210 BTU.







ENTHALAPY OF SULFURIC ACID SOLUTIONS



T.T. BUMP AND W.L. SIBBITT, •AQUEOUS SOLUTIONS OF NITRIC ACID AND OF SUL-FURIC ACID," INDUSTRIAL AND ENGINEERING CHEMISTRY, 47 (1955), PP. 1665-1670.

SPECIFIC HEAT OF SULFURIC ACID SOLUTIONS





Density-Strength Conversion Table for Sulfuric Acid Solutions at 60° F

When density has been determined at temperatures other than 60° F, a temperature correction must be made. The correction procedure and necessary correction factors are given starting on page 20.

Degrees Baumé	Specific Gravity At 60°/60° F	Density In Pounds per Gallon	Per Cent H ₂ SO ₄	Per Cent 66° Bé Acid	Degrees Baumé	Specific Gravity at 60°/60° F	Density In Pounds Per Gallon	Percent H ₂ SO ₄	Per Cent 66° Bé Acid
0.0	1.0000	8.338	0.00	0.00	23.5	1.1934	9.939	26.42	28.34
1.0	1.0069	8.395	1.02	1.09	23.6	1.1944	9.947	26.54	28.48
2.0	1.0140	8.455	2.08	2.23	23.7	1.1954	9.955	26.66	28.61
3.0	1.0211	8.515	3.13	3.36	23.8	1.1964	9.963	26.79	28.74
4.0	1.0284	8.575	4.21	4.52	23.9	1.1974	9.972	26.91	28.86
5.0	1.0357	8.636	5.28	5.67	24.0	1.1983	9.980	27.03	29.00
6.0	1.0432	8.697	6.37	6.84	24.0	1.1983	9.988	27.05	29.00
7.0	1.0507	8.734	7.45	7.99	24.2	1.2003	9.996	27.28	29.15
8.0	1.0584	8.825	8.55	9.17	24.2	1.2003	10.005	27.40	29.40
9.0	1.0662	8.890	9.66	10.37	24.4	1.2023	10.013	27.53	29.54
10.0	1.0741	8.956	10.77	11.56	24.5	1.2033	10.021	27.65	29.67
11.0	1.0821	9.023	11.89	12.76	24.6	1.2043	10.030	27.78	29.80
12.0	1.0902	9.091	13.01	13.96	24.7	1.2053	10.038	27.90	29.94
13.0	1.0985	9.159	14.13	15.16	24.8	1.2063	10.046	28.03	30.07
14.0	1.1069	9.230	15.25	16.36	24.9	1.2073	10.055	28.15	30.21
15.0	1.1154	9.300	16.38	17.58	25.0	1.2083	10.063	28.28	30.34
16.0	1.1240	9.372	17.53	18.81	25.1	1.2093	10.072	28.40	30.47
17.0	1.1328	9.445	18.71	20.08	25.2	1.2104	10.080	28.53	30.61
18.0	1.1417	9.520	19.89	21.34	25.3	1.2114	10.089	28.65	30.74
19.0	1.1508	9.596	21.07	22.61	25.4	1.2124	10.097	28.78	30.88
20.0	1.1600	9.661	22.25	23.87	25.5	1.2134	10.105	28.90	31.01
20.1	1.1609	9.669	22.37	24.00	25.6	1.2144	10.114	29.03	31.15
20.2	1.1619	9.676	22.49	24.12	25.7	1.2154	10.122	29.15	31.28
20.3	1.1628	9.684	22.60	24.25	25.8	1.2164	10.131	29.28	31.41
20.4	1.1637	9.692	22.72	24.38	25.9	1.2175	10.139	29.40	31.55
20.5	1.1647	9.700	22.84	24.50	26.0	1.2185	10.148	29.53	31.69
20.6	1.1656	9.708	22.96	24.63	26.1	1.2195	10.157	29.66	31.82
20.7	1.1665	9.716	23.08	24.76	26.2	1.2205	10.165	29.78	31.96
20.8	1.1675	9.723	23.19	24.89	26.3	1.2216	10.174	29.91	32.09
20.9	1.1684	9.731	23.31	25.01	26.4	1.2226	10.182	30.03	32.23
21.0	1.1694	9.739	23.43	25.14	26.5	1.2236	10.191	30.16	32.36
21.1	1.1703	9.747	23.55	25.27	26.6	1.2247	10.199	30.29	32.50
21.2	1.1712	9.755	23.67	25.39	26.7	1.2257	10.208	30.41	32.63
21.3	1.1722	9.763	23.78	25.52	26.8	1.2267	10.217	30.54	32.77
21.4	1.1731	9.771	23.90	25.65	26.9	1.2278	10.225	30.66	32.90
21.5	1.1741	9.779	24.02	25.77	27.0	1.2288	10.234	30.79	33.04
21.6	1.1750	9.787	24.14	25.90	27.1	1.2299	10.242	30.92	33.17
21.7	1.1760	9.794	24.26	26.03	27.2	1.2309	10.251	31.04	33.31
21.8	1.1769	9.802	24.37	26.16	27.3	1.2319	10.260	31.17	33.44
21.9	1.1779	9.810	24.49	26.28	27.4	1.2330	10.269	31.29	33.58
22.0	1.1789	9.818	24.61	26.41	27.5	1.2340	10.277	31.42	33.71
22.1	1.1798	9.826	24.73	26.54	27.6	1.2351	10.286	31.55	33.85
22.2	1.1808	9.834	24.85	26.67	27.7	1.2361	10.295	31.67	33.98
22.3	1.1817	9.842	24.97	26.79	27.8	1.2372	10.304	31.80	34.12
22.4	1.1827	9.850	25.09	26.92	27.9	1.2383	10.312	31.92	34.25
22.5	1.1836	9.858	25.21	27.05	28.0	1.2393	10.321	32.05	34.39
22.6	1.1846	9.866	25.33	27.18	28.1	1.2404	10.330	32.18	34.53
22.7	1.1856	9.874	25.45	27.31	28.2	1.2414	10.339	32.31	34.66
22.8	1.1866	9.882	25.57	27.43	28.3	1.2425	10.348	32.43	34.80
22.9	1.1876	9.890	25.69	27.56	28.4	1.2436	10.357	32.56	34.94



Degrees Baumé	Specific Gravity At 60°/60° F	Density In Pounds per Gallon	Per Cent H ₂ SO ₄	Per Cent 66° Bé Acid	Degrees Baumé	Specific Gravity at 60°/60° F	Density In Pounds Per Gallon	Percent H ₂ SO ₄	Per Cent 66° Bé Acid
23.0	1.1885	9.898	25.81	27.69	28.5	1.2446	10.366	32.69	35.07
23.1	1.1895	9.906	25.93	27.82	28.6	1.2440	10.375	32.82	35.21
23.2	1.1905	9.914	26.05	27.95	28.7	1.2468	10.384	32.95	35.35
23.3	1.1915	9.923	26.18	28.08	28.8	1.2408	10.392	33.07	35.49
23.4	1.1924	9.931	26.30	28.21	28.9	1.2489	10.401	33.20	35.62
29.0	1.2500	10.410	33.33	35.76	35.5	1.3242	11.029	41.95	45.01
29.1	1.2511	10.419	33.46	35.90	35.6	1.3254	11.039	42.09	45.16
29.2	1.2522	10.428	33.59	36.04	35.7	1.3266	11.049	42.22	45.30
29.3	1.2532	10.437	33.72	36.18	35.8	1.3278	11.059	42.36	45.45
29.4 29.5	1.2543 1.2554	10.447 10.456	33.85 33.98	36.32 36.46	35.9 36.0	1.3291 1.3303	11.069 11.079	42.49 42.63	45.59 45.74
29.6	1.2565	10.465	34.11	36.60	36.1	1.3315	11.089	42.77	45.89
29.7	1.2576	10.474	34.24	36.74	36.2	1.3327	11.100	42.90	46.03
29.8 29.9	1.2587 1.2598	10.483	34.37	36.88 37.02	36.3 36.4	1.3339	11.110	43.04	46.18 46.32
29.9	1.2398	10.492	34.50	57.02	30.4	1.3352	11.120	43.17	40.32
30.0	1.2609	10.501	34.63	37.16	36.5	1.3364	11.130	43.31	46.47
30.1	1.2620	10.510	34.76	37.30	36.6	1.3376	11.140	43.45	46.62
30.2	1.2631	10.519	34.89	37.44	36.7	1.3389	11.151	43.58	46.76
30.3	1.2642	10.529	35.02	37.58	36.8	1.3401	11.161	43.72	46.91
30.4	1.2653	10.538	35.15	37.72	36.9	1.3414	11.171	43.85	47.05
30.5	1.2664	10.547	35.28	37.85	37.0	1.3426	11.181	43.99	47.20
30.6	1.2675	10.556	35.41	37.99	37.1	1.3438	11.192	44.13	47.35
30.7	1.2686	10.565	35.54	38.13	37.2	1.3451	11.202	44.26	47.49
30.8	1.2697	10.574	35.67	38.27	37.3	1.3463	11.213	44.40	47.64
30.9	1.2708	10.583	35.80	38.41	37.4	1.3476	11.223	44.53	47.78
31.0	1.2719	10.593	35.93	38.55	37.5	1.3488	11.233	44.67	47.93
31.1	1.2730	10.602	36.06	38.69	37.6	1.3501	11.244	44.81	48.08
31.2	1.2742	10.611	36.20	38.84	37.7	1.3514	11.254	44.94	48.22
31.3	1.2753	10.621	36.33	38.98	37.8	1.3526	11.264	45.08	48.37
31.4	1.2764	10.630	36.46	39.12	37.9	1.3539	11.275	45.21	48.51
31.5	1.2775	10.640	36.59	39.26	38.0	1.3551	11.286	45.35	48.66
31.6	1.2787	10.649	36.73	39.41	38.1	1.3564	11.296	45.49	48.81
31.7	1.2798	10.659	36.86	39.55	38.2	1.3577	11.307	45.62	48.95
31.8	1.2809	10.668	36.99	39.69	38.3	1.3590	11.318	45.76	49.10
31.9	1.2821	10.677	37.13	39.84	38.4	1.3602	11.328	45.90	49.25
22.0	1 2922	10 (97	27.0(20.09	20 E	1 2/15	11 220	46.02	40.20
32.0 32.1	1.2832 1.2843	10.687 10.696	37.26 37.39	39.98 40.12	38.5 38.6	1.3615 1.3628	11.339 11.350	46.03 46.17	49.39 49.54
32.2	1.2855	10.706	37.52	40.26	38.7	1.3641	11.360	46.31	49.69
32.3	1.2866	10.715	37.66	40.41	38.8	1.3653	11.371	46.45	49.84
32.4	1.2877	10.725	37.79	40.55	38.9	1.3666	11.381	46.58	49.98
32.5	1.2889	10.734	37.92	40.69	39.0	1.3679	11.392	46.72	50.13
32.5 32.6	1.2889	10.734	37.92	40.83	39.0 39.1	1.3692	11.392	46.72	50.13 50.28
32.0	1.2900	10.753	38.18	40.97	39.2	1.3705	11.405	40.00	50.28 50.43
32.8	1.2923	10.763	38.32	41.12	39.3	1.3718	11.425	47.13	50.57
32.9	1.2935	10.772	38.45	41.26	39.4	1.3731	11.436	47.27	50.72
33.0	1.2946	10.782	38.58	41.40	39.5	1.3744	11.447	47.41	50.87
33.1	1.2958	10.791	38.71	41.54	39.6 30.7	1.3757	11.458	47.55	51.02
33.2 33.3	1.2970	10.801	38.85	41.69	39.7 30.8	1.3770	11.469	47.69	51.17 51.31
33.3 33.4	1.2981 1.2993	10.811 10.821	38.98 39.12	41.83 41.97	39.8 39.9	1.3783 1.3796	11.479 11.49	47.82 47.96	51.31 51.46
	1.4773	10.021	37.14	71.77	57.7	1.5770	11.77	τ <i>ι.</i> 90	51.40
33.5	1.3004	10.830	39.25	42.11	40.0	1.3810	11.501	48.10	51.61
33.6	1.3016	10.840	39.38	42.26	40.1	1.3823	11.512	48.24	51.76
33.7	1.3028	10.850	39.52	42.40	40.2	1.3836	11.523	48.37	51.90
33.8	1.3040	10.860	39.65	42.54	40.3	1.3849	11.534	48.51	52.05
33.9	1.3051	10.869	39.79	42.69	40.4	1.3862	11.545	48.65	52.20



Degrees Baumé	Specific Gravity at 60°/60° F	Density In Pounds per Gallon	Per Cent H ₂ SO ₄	Per Cent 66° Bé Acid	Degrees Baumé	Specific Gravity at 60°/60° F	Density In Pounds Per Gallon	Percent H2SO4	Per Cent 66° Bé Acid
34.0	1.3063	10.879	39.92	42.83	40.5	1.3876	11.556	48.78	52.34
34.1	1.3075	10.889	40.05	42.97	40.6	1.3889	11.567	48.92	52.49
34.2	1.3087	10.899	40.19	43.12	40.7	1.3902	11.578	49.06	52.64
34.3	1.3098		40.32	43.26	40.8	1.3916	11.589	49.20	52.79
34.4	1.3110	10.919	40.46	43.41	40.9	1.3929	11.600	49.33	52.93
34.5	1.3122	10.929	40.59	43.55	41.0	1.3942	11.611	49.47	53.08
34.6	1.3134	10.939	40.73	43.70	41.1	1.3956	11.623	49.61	53.23
34.7	1.3146	10.949	40.86	43.84	41.2	1.3969	11.634	49.75	53.38
34.8	1.3158	10.958	41.00	43.99	41.3	1.3983	11.645	49.89	53.53
34.9	1.3170	10.968	41.13	44.13	41.4	1.3996	11.657	50.03	53.68
35.0	1.3182	10.978	41.27	44.28	41.5	1.4010	11.668	50.17	53.83
35.1	1.3194	10.988	41.41	44.43	41.6	1.4023	11.679	50.31	53.98
35.2	1.3206	10.998	41.54	44.57	41.7	1.4037	11.690	50.45	54.13
35.3	1.3218	11.008	41.68	44.72	41.8	1.4050	11.702	50.59	54.28
35.4	1.3230	11.000	41.81	44.86	41.9	1.4064	11.702	50.73	54.43
42.0	1.4078	11.724	50.87	54.58	48.5	1.5026	12.514	60.03	64.41
42.1	1.4091	11.736	51.01	54.73	48.6	1.5041	12.527	60.18	64.57
42.2	1.4105	11.747	51.15	54.88	48.7	1.5057	12.540	60.32	64.72
42.3	1.4119	11.759	51.29	55.03	48.8	1.5073	12.553	60.46	64.87
42.4	1.4133	11.770	51.43	55.18	48.9	1.5088	12.566	60.61	65.03
42.5	1.4146	11.782	51.56	55.32	49.0	1.5104	12.579	60.75	65.18
42.6	1.4160	11.793	51.70	55.47	49.1	1.5120	12.592	60.89	65.33
42.7	1.4174	11.805	51.84	55.62	49.2	1.5136	12.605	61.04	65.49
42.8	1.4188	11.816	51.98	55.77	49.3	1.5152	12.619	61.18	65.64
42.9	1.4202	11.828	52.12	55.92	49.4	1.5167	12.632	61.32	65.80
43.0	1.4216	11.839	52.26	56.07	49.5	1.5183	12.645	61.46	65.95
43.1	1.4230	11.851	52.40	56.22	49.6	1.5199	12.658	61.61	66.10
43.2	1.4244	11.863	52.54	56.37	49.7	1.5215	12.672	61.75	66.26
43.3	1.4258	11.874	52.68	56.52	49.8	1.5231	12.685	61.89	66.41
43.4	1.4272	11.886	52.82	56.67	49.9	1.5247	12.698	62.04	66.57
10.5	1.1007	11.000	50.07	54.00	50.0	1.50(2	40 744	(2.10	((= 2
43.5	1.4286	11.898	52.96	56.82	50.0	1.5263	12.711	62.18	66.72
43.6	1.4300	11.909	53.10	56.98	50.1	1.5279	12.725	62.33	66.88
43.7	1.4314	11.921	53.24	57.13	50.2	1.5295	12.739	62.48	67.04
43.8	1.4328	11.933	53.38	57.28	50.3	1.5312	12.752	62.62	67.20
43.9	1.4342	11.944	53.52	57.42	50.4	1.5328	12.766	62.77	67.36
44.0	1.4356	11.956	53.66	57.58	50.5	1.5344	12.779	62.92	67.51
44.1	1.4371	11.968	53.80	57.73	50.6	1.5360	12.793	63.07	67.67
44.2	1.4385	11.980	53.94	57.88	50.7	1.5376	12.806	63.22	67.83
44.3	1.4399	11.992	54.08	58.03	50.8	1.5393	12.820	63.36	67.99
44.4	1.4414	12.004	54.22	58.18	50.9	1.5409	12.834	63.51	68.15
44.5	1.4428	12.016	54.36	58.33	51.0	1.5426	12.847	63.66	68.31
44.6	1.4442	12.028	54.51	58.49	51.1	1.5442	12.861	63.81	68.47
44.7	1.4457	12.040	54.65	58.64	51.2	1.5458	12.875	63.95	68.66
44.8	1.4471	12.052	54.79	58.79	51.3	1.5475	12.888	64.10	68.78
44.9	1.4486	12.064	54.93	58.94	51.4	1.5491	12.902	64.25	68.94
45.0	1.4500	12.076	55.07	59.09	51.5	1.5508	12.916	64.39	69.10
45.0 45.1	1.4515	12.078	55.21	59.24	51.5	1.5525	12.930	64.59	69.26
45.2	1.4515	12.008	55.35	59.39	51.0	1.5525	12.943	64.69	69.42
45.2 45.3	1.4529	12.100	55.35 55.49	59.59 59.54	51.7	1.5558	12.943	64.89 64.84	69.42 69.57
45.5 45.4	1.4544	12.112	55.63	59.54 59.69	51.8 51.9	1.5558	12.957	64.84 64.98	69.57 69.73
	1.1550	10.100	55.65	57.07	51.7	1.0010		51.20	
45.5	1.4573	12.137	55.77	59.84	52.0	1.5591	12.985	65.13	69.89
45.6	1.4588	12.149	55.92	60.00	52.1	1.5608	12.999	65.28	70.05
45.7	1.4602	12.161	56.06	60.15	52.2	1.5625	13.013	65.43	70.21
45.8	1.4617	12.173	56.20	60.30	52.3	1.5642	13.027	65.58	70.37
45.9	1.4632	12.185	56.34	60.45	52.4	1.5659	13.041	65.73	70.53



Degrees Baumé	Specific Gravity at 60° /60° F	Density In Pounds per Gallon	Per Cent H ₂ SO ₄	Per Cent 66° Bé Acid	Degrees Baumé	Specific Gravity at 60° /60° F	Density In Pounds Per Gallon	Percent H ₂ SO ₄	Per Cent 66° Bé Acid
	00 / 00 1	Gallon		Telu		00 700 1	Galoli		ricid
46.0	1.4646	12.198	56.48	60.60	52.5	1.5676	13.055	65.88	70.69
46.1	1.4661	12.210	56.62	60.75	52.6	1.5693	13.070	66.03	70.86
46.2	1.4676	12.223	56.76	60.91	52.7	1.5710	13.084	66.18	71.02
46.3	1.4691	12.235	56.91	61.06	52.8	1.5727	13.098	66.33	71.18
46.4	1.4706	12.248	57.05	61.21	52.9	1.5744	13.112	66.48	71.34
46.5	1.4721	12.260	57.19	61.36	53.0	1.5761	13.126	66.63	71.50
46.6	1.4736	12.272	57.33	61.52	53.1	1.5778	13.141	66.78	71.66
46.7	1.4751	12.285	57.47	61.67	53.2	1.5795	13.155	66.93	71.82
46.8	1.4766	12.297	57.62	61.82	53.3	1.5812	13.169	67.08	71.98
46.9	1.4781	12.301	57.76	61.98	53.4	1.5830	13.184	67.23	72.14
47.0	1.4796	12.322	57.90	62.13	53.5	1.5847	13.198	67.38	72.30
47.1	1.4811	12.335	58.04	62.28	53.6	1.5864	13.213	67.53	72.47
47.2	1.4826	12.348	58.18	62.43	53.7	1.5882	13.227	67.68	72.63
47.3	1.4841	12.360	58.33	62.59	5.38	1.5899	13.241	61.83	72.79
47.4	1.4857	12.373	58.47	62.74	53.9	1.5917	13.256	67.98	72.95
47.5	1.4872	12.386	58.61	62.89	54.0	1.5934	13.271	68.13	73.11
47.6	1.4887	12.398	58.75	63.04	54.0	1.5954	13.285	68.28	73.27
47.7	1.4902	12.398	58.89	63.19	54.2	1.5952	13.300	68.43	73.44
47.8	1.4918	12.424	59.04	63.35	54.3	1.5987	13.314	68.59	73.60
47.9	1.4933	12.430	59.18	63.50	54.4	1.6004	13.329	68.74	73.76
48.0	1.4948	12.449	59.32	63.65	54.5	1.6022	13.344	68.89	73.92
48.1	1.4964	12.462	59.46	63.80	54.6	1.6040	13.359	69.04	74.09
48.2	1.4979	12.475	59.61	63.96	54.7	1.6058	13.373	69.19	74.25
48.3	1.4995	12.488	59.75	64.10	54.8	1.6075	13.388	69.35	74.41
48.4	1.5010	12.501	59.89	64.26	54.9	1.6093	13.403	69.50	74.58
55.0	1.6111	13.148	69.65	74.74	61.0	1.7262	14.376	79.43	85.23
55.1	1.6129	13.433	69.80	74.90	61.1	1.7282	14.393	79.62	85.43
55.2	1.6147	13.448	69.95	75.07	61.2	1.7303	14.411	79.80	85.63
55.3	1.6165	13.463	70.11	75.23	61.3	1.7324	14.428	79.99	85.83
55.4	1.6183	13.478	70.26	75.39	61.4	1.7344	14.445	80.18	86.03
55.5	1.6201	13.493	70.41	75.55	61.5	1.7365	14.463	80.36	86.23
55.6	1.6219	13.508	70.56	75.72	61.6	1.7386	14.480	80.55	86.44
55.7	1.6237	13.523	70.71	75.88	61.7	1.7407	14.497	80.74	86.64
55.8	1.6256	13.538	70.87	76.04	61.8	1.7428	14.515	80.93	86.84
55.9	1.6274	13.553	71.02	76.21	61.9	1.7449	14.532	81.11	87.04
56.0	1.6292	13.568	71.17	76.37	62.0	1.7470	14.549	81.30	87.24
56.1	1.6310	13.584	71.33	76.54	62.1	1.7491	14.567	81.50	87.46
56.2	1.6329	13.599	71.49	76.71	62.2	1.7512	14.585	81.71	87.68
56.3	1.6347	13.615	71.64	76.88	62.3	1.7533	14.603	81.91	87.90
56.4	1.6366	13.630	71.80	77.05	62.4	1.7554	14.620	82.12	88.12
56.5	1.6384	13.645	71.96	77.23	62.5	1.7576	14.638	82.32	88.33
56.6	1.6403	13.661	72.12	77.39	62.6	1.7597	14.656	82.52	88.55
56.7	1.6421	13.676	72.28	77.56	62.7	1.7618	14.674	82.73	88.77
56.8	1.6440	13.692	72.43	77.73	62.8	1.7640	14.691	82.93	88.99
56.9	1.6459	13.707	72.59	77.90	62.9	1.7661	14.709	83.14	89.21
57.0	1.6477	13.722	72.75	78.07	63.0	1.7683	14.727	83.34	89.43
57.1	1.6496	13.738	72.91	78.24	63.1	1.7705	14.745	83.57	89.68
57.2	1.6515	13.754	73.07	78.41	63.2	1.7726	14.763	83.80	89.93
57.3	1.6534	13.770	73.23	78.59	63.3	1.7748	14.781	84.04	90.18
57.4	1.6553	13.786	73.39	78.76	63.4	1.7770	14.799	84.27	90.43



Degrees Baumé	Specific Gravity at 60° /60° F	Density In Pounds per Gallon	Per Cent H2SO4	Per Cent 66° Bé Acid	Degrees Baumé	Specific Gravity at 60° /60° F	Density In Pounds Per Gallon	Percent H ₂ SO ₄	Per Cent 66° Bé Acid
57.5	1.6571	13.802	73.55	78.93	63.5	1.7791	14.818	84.50	90.67
57.6	1.6590	13.817	73.72	79.10	63.6	1.7813	14.836	84.73	90.92
57.7	1.6609	13.833	73.88	79.27	63.7	1.7835	14.854	84.96	91.17
57.8	1.6628	13.849	74.04	79.45	63.8	1.7857	14.872	85.20	91.42
57.9	1.6648	13.865	74.20	79.62	63.9	1.7879	14.890	85.43	91.67
58.0	1.6667	13.881	74.36	79.79	64.0	1.7901	14.908	85.66	91.92
58.1	1.6686	13.897	74.52	79.96	64.1	1.7923	14.927	85.90	92.17
58.2	1.6705	13.913	74.69	80.14	64.2	1.7946	14.946	86.17	92.47
58.3	1.6724	13.929	74.85	80.31	64.3	1.7968	14.964	86.43	92.75
58.4	1.6744	13.945	75.01	80.49	64.4	1.7990	14.983	86.72	93.06
58.5	1.6763	13.961	75.17	80.66	64.5	1.8012	15.002	87.03	93.39
58.6	1.6782	13.977	75.34	80.84	64.6	1.8035	15.020	87.33	93.71
58.7	1.6802	13.993	75.50	81.01	64.7	1.8057	15.039	87.65	94.06
58.8	1.6821	14.009	75.66	81.19	64.8	1.8080	15.058	88.05	94.48
58.9	1.6841	14.025	75.83	81.36	64.9	1.8102	15.076	88.31	94.76
59.0	1.6860	14.041	75.99	81.54	65.0	1.8125	15.095	88.65	95.13
59.1	1.6880	14.058	76.16	81.72	65.1	1.8148	15.114	88.98	95.48
59.2	1.6900	14.075	76.33	81.90	65.2	1.8170	15.133	89.30	95.83
59.3	1.6919	14.091	76.49	82.08	65.3	1.8193	15.152	89.76	96.32
59.4	1.6939	14.108	76.66	82.26	65.4	1.8216	15.171	90.17	96.76
59.5	1.6959	14.124	76.83	82.44	65.5	1.8239	15.190	90.60	97.22
59.6	1.6979	14.141	77.00	82.63	65.6	1.8262	15.209	91.06	91.71
59.7	1.6999	14.157	77.17	82.81	65.7	1.8285	15.228	91.54	98.23
59.8	1.7019	14.174	77.33	82.99	65.8	1.8308	15.248	92.07	98.80
59.9	1.7039	14.191	77.50	83.17	65.9	1.8331	15.267	92.60	99.37
60.0	1.7059	14.207	77.67	83.35	66.0	1.8354	15.286	93.19	100.00
60.1	1.7079	14.224	77.85	83.54	66.1	1.8381	15.327	94.00	100.87
60.2	1.7099	14.241	78.02	83.73	66.2	1.8407	15.348	95.00	101.94
60.3	1.7119	14.258	78.20	83.91	66.3	1.8427	15.365	96.00	103.01
60.4	1.7139	14.275	78.37	84.10	66.4	1.8437	15.374	97.00	104.09
60.5	1.7160	14.292	78.55	84.29	66.4	1.8437	15.374	98.00	105.16
60.6	1.7180	14.309	78.73	84.48	66.3	1.8424	15.363	99.00	106.24
60.7	1.7200	14.325	78.90	84.67	66.2	1.8391	15.335	100.00	107.31
60.8	1.7221	14.342	79.08	84.85					
60.9	1.7241	14.359	79.25	85.04					

*SPECIFIC GRAVITY IS NOT A TRUE INDICA-TION OF ACIDITY OF SULFURIC ACID AT SOLUTION STRENGTHS BETWEEN 93 PER-CENT AND 100 PERCENT."CHEMICAL PLANT CONTROL DATA HANDBOOK," 9TH ED., CHEMICAL CONSTRUCTION CORP. (1976)



METHODS OF ANALYSIS OF SULFURIC ACID AND OLEUM

TOTAL ACIDITY DETERMINATION

This procedure is based on titration of the sample with standard sodium hydroxide solution.

SAFETY

Safety clothing, i.e., rubber gloves and apron, should be worn when working with sulfuric acid or sodium hydroxide. All pipetting should be done using a rubber bulb. Eye protection must also be worn.

APPARATUS

100 ml. burette, class A, with Teflon stopcock; 500 ml. bottle or Erienmeyer flask with stopper; magnetic stirrer; Teflon magnetic stirring bar, indicator dropper bottle with fitting eyedropper; analytical balance or scales accurate to 1 mg.; weighing dish; washing bottle; analytical ampoules of diameter 20-21 mm.; glass rod capable of crushing stem of ampoule inside 500 ml. bottle; bunsen burner; rigid polyethylene or lead sheet circle with 1/8 inch hole to place over sample bottle.

REAGENTS

- 1. Certified 1N Sodium Hydroxide Solution (NaOH).
- Phenolphthalein Indicator Solution (.5g. phenolphthalein in powder in 60 ml. methanol, then distilled H₂0 to bring final volume to 100ml.).
- Primary Standard Potassium Acid Phthalate (PAP) National Bureau of Standards certified acid metric standard. Crust to 100 mesh fineness, and dry at 120° C for 2 hours. Cool and store in dessicator.
- 4. Distilled water, boiled at least 15 minutes to remove CO_2 , then neutralized to pH 7, and cooled to 5° C.

PROCEDURE

- 1. Weigh approximately 16 g. of potassium acid phthalate to the nearest mg. Work quickly to avoid moisture absorption.
- Transfer to the bottle or flask and dissolve in 250 ml. distilled, CO₂free water, using a magnetic stirrer. Be sure bottle is stoppered and when PAP is dissolved rinse all moisture from stopper and on sides of bottle into solution. Stopper until solution is ready to be titrated.
- Add 6 drops of phenolphthalein indicator and titrate with the NaOH solution to a faint permanent pink color (pH 8.6) using the 100 ml. burette. Care should be exercised as follows:
 - a) Spout on container of NaOH solution should be opened a couple times quickly to remove any crystals that might have formed before titrating is collected.
 - b) Allow one minute for all NaOH solution to drain from sides of burette into titrating solution before starting titration.
 - c) All NaOH solution, when not being used in actual titration, should be covered.





- d) Titrate solution no faster than .25 ml. per second
- e) Final volume of water in bottle should be consistent with all future titrations.
- 4. Calculate precise normality of NaOH solution as follows:

Correct ml. NaOH used to amount used at 25° C using chart. Normality of NaOH = $\frac{(Weight of PAP) (assay value of PAP)}{(.2042) (ml. NaOH in titration)}$

Repeat twice and take an average.

- 5. Break off only enough of the ampoule stem to leave behind enough stem on the ampoule to reach through the circle into the acid bottle and well into the acid. Make as clean of break as possible, not leaving any jagged edges or cracks. After weighing and recording weight, rotate the bulb of the ampoule in a bunsen burner until flame appears yellow around the bulb, wait 5 seconds, and insert stem into acid through the hole in the circle. Draw up as much acid as needed into ampoule. Immediately insert end of stern into flame and heat just enough to close off the stem. Rinse the ampoule and dry. Be sure ampoule is at room temperature at this point. Weigh again to find the grams of acid inside the ampoule.
- 6. Place the ampoule inside of a bottle containing 200 ml. of the CO₂-free pH 7 water and tightly stopper. Break the ampoule. If the acid is 98% or stronger, shake the bottle until all of the gases are dissolved. Carefully remove the stopper and rinse all moisture from the stopper and the sides of the bottle into the acid solution. Take the stirring rod and, starting from the closed end, crush the stem completely. Rinse the water on the stirring rod and, starting rod into the solution. Keep stoppered until ready for titration.
- 7. Titrate with the standardized NaOH solution as detailed in the standardized procedure.
- 8. Determine the strength of the acid as follows: %H2SO4= (temp. adj ml. NaOH used) (Norm of NaOH) (.04904) (100) Weight of sample in grams
- 9. Duplicate tests are required for best results.



IRON DETERMINATION IN SULFURIC ACID AND OLEUM THIOCYANATE COLORMETRIC METHOD

SAFETY

Acids can cause severe burns. Do not get in eyes, on skin, or on clothing. In case of spillage flush with plenty of water and remove contaminated clothing.

APPARATUS

- 1. Spectrophotometer capable of readings at 458 nm.
- 2. 1 cm. cells
- 3. 250 ml. Erlenmeyer flask
- 4. 10 ml. burette.
- 5. Graduated 5 or 10 ml. pipettes for sulfuric acid, 10 ml. graduated cylinder (calibrated "to deliver") for oleum.

REAGENTS

- 1. C.P. Sulfuric Acid.
- 2. C.P. Hydrochloric Acid.
- 3. 0.1 N Potassium Permanganate Solution.
- 4. Ammonium Thiocyanate Solution. Dissolve 298 g. of ammonium thiocyanate in 2000 ml. of water. Store in dark.
- 5. Iron Standard Solution 1000 ppm.
- 6. Working Iron Solution. Pipette 10 ml. of iron into a 100 ml. volumetric flask, add 1 ml. of hydrochloric acid and dilute to volume with distilled water.

PROCEDURE

- 1. Prepare calibration curve using the steps numbered below, but with the following exceptions:
 - a) Instead of sample, use 4.8 ml. of C.P. sulfuric acid.
 - b) In each separate flask, use 1,2,4,7, and 10 ml. of the working iron solution for, respectively, 100, 200, 400, 700, and 1000 ug of iron.
 - c) Reduce enough of the distilled water added to each flask so that final volumes are constant. Measure % transmittance of each sample and prepare curve from data obtained (@ 485 nm).
- 2. Add 100 ml. of distilled water to a 250 ml. flask.
- For sulfuric acid, pipette enough of the acid so that sulfate concentration is equivalent to that of standards. With oleum, dilution by 80% sulfuric acid will be necessary.



- 4. Add 1 N potassium permanganate dropwise to flask until a faint permanent pink color is obtained, then add 2-3 drops in excess.
- 5. Add 10 ml. ammonium thiocyanate solution.
- 6. Add 85 ml. distilled water and swirl.
- 7. Let the color develop for three minutes and read the transmittance of the sample of 485 nanometers. Distilled water is sufficient for a reagent blank for clear acid samples.
- 8. From the prepared calibration curve, determine the micrograms of total iron found in the analyzed sample. Parts per million iron can then be calculated as follows:

ppm Fe by weight =	Micrograms Fe Found From Graph						
ppin re by weight –	ml. of sample	Х	spec. gravity of				
	before dilution		sample before dilution				



SAFETY, HEALTH, AND ENVIRONMENTAL INFORMATION

INTODUCTION

Sulfuric acid is an extremely corrosive material, but it can be handled safely if the proper precautionary measures are observed. This section presents a review of the hazardous properties of sulfuric acid and the precautionary measures which should be followed. It is recommended that all personnel who handle or are potentially exposed to sulfuric acid become familiar with this information. It is also recommended that a qualified safety professional review operations involving the handling of sulfuric acid to ensure that all possible hazards have been eliminated and to ensure that all necessary precautionary measures have been

implemented. All applicable Federal/State regulations should be followed.

Unless otherwise indicated, the information in this section applies to both concentrated sulfuric acid and oleum.

SAFETY CONSIDERATIONS

Training

Individuals who handle sulfuric acid should receive training regarding work practices, maintenance procedures, emergency procedures, cleanup methods, and special hazards which are unique to their job. Individuals who may be potentially exposed to sulfuric acid in emergency situations should know how to correctly use respiratory equipment and protective clothing.

Hazard Communications

Before any person handles or works near sulfuric acid, they must be trained in accordance with the OSHA Hazard Communication Standard, 29 CFR 1910.1200, also known as the "Right to Know" Law.

Hazardous Materials Transportation

Any person involved in the transportation of sulfuric acid must be trained in accordance with DOT Hazardous Materials Employee Training (HM-126F), 49 CFR 172, Subpart H.

Precautions

The basic rules for safely handling sulfuric acid are as follows:

Always avoid direct personal contact with sulfuric acid

Always wear the required protective equipment

Always ensure the immediate availability of an adequate water supply

Always avoid ignition sources

Always add acid to water, not water to acid







PROTECTIVE EQUIPMENT

General

Personal protective equipment should not be considered as a substitute for safe working conditions where better engineering controls and safer work practices can be implemented. It is, however, in certain instances, the only practical means of protecting the worker.

The personal protective equipment described in this section should be worn whenever skin contact with sulfuric acid is possible or whenever the airborne concentration of sulfuric acid may exceed acceptable exposure limits.

The protective equipment to be used includes:

- Chemical safety goggles and face shield (eight-inch high minimum);
- Rubber or polyvinyl chloride gloves with gauntlets large enough to cover the forearm;
- Rubber or polyvinyl chloride high-top safety toe shoes or boots, the tops of which should be covered by the trousers;
- Acid-proof outer clothing that fits snugly at neck and wrists. The wrists of the outer clothing should be positioned to prevent drainage of acid into the gloves. Jackets with attached gloves are available;
- Hard hat or other form of head protection (e.g., full cover acid hood);
- A respirator for protection against airborne concentrations of sulfuric acid.

(See section on Respiratory protection that follows.)

Respiratory protection

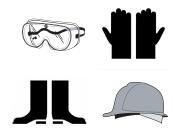
If the airborne concentrations of sulfuric acid cannot be controlled to acceptable levels, potentially exposed individuals should be protected through the use of NIOSH/MSHA-approved respiratory equipment. Care should be taken in choosing and using a respirator. All aspects of any respiratory protection program should be thoroughly reviewed and approved by a competent safety or health professional.

The Occupational Health Guidelines for Chemical Hazards, published by the U.S. Department of Health and Human Services and the U.S. Department of Labor (DHHS (NIOSH) Publication No. 81-123) provides information regarding the proper selection of respiratory equipment (refer to section entitled "Occupational Health Guidelines for Sulfuric Acid").

Respiratory protections should be used whenever there is potential exposure to oleum. Oleum will liberate sulfur trioxide (SO_3) gas, exposure to which can be extremely damaging to the lungs.



CAUTION: CONTACT LENSES SHOULD NEVER BE WORN WHILE HANDLING SULFURIC ACID





Water supply-safety showers and eyewash fountains

Rapid action freeze-proof safety showers and eyewash fountains that are readily accessible and well-identified should be installed in all areas where individuals may come in contact with sulfuric acid.

When unusual or non-routine circumstances may result in individuals being potentially exposed to sulfuric acid at work stations where safety showers and eyewash fountains are not available, a hand-held drench hose or other adequate source of continuous water flow should always be available.

Recommendations regarding equipment design and installation are given in ANSI A358.1- American National Standard for Emergency Eyewash and Shower Equipment.

Fire and explosion hazards

Sulfuric acid is not flammable; however, under some conditions, it can cause the ignition of other combustible materials if it is allowed to come in contact with these materials. In general, concentrated sulfuric acid should be isolated from organic materials and nitrates, chlorates, carbides, and metallic powders.

Hydrogen, a highly flammable, odorless, colorless gas, is generated by the corrosive action of acid on most metals. Consequently, hydrogen can be generated inside a drum, tank car, tank truck, or metal storage tank containing sulfuric acid. As hydrogen will form flammable mixtures with air over a wide range of concentrations, ignition sources should not be permitted near drums, storage tanks, tank cars, or tank trucks, particularly when these containers are being opened after an extended period of being closed.

Dilution

Except when large quantities of water are used to dilute spills (See section on Spill cleanup below), water or alkaline solutions containing water should never be added to sulfuric acid. A violent reaction will take place which may cause spattering to occur. If it is necessary to mix acid and water, the acid should always be slowly and cautiously added to the water.

Emergency Response

Adequate protective and rescue equipment should be readily available for each individual who may be required to respond to an emergency situation involving sulfuric acid. This equipment should include a hood-type face shield, chemical safety goggles, impervious gloves, rubber or polyvinyl chloride gloves, head protection, and self-contained/supplied-air respiratory protective equipment operated in pressure demand or other positive pressure mode. This equipment should be routinely maintained. Where confined space entry is possible, those items specifically required for safe entry should also be readily available (See section on Confined space entry that follows). Review pertinent emergency response requirements in 29 CFR 1910.120. Other Federal and State regulations should also be followed.

Confined Space Entry

Precautions must be taken before any individual is allowed to enter a confined space (e.g., tank truck, rail car, storage tank, process equipment, open sump) which has contained sulfuric acid. Requirements for the elements of a proper confined space entry permit procedure are given in 29 CFR 1910.146. Applicable State regulations should also be followed.

CAUTION: SAFETY SHOWERS, EYE-WASH FOUNTAINS, AND OTHER DESIGNATED WATER SUPPLIES SHOULD BE ROUTINELY TESTED, AT LEAST WEEKLY. WHERE APPROPRIATE, THESE FACILITIES SHOULD ALWAYS BE TESTED BEFORE EACH JOB.







Spill Cleanup

Sulfuric acid should always be handled with extreme caution. In the event of a sulfuric acid spill, entry to the contaminated area should immediately be closed to personnel unless they are wearing the required protective equipment.

The spilled acid should be contained and cautiously diluted (See section above on Dilution) to a minimum of 5 or 6 times its original volume with water. The water should be added gradually to the acid to minimize spattering.* During the dilution process, personnel should stand as far away as possible from the spill to avoid contact with the acid and with the corrosive fumes that will be emitted. In the case of an oleum spill, highly toxic sulfur trioxide gas will be emitted.

A qualified person should determine the degree of dilution and the need for neutralizing the acid before recovering it or discharging it to a wastewater treatment facility.

Care should be taken to ensure that acid leaks, spills, or drainage do not come in contact with sulfide materials, because of the danger of evolving toxic and flammable hydrogen sulfide gas.

Once the spilled acid has been cleaned up, the contaminated area should be thoroughly washed or saturated with water. When the washwater can be collected, it should be discharged to a wastewater treatment facility designed to neutralize acidic wastes. Applicable Federal/State regulations should be followed.

HEALTH EFFECTS

Toxicity

Sulfuric acid is highly corrosive; direct contact with eyes or skin, or ingestion can cause severe tissue injury, blindness, and death. Sulfuric acid is highly toxic via inhalation and moderately toxic by ingestion. Chronic conjunctivitis, frequent respiratory infections, emphysema, digestive disturbances, erosion and/or discoloration of teeth have been reported in persons exposed to sulfuric acid over the course of many years.

The acute oral LD_{50} in rats (lethal ingested dose for 50% of the animals) has been found to be 2140 mg/kg. Accidental ingestion of an unspecified quantity of 50% w/w sulfuric acid (presumably aqueous) by humans has resulted in necrosis (death of tissue) at all areas of contact with acid, including the digestive tract. Healing is slow, with stricture and scar formation.

Exposure to concentrations in air of 18 to 60 Mg/M³ for 18 hours has been found to be lethal to guinea pigs. Lower concentrations in guinea pigs have been shown to produce damage to the respiratory tract, with severity of damage depending largely on concentration and duration of exposure. Studies have also indicated that aerosol size and humidity may affect the toxicity of sulfuric acid.

In humans, concentrations of about 5 mg/M^3 may be very objectionable, usually causing cough, with marked alterations in respiration. Inhalation exposure to sulfuric acid by splash or spray has resulted in pulmonary edema and chronic pulmonary fibrosis, bronchial lesions, and pulmonary emphysema.

CAUTION: CARE MUST BE TAKEN WHEN ADDING SODA ASH OR LIME TO SULFURIC ACID BECAUSE OF THE HEAT LIBERATED BY THE REACTION.



Carcinogenicity

The International Agency for Research on Cancer (IARC) has concluded that long-term exposure to strong inorganic acid mists containing sulfuric acid is carcinogenic to humans (Category 1 human carcinogen). This conclusion is based on epidemiology studies and studies in laboratory animals which suggest that an increased risk of cancer can result from prolonged and repeated exposure to mists of sulfuric acid.

A more detailed discussion on the health effects of sulfuric acid can be found in the following references:

- NIOSH. Review and Evaluation of Recent Literature. Occupational Exposure to Sulfuric Acid. 1981, DHHS (NIOSH) Publication No. 82-104.
- 2. NIOSH. Criteria for a Recommended Standard. Occupational Exposure to Sulfuric Acid. 1974, HEW Publication No. (NIOSH) 74-128.
- American Conference of Governmental Industrial Hygienists. Documentation of the Threshold Limit Values. Fourth Edition, 1980, ACGIH, Cincinnati.

Workplace Exposure Criteria

The American Conference of Governmental Industrial Hygienists (ACGIH) has established a threshold limit value* of 1 mg/M³ for sulfuric acid (1 milligram of sulfuric acid per cubic meter of air). The Occupational Safety and Health Administration (OSHA) has adopted the same value as their permissible exposure to limit (PEL).

Exposure Measurements

The actual concentration of sulfuric acid to which an individual is exposed can be determined by measuring the acid concentration in the ambient air which the individual breathes. Tests of this nature are generally conducted by an industrial hygienist, who is trained in the recognition, evaluation and control of occupational health hazards. *SPATTERING MAY BE REDUCED THROUGH THE USE OF A WATER SUPPLY





Contact with concentrated sulfuric acid or oleum is an emergency requiring immediate treatment since the corrosive action of the acid occurs within seconds.

Remove the victim from exposure immediately.

Medical help should be obtained as quickly as possible.

Contact With Eyes

- 1. Check for contact lenses and if individual is wearing them, remove them immediately.
- 2. Wash out eyes with large amounts of water or an isotonic eye wash for at least 15 minutes. If pain is still present, continue the washing out for an additional 15 minutes. During the washing, spread apart the eyelids as much as possible to ensure contact of the water or eye wash with all the surfaces of the eyes and lids.
- 3. Send individual to a doctor, preferably an eye specialist, as soon as the washing out has been completed.

Contact With Skin and Mucous Membranes

- 1. Remove contaminated clothing as quickly as possible.
- 2. Immediately flush the contaminated skin and membranes with large quantities of water. This can be done by placing the individual under a safety shower or other continuous water supply. Further help can be obtained by simultaneously washing the exposed areas with soap for approximately 15 minutes.

Inhalation

- 1. Remove individual from contaminated area immediately.
- 2. If breathing has ceased, start artificial respiration or mouth-to mouth resuscitation.
- 3. Remove all tight clothing and cover individual with a blanket.
- 4. Call a doctor immediately to assume responsibility for further respiratory treatment and handling of the individual.

Ingestion

- 1. Do not induce vomiting or administer a strong base or bicarbonate to neutralize the acid.
- 2. Remove individual quickly from the contaminated area to one which is quiet and well-ventilated for protection and comfort. This will also help calm the individual and make attempts to dilute and neutralize the acid easier.
- 3. Rinse mouth and throat with cold water to reduce injury and relieve pain.





- 4. If conscious and able to swallow, have the individual drink 4 8 ounces (no more than 1 glassful) of either
 - a) Milk
 - b) Soap solution
 - c) Milk of magnesia
 - d) Lime water (calcium Hydroxide)
 - e) Oil (olive or mineral oil) or eggs Avoid water unless none of the above are available because of the heat produced by the chemical reaction with concentrated sulfuric acid.
- 5. Remove tight clothing as soon as possible and cover individual with a blanket.
- 6. Place individual in a lying down position with his legs elevated.
- 7. If individual is vomiting, lower his head and if lying down, turn him on his left side to prevent inhaling the vomit into his lungs.
- 8. Remove individual to a medical facility as soon as possible for more complete treatment.

ENVIRONMENTAL CONSIDERATIONS

Sulfuric acid, depending upon the quantities involved and its utilization, is regulated by numberous Environmental Protection Agency (EPA) affiliated programs. Regulations pursuant to laws and regulations including, but not limited to, the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), Clean Air Act, Clean Water Act, Emergency Planning and Community Right-to Know Act (EPCRA-SARA Title III), Resource Conservation Recovery Act (RCRA) must be reviewed and complied with, if required.



UNLOADING SULFURIC ACID

INTRODUCTION

The following is a general guide to unloading sulfuric acid from tank cars and tank trucks. The primary intention of this section is to present typical procedures for unloading sulfuric acid by air pressure and pump. The information given is by no means complete, and actual unloading conditions will necessitate the use of more detailed procedures to guarantee safe unloading.

Detailed sulfuric acid unloading procedures and safety regulations can be found in the following industry and government publications:

49 CFR 171-181 Code of Federal Regulations, Department of Transportation (DOT)

29 CFR 1910 Code of Federal Regulations, Department of Labor (OSHA)

The above applicable sections of the Federal Regulations should be reviewed prior to the installation of unloading facilities or the drafting of detailed unloading procedures for your site. Particular attention should be given to the following and other applicable sections of the Federal Regulations:

CFR TITLE 49 DEPARTMENT OF TRANSPORTATION

171.15	Spill and Leak Reporting
172.508 & .510	Placards, Rail Car
172.514	Placards, Cargo Tanks
172, Subpart H	Employee Training, Hazardous Materials
173.29	Empty Containers
173.242	Authorized containers, Sulfuric Acid
174.67	Unloading Tank Cars
177.834	Unloading Cargo Tanks
177.839	Securing Closures, Highway
179, Subpart C &D	Containers Specification, Tank Cars
179.100-15 & .200-18	Safety Relief Devices, Tank Cars
180, Subpart E	Qualification and Maintenance of Cargo Tanks







CFR TITLE 29 DEPARTMENT OF LABOR

1910.24 (b)	Stairs, Walkways, Work Platforms
1910.120	Emergency Response
1910.132 (c)	Personal Protective Equipment
1910.134	Respiratory Protection
1910.151 (c)	Safety Showers, Eye Wash Stations
1910.1200	Hazard Communication

SHIPPING INFORMATION

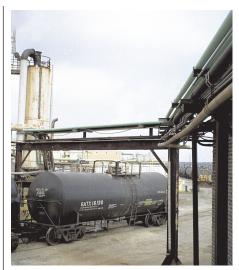
Sulfuric acid having a concentration greater than 65.25% may be shipped in the following Department of Transportation (DOT) authorized tank cars and tank trucks:

<u>Tank Cars</u>	<u>Tank Trucks</u>
103A-103AW-103CW	MC-310
105A300W	MC-311
111A60W2-111A100W2	MC-312
111A100W6-111A100F2	

The above is only a partial list of authorized containers. Consult 49 CFR 173.242 for more details.

Tank cars and tank trucks vary in design and piping arrangement, and require different unloading procedures. For specific unloading procedures, refer to information sources listed previously.

Tank cars and tank trucks may be insulated, insulated and coiled (internal or external), for shipments of acid concentrations that will freeze at ambient temperatures. High quality acid is usually shipped in containers that are lined to reduce contamination from container corrosion during transit. Some authorized tank cars for shipment of heavy (sludge spent) acids are equipped with bottom outlets. The procedures for safely handling this type acid depend on too wide a quality range to be included in this section.





UNLOADING TANK CARS AND TANK TRUCKS

Southern States tank cars and tank trucks are shown in Figures 1 and 2. They are available in 100 ton tank cars, and approximately 24 ton tank trucks.

Figure 1.



Figure 2.



The following unloading procedures are intended to familiarize the reader with the important steps and considerations necessary to safely unload a typical sulfuric acid shipping container with air pressure or by pump. No attempt is made here to show the specific procedures required for unloading each of the various specifically authorized containers. If more detailed information is required, please contact your Shipper.



GENERAL PRECAUTIONS

Please refer to the Safety Section of this Handbook for the proper protective clothing to use when making or disassembling connections of sulfuric acid piping. When disassembling acid piping connections that are above head high, or that are expected to contain acid, a full cover acid hood is also recommended.

Attendance by a qualified person during the entire unloading procedure is required. This person must be thoroughly familiar with the hazards of sulfuric acid, and all operating, emergency, safety and first aid procedures recommended for its safe handling.

A large supply of cold water should be available at the unloading site for reasons of personal safety, as described in the Safety Section, and to wash down any acid that may be spilled.

A remote backup safety shower is recommended, along with a signal system designed to get assistance if an emergency should arise. The signal system and safety showers should be tested routinely. Signs restricting access to the area during unloading should be used.

Piping should be arranged to allow draining by the use of vent and drain valves. Connections should be made below waist high. Trapping acid between valves should be avoided. Storage tanks must be adequately vented to avoid pressure build up during unloading. Overflow piping should be directed to a harmless area.

The unloading site should be designed to provide good ventilation, lighting, and OSHA-approved access to all connection points. Non-sparking tools should be used when working around sulfuric acid piping and containers, since hydrogen gas can be produced by corrosion. Open flame, smoking, and other ignition sources must not be allowed in the unloading area.

Avoid exposure to vapors and acid mist when handling sulfuric acid or oleum. Pump unloading with a vapor recovery system is recommended for oleum.

Respiratory equipment should be available in the event of spills or other fume problems.

See Figures 3 and 4 showing a typical air supply and shipping container configurations.

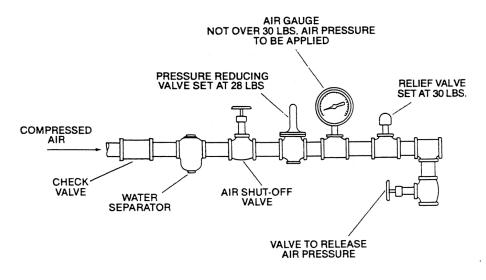




Figure 3. Air piping arrangement for unloading acid tank cars.



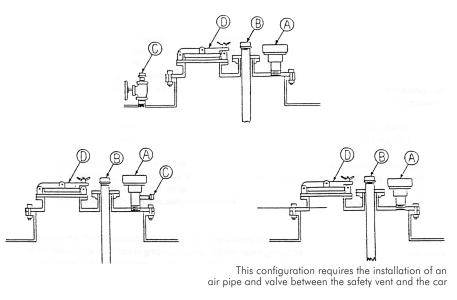


Figure 4. Tank car dome with air inlet and liquid eduction valves.

A. Safety Vent B. Eduction Pipe C. Air Pipe D. Fill-Hole Cover

TYPICAL UNLOADING PROCEDURE USING AIR PRESSURE FOR EDUCTION TUBE TOP UNLOADING CONTAINERS.

- Verify the contents of the container by checking the shipping papers and container stenciling and/or placarding. Make sure the receiving storage tank has sufficient outage to hold the contents of the container being unloaded. Inspect the shipping container for leaks or damage which could interfere with safe unloading. Set the brakes and block the wheels of the shipping container. For rail cars, install the required warning signs.
- 2. Carefully open the air connection valve and vent any pressure in the container. If no venting occurs, check to make certain the air connection and valve are unobstructed. Carefully loosen the fill hole cover bolt (do not disengage), and pry up slightly. If any pressure is present, let it bleed off slowly until no pressure is in the tank, then open the fill hole cover. Samples may be taken at this point.

CAUTION: All pressure must be vented before any container fittings are removed because of the danger of acid spray. Do not remove the cap from the liquid eduction pipe until it is definitely known that all pressure has been vented from the container, and that the valve is closed (if so equipped).

- 3. Close the liquid eduction valve (if so equipped), and carefully remove the cap from the valve, or from the liquid eduction pipe (if no valve is provided). Inspect the condition of the threads, gaskets and liquid eduction pipe carefully. Pipe threads in sulfuric acid service deteriorate rapidly, especially when exposed to acid and atmospheric moisture. Make a two inch or larger steel pipe connection to the line leading to the acid storage tank. Swivel connections are available which can be used to facilitate such connections. Flexibility can also be provided by the use of an armored hose approved for sulfuric acid or oleum use. A valve located as close as possible to the liquid eduction pipe is an excellent safety item. If quick couplings are used, the cams or ears should be tied and locked in their closed position.
- 4. Connect the air line to the air connection valve. The air supply must have a check valve; a pressure reducing valve set at about 28 pounds per square inch gauge pressure; and a safety relief valve set at not over 30 pounds per square inch guage pressure. A pressure gauge and bleed valve are also required to insure proper pressure is maintained, and to bleed off the



pressure when the container is empty. The air should be non-contaminated, free from oil, moisture and foreign matter. A suitable inert gas may be substituted for the unloading air if desired.

Close the fill hole cover and inspect the system to be sure that it is tight, that all vent and bleed valves on the unloading line are closed, and that all valves are properly positioned to receive acid.

5. Slowly apply air pressure to the container by opening a valve on the air supply line located outside of the probable spray area, being careful not to exceed 28 pounds per square inch guage pressure. If the air pressure is to high, the safety relief valve on the supply line will open. If pressure cannot be maintained, check for leaks or a broken frangible disc. Do not replace the frangible disc with other than an authorized, properly rated disc.

If leaks develop or acid is spilled, promptly wash down any surface where acid has spilled with an excess of water, being extremely careful to avoid getting water into the container, piping or into large accumulations of acid in holes or trenches. If the valving is properly set, acid will flow to the storage tank. If flow cannot be established or maintained, the use of an unloading pump may be required.

When the container is empty, a drop in the pressure and the sound of air rushing through the discharge pipe will be heard. Shut off the air supply, then shut off the valve in the unloading line near the connection point. Allowing some air to flow through the unloading line will help clear the acid from the line.

- 6. Vent all pressure from the shipping container through the air supply line bleed valve. Carefully open the fill hole cover, and inspect the container to insure unloading is complete. Disconnect the air supply line. When the unloading line has been completely drained, carefully disconnect it (highest point first) from the shipping container. If the unloading line is not equipped with a drain and vent valve, acid should be expected to spill out at the disconnect points.
- Close the fill hole cover tightly and replace the caps on the air connection valve and liquid eduction line. Replace the valve covers. Inspect the container and place proper DOT required placards on the container for the return shipment.

TYPICAL UNLOADING PROCEDURE USING PUMPS.

- 1. Use the same preparation and procedural Steps 1 through 3 listed on page 43 for unloading by air pressure apply to unloading by pump.
- 2. A self-priming pump is recommended. Vent the container to the atmosphere by leaving the fill hole cover open. Start the pump from outside the probable spray area. If auxiliary priming is required, follow the air unloading procedure, applying only sufficient pressure to start the flow of acid to the pump, and in no case more than 28 pounds per square inch. It is essential that adequate vacuum relief be provided to avoid collapse of the shipping container. A positive pressure can be maintained during the unloading if desired. When unloading is complete, a noticeable change in the pump load and sound will occur.
- 3. Close the pump suction valve and shut off the pump. Proceed with Steps 6 and 7 of the procedure for unloading with air pressure.



FROZEN ACID

Some acid concentrations freeze at ambient temperatures and require heating before unloading is possible. This acid should be shipped in containers that are insulated and have provisions for heating. The following precautions and/or actions should be taken when heating containers.

- a) Be sure the container is vented by opening the fill cover using Step 2 of the procedure for unloading with air pressure.
- b) After the container is vented, the heating coil caps may be cautiously removed. Because of the possibility of leaked in the heating coils, there may be considerable acid pressure present.
- c) Do not add water to the containers containing sulfuric acid.
- d) Never use open flames to heat sulfuric acid.
- e) Sulfuric acid expands when heated and can cause container overflows.
- f) To liquefy containers that are not equipped with insulation or coils, the container will have to be enclosed or placed in an enclosure where the temperature is above the freezing point of the acid.

EMPTY TANK CARS AND TRUCKS

No internal washing should ever be attempted with empty tank cars. Water or other liquids must never be introduced into a tank car. No employee should ever be permitted to enter and empty tank car or tank truck for any purpose.

When a tank car is empty, the DOT corrosive rail placards must be reversed. Heating coil caps must be removed to allow drainage during the return trip. All closures and valves must be closed and free of leaks. The shipping container and carriage must be inspected and the shipper and carrier notified of and defects.



MATERIALS OF CONSTRUCTION

The conditions under which sulfuric acid must be handled depend upon acid strength, temperature, velocity, and a multitude of factors that vary from one installation to the next. The presence of suspended carbon or other impurities may greatly affect corrosion rates and must be carefully considered. The recommendations given here represent a consensus of our own plant experience. They are, therefore, of a general nature and are intended only as guide to assist acid users in the selection of the most suitable materials for their particular requirements. Except where otherwise stated, the recommendations given here are for 75 percent to 100 percent acid or oleum above 104 percent at ambient temperatures.

TANKS

Sulfuric acid storage tank size is determined by the type of acid delivery (tank truck or tank car) and by the expected weekly or monthly acid requirements. Good practice is to have capacity at least 50 percent greater than the delivery unit but not less than two weeks' operating requirements. As the life expectancy of a properly designed storage tank is typically at least 15 years, tank installation should be sized not only for the immediate requirements but also for anticipated future requirements.

Sulfuric acid less than approximately 68% strength (54° Bè) should be stored in phenolic-lined tanks or chemical lead-lined tanks. Above 77% (60° Bè) mild steel is recommended as long as temperatures stay below 100°F. Strengths of sulfuric acid between approximately 79% (61° Bè) and 93% (66° Bè) and between 99% and 101.5% are much more corrosive to steel, especially when the acid is hot. Because of this, these strengths should be stored in steel only when the temperature is kept below 75°F and moist air is excluded from the tank.

Even with these limitations, allowance should be made for high corrosion rates to the tank wall. A.P.I. Standard 653 provides guidance for recommended tank inspection methods and frequencies.

High quality grades of sulfuric acid, such as electrolytic grade acid, should be kept free from contamination with iron and other corrosion products. To guard against these impurities, this acid typically is shipped and stored in resin-lined tanks.¹ Electrochemical corrosion control equipment is also commercially available for maintenance of high quality acid. ² Welded construction is preferable for all sulfuric acid tanks. Generally, prefabricated tanks of up to 27,500 gallons capacity are available. Larger tanks are usually built to order.³ For tanks of less than 10,000 gallons capacity, 3/8 inch steel plate is typically used. Larger tanks require thicker steel plate and should be designed according to A.P.I. Standard 650 with additional wall thickness allowance for corrosion based on expected service life. A bottom-discharge sulfuric acid tank may be provided with a valve of Alloy 20 steel or tetraflouroethylene-lined plug valve for position shut-off.

Sulfuric acid storage tanks are usually installed on concrete piers with steel grillage so that the entire tank is visible for inspection and leak correction. The tank elevation, however, should be low enough, in relation to the elevation of the incoming shipments, so that the hydrostatic head developed by the storage tank is reasonably below the unloading pump discharge pressure. This will permit off-loading of incoming acid within a reasonable time and minimize the possibility of transport equipment detention charges.



- 1 ONE SUCH RESIN IS PROVIDED BY HERESITE AND CHEMICAL COMPANY, MANITOWOC, WISCONSIN.
- 2 CHEMETICS INTERNATIONAL LTD., TORONTO, ONTARIO, CANADA.
- 3 THERE ARE NUMEROUS SUPPLIERS OF PREFABRICATED AND CUSTOM BUILT TANKS. LOCAL INQUIRY WILL GENERALLY FIND SUITABLE SUPPLIER.



Air vents are necessary on sulfuric acid tanks to allow normal breathing with temperature changes, to handle surges of air from compressed air unloading, and to prevent the accumulation of highly flammable and potentially explosive hydrogen gas which is evolved by the reaction of the acid with the metal tank. Polyvinyl chloride-lined steel is frequently used for these vents, which must be sized not only for the normal breathing of the tank due to volumetric changes in the acid level and temperature, but also for the rush of compressed air at the completion of unloading of an acid receipt. Because the hydrogen gas evolved in the tank will also be carried off by this vent, open lights and smoking should be excluded from this area. Tank vents and openings must be arranged to exlude rain and snow.

Access: A platform with a stair from the ground should be provided at the top of the tank. This should allow access to the top nozzles, vent and manway for sampling and, if no other liquid-level indicator is used, as a means of measuring liquid volume. Unloading lines should be placed away from the side of the tank to reduce corrosion to the side wall.

A side wall manway should also be provided near the bottom of the tank for tank entry to facilitate inspection and cleaning.

Agitation: To avoid the accumulation of weak acid on the surface and resulting increased tank corrosion at the vapor-liquid interface, acid stored in a tank not used for an extended period should be periodically agitated. This may be accomplished by recirculation with a pump or agitation through an open ended pipe, preferably with nitrogen, if it is available, or with dry compressed air.

Cleaning: Tank washing is typically required every 5 years to remove accumulated iron sulfate. A bottom outlet expedites this occasional cleaning. To minimize corrosion, the tank should be filled with strong acid as soon after washing as practical.

Painting: The outside of the storage tank is commonly covered with a lightcolored paint to reflect the sun's heat and to maintain the lowest possible tank shell temperature. Vinyl-based coatings have been used in areas subject to strong acid exposure.

Stand Pipe: To minimize the possibility of pumping significant quantities of iron sulfate solids that may have settled to the bottom of the storage tank, a stand pipe suction line placed several inches above the tank bottom should be considered.

Inlet Piping: Inlet piping should extend into the tank, terminating no less than two feet from the tank bottom. Incoming acid should not be allowed to run down the inside wall of the tank to avoid corrosion to the sidewall. Internal lines should be designed to prevent syphoning.

PIPING

All-welded schedule 80 black steel pipe with forged welding fittings and series 15 welding-neck or slip on weld flanges are commonly used in sulfuric acid service. Less expensive schedule 80 threaded steel pipe and threaded malleable iron fittings (while not recommended) may be used where occasional leaks and downtime for repairs can be tolerated.







Lines should be kept full of acid to minimize corrosion and excessive sulfation. Venting of acid lines is suggested to avoid pressure build-up and possible rupture from hydrogen gas or thermal expansion.

Highly flammable hydrogen gas is generated by the action of sulfuric acid on metal pipes. The valves at each end of a line should not be closed unless there is a vent, preferably back to the vented storage tank. This will prevent hydrogen gas from accumulating in the line.

It is occasionally necessary to empty acid lines for maintenance purposes, so the lines should have sufficient pitch to permit complete draining. If nitrogen is available, it may be used to facilitate pipe drainage. Sulfuric acid pipes should not be blown with air unless absolutely necessary. If air blowing is necessary, dry air should be used to minimize corrosion.

Cast iron pipe is generally preferred to steel for use with hot acid up to the temperature limit for such materials. Extra-heavy cast iron flanged pipe and 125-pound American Standard cast iron fittings are generally acceptable. Since cast iron pipe is fragile, it must be handled with care and well supported when installed.

Since iron sulfate forms during sulfuric acid storage, care must be taken to avoid clogging pipe lines. Generally, pipe larger than one inch in diameter is necessary. Also, as the pipe diameter is enlarged, acid velocity is reduced and corrosion rates are substantially lower.

VALVES

In selecting valves for sulfuric acid and oleum service, it is necessary to consider acid concentration, temperature, and the service application for the valve.

For block valve service, the selections are usually gate valves or plug valves. For temperatures up to about 190°F, Alloy 20 valves with Teflon® gaskets and packing are frequently used. However, in the use and manufacture of sulfuric acid, in recent years the temperatures have gone considerably above 200° F. and have necessitated special materials of construction. At least one manufacturer has developed a gate valve specifically for this type of service which employs an iron alloy, heavy walled section body with Lewmet⁴ for the discs, seat rings, and back seat bushings. Normally, plug valves with Teflon soft seats would be satisfactory at 190° F. and below, especially when acid contains no abrasives. However, hot acid in some operations frequently contains brick fines and tower packing chips which can cause cutting of the Teflon seats, resulting in the failure of the valve to shut off properly.

For control valve service, the usual choice is a glove or a butterfly valve. The globe valves can be manually operated or fitted with actuators. In some cases, sophisticated automated control valves with single or double ports are employed for this service. Butterfly valves used for control service can also be fitted with actuators. There are several manufacturers of soft seat Teflon butterfly valves with Alloy 20 discs. There is also available a metal-to metal seated butterfly valve of Lewmet alloy for this type of control service.

^{4 &}quot;LEWMET" IS A PATENTED ALLOY OF CHAS S. LEWIS & CO., INC.

TEFLON IS A REGISTERED TRADE MARK OF E.I. DU PONT DE NEMOURS & CO., INC.



PUMPS

In selecting pumps for sulfuric acid and for oleum service, one should always consider the acid concentration, temperature and the service application.

Horizontal centrifugal pumps or heavy duty vertical, submerged, centrifugal pumps are typically used for most sulfuric acid pumping requirements. In sulfuric acid producing plants, heavy duty vertical, submerged pumps are typically used for hot acid service. These pumps have heavy walled section, iron alloy castings with Lewmet internal parts.

The primary advantages of vertical, submerged pumps over horizontal pumps is the inherent safety in their being submerged and fact that there is no stuffing box leakage.

In transfer service of ambient temperature 66° Bè acid from storage tanks, Alloy 20 horizontal centrifugal pumps are commonly used with packing or mechanical seals. CAUTION: Packing of asbestos or Teflon types can allow some leakage which could cause acid spray in the vicinity of the horizontal pump. For this reason mechanical seals of Alloy 20 or higher alloy construction are usually employed to minimize this type of problem.

Pumps for oleum service usually employ similar materials of construction even though the temperatures normally are not as high as those encountered with 98% sulfuric acid.

Special applications require the use of other types of pumps. Controlled volume pumps are particularly well-adapted to the metering or feeding of sulfuric acid at predetermined constant or variable rates. Typical materials of construction include Alloy 20 plunger, cast iron or Alloy 20 wetted end, tetrafluoroethylene plastic chevron packing, "Hastelloy C"⁵ ball checks, and Stellite seats.

GASKETS

Ring gaskets for use with sulfuric acid are generally made from either 1/16 inch sheets of tetrafluoroethylene or chlorotrifluoroethylene. These material are particularly recommended for service with hot acid. (450° F max.)

SAFETY SHOWERS AND EYE WASH FOUNTAINS

Readily accessible, well marked, rapid action safety showers and eye wash fountains (preferably with warm potable water supply) must be available in the areas where sulfuric acid is being handled. ⁶ Showers should have deluge type heads, be easily accessible, plainly marked, and controlled by quick-opening valves of the type that stay open. They should be capable of supplying large quantities of water under moderately high pressure. Blankets should be located near the safety showers. Eye wash fountains, or a ready source of running tap water, such as drinking fountain or hose with a gentle flow of water should be immediately available for eye irrigation. All safety equipment should be inspected and tested at regular intervals, preferably daily and especially during freezing weather, to make sure it is in good working condition at all times. Heat-traced safety showers, eye washes, and their water supply lines are highly recommended in environments with freezing temperatures.

CAUTION: PACKING OF ASBESTOS OR TEFLON TYPES CAN ALLOW SOME LEAKAGE WHICH COULD CAUSE ACID SPRAY IN THE VICINITY OF THE HORIZONTAL PUMP.



- 5 "HASTELLOY" IS A TRADE NAME OF THE HIGH TECH-NOLOGY MATERIALS DIVISION, CABOT CORPORA-TION.
- 6 TITLE 29 CODE OF FEDERAL REGULATIONS PART 1910.151 (C), OCCUPATIONAL SAFETY AND HEALTH ADMINISTRATION.