

SODA ASH



 *General Chemical*
Industrial Products

1 Introduction

Soda ash, the common name for sodium carbonate (Na_2CO_3), has significant economic importance because of its applications in manufacturing glass, chemicals, paper, detergents and many other products. It has been used since ancient times. The Egyptians, for instance, made glass containers from soda ash as early as 3500 BC. And the early Romans expanded its use beyond glass as an ingredient in medicinals and bread.



Figure 1-1: Green River Soda Ash Facility

Much of the world's supply of natural soda ash comes from trona ore. The largest known trona deposits are found in the Green River Basin, a prehistoric alkaline lakebed in southwest Wyoming known to geologists as the Gosiute Lake. It is here, on 67 billion tons of trona deposits, that General Chemical established its Green River facility in 1968. This facility has been expanded over the years and now has a nameplate capacity of 2.8 million tons.

The Green River facility converts trona ore to soda ash in a multi-step purification process. First, crushed

trona is heated in a kiln to drive off unwanted gases. This transforms the ore to crude sodium carbonate. Water is added, and the solution is filtered to remove impurities. The liquid is then boiled off to form crystals, which are separated in a centrifuge and dried.

Soda ash and materials made from it can be found in most industries. It is such a basic material that government and commerce agencies use soda ash production statistics to gauge the health of the economy. U.S. industry consumes soda ash in approximately the ratios shown in Figure 1-2 below:

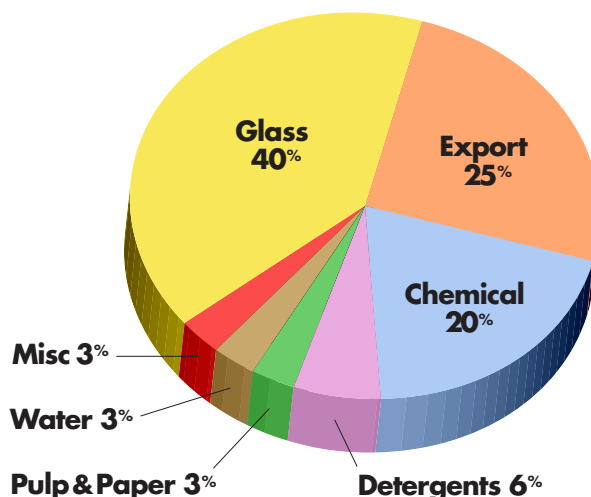


Figure 1-2: US Soda Ash Markets Sectors

General Chemical has been a soda ash producer for more than 100 years and uses the practical experience gained during this time to support its customers in shipping, storing and using soda ash. The following document offers an overview of this knowledge base.

2 Properties

Soda ash is a white, anhydrous, powdered or granular material containing more than 99% sodium carbonate (Na_2CO_3) when shipped. The accepted commercial standard for soda ash is expressed in terms of the equivalent sodium oxide (Na_2O) content. A 99.5% soda ash is equivalent to 58.2% Na_2O (the conversion equation is: % $\text{Na}_2\text{CO}_3 \times 0.585 = \% \text{Na}_2\text{O}$).

Soda ash is an alkali that has a high pH in concentrated solutions. It can irritate the eyes, respiratory tract and skin. It should not be ingested, because it can corrode the stomach lining.

Soda ash is made in three main grades — light, intermediate and dense. These differ only in physical characteristics, such as bulk density and particle size and shape (which affects flow characteristics and angle

of repose). Other physical and chemical properties are common to all grades, whether in solid or liquid form. These are similar to those given for pure sodium carbonate in standard reference books and other sources from which much of the data that follow are derived,

Temperature, °C	Density, kg/m ³
851 (melting point)	1970
900	1940
1000	1910

Table 2-2: Densities at Temperatures Above the Melting Point ²

e.g., the properties in Table 2-1 and the densities, dissociation pressures and heats for formation, hydration and solution in Tables 2-2 through 2-5.

Decomposition on Heating

Anhydrous sodium carbonate loses weight when heated due to dissociation and volatilization according to the following reaction:



Dissociation pressure rises with increasing temperature (Table 2-3).

Temperature, °C	Disassociation Pressure, mm Hg
1000	1.5 ±0.5
1100	5.5 ±1.0
1200	14.0 ±0.5
1300	20.0 ±1.0
1400	66.0

Table 2-3: Disassociation Pressures of Soda Ash ³

Chemical formula and name:	Na_2CO_3 Sodium carbonate, anhydrous
Molecular weight:	105.989
Absolute density:	2533 g/L @ 25°C (See also Table 2-2)
Melting point:	851°C (1564°F)
Decomposition on heating:	See Table 2-3
Specific heat:	1042 J/kg•K (0.249 cal/g•°C or Btu/lb•°F)
Heat of fusion:	315,892 J/kg ⁽¹⁾ (75.5 cal/g or 136 Btu/lb)
Heat of formation from the elements:	10.67×10^6 J/kg (2550 cal/g or 4590 Btu/lb)
Heat of hydration:	See Table 2-4
Heat of solution:	See Table 2-5
Stability in air:	Slowly absorbs moisture and carbon dioxide to form sodium sesquicarbonate ($\text{Na}_2\text{CO}_3 \cdot \text{NaHCO}_3 \cdot 2\text{H}_2\text{O}$)
Bulk density:	See Table 2-12

Table 2-1: Properties of Soda Ash

Hydrates of Sodium Carbonate

Sodium carbonate has three hydrate forms: Sodium carbonate monohydrate, heptahydrate and decahydrate.

Sodium carbonate monohydrate ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) contains 85.48% Na_2CO_3 and 14.52% water of crystallization. It separates as small crystals from saturated

Properties

aqueous solutions above 35.4°C (95.7°F). It can be formed by wetting soda ash with a calculated quantity of water at or above this temperature. It loses water on heating, and its solubility decreases slightly with increasing temperature. It converts to Na_2CO_3 upon contact with its saturated solution at 109°C (228°F).

Sodium carbonate heptahydrate ($\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$) contains 45.7% Na_2CO_3 and 54.3% water of crystallization. It is of no commercial interest because its stability range only extends from 32.0° to 35.4°C (89.6° to 95.7°F).

Sodium carbonate decahydrate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$), commonly called “sal soda” or “washing soda,” usually forms large, transparent crystals containing 37.06% Na_2CO_3 and 62.94% water of crystallization. It can be crystallized from saturated aqueous solutions between -2.1 and 32.0°C (28.2 and 89.6°F) or by wetting soda ash with a calculated quantity of water in this temperature range. The crystals readily effloresce in dry air, forming a residue of lower hydrates (principally the monohydrate form).

Heat of Solution

Heat is released when anhydrous or monohydrate sodium carbonate dissolve in water. Heat is absorbed when the heptahydrate or decahydrate forms dissolve in water. The stronger the concentration, the greater the heat released or absorbed per unit of Na_2CO_3 dissolved. Use Fig. 2-1 to calculate the heat absorbed when diluting a sodium carbonate solution. For example, when a 25% solution is diluted to 10%, temperature decreases through the absorption of:

$$131.7 - 114.3 = 17.5 \text{ Btu/lb. Na}_2\text{CO}_3.$$

When soda ash is dissolved in water to form a 32% saturated solution, 135 Btu/lb. of heat is released (Fig. 2-1). As additional soda ash is added to the saturated solution, monohydrate crystals are formed. This heat of formation is 54 Btu/lb (Table 2-4). If equal weights of soda ash and water are mixed, forming a

	Monohydrate $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	Heptahydrate $\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$	Decahydrate $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$
Heat of formation from the elements, J/kg cal /g Btu / lb	11.531 x 10 ³ 2756 4960	13.790 x 10 ³ 3296 5932	14.263 x 10 ³ 3409 6138
Heat of hydration, J/kg cal /g Btu / lb	125.5x 10 ³ 30.0 54.0	654.4 x 10 ³ t 56.4 281.5	873.6x10 ³ 208.8 375.8

Table 2-4: Heat of Formation and Hydration at 25°C⁴

Solute			Heat Quantity		
Formula	grams	% Na_2CO_3	J/kg ⁽⁴⁾ Na_2CO_3	cal/g Na_2CO_3	Btu/lb Na_2CO_3
Na_2CO_3	105.989	2.86	231.8 x 10 ³	55.4	99.7
$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	124.004	2.84	106.2 x 10 ³	25.4	45.6
$\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$	237.094	2.76	-422.6 x 10 ³	-101.0	-181.9*
$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$	286.139	2.73	-641.8 x 10 ³	-153.4*	-276.1

Table 2-5: Heat of Solution at 25°C
(One Mole of Sodium Carbonate Dissolved in 200 Moles H_2O)

50% slurry, about 42% of the soda ash dissolves to form a saturated 32% solution. The remaining 58% soda ash forms monohydrate crystals. The total heat developed in preparing a 50% slurry is:

$$(0.42 \times 134) + (0.58 \times 54) = 88 \text{ Btu/lb. of soda ash.}$$

When more water is added to the slurry, monohydrate dissolves to saturate the water. For example, when one gallon of water is added to soda ash slurry, about 4 pounds of soda ash will dissolve (4.7 lbs. of monohydrate). Subtracting the heat of formation from the heat of solution gives the net heat released by dissolving a saturated monohydrate slurry with 1 gallon of water:

$$4 \times (134 - 54) = 282 \text{ Btu of heat.}$$

Properties

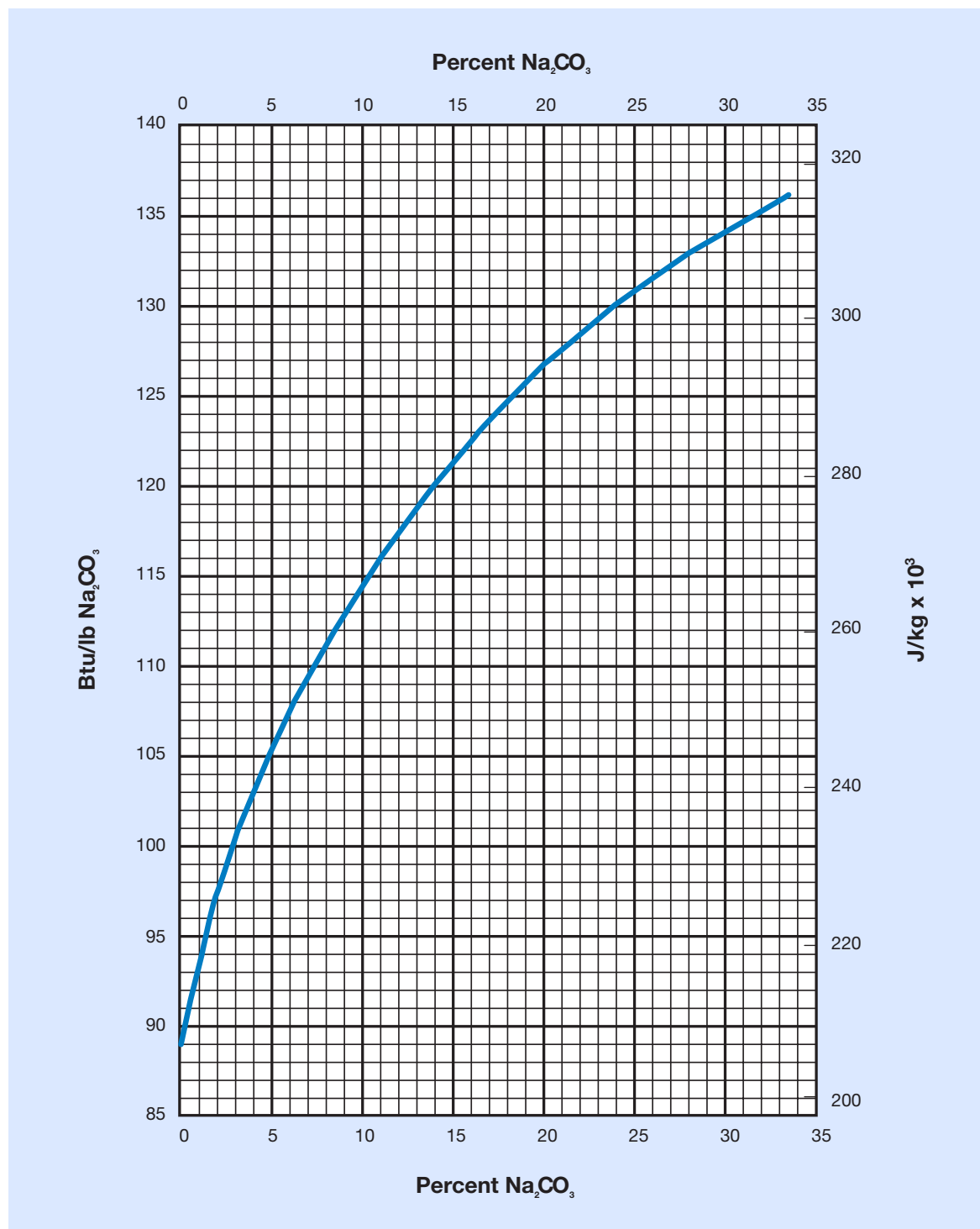


Figure 2-1: Heat Evolved in Dissolving Soda Ash to Form Solutions of Varying Concentrations @ 25°C³

Properties

Solubility

Sodium carbonate, although readily soluble in water, is unusual in that it reaches maximum solubility at the relatively low temperature of 35.4°C (95.7°F). At this point, 100 parts of water dissolves 49.7 parts of Na_2CO_3 to yield a 33.2% solution by weight. Solubility decreases above this temperature, so there are two saturation temperatures for concentrations between about 29% and 33.2%. The phase diagram (*Fig. 2-2*) portrays this relationship by tracing solubility (as % Na_2CO_3) between -2.1 and 109°C (28.2 and 228.2°F).

Solubility data above about 105°C represent solutions held above atmospheric in order to prevent boiling, since the boiling point-concentration curve crosses the solubility curve at about 105°C. Unsaturated solutions exist in the area above and to the left of this curve.

The region below and to the right of the curve contains either undissolved solids in contact with saturated solutions or solids alone (*Table 2-6*).

This diagram helps trace the effects of cooling, heating and changing concentrations. For example, a 20% Na_2CO_3 solution is unsaturated at all temperatures above 22.4°C (72.3°F), which is where the 20% concentration line crosses the saturation curve. Below this temperature, the solid phase ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$) begins to form, increasing in amount as temperature falls. This phase change causes the concentration of the saturated solution in contact with the crystals to decrease, until at -2.1°C (28.2°F), the liquid phase disappears leaving only a mixture of solid $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and ice.

Area	Percent Soda Ash		Temperature, °C			Solid Phases	Liquid Phase Percent Soda Ash in Solution		Transition Points	
	from	to	At and Below	from	to		from	to	Percent Soda Ash	°C
A	0	5.8		0	-2.1	ICE	0	5.8		
B	0	37.0	-2.1			ICE + $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$	None		5.8	-2.1
C	5.8	37.0		-2.1	32.0	$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$	5.8	31.2		
D	37.0	45.7	32.0			$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ + $\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$	None		31.2	32.0
E	31.2	45.7		32.0	35.4	$\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$	31.2	33.2		
F	45.7	85.5	35.4			$\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$ + $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	None		33.2	35.4
G	30.8	85.5		35.4	109.0	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	33.2	30.8		
H	85.5	100.0	109.0			$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ + Na_2CO_3	None		30.8	109.0
I	30.8	100.0		109 and above		Na_2CO_3	30.8			

Table 2-6: Phase Diagram Defined (see Figure 2-2)

Properties

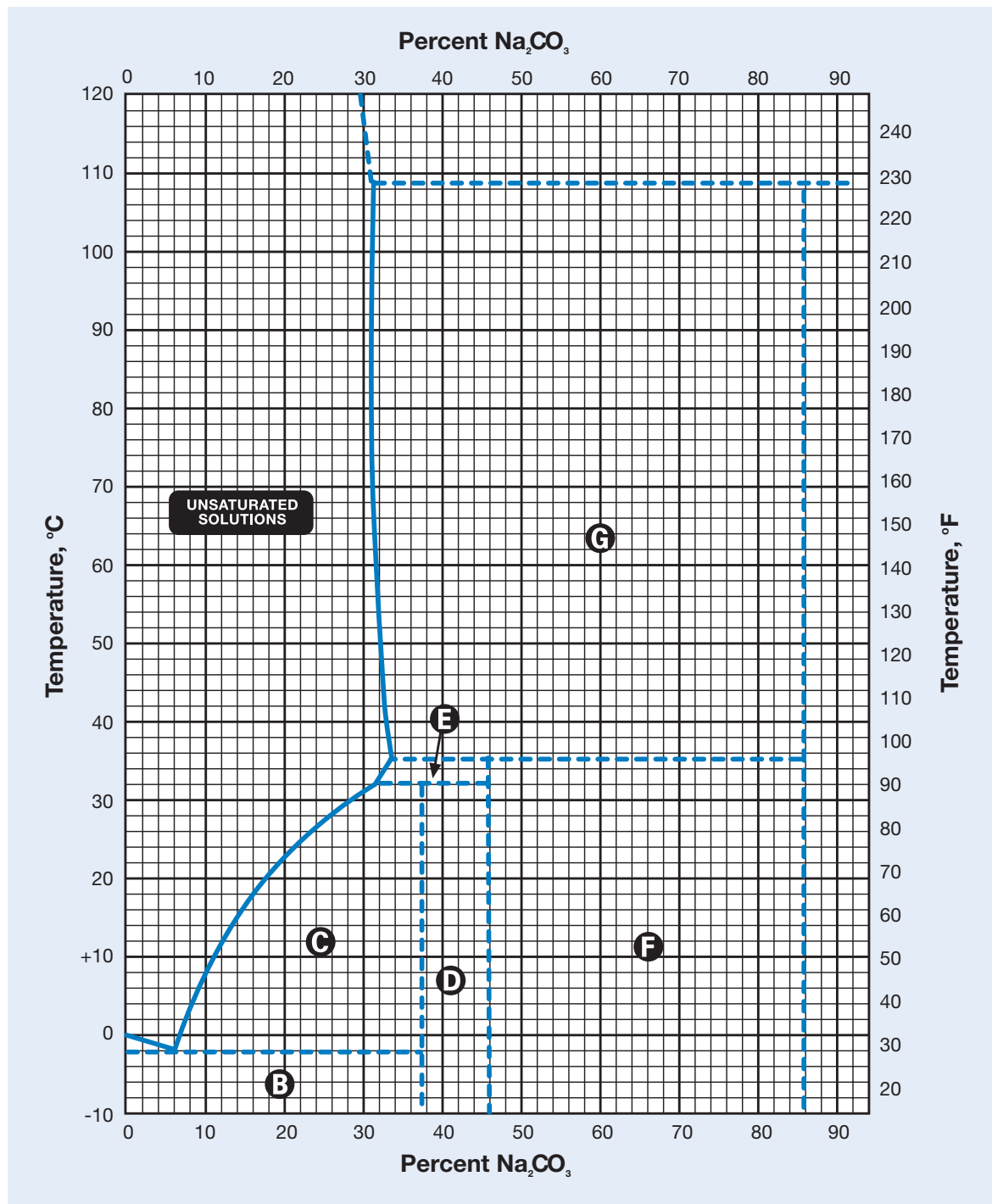


Figure 2-2: Phase Diagram for Aqueous Soda Ash Solutions

Properties

The composition of such a solidified mass may be calculated from:

$$\frac{(A - C)}{(B - C)} \times 100 = W$$

where: A = % Na_2CO_3 in the original mixture.

B = % Na_2CO_3 in the richer component of the mixture at the final temperature.

C = % Na_2CO_3 in the leaner component of the mixture at the final temperature.

W = weight % of the richer component in final mixture.

In the above example, where a 20% Na_2CO_3 solution is cooled below -2.1°C so it solidifies, the richer component of the final mixture ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$) has 37.0% Na_2CO_3 and the leaner component (ice) has 0% Na_2CO_3 . The final mixture then contains:

$$\frac{(20 - 0)}{(37 - 0)} \times 100 = 54.1\% \text{ Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$$

and the rest is solid ice.

Referring to the phase diagram (Fig. 2-2), a mixture of 40% Na_2CO_3 and water at 50°C contains $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ crystals (85.5% Na_2CO_3) in equilibrium with its saturated solution (32% Na_2CO_3). The physical composition of the mixture is:

$$\frac{(40 - 32)}{(85.5 - 32)} \times 100 = 15\% \text{ Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$$

and 85% is saturated solution.

Cooling this mixture to 35°C changes the solid phase from $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ to solid $\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$ containing 45.7% Na_2CO_3 in contact its saturated solution (33% Na_2CO_3). The mixture now consists of:

$$\frac{(40 - 33)}{(45.7 - 33)} \times 100 = 55\% \text{ Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$$

and 45% is saturated solution.

If the mixture is cooled below 32°C , it solidifies to a mixed solid, which contains $\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$ (45.7% Na_2CO_3) and $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ (37.0% Na_2CO_3) in the proportion of:

$$\frac{(40 - 37.0)}{(45.7 - 37.0)} \times 100 = 34.5\% \text{ Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$$

and 65.5% $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$.

The solubility of soda ash in the presence of appreciable amounts of foreign salts, such as sodium chloride, sodium sulfate and sodium nitrate, changes how well the phase diagram in Fig. 2-2 applies. See the International Critical Tables or the Solubilities of Inorganic and Metal Organic Compounds by A Seidell, 4th Edition, 1958 (Vol. 1) and 1965 (Vol. 11) for the effects of salts on the solubility of sodium carbonate.

Solution Specific Gravity and Density

Soda ash has a solubility limit of 14.5% Na_2CO_3 at 15.6°C (60°F). Table 2-7 lists densities at 15.6°C and specific gravities for concentrations of sodium carbonate up to 14.0% (from the International Critical Tables⁵).

Higher concentrations are possible above 15.6°C . The specific gravities of saturated solutions above 15.6°C lie on a smooth curve (Fig. 2-3)⁶. Table 2-8 lists values at saturation for concentrations of 15% and above. Crystallization occurs when temperature falls below that shown for these solutions.

The specific gravity of sodium carbonate solutions decreases with increasing temperature. Concentration can be determined if solution specific gravity and temperature are known (Table 2-8 and Fig. 2-4).

Properties

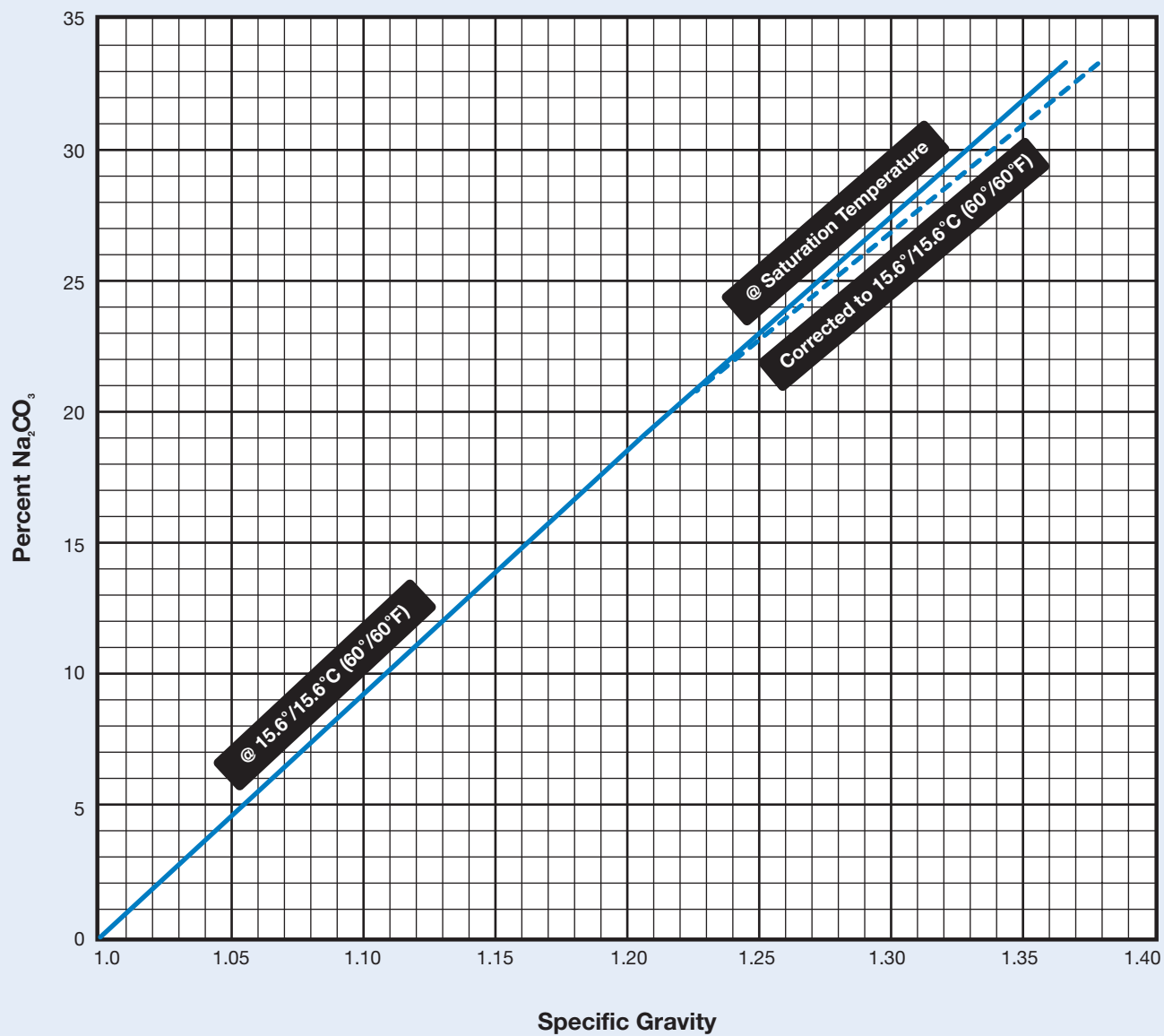


Figure 2-3: Specific Gravity of Soda Ash Solutions

Properties

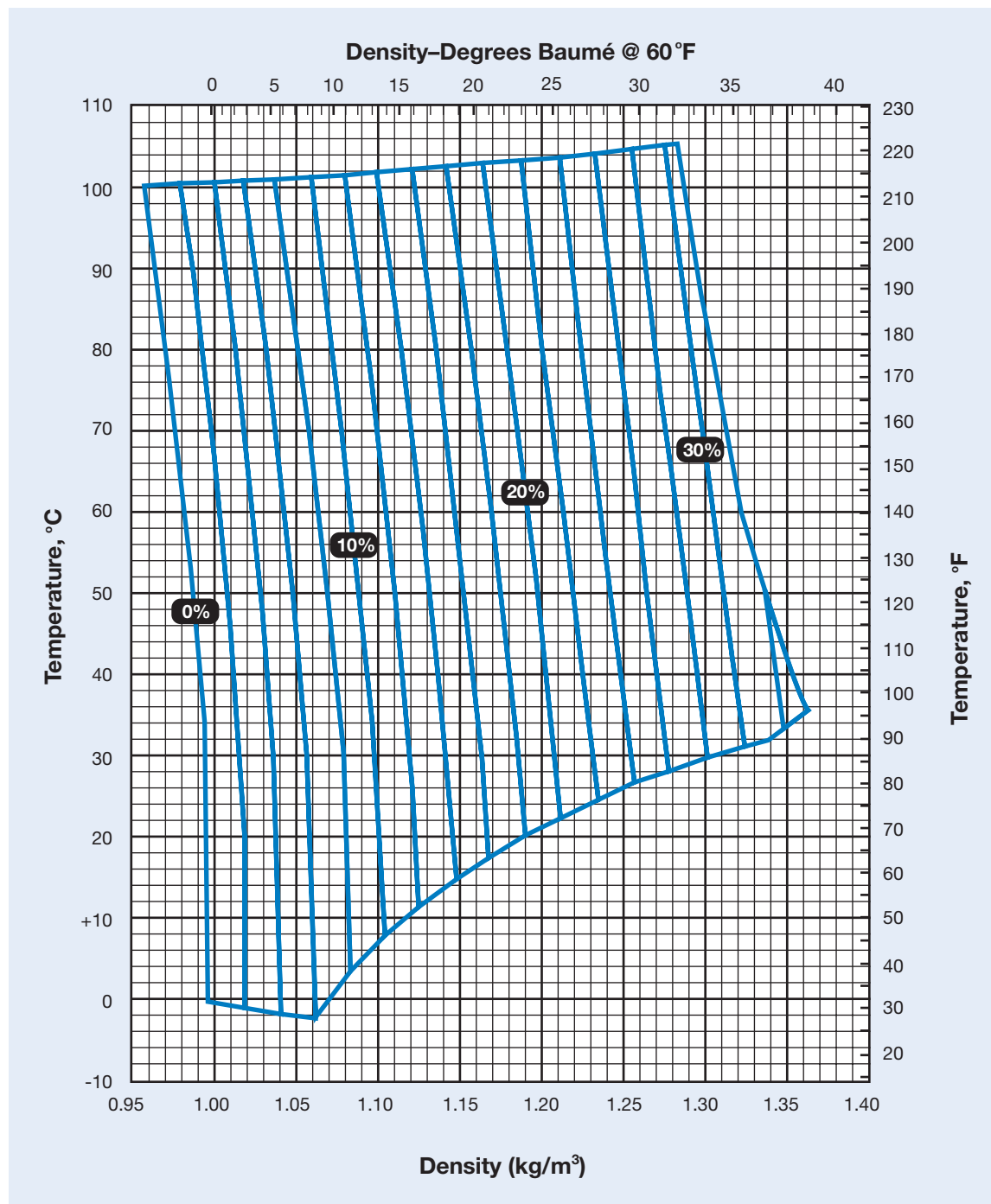


Figure 2-4: Concentration of Sodium Carbonate Solutions at Varying Densities and Temperatures

Properties

Specific Heat

Figure 2-5 shows the specific heat of sodium carbonate solutions at 20°C⁷ and at 30 and 76.6°C⁸. For example, the heat required to raise the temperature of 1000 gallons of 10% Na₂CO₃ solution from 68°F to 170°F is

calculated from:

$$A \times B \times C = Q$$

where A = weight of solution = 9924 lb.

$$B = \text{temperature rise} = 170 - 68 = 102^{\circ}\text{F}$$

C = mean specific heat (Btu/lb.°F)

Q = quantity of heat in Btu's

A = (gal. of Na₂CO₃ solution) x (8.34 lb./gal.) x
(SpG of Na₂CO₃ solution) = lb. of Na₂CO₃
solution, or

$$1000 \times 8.34 \times 1.106 = 9224 \text{ lb. Na}_2\text{CO}_3 \text{ solution}$$

$$C = \frac{0.902 (\text{sp. heat @ } 68^{\circ}\text{F}) + 0.945 (\text{sp. heat @ } 170^{\circ}\text{F})}{2} = 0.924 \text{ Btu/lb.}^{\circ}\text{F}$$

$$Q = 9224 \times 102 \times 0.924 = 869 \times 10^3 \text{ Btu}$$

Percent Sodium Carbonate (Na ₂ CO ₃)	Percent Equivalent Sodium Oxide (Na ₂ O)	Specific Gravity at 15.6°/15.6°C	Kilograms Sodium Carbonate per Cubic Meter at 15.6°C	Degrees Baumé at 60° F (Am.Std.)	Weight of One Gallon at 60° F (Pounds)	Pounds Sodium Carbonate per Gallon at 60° F
1	0.6	1.010	10.1	1.4	8.42	0.08
2	1.2	1.021	20.4	3.0	8.51	0.17
3	1.8	1.032	30.9	4.4	8.60	0.26
4	2.3	1.042	41.6	5.9	8.69	0.35
5	2.9	1.053	52.6	7.2	8.78	0.44
6	3.5	1.063	63.7	8.6	8.86	0.53
7	4.1	1.074	75.1	10.0	8.95	0.63
8	4.7	1.085	86.7	11.3	9.05	0.72
9	5.3	1.095	98.4	12.6	9.13	0.82
10	5.8	1.106	110.5	13.9	9.22	0.92
11	6.4	1.116	122.6	15.1	9.30	1.02
12	7.0	1.127	135.1	16.4	9.40	1.13
13	7.6	1.138	147.8	17.6	9.49	1.23
14	8.2	1.149	160.7	18.8	9.58	1.34

Table 2-7: Specific Gravities and Densities of 0-14% Soda Ash Solutions at 15.6°C (60°F)

Percent Sodium Carbonate (Na ₂ CO ₃)	Percent Equivalent Sodium Oxide (Na ₂ O)	Temperature at Saturation		Specific Gravity at Saturation	Kilograms Sodium Carbonate per Cubic Meter	Equivalent Degrees Baumé	Weight of One Gallon (Pounds)	Pounds Sodium Carbonate per Gallon
		°C	°F					
15	8.8	16.2	61.2	1.160	173.8	20.0	9.67	1.45
16	9.4	17.5	63.5	1.170	187.0	21.1	9.75	1.56
17	9.9	18.8	65.8	1.181	200.5	22.2	9.85	1.67
18	10.5	20.1	68.2	1.192	214.3	23.4	9.94	1.79
19	11.1	21.3	70.3	1.203	228.3	24.5	10.03	1.91
20	11.7	22.4	72.3	1.214	242.5	25.6	10.12	2.02
21	12.3	23.5	74.3	1.225	257.0	26.6	10.21	2.14
22	12.9	24.5	76.1	1.236	271.6	27.7	10.30	2.27
23	13.4	25.5	77.9	1.247	286.4	28.7	10.40	2.39
24	14.0	26.4	79.5	1.258	301.5	29.7	10.49	2.52
25	14.6	27.2	81.0	1.269	316.9	30.7	10.58	2.65
26	15.2	28.0	82.4	1.280	332.4	31.7	10.67	2.77
27	15.8	28.8	83.8	1.292	348.4	32.8	10.77	2.91
28	16.4	29.6	85.3	1.304	364.7	33.8	10.87	3.04
29	17.0	30.4	86.7	1.315	380.8	34.7	10.96	3.18
30	17.5	31.1	88.0	1.326	397.4	35.6	11.05	3.32
31	18.1	31.8	89.2	1.338	414.3	36.6	11.15	3.46
32	18.7	33.2	91.8	1.350	431.5	37.6	11.25	3.60
33	19.3	34.8	94.6	1.361	448.7	38.5	11.35	3.74

Table 2-8: Specific Gravities and Densities of 15% and Higher Soda Ash Solutions @ Saturation Temperatures.

Properties

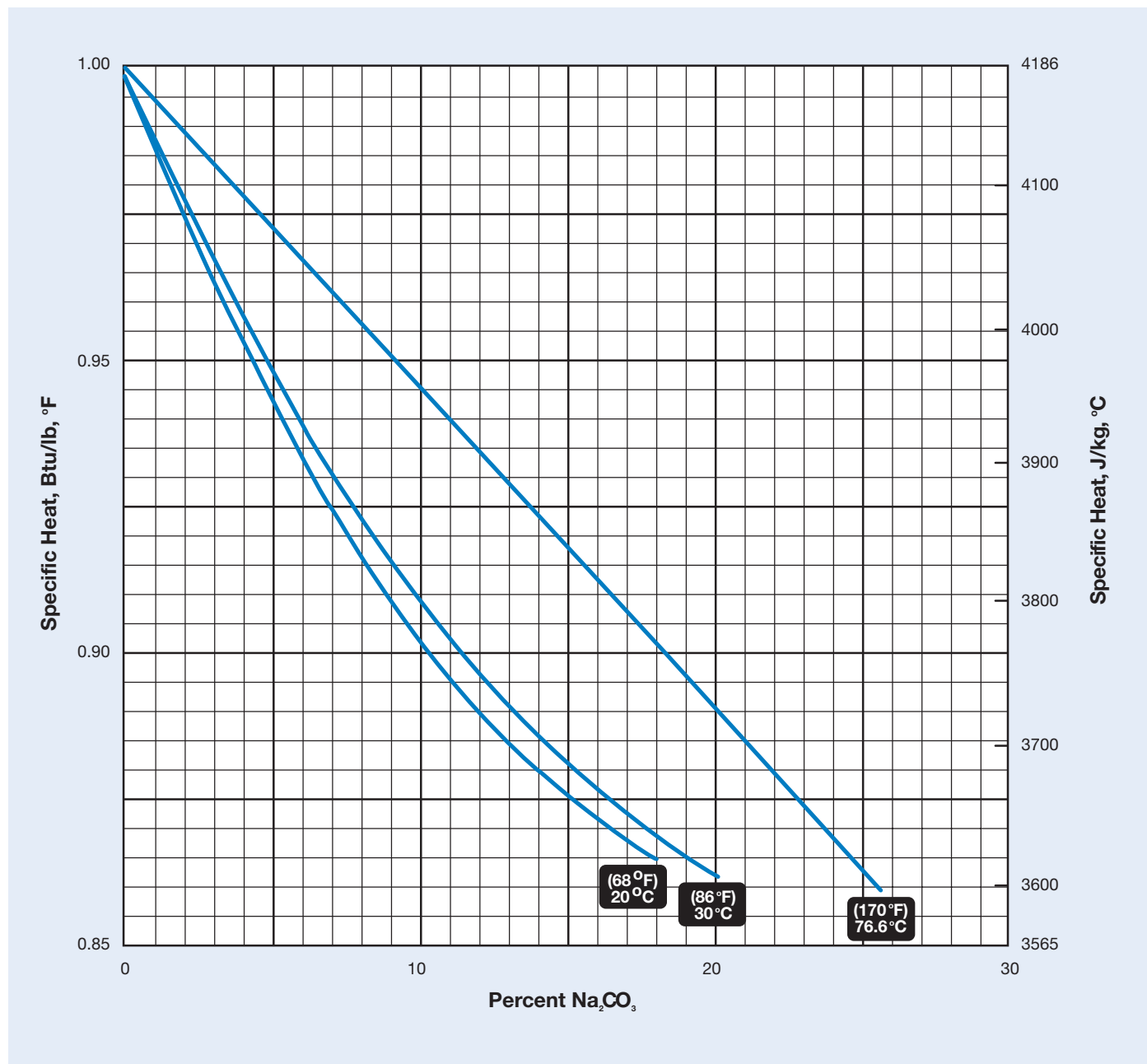


Figure 2-5: Specific Heat of Soda Ash Solutions

Properties

Vapor Pressure

Table 2-10 shows vapor pressures and boiling points for sodium carbonate solutions⁹ with values for saturated solutions in parentheses¹⁰. Approximate values for vapor pressures in the upper temperature ranges may be interpolated using Fig. 2-6 by extending a straight line from the temperature line through the % Na₂CO₃ line to the vapor pressure line. The example in Fig. 2-6 (dashed line) shows the vapor pressure of a 10% sodium carbonate solution at 90°C (194°F) to be 67,500 Pascals (506 mm of mercury).

Viscosity

Viscosity is important for designing pumping and piping systems and for calculating heat transmission and gas absorption in chemical processes. The viscosity of a soda ash solution is determined from Fig. 2-7 by extending a straight line from the % soda ash line through the temperature curve to the viscosity line. The example in Fig. 2-7 (dashed line) shows the viscosity of a 22% sodium carbonate solution of 24°C (75°F) to be approximately 4.0 centipoises (0.004 Pascal seconds).

Percent Na ₂ CO ₃	Temperature					
	0°C (32°F)	20°C (68°F)	40°C (104°F)	60°C (140°F)	80°C (176°F)	100°C (212°F)
0	1000	998	992	983	971	958
1	1010	1009	1003	995	983	970
2	1022	1020	1015	1006	994	982
3	1033	1031	1025	1016	1004	992
4	1044	1042	1035	1026	1014	1002
5	1054	1052	1045	1036	1024	1011
6	1065	1062	1055	1046	1034	1020
7		1072	1065	1055	1043	1030
8		1083	1076	1065	1053	1040
9		1093	1086	1075	1063	1050
10		1104	1096	1086	1073	1060
11		1115	1107	1096	1084	1071
12		1126	1117	1106	1094	1082
13		1137	1128	1117	1104	1092
14		1148	1139	1128	1115	1102
15		1158	1150	1138	1126	1113
16		1170	1161	1149	1137	1124
17		1181	1172	1160	1147	1134
18		1192	1183	1171	1158	1145
19			1194	1182	1170	1157
20			1205	1193	1181	1168
21			1216	1204	1192	1180
22			1227	1215	1203	1191
23			1238	1226	1214	1203
24			1250	1238	1226	1214
25			1261	1249	1237	1225
26			1273	1261	1249	1237
27			1285	1272	1260	1248
28			1297	1284	1272	1259
29			1309	1296	1283	1269
30			1320	1307	1294	1270
31			1332	1318	1304	
32			1345			

Table 2-9: Densities of Soda Ash Solutions (kg/m³)

Properties

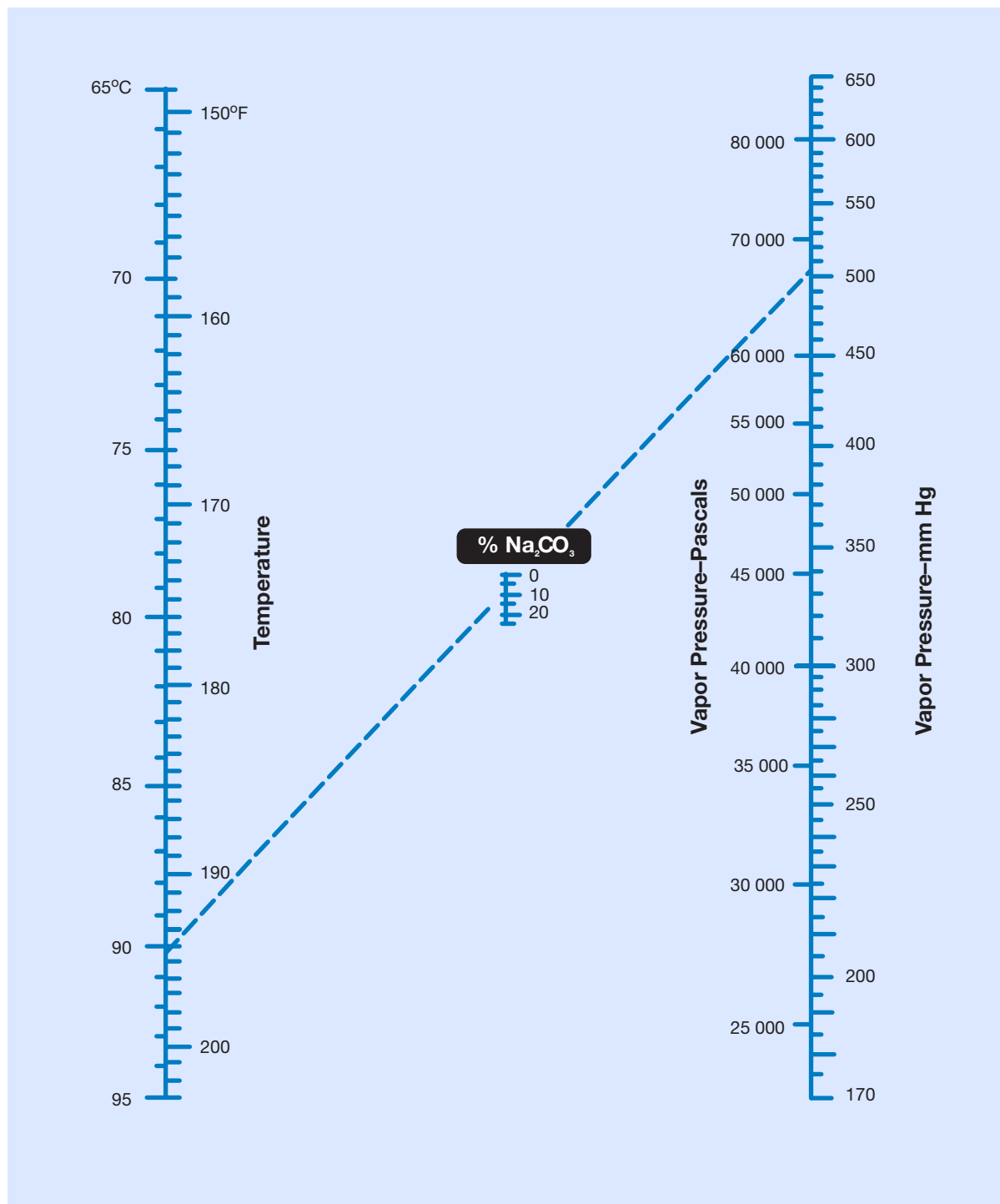


Figure 2-6: Vapor Pressure of Soda Ash Solutions ¹¹

Properties

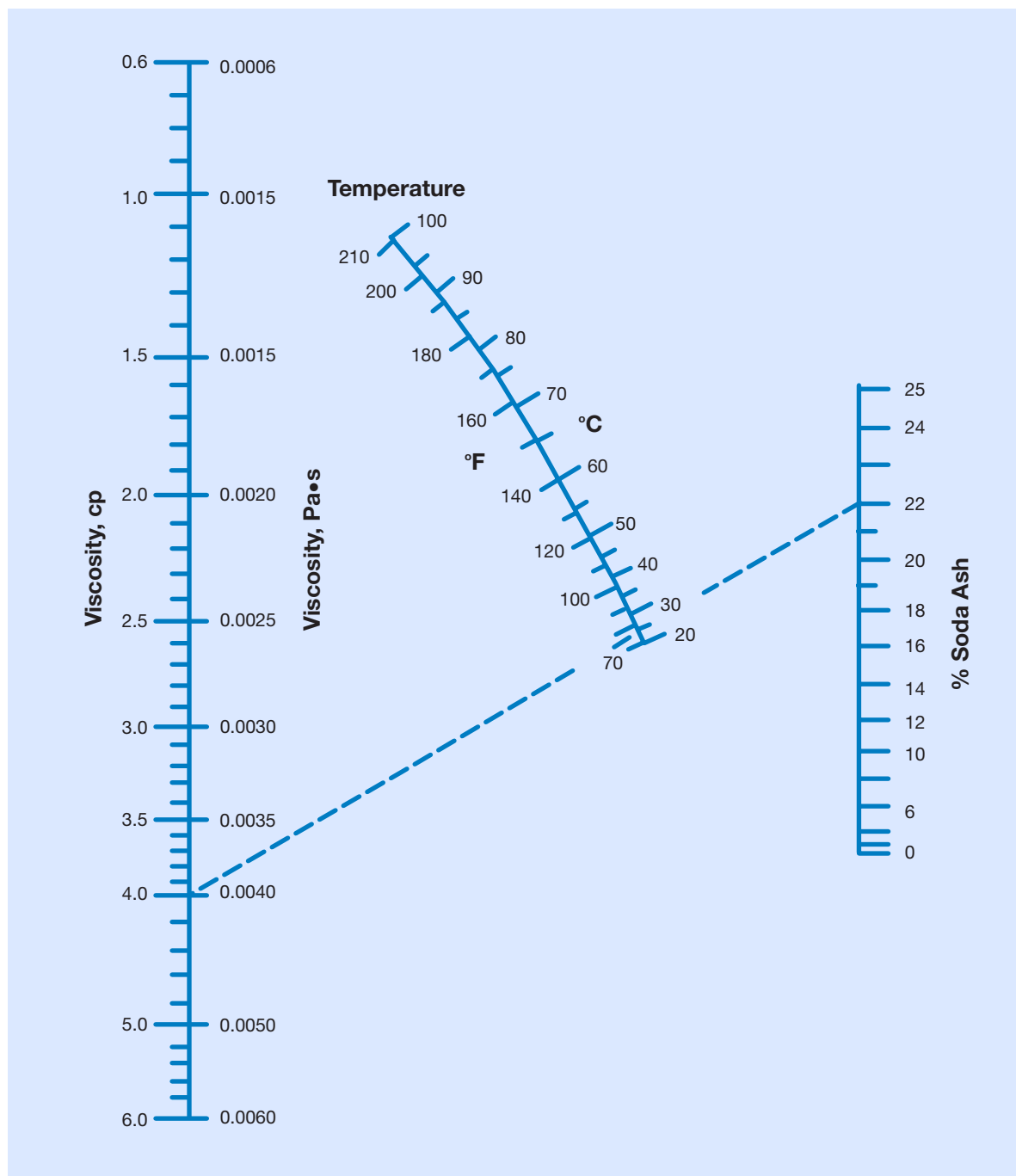


Figure 2-7: Viscosity of Soda Ash Solutions ¹²

Properties

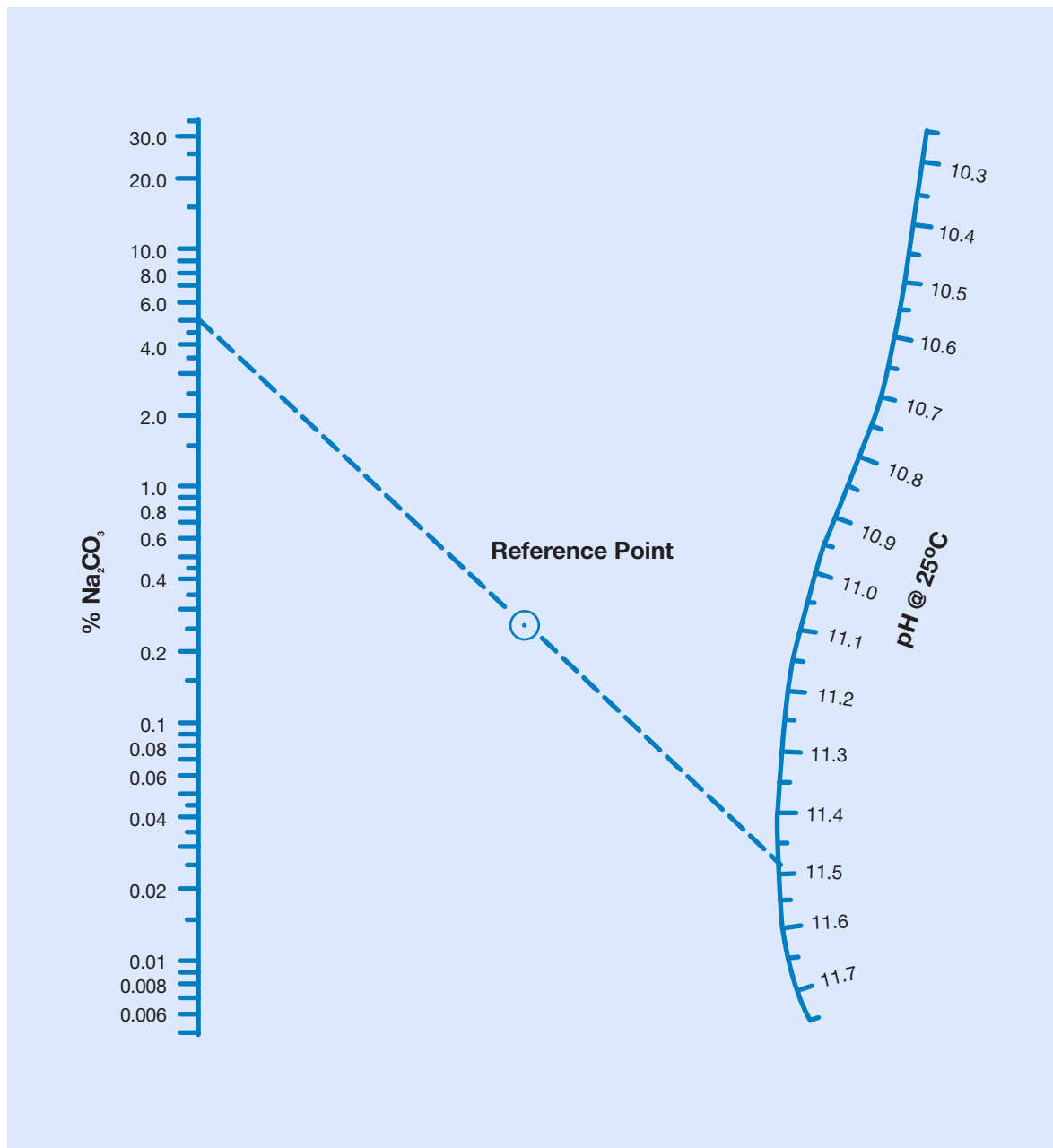


Figure 2-8: *pH of Soda Ash Solutions*¹³

Properties

Temperature		Percent Soda Ash Solution													
°C	°F	0.0%		5.0%		10.0%		15.0%		20.0%		25.0%		30.0%	
		mmHg	pascals	mmHg	pascals	mmHg	pascals	mmHg	pascals	mmHg	pascals	mmHg	pascals	mmHg	pascals
0	32	4.5	610	4.5	600										
10	50	9.2	1 230	9.0	1 200	8.8	1 170								
20	68	17.5	2 330	17.2	2 290	16.8	2 240	16.3	2 170						
30	86	31.8	4 240	31.2	4 160	30.4	4 050	29.6	3 950	28.8	3 840	27.8	3 710	(26.5 @ 28.6%)	(3 530 @ 28.6%)
40	104	55.3	7 370	54.2	7 230	53.0	7 070	51.6	6 880	50.2	6 690	48.4	6 450	46.1 (43.2 @ 32.6%)	6 150 (5 760 @ 32.6%)
50	122	92.5	12 330	90.7	12 090	88.7	11 830	86.5	11 530	84.1	11 210	81.2	10 830	77.5	10 330
60	140	149.5	19 930	146.5	19 530	143.5	19 130	139.9	18 650	136.1	18 150	131.6	17 550	125.7	16 760
70	158	239.8	31 970	235.0	31 330	230.5	30 730	225.0	30 000	219.0	29 200	211.5	28 200	202.5	27 000
80	176	355.5	47 400	348.0	46 400	342.0	45 600	334.0	44 530	325.0	43 330	315.0	42 000	301.0	40 130
90	194	526.0	70 130	516.0	68 790	506.0	67 460	494.0	65 860	482.0	64 260	467.0	62 260	447.0	59 590
100	212	760.0	101 320	746.0	99 460	731.0	97 460	715.0	95 330	697.0	92 930	676.0	90 130	648.0	86 390
Boiling temperature at 760 mm															
°C		100.0		100.6		101.2		101.9		102.7		103.8		105.1	
°F		212.0		213.1		214.2		215.4		216.9		218.8		221.2	
		(Extrapolation of the above vapor pressure values)													

Table 2-10: Vapor Pressures and Boiling Points of Soda Ash Solutions

Hydrogen Ion Concentration (pH)

Precise and accurate determination of pH values for sodium carbonate solutions requires electrometric measurement with glass electrodes. These should be designed for use in alkaline sodium salt solutions and measure over the entire pH range. Organic color indicators are not recommended for pH measurement in sodium carbonate solutions (see Section 9). The pH of sodium carbonate solutions can be interpolated from Fig. 2-8¹³ by extending a straight line from the % Na₂CO₃ line through the reference point to the pH line as shown by the dashed line on the chart.

Electrical Conductivity

Electrical conductivity instruments may be designed for the measurement and control of sodium carbonate concentrations, particularly when applied to concentrations in the lower ranges (Table 2-11).

Specific Conductance—Siemens (mhos) /cm				
Concentration	0°C	18°C	25°C	35°C
0.010N	0.00054 ⁽¹⁴⁾	0.00096 ⁽¹⁴⁾	0.00108 ⁽¹⁴⁾	0.00132 ⁽⁶⁾
0.100N	0.00437 ⁽¹⁴⁾	0.00728 ⁽¹⁴⁾	0.00853 ⁽¹⁴⁾	0.01050 ⁽⁶⁾
1.000N	0.0270 ⁽¹⁴⁾	0.0454 ⁽¹⁴⁾	0.0534 ⁽¹⁴⁾	0.0649 ⁽⁶⁾
2.000N		0.0690 ⁽¹⁴⁾	0.08088 ⁽⁶⁾	0.1000 ⁽⁶⁾
3.000N		0.0810 ⁽¹⁴⁾	0.09656 ⁽⁶⁾	0.1211 ⁽⁶⁾
4.000N			0.1027 ⁽⁶⁾	0.1311 ⁽⁶⁾
5.000N			0.1016 ⁽⁶⁾	0.1298 ⁽⁶⁾
6.000N				0.1250 ⁽⁶⁾
Concentration	Temperature °C	Specific Conductance Siemens (mhos) /cm		
Saturated	25	0.103 ⁽¹⁵⁾		
"	30	0.099 ⁽¹⁵⁾		
"	35	0.102 ⁽¹⁵⁾		
"	43	0.134 ⁽¹⁵⁾		
"	50	0.167 ⁽¹⁵⁾		
"	64	0.234 ⁽¹⁵⁾		
"	75	0.286 ⁽¹⁵⁾		
"	90	0.366 ⁽¹⁵⁾		

Table 2-11: Electrical Conductivity of Soda Ash Solutions

Properties

Bulk Density

The bulk density (weight of dry soda ash per unit volume) varies with the form of ash and the handling it receives. Typical bulk densities are shown in Table 2-12.

	Loose; carefully poured		Vibrated and jarred		For engineering purposes			
					Capacity of bins		Calculation of strength of structure	
	lbs/ft ³	kg/m ³	lbs/ft ³	kg/m ³	lbs/ft ³	kg/m ³	lbs/ft ³	kg/m ³
Light Soda Ash	35	560	47	760	33	530	46	740
Natural Light	48	770	61	970	45	720	63	1010
Natural Light HA	45	720	57	910	42	670	59	950
Dense Soda Ash	65	1040	76	1220	60	960	85	1370
Volumes occupied	<p>One short ton of dry soda ash in bulk storage:</p> <p>Light soda ash60 ft³</p> <p>Natural Light Soda Ash45 ft³</p> <p>Natural Light HA Soda Ash48 ft³</p> <p>Dense soda ash34 ft³</p> <p>One metric ton of dry soda ash in bulk storage:</p> <p>Light soda ash1.9 m³</p> <p>Natural Light Soda Ash1.4 m³</p> <p>Natural Light HA Soda Ash1.5 m³</p> <p>Dense soda ash1.1 m³</p>							

Table 2-12: Bulk Density of Dry Soda Ash

References

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3 Shipments

Paper Bags

Light and dense soda ash are normally packaged in plastic or polyethylene-lined, multi-wall paper bags holding 25 kg or 50 lb. (22.7 kg). The product is typically shipped in boxcars, closed van-type trucks or on flatbed trucks, if carefully protected from the weather with tarpaulins.

Bags are shipped on non-returnable wood pallets and stacked in an interlocking pattern for stability in transit and handling. A typical unit load has 45 to 55 bags, depending on packaging configuration and bulk density, and measures no more than three to four feet in length or width. The number of bags may vary from less than 18 to more than 24 per pallet.

Bulk Railcars

Covered hopper cars with bottom discharge are the most common rail cars used for bulk soda ash. They have weather-tight steel roofs fitted with hinged circular or trough hatches for loading. Their sloping bottoms are essentially self-cleaning. They usually have three compartments, each with a single outlet on the centerline of the car. The outlets are closed by tight-fitting, horizontal slide gates opened by a rack-and-pinion or scissors type mechanism. Hopper car sizes appear in Table 3-1.

Most covered hopper cars discharge by gravity. General Chemical Industrial Chemicals also offers pneumatic discharge (PD) railcars, if a pressurized off-loading is necessary.

	Volume capacities	Weight capacities
	ft ³	lbs
Light soda ash	4700-5700	160,000-185,000
Dense soda ash	4700-4750	195,000-200,000

Table 3-1: Hopper Car Capacity Ranges

Bulk Trucks

Trucks used for bulk soda ash are like those for cement and other dry bulk materials. The most popular are self-unloading, pneumatic trailers such as hopper pressure tanks. These have air compressors that can blow the 15 to 25-short-ton (14 to 23 metric-ton) load they carry into storage bins in one to two hours. Gravity-unload hopper trucks are also available. Maximum weight loads depend on local regulations.

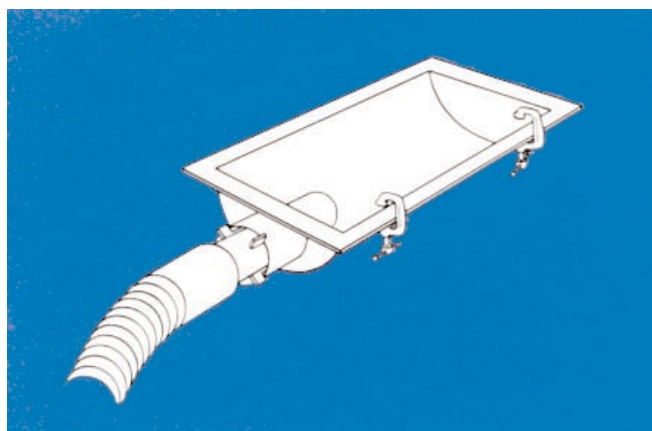


Figure 3-1: Pneumatic Unloading Adapter

4 Unloading

When unloading soda ash from railcars or trucks, it is important to consider the end use of the product where particle degradation or attrition is of concern, belts, screws and elevators should be used to minimize particle damage. Where particle size is less of a concern, pneumatic transport can be considered.

Dry soda ash storage vessels should be designed for plug flow (first in-first out). This minimizes buildup on bin walls should the soda ash absorb moisture when the walls sweat. In addition, the roof should be designed to shed water and be sealed to prevent rain and other sources of water from entering the bin.

Bagged Soda Ash

Bagged soda ash is usually unloaded from railcars or trucks using a warehouse forklift. The forklift should have rubber-tires, antifriction bearings and be free from protuberances and rough spots that might tear the bags. The load should overhang the ends of the forks by six or eight inches

Store bagged product on a firm, smooth floor.

When moving bags by hand, lift them from both ends to avoid undue strain on the body of the bag. Do not drop them, place them across a sharp edge or drag them across the floor. Repeated handling and rough treatment may damage a bag and cause it to leak.

Bulk Soda Ash

Hopper Cars. Covered hopper cars are unloaded by spotting a car hopper over an unloading point and opening the discharge gate or gates with a crowbar so the bulk material flows from the bottom outlet. If the soda ash has compacted due to vibration during transit, flow can be improved by poking the load with a long rod from one of the top hatches, through the open gate, or by rapping on the outside of the outlet. Most covered hopper cars have a standard grooved bracket beside each outlet so an electrical or pneumatic vibrator can be attached to assist flow. Do not operate vibrators if the soda ash is not flowing because this will compact the material.

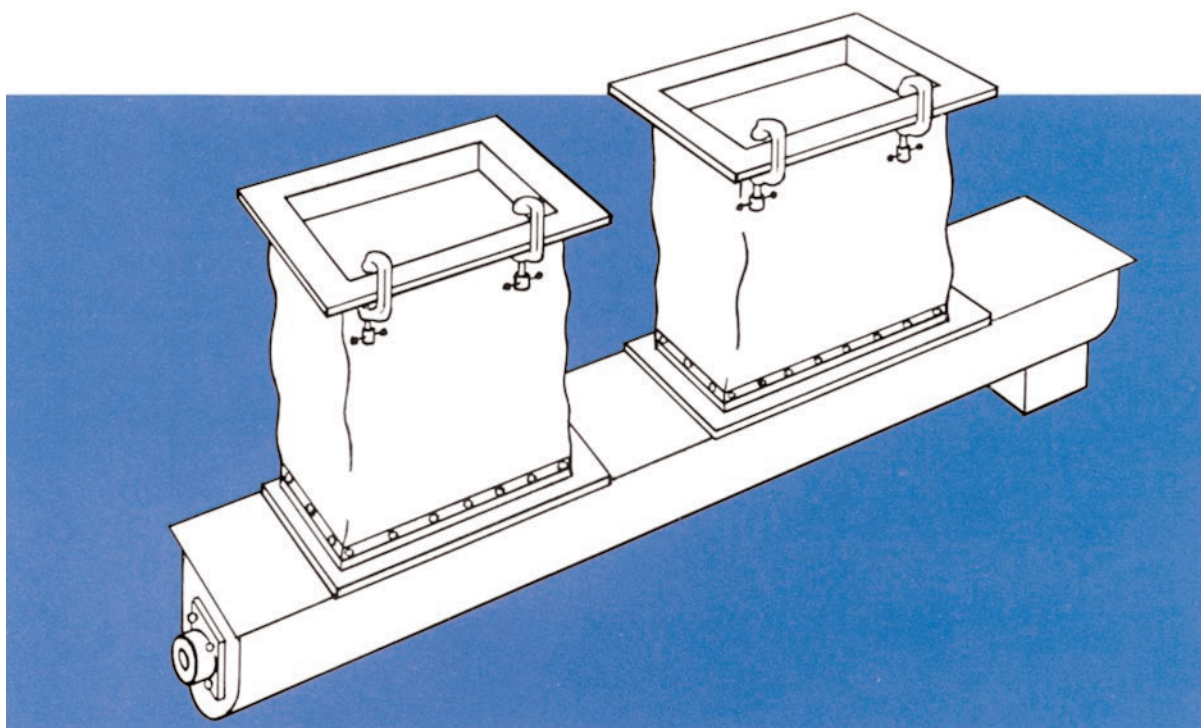


Figure 4-1: Dust Shrouds for Gravity Unloading

Unloading

The location and spacing of outlets in covered hopper cars can differ from car to car, so it is customary to unload one compartment at a time. When a compartment is empty, the car is moved to the next outlet or pair of outlets with a car-moving jack or capstan and rope.

Clearance under the car ranges from 4 to 11 in. (0.102 to 0.279 m) above the top 'I' of the rails, depending upon the car, its lading, and the condition of its wheels, journals and springs. Provision for moving

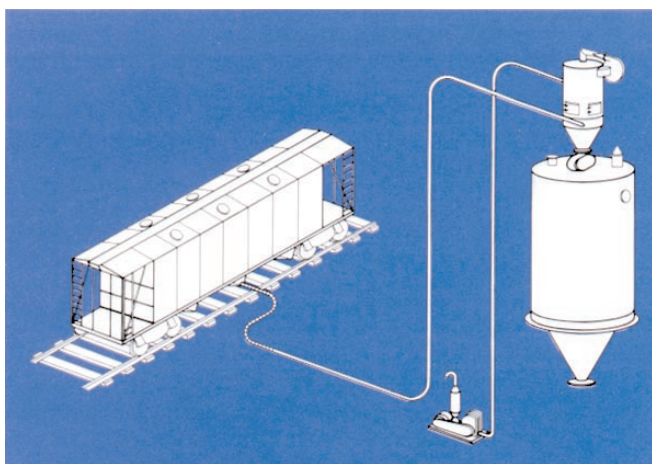


Figure 4-2: Vacuum Unloading System

soda ash away from a car as it unloads is usually placed beneath the track, although over-track belt conveyors are also available. The latter fit in the narrow space between outlet and rails and are usually slide-belt conveyors with the belt supported on a pan rather than on rollers. Unloading rates with such conveyors may be restricted by low under-car clearance. Over-track screw conveyors are also available, but unloading rates are limited by the small diameter.

One method for controlling dust when discharging soda ash from the bottom of a hopper car is to attach flexible shrouds to the outlets before opening the slide gates. This can be done with a pair of canvas or plastic sleeves fastened to rectangular steel frames attached to the undersides of the car outlets with C-clamps (many car outlets have built-in ledges that allow for this). The

lower ends of the shrouds are permanently attached to the cover of the chute, conveyor or other means used to move the soda ash (*Figure 4-1*).

Covered hopper cars can be unloaded pneumatically, if nozzles designed for this purpose are present on the cars (*Figure 4-2*). For cars without such nozzles, special attachments may be constructed to allow pneumatic unloading.

Trucks. Trucks that discharge by gravity are handled in a similar manner to hopper cars. Discharge points on these vehicles are generally so low that transfer equipment must be located below grade.

Self-unloading, pneumatic trucks discharge through a hose, usually at the rear of the truck, to a permanent pipeline. The tractor and trailer must have sufficient access to the conveying pipe so a minimum length of hose (9 ft.) can connect the truck discharge to the unloading pipe.

The conveying pipe should have a vertical, male, quick-connect fitting, usually 4-in. standard pipe size, about 3 ft. (1 m) off the ground. This allows the hose to be attached without tight bends or kinks. A female 4-in. quick disconnect dust cap is attached on the inlet of the conveying pipeline when it is not in use.

Aside from the inexpensive conveying pipe, pneumatic trucks contain all necessary unloading equipment and are operated by the truck driver. The customer only has to ensure the storage bin has enough room for the load.

Conveyors. Transporting bulk soda ash from the unloading area often involves gravity or powered conveyors that can move large amounts over short distances. Gravity conveyors may be a simple inclined chute or a roller conveyor that travels around corners and inserts into a car. Powered conveyors are usually of the belt type and may have cleats for conveying up steep inclines.

5 Handling Bulk

Dust Control

Soda ash is a fragile, crystalline product subject to breakage from conveying equipment. Abrasion produces undesirable soda ash fines or dust that can impair its physical characteristics. Soda ash should thus be handled as gently as possible to minimize grinding or abrading effects.

Although soda ash is not considered a toxic substance (see Section 10), the dust that inevitably arises during bulk handling can be a serious annoyance, especially in congested areas. Air quality regulations require that reasonable measures to control dust be considered where bulk soda ash is handled.

When a stream of bulk soda falls freely, it entrains air in proportion to the amount of soda ash and height (hence velocity) of the fall. At the terminal point, the entrained air disengages turbulently, generating appreciable velocity pressure and carrying finely-divided soda ash dust with it¹.

Freefall should be kept short as possible. A better practice is to use an inclined slide or chute instead of a clear, straight drop. This reduces velocity pressure, compacts the flow and entrains less dust. Gravity movement should terminate in a tightly sealed enclosure with a somewhat constricted inlet.

Dusting from handling and processing equipment can be reduced or eliminated by using tight covers and, if possible, by placing the equipment under slight negative pressure so air leaks inward. (Localized velocity pressures may occur that overcome the negative system pressure and leak dust from otherwise sealed equipment. This can usually be corrected with minor equipment modifications.)

Air exhausted from soda ash-handling equipment should be cleaned in a dust collector before it is discharged to the atmosphere. Dust collector size can be reduced by connecting air from various pieces of handling equipment in series using dust jumpers and applying suction at the final stage.

Soda ash generates micron-sized particles that require high-efficiency collectors. Bag or cloth filter collectors are recommended for dry soda ash dust. This dust has comparatively little tendency to blind filter media. Cloth collectors should handle up to 10 cu. ft. of air per minute per sq. ft. of fabric (3 cu. m of air per minute per sq. m), depending upon the type and condition of the filter medium and the pressure drop.

Cotton sateen cloth is a satisfactory filter medium for soda ash dust. Wool felt can be used in high-performance collectors. If a filter is used intermittently, soda ash accumulated in the weave of the cloth may absorb atmospheric moisture and crystallize, which can stiffen the fabric and lead to cracks and pinholes. To avoid this, either remove the bags and store them in a dry place when not in use or launder them. If this is impractical, nylon can be used instead of cotton, especially in sections where cracking is severe.

When a soda ash end-use involves a solution feed, cyclone wet scrubbers may be an economical dust control option because soda ash dust is readily soluble. The soda ash scrubber liquor may be returned to the process as make-up water. This is an attractive option when the scrubber solution can be used or disposed of in a neutralization step. If this is not the case, wet collection may be an uneconomical alternative.

Materials of Construction and Design Considerations

Iron, steel, and most other metals are commonly used in soda ash service. The 5000 series aluminum alloys may be used in dry soda ash service. Brass or high-zinc bronze is undesirable if wetting occurs, because it dezincifies. Plastics may be used within the limitations of temperature and structural strength established by the manufacturer. Equipment should be designed using out-board bearings to reduce maintenance where dry soda ash may otherwise come in contact with in-board bearings.

Handling Bulk

Gravity Conveying

Bulk soda ash is best moved by gravity, which calls for careful design to minimize dusting. Chutes should be totally enclosed and have dust-tight hand holes for clearing stoppages. Chute slope should be at least 45° below the horizontal. Short chutes are preferably rectangular and wide enough so the soda ash spreads to a thin stream. This will keep the soda ash from grinding on itself and reduce air entrainment.

The bottom of a chute is a good place to install a permanent magnet to capture tramp iron, such as car seals and nuts and bolts from handling machinery. Magnets are more effective when installed just above an apron or suspended flap gate that checks the flow and allows for good contact between the magnet and the soda ash. (Magnetic power is reduced by an air gap.) Flap gates also trap turbulent, dusty air drawn down the chute with the solids.

Belt Conveyors

Belt conveyors work well with bulk soda ash because the material rests on the belt so there is little or no abrasion and dusting. Proper design of chutes and slides, including dust collection, can minimize dusting at transfer points.

Spillage usually occurs, however, because some material sticks to the return side of the belt. This material can be recovered by use of belt cleaners. Belts running outdoors or through drafty locations should be enclosed. Bearings and idlers should be of the anti-friction, dustproof type. If the soda ash is likely to be hot, use of temperature-resistant rubber and fabrics will prolong belt life.

Screw Conveyors

Screw conveyors are simple, compact, and widely used to convey soda ash horizontally. Screws are not as gentle as belts, so they are generally found where degradation of soda ash particles is of little concern.

The design of a screw conveyor is affected by how it will be used. It should be designed for about 20% volumetric loading in heavy, continuous service and up to 40% loading for light, intermittent duty. It should be driven from the discharge end and have roller bearing end thrusts. Intermediate bearings should be of the dry type to prevent soda ash contamination of the lubricant. For heavy duty, these bearings may be of Stellite™ (a trademark of Cabot Corporation, Kokomo, IN) wear-resistant alloys, while hard iron is satisfactory for light duty. Steel-frame bearing hangers are preferable to cast types because they interfere less with the flow.

The conveyor trough should be closed with a tight cover. Increasing conveyor trough depth a few inches allows air to pass through the system for dust control. When feeding a large bin, a bottomless conveyor can be extended across its entire length, thus becoming jam proof and self-trimming. The screw and casing should be designed to expand independently when conveying hot soda ash for long distances.

Vibrating Conveyors

Vibrating conveyors have circular or rectangular chutes, usually set at a slight incline below the horizontal, and are vibrated mechanically or by pulsating electromagnets. The conveyors should be isolated from other machinery that might dampen the vibration.

Transfer points should be closed with tight-fitting flexible seals to limit dusting, although they can become difficult to maintain. The amplitude of vibration, and hence the conveying rate, is controlled by varying the power input. This allows these conveyors to be used as feeders.

En Masse Conveyors

En masse conveyors allow dry materials to be conveyed vertically (on an incline) or horizontally using a single piece of equipment. They can, for example, convey bulk soda ash from a track hopper under a car to a

Handling Bulk

higher elevation in an adjacent building. They have a continuous chain with solid or skeleton flights attached at intervals that push the material along in slugs. The entire assembly is enclosed in a casing that fits the flights closely and has feed and discharge openings. It operates at slow speeds with little product degradation, so it is usually dustless.

Elevators

Dry bulk soda ash is usually moved vertically in bucket elevators, most often centrifugal-discharge and continuous-discharge types. Centrifugal-discharge elevators operate at relatively high speeds, e.g., 200 to 500 ft. (60 to 150 m) per minute. They contain spaced, cup-shaped buckets fastened to a belt or chain. The buckets scoop soda ash from the elevator boot and throw out their contents tangentially upon passing over the head shaft, abrading the soda ash. The high speed of the centrifugal-type elevator causes velocity pressures similar to that of a fan, so a relieving duct is needed to control dusting. Attrition is greater in centrifugal-discharge elevators than in en masse or continuous discharge elevators.

Continuous-discharge elevators operate at up to 120 ft. (140 m) per minute and have buckets mounted on a chain adjacent to each other. The material continuously flows into the bucket at the elevator boot and slides out of the spilling bucket at the headshaft. At this point, it flows onto the sloping bottom of the inverted bucket immediately below it, minimizing attrition.

The discharge spout in both types of elevators requires special attention, because soda ash has a tendency to cling to the buckets when they are inverted. Two or three extra feet of discharge height are sometimes recommended so the buckets can free themselves completely and avoid “back-legging,” excessive dusting and loss of capacity.

Pneumatic Conveying

Soda ash can be pneumatically conveyed using air. This is an attractive method when particle degradation is not a concern, e.g., when the product is to be slurried or dissolved. Soda ash settles rapidly, so low solids-to-air ratios are required to prevent pluggage, especially in horizontal runs. Soda ash is typically unloaded using 750 scfm of air through a 4-in. line at a velocity of 15,000 ft./min. velocity. This can cause significant attrition. Long-sweep elbows should be used and the unloading line should be as short as possible. Pressure relief protection is recommended for all tanks or bins present in pneumatic conveying service. Because pneumatic conveying requires a high volume of air for a relatively small amount of material, filters or cyclones are needed to separate the fine dust from the exhausting air.

This type of conveying involves low pressures, so thin-walled pipe or tubing is adequate (sometimes with reinforced sections at bends). Sixteen-gauge steel tubing or aluminum pipe is usually used. Pipe sections are butted tightly and fastened with Morris or Dresser-type, clamp-on couplings. Runs should be as short and direct as possible and have a minimum of bends or inclines. Bend radius should be at least of 10 or 12 pipe diameters. The pipeline should discharge into the roof, rather than from the side or tangentially. The conveying pipe should rise straight up from the unloading point to the top of the bin.

The volume of conveying air used for unloading is normally on the order of 600 scfm (17 mcm) in a 4-in. diameter pressure pipeline for trucks and 1000 scfm (28 mcm) in a 5 or 6-in. vacuum pipeline for railcars. These volumes can convey as much as 2 lb. soda ash/scfm (32 kg/mcm) in a simple, vertical system or as little as 0.25lb./scfm (4 kg/mcm) for complex pipelines with long horizontal runs.

The receiving bin must have an adequately sized vent to prevent pressure build-up during unloading. A

Handling Bulk

nominal 12-in. vent provides adequate pressure relief and reduces the entrainment velocity of the exhaust air sufficiently so only a small dust collector is required. It is recommended that a separate vacuum-pressure relief device be installed as added precaution. For pneumatic truck unloading systems, a simple sock made of 14 sq. yds. (12 sq.m) of woven fabric sewn into a sleeve and attached to the bin vent has been effective. The sock can be shaken back into the bin or emptied after unloading. Commercial small-bag collectors and cyclone scrubbers are available and provide higher efficiency recovery.

Railcars are typically unloaded using vacuum-pressure systems in which a vacuum pulled on the hopper sucks soda ash into an unloading tank. When the tank is full, the valves switch and the tank is pressurized and the soda ash is blown to the final storage tank. This process continues until the hopper and railcar are emptied. High-efficiency dust collectors are needed if the exhaust air is recycled to the vacuum pump,.

Slurry and Solution Handling

When soda ash can be stored as a slurry, it is sometimes convenient to pump it directly from the unloading point to the storage tank. Slurries having up to 35 to 40% suspended solids by weight (50 to 60% total soda ash) can be pumped, although 10 to 20% is the more usual. Weak solutions (5 or 6%) can be handled as if they are water. The temperature of slurries or solutions must be maintained above 100°F to avoid crystallization

or the formation of unwanted hydrates. When hard water (more than 120 ppm hardness) is used in make-up, the soda ash will react with the calcium and magnesium present to form scale in pipelines and storage vessels, where it will settle out. These solids must be removed periodically.

Pipelines carrying strong soda ash solutions should be well insulated. Long pipelines should be heat traced with low-pressure steam or electricity designed to avoid excessive heating. The heat source should be secured at the top or sides of the pipe to prevent the formation of solid anhydrous scale if the line experiences overheating. If the use point is distant from the storage tank and the use rate is low or intermittent, the pipeline should be constructed as a continuous loop so most of the solution recirculates back to the tank.

References

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6 Storage

Bag Storage

Soda ash tends to cake when exposed to moisture or the atmosphere for a long time. Dense soda ash does not cake as readily as lighter density products. Typically, the soda ash layer at the bag surface will begin to dissolve in a bag exposed to adverse conditions. Caking occurs because not enough water is present to dissolve the soda ash completely. Because caked soda ash has less surface area than the powdered product, the caked product does dissolve readily.

Normal warehouse storage of soda ash seldom presents caking problems, especially if the oldest stock is used first. For best results, do not store soda ash in a damp or humid place or where there is excessive air circulation. When storing soda ash for an extended time under adverse conditions, cover the bags with a tight-fitting, impermeable sheet.

Palletized bags on slipsheets or other disposable cardboard pallets should be transferred to double-faced hardwood pallets before tiering. Some types of disposable pallets should not be stacked more than two or three high so the bottom pallet does not collapse.

Warehouse floors should be dry, smooth, free of breaks and able to support concentrated loads, especially when bags are tiered or handled with forklift trucks (nearly the entire weight of a loaded forklift falls on the two front wheels). Table 6-1 lists approximate floor areas, space requirements and floor loadings for warehousing soda ash in bags.

Bag Dimensions (one bag lying flat)	Light Soda Ash		Dense Soda Ash	
	In.	M	In.	M
Length	31 to 34	0.79 to 0.86	24 to 26	0.61 to 0.66
Width	18 to 20	0.46 to 0.51	17 to 20	0.43 to 0.51
Thickness	6 to 7	0.15 to 0.18	6 to 7	0.15 to 0.18
Floor Area (one pallet)	Ft ²	M ²	Ft ²	M ²
48" x 48" (24 bags)	20.25	1.88		
48" x 36" (18 bags)	13.5	1.25		
44" x 44" (24 bags)			14.7	1.37
Volume (includes pallet)	Ft ³	M ³	Ft ³	M ³
48" x 48" (24 bags)	81	2.3		
48" x 36" (18 bags)	53	1.5		
44" x 44" (24 bags)			56	1.6
Floor Loading (includes pallet)	lbs/ft ²	kg/m ²	lbs/ft ²	kg/m ²
48" x 48" 2400 lbs/1088 kg	121	590		
48" x 36" 1800 lbs/816 kg	138	670		
44"x44" 2400lbs/1088kg			167	810

Table 6-1 Storage of Soda Ash in 100 lb (45.4 kg) Paper Bags

Dry Bulk Storage

The "shelf life" of soda ash is dictated by the storage environment, because it is slightly hygroscopic and absorbs moisture from the air. It should remain dry and free flowing below a relative humidity of 85%, but will have an increased tendency to cake increases above this. The cooling of hot, humid air can cause "bin sweating" and form unwanted scale or lumps.

It is recommended that at least a 10-day inventory in the form of on-site and railcar storage be maintained as insurance against delivery delays. The minimum storage requirement is the volume of an entire shipment. Where delivery is via waterways that may close during the winter, inventory should be sufficient to last through the closed season, unless other modes of delivery are available.

Storage

Warehouses can store bulk soda ash for a short time and then returned to general warehouse use after the soda ash is removed. Soda ash can be stored in bulk as a pile on the floor and reclaimed by a front-end loader or other bulk-handling machine. The building must be suitable for storage, with no roof or other leaks, closed to prevent the free circulation of atmospheric air, and the floor (if on grade) should be protected by a membrane to prevent moisture penetration. The pile should be protected from contamination, especially from vehicular traffic that might track mud or dirt. Dusting may become a problem with bulk pile storage.

More typically, soda ash is stored in closed bins, bunkers or silos. Large bunkers are often an integral part of a building, conforming to its proportions and supported by its structure. Such bunkers work well for storing large amounts of soda ash, because the bin can be made longer than it is wide, a shape that experience shows is easier to fill and empty than square or circular bins.

Bunkers can be filled with one or more open-bottom screw conveyors set longitudinally across the top and designed to load it progressively from one end to the other. The bunker's cover or roof should be tight and have observation or access hatches that can be sealed when closed and guarded with removable grates when open. Dust control for the entire conveying system can be placed at the bunker, which can serve as an ideal settling chamber for the dust.

The bunker's discharge hopper should be have sloping sides. The preferred slope is 60° below the horizontal, but it is recommended that it never be less than 45° below the horizontal. If a bunker is longer than it is wide, it should have a series of outlets separated by auxiliary transverse slope sheets (set as inverted V's between each pair of outlets to eliminate dead space) connected to a reclaiming conveyor. Rack-and-pinion slide gates on each outlet can minimize sifting and dusting, allow selected withdrawal from the bunker, and make maintenance of the conveyor easier.

Although rectangular bunkers may be superior, many steel and concrete bins and silos are circular because they are less expensive. These should also have a wedge or V-shaped bottom to minimize arching and bridging. Simple and less-costly conical bottoms perform satisfactorily, if provision is made to overcome possible "bridging" or "rat-holing".

Storage bins exhibit either mass flow or funnel flow characteristics. In mass flow, all of the material in the vessel flows unassisted whenever any is withdrawn. Mass flow bins are designed to exert sufficient internal forces to prevent bridges from forming. The result is uniform, non-segregated, constant density flow of deaerated soda ash. Mass flow bins require more space and are more expensive to build than funnel flow bins.

In funnel flow, a portion of the material usually moves in a channel or "rathole" down the center.¹ Funnel flow bins have smaller outlets and bottoms that are not as steep-sided as mass-flow bins. This type of flow is an erratic, first-in/last-out movement that allows the product to segregate. The product tends to bridge and rathole, flowing directly from the top center vertically down to the outlet, and density will vary depending on the segregated product being fed at the time (from coarse to powder). Product near the walls eventually falls to the center until the bin is refilled, which refills the rathole. When a powder bridge is broken, the material may fluidize and its flow may be difficult to check. A positive shutoff is recommended for funnel flow bins.

Bridging occurs when outlet devices are too small. If outlet size is constrained by building height or narrow conveying equipment, auxiliary equipment can be helpful in combatting bridging and channelling.

One simple method, which can cause some dusting, is to use a poke-hole near the bin outlet that is capped when not in use. A 2-in. pipe nipple at least 4-in. long mounted horizontally to the sloping bottom is adequate to account for the angle of repose and prevent soda ash from flowing out the open nipple.

Storage

Another method is to introduce jets of clean, dry, compressed air through small-diameter pipe connections into the mass of soda ash near the bottom of the bin.

An electric or pneumatic vibrator can be mounted on the exterior of the bin bottom, somewhat above the outlet. Vibrators will tend to further compact the soda ash if used when no flow is occurring. Excessive vibration can degrade and segregate soda ash particles. The use of vibrators is not recommended when it is important to preserve the particle size. Devices designed to break the bridge are often electrically interlocked with the reclaim conveyor so they only operate when the conveyor is running.

Live-bottom bin devices can also reduce bridging and channeling. As with vibrators, they tend to fracture dense soda ash particles if used frequently.

Liquid Soda Ash

Soda ash solution is an excellent option if dry handling systems cannot be used but the economies of soda ash are desired. General Chemical Industrial Products provides a 30% liquid soda ash solution using its GCH[®] Hydrator technology, so customers do not have to invest in and operate dry-to-liquid conversion systems. Water used in the hydrator is usually preheated and the solution is delivered ready to use at 110 to 125°F (43 to 52°C). This solution may require some heating to prevent crystallization (which occurs at 90°F (32°C)). Outdoor tanks are usually insulated, especially if throughput is low. In addition to the methods cited above, many other practical slurry unloading and storage set-ups exist. General Chemical Industrial Products can help in selecting the plan that best fits a particular need.

Liquid Soda Ash Storage

When soda ash is used as a solution, it may be convenient to store it in this form in a tank. Soda ash can be dissolved to a known concentration and dispensed volumetrically in simple and relatively inexpensive pumps and pipelines. The concentration of the solution is commonly maintained at 20% or some lower value to avoid any risk of crystallization. Because of the excessive tankage required for storage of substantial amounts of soda ash, this method is usually limited to intermediate, short-term use. If large quantities of soda ash are involved, it has been found more practical to store it as a slurry.

Solution Storage

Soda ash is usually delivered in 24-ton trucks or 10-ton railcars. Trucks have a minimum capacity of 15,000 gallons and railcars a minimum of 62,000 gallons.

One strategy for storing soda ash liquid is to fill a storage tank to a high concentration and draw off liquid to the process. This liquid is diluted after the process feed pump discharge to the concentration needed. As liquid is withdrawn from the tank, process water fills the tank to maintain a constant upper liquid level. The solution in storage is diluted until the concentration approaches that needed for the process. At or before that point, new soda ash is added to the tank to restore the original concentration and increase soda ash inventory.

A day tank is recommended so the soda ash liquid feeding the process is not interrupted by new deliveries of dry soda ash. A stilling period is recommended after unloading dry soda ash so solids can dissolve or settle, which prevents two-phase flow to the process and subsequent overfeeding of soda ash.

Storage

For example, a truck unloaded into a 15,000-gallon tank and mixed to a 30% concentration will provide about a 10-hour inventory if the desired process use concentration is 18%. Figure 6-1 shows estimated operating hours of concentrated soda ash liquid given a process-use feed rate of 10 to 25 gpm. Twice the volume will give twice the time, e.g., 60,000 gallons will last about 20 to 24 hours.

A truckload can be received after the concentration in the tank falls below 22%. The solution in the tank will saturate to 32%. If the tank cools to below 95°F, solids will form and may account for up to 5% of the tank volume. These solids should readily dissolve as make-up water replenishes the tank level.

Soda ash can be unloaded to solution tanks from pneumatic trucks in about 2 to 2.5 hours. The preferred method for unloading dry soda ash to storage call for a

mixing tee. Recirculated solution from storage serves as a spray to wet the incoming dry soda ash. This reduces dust emissions during unloading, although a wet scrubber or baghouse is needed to control dust emissions carried by the pneumatic airflow through the tank (typically 600 cfm). General Chemical can provide design information for the mixing tee configuration.

A second strategy for liquid storage when deliveries are by rail uses the GCH® Hydrator. This unloading system offers several advantages where higher soda ash usage demand exists.

1. The soda ash is slurried through the hydrator as it unloads the railcar, eliminating a possible fugitive emission point. i.e., no dry solids are emitted.
2. The hydrator unloads about 8 to 10 tons per hour, so a railcar can be unloaded in less than two shifts. The hydrator, which is designed to unload each railcar

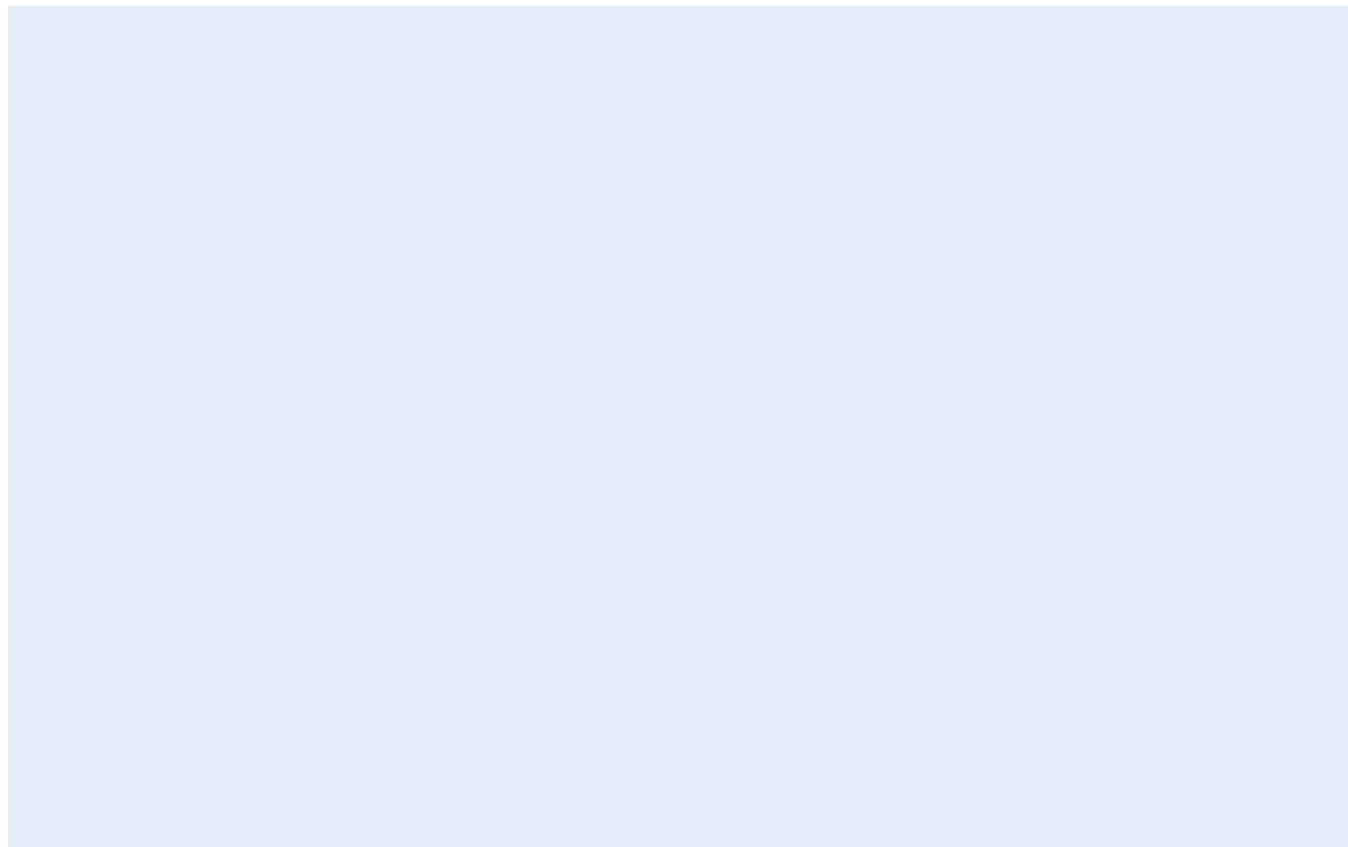


Figure 6-1 Decreasing Concentration vs. Hours Drawdown

Storage

hopper, can be conveniently interrupted without incident so volumes of less than 100 tons can be unloaded to storage. The railcar itself thus becomes an added source of soda ash inventory, so the product it contains does not have to go to a silo.

3. Concentration is measured using a hydrometer, density meter or differential pressure cell at constant tank level. (This actually measures the change in weight of the liquid, from which concentration is inferred.) With experience, temperature can aid in predicting concentration.

Slurry Storage

When similar quantities of soda ash and water are mixed, part of the soda ash will, of course, dissolve to make a saturated sodium carbonate solution. The undissolved portion will form crystals of sodium carbonate monohydrate that settle out as a fluid, non-hardening slurry. The slurry has a considerably higher apparent density than the dry soda ash from which it was made, so more soda ash can be stored in a given volume as a slurry than in the dry form.

Sodium carbonate solution is readily recovered from storage by skimming or decanting from the clear liquid layer on top of the slurry. The concentration of saturated solution in contact with monohydrate crystals is remarkably uniform at all temperatures between 35°C (96°F) and the boiling point. This frequently makes it possible to dispense soda ash volumetrically with acceptable accuracy by use of ordinary liquid metering devices.

Solution withdrawn from storage is replenished by simply adding water to the slurry, which dissolves some of the settled crystals to form fresh saturated solution.

The slurry is replenished when necessary by adding dry soda ash.

Dry soda ash mixed with saturated solution forms a bed occupying about 200 gallons of apparent space per ton of dense ash. Tanks should contain at least that much clear solution before dry soda ash is added. In addition, soda ash displaces 80 gallons of total volume per ton, so tanks should have at least this much space above the surface before the soda ash is added.

Experience shows that in a moderately sized system, the settled solids should occupy no more than about 85% of the stored volume to facilitate decanting the clear supernatant solution. This corresponds to an overall concentration of approximately 9.5 pounds per gallon of soda ash. (*Figures 6-2 and 6-3 show concentrations and densities for soda ash slurries.*)

Essentially, a slurry storage system consists of a tank, a way to slurry the bulk soda ash and transfer it to storage, and the means to reclaim solution from the tank and replenish it with water.

Crystals in the tank rapidly settle from the liquid, which is decanted from near the surface and recirculated to make up fresh slurry. Clear saturated solution for use is similarly decanted, although a brief settling period is needed after unloading to avoid turbidity. As supernatant solution is withdrawn, it is replaced with water through a perforated pipe manifold in the bottom of the tank. The water dissolves the sodium carbonate crystals as it rises through the slurry bed. Table 6-2 shows that a 30,000-gal. storage tank can hold 48 tons of soda ash as saturated solution and up to 116 tons as an 80% slurry.

Storage

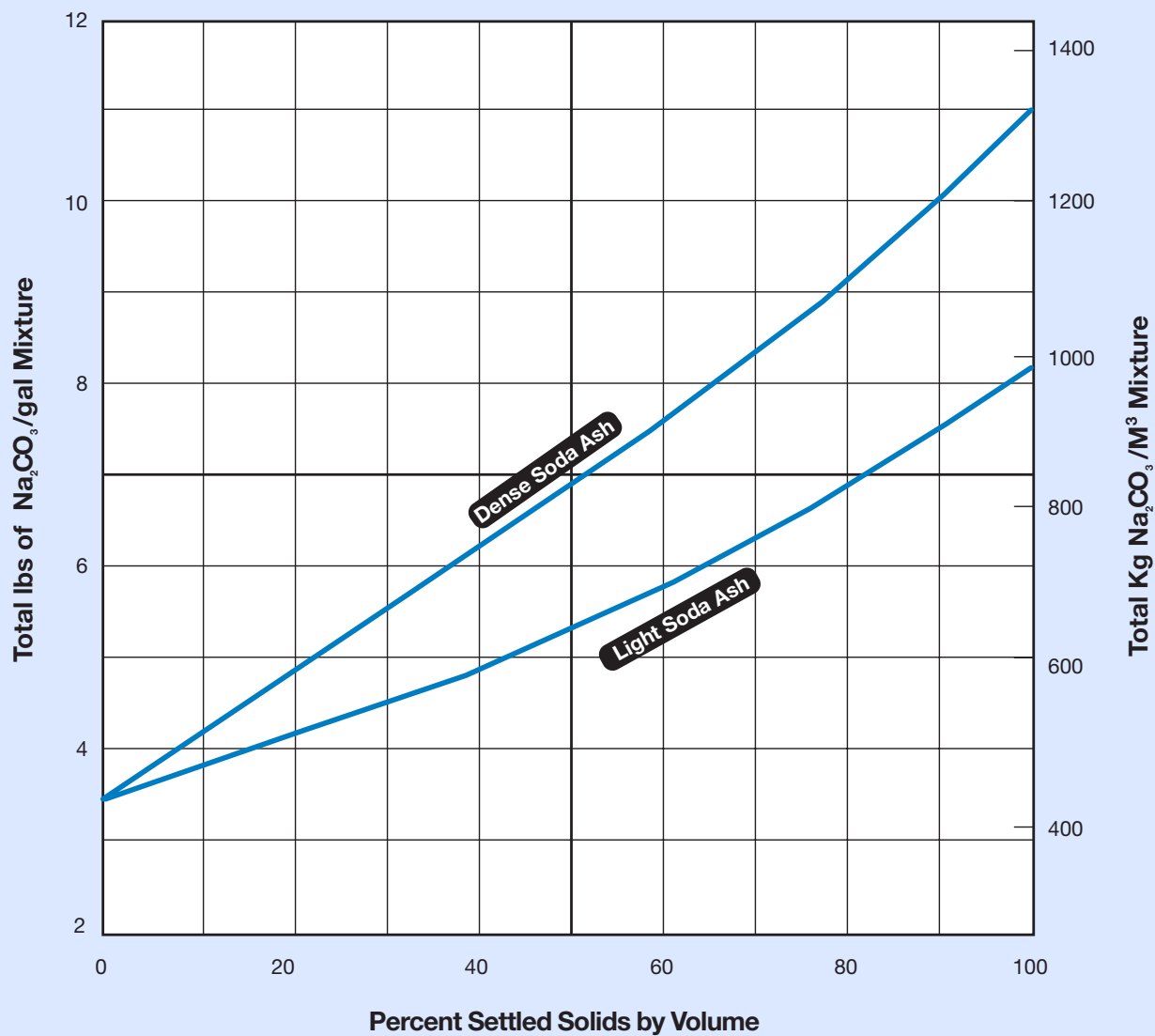


Figure 6-2 Concentrations of Soda Ash Slurries

Storage

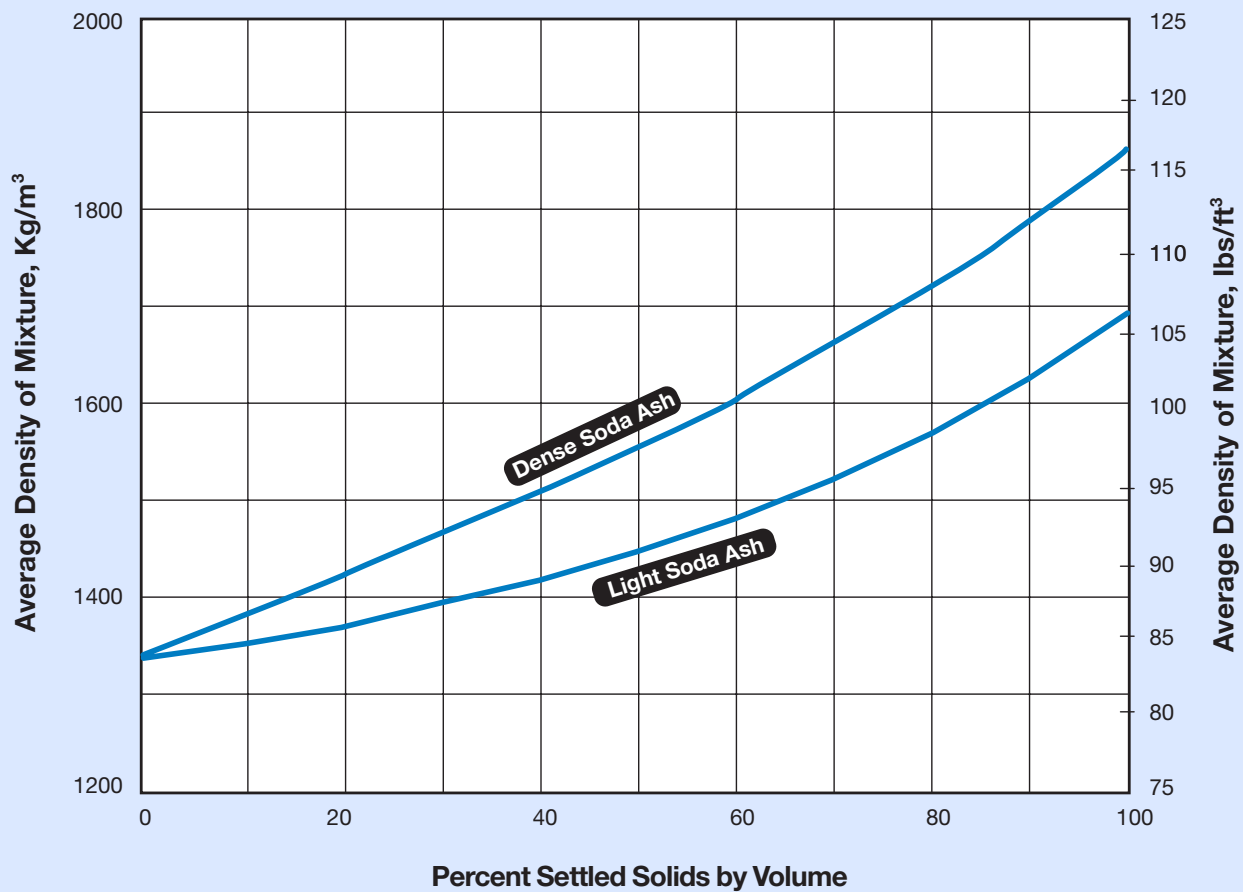


Figure 6-3 *Density of Soda Ash Slurries*

Storage

Tons Soda Ash per Active Volume (Active Volume = 85% of Tank Volume)									
Tank Vol.	Sat. Soln.	10%	20%	30%	40%	50%	60%	70%	80%
3000	5	5	6	7	8	9	10	11	12
4000	6	7	8	9	11	12	13	14	15
5000	8	9	10	12	13	15	16	18	19
10000	16	18	21	23	27	29	32	35	39
15000	24	27	31	35	40	44	48	53	58
20000	32	36	41	47	53	58	64	70	77
30000	48	54	62	70	80	87	96	106	116
40000	65	72	82	94	106	116	128	141	155
50000	81	90	103	117	133	145	160	176	193
60000	97	108	123	141	159	174	192	211	232
70000	113	126	144	164	186	203	224	246	271
80000	129	144	164	188	212	232	256	282	309
90000	145	162	185	211	239	261	288	317	348
100000	161	180	205	235	265	290	320	352	387
110000	178	198	226	258	292	319	352	387	425
120000	194	216	246	282	318	348	385	422	464

Inventory as Monohydrate = Tons per Active Vol. Minus Sat. Soln.

Table 6-2: Tons Soda Ash per Active Volume

Storage Systems

Temperature control is one of the most important requirements for successfully storing soda ash solutions and slurries. Solids form as a saturated solution cools below 95°F. These solids expand as they crystallize and form a hard, dense mass that is difficult to redissolve.

The actual heat requirement of a slurry system is usually low, because both the hydration of dry soda ash to form monohydrate and the dissolving of the monohydrate to form solution are exothermic. Mixing dry soda ash with recirculated saturated solution to produce settled slurry generates enough heat to raise the temperature of the mixture approximately 35°F. Theoretically, water added to the slurry to dissolve crystals and form saturated solution can be approximately 38°F below the

temperature of the slurry without cooling the mixture. In practice, care must be taken to distribute the water to avoid localized chilling that could cause undesirable hydrates to form.

Water used to operate the system is preferably preheated. Live steam may be injected directly into the bottom of the slurry bed. This also supplies some of the make-up water in addition to heat, although this can cause the tank to overflow if it is inactive for an extended period.

Heat should be conserved as much as possible in a slurry storage system for economy and to avoid undesirable crystallization. Outdoor storage tanks are generally insulated against heat loss, particularly if the throughput rate is low.

Storage

GCH Hydrator

General Chemical's GCH Hydrator System is an economical way to unload soda ash solution without dusting, at low noise and using little labor. The hydrator, when combined with the Company's large railcar fleet, enables General Chemical to ship dry soda ash to any location and dissolve it rapidly on-site to liquid or slurry form.

The GCH System has four major components: an eductor (or jet pump), a cone-shaped mixer, a 3-in. flexible vacuum hose with an aluminum nozzle, and a universal pan specially designed to fit under any railroad hopper car used for soda ash delivery.

The hydrator draws soda ash into the mixing chamber using Bernoulli's principle, i.e., the increase in speed of flow of the motive fluid (solution or water) through the eductor jet causes a decrease in pressure (or suction)¹. This makes the unit easy to use, since it seeks its own equilibrium (or steady state) under all operating conditions and no adjustments are needed during operation.

A centrifugal pump draws solution from a plant's liquid storage tank and pumps it through the eductor, creating a vacuum at the base of the cone-shaped mixer. The vacuum hose draws soda ash from the pan beneath the railcar and feeds it into the top of the mixer. Here it is mixed, or slurried, with the soda ash solution and transferred back to the storage tank through the eductor discharge.

Depending on the temperature and density of the solution, as well as pump and pipe sizing, the hydrator dissolves 8 to 10 tons of soda ash per hour. The GCH system is quiet and efficient compared to conventional unloading systems. There is little spillage, no plugging, and, since there is little dust, no scrubbers are needed.

References

1. Robert H. Perry, Don Green, Perry's Chemical Engineering Handbook, Sixth Edition, McGraw Hill, 1984

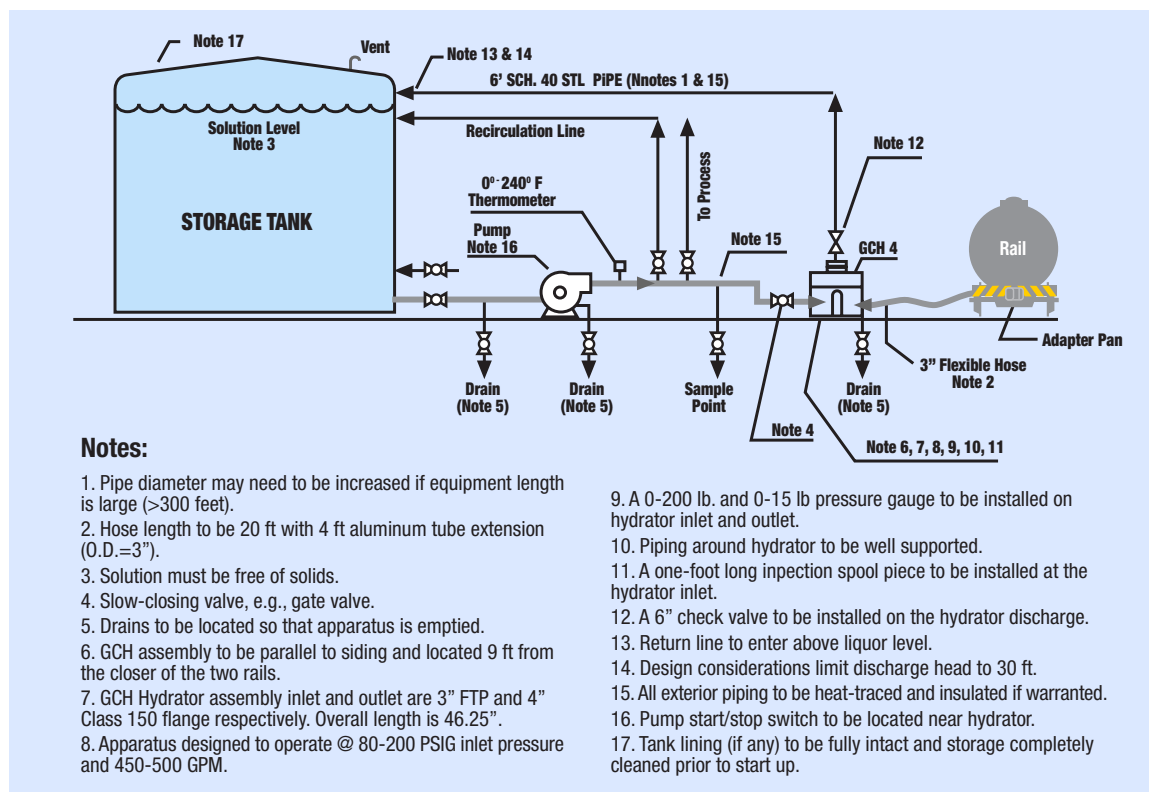


Figure 6-4: GCH® Hydrator System

7 Weighing & Feeding

Shipping Weights

It is vital to maintain strict control over shipping weights, especially if the invoiced weight of soda ash received is a primary indicator of raw material consumption in controlling production and evaluating process yields. Many sites formulate mixes or reactions using package count or depend on a secondary variable, such as pH, to control use rate or quantity.

The alkali industry uses automatic machinery to bag soda ash in multi-wall paper bags. These machines are rugged, dependable and have a high degree of reliability. Trucks that carry soda ash are weighed on truck scales before and after loading. If they are reweighed at a destination, fuel consumption must be subtracted. The tractors hauling self-unloading bulk trailers may be changed between loading and delivery, so gross shipping weight at the origination and delivery points may not be comparable. (For a comprehensive discussion of the design and application of scales see Industrial Weighing by Douglas M. Considine; Reinhold Publishing Corp., 1948.)

Weighing

Measurement of dry soda ash should be based on weight, not volume. Even measurements made by package count are based on weight, because packages are preweighed. Most industrial scales use gravity in a counterbalancing system of levers and linkages. Inaccuracies due to friction are generally on the order of 0.1% of scale capacity for new and well-maintained scales.

For maximum accuracy, scale capacity should match as closely as possible the weight to be determined. One hundred pounds can be weighed on a 100-lb. capacity scale to accuracy of 1/10 lb. or better, but only to 1 lb. on a 1000-lb. scale. Accuracy is not related to the precision with which the scale may be read or set. A 250,000-lb. capacity railroad scale read to the nearest 10 lb. may still have an inherent accuracy no better than

0.1% of its capacity, or 250 lb. (Railroad rules permit a difference of up to 1% of the lading between any two railroad scales, with a maximum of 500 lb.)

The levers in mechanical scales can actuate trips, alarms and control devices. They also can be adapted to automatic weighing (see *below*). Attachments available for most mechanical scales allow them to print, record or transmit a signal. Photoelectric cells or magnets can permit this without mechanical contact.

Non-mechanical scales use hydraulic, pneumatic or electronic load cells rather than mechanical linkages to generate signals proportional to the forces involved. They are useful for large quantities, such as bulk storage bins or tanks. Load cells are sturdy and compact so they are often mounted on crane hooks and lift trucks to read the load handled. They give continuous, full-range signals that can be amplified and converted. This is more useful than the simple over-under signal from many mechanical scales. Load cells require calibration before use and may have a comparable accuracy to mechanical scales at a lower installed cost. They do not have the same kind of progressive wear as mechanical scales.

Batch Weighing

Manual batch weighing allows the scale operator to put or take so as to trim the scale balance to the desired weight. The accuracy of manual batch weighing is affected by the mechanical interferences described above and by the feed system to the scale. Batch-type scales operate on the basis that a flowing stream of material has constant density. The stream of material flowing to the scale must be cut off far enough in advance so the weight on the scale places it in proper balance. This requires a uniform flow to the scale. One possibility is to keep the flow rate relatively low to minimize the effect of flow variations. Since this slows down the operation, two rates of feed are often used to charge automatic batch weighers: most of the

Weighing & Feeding

charge is fed rapidly and then the flow is reduced to a dribble for finishing.

Many types of manual and automatic batching scales are available. Most of them have a hopper to contain the soda ash. The hopper can be movable or resting on a dormant or built-in scale with the platform flush with the floor level. It also can be suspended from a trolley on an overhead track scale. Such arrangements are frequently used when soda ash is combined with other dry ingredients at a central mixing station. In most cases, the hopper is an integral part of the scale, and the dry soda ash is fed to the hopper and removed by gravity or mechanical conveyors.

Continuous scales used for batching should have an integrating device to register the flow of a predetermined amount, but this type is more properly classed as a feeder.

Continuous Weighing

Continuous weighing involves a device that is sensitive to the amount of material flowing and to changes in the flow. Continuous weighing scales use a section of a belt conveyor over which the material to be weighed passes. The belt is mounted on a weight-sensitive platform, typically equipped with load cells, that can detect minute changes in the weight of material passing over the belt. The load cell output is integrated over short time intervals to give a rate of flow and total flow.¹

All scales require continuous monitoring to assure that the desired set weight is maintained and does not drift off because of changes in bulk-density or flowability. They must be clean, carefully maintained and checked regularly with test weights. Dust or other foreign matter should not accumulate on levers or pivots.

Feeding Dry Soda Ash

Dry soda ash can be continuously fed based on volume or weight. Weigh feeders are not subject to error when material density changes as are volumetric feeders.

Continuous feeding is different from simple conveying or batching because the flow of material must be maintained at a controlled rate. Almost any type of conveyor can be a feeder if it has a reasonably uniform discharge rate that can be varied by changing conveyor speed or amplitude or by throttling the flow to the feeder. Some of the better known feeders are: variable speed screw feeders; rotary pocket, star and vane feeders; belt or apron feeders; oscillating tray, pan or undercut gate feeders with adjustable throws; vibratory feeders; and revolving table or disk feeders with adjustable scraper blades. These volumetric feeders can be calibrated by measuring discharge over a set time and are subject to error when the density of the material varies.

Gravimetric feeders measure the material being continuously fed to the process and have an endless conveyor partially or entirely supported on a scale. Scale position is transmitted to a primary feeder controlled to keep the weight of the material on the belt constant. Belt speed controls the discharge rate. The amount discharged is measured by integrating the travel of the belt.

The loss-in-weight feeder is a special case. This hopper scale has a discharge mechanism controlled by the scale beam that always keeps it in balance. A controllable drive moves the poise down the weigh-beam at a predetermined rate, which continuously unbalances the scale. Balance is restored by controlling the hopper's discharge rate. When the hopper is nearly empty, discharge ceases while the hopper is refilled. This system requires dual units for continuous flow.

Feeders should be checked dynamically by calibrating their delivery against a scale of known accuracy.

Weighing & Feeding

Solution Feeding

Soda ash solutions have uniform physical characteristics and are much simpler to meter than dry ash. When concentration, which is readily determined, is known, measurement can be made by volume as easily as weight. As a result, most liquid metering systems are volumetric.

The metering equipment chosen depends on the accuracy required. The concentration of batch quantities can be determined by its level in a tank, although the design should consider possible scaling effects and crystallization tendencies. A manual or automatic valve in the pipeline followed by a pressure gauge can be used to detect variations in flow (a rough correlation exists between pressure and flow rate).

Volumetric or proportioning pumps are commonly used when flow rate is low and measurement is secondary to flow control. This can involve any positive-displacement pump, such as a gear pump. Precision feeding usually calls for a special diaphragm pump

(proportioning pump) that has an adjustable stroke and is protected by a strainer.

Various volumetric flow instruments, such as rotameters, orifices, magnetic flow meters and turbine meters, indicate flow directly and continuously. They also can integrate quantity to provide direct or auxiliary control. Displacement meters, similar to those used for water and similar liquids, integrate flow quantity but may not give flow rate directly. Consult the meter manufacturer when procuring such equipment.

Mass flow meters can measure flow rate, mass (weight), specific gravity and temperature. These meters can be alarmed to indicate changes in flow rate, mass flow (two phase flow caused by crystallization) and changing liquid concentration.

References

1. Chemical Engineers' Handbook, Fifth Edition, 1973.

8 Sampling

Sampling Bulk Shipments

Soda ash can be hard to sample because it absorbs moisture and carbon dioxide from the air through its shipping container and because vibrations in transit can cause particles in bulk dense ash to segregate by size. Soda ash specifications often allow for moisture absorption, e.g., ASTM D 458-74 and Federal Specification O-S-57 1f.

Details of good sampling depend on the type of shipment (container or bulk), the type of product (light or dense soda ash) and the type of analysis desired. The latter involves chemical and physical analyses and whether the quality to be determined is “as shipped,” “as received,” or “as used.”

Although bulk soda ash shipments are normally in transit a relatively short time, some absorption of moisture and carbon dioxide by surfaces exposed to the atmosphere is likely. In sampling a hopper car for chemical analysis, brush the surface layer aside to a depth of about 4 in. and take product from the exposed area. Samples from various parts of the car can be taken and composited.

For physical tests, such as sieve analysis, sample at a depth of at least 12 in. The best sampling points are near the discharge points of hopper cars and bulk pneumatic transports. Conveying systems, especially pneumatic conveyors, tend to break down particle size and so can cause the absorption of moisture and carbon dioxide to increase.

A representative sample usually involves collecting about 1 cu. ft. in total per carload. Never fill the sampling cup or other container more than level-full so larger grains do not roll off. The composite sample should be reduced in volume by “quartering” to a suitable size for analysis.

Sampling Containers

Sample packaged soda ash by taking samples near the center of the package. This can be done by removing the top 6 to 8 in. from the package and sampling from the center of the remaining portion. Such a sample is generally representative except when storage was lengthy or involved damp conditions.

Packaged soda ash stored for an extended time can be sampled only after emptying the package and mixing the contents thoroughly. The sample taken is likely to represent only the package sampled and not the entire stock of packages, because those in the outer layer of a storage pile most likely absorb more moisture and carbon dioxide than those deeper in the stock.

To estimate how much moisture and carbon dioxide absorption has occurred, compare the weight of the full container to the original gross weight. Absorption increases weight and reduces the percent of sodium carbonate present, although the total alkali content remains the same. Some of the sodium carbonate may be converted to sodium bicarbonate due to absorption, but this is not important in most cases.

9 Analytical Procedures

CAUTION!

Many of the procedures described below involve reagents, indicators and standard solutions that may be dangerous. These procedures should only be used by competent analysts who have full knowledge of how to handle these materials safely. All water used in these procedures should be distilled or deionized.

The analysis methods below are used at General Chemical's soda ash facility to monitor soda ash quality. Other methods are available upon request from our Technical Service Department. All reagents listed are discussed in the last portion of this section.

1. Sodium Carbonate Reagents

- Methyl orange indicator
- Modified methyl orange indicator
- Sulfuric acid, 1 N

Accurately weigh 5.2 ± 0.02 g of a representative sample to the nearest 0.0001 g on an analytical balance, and transfer to a 500-mL Erlenmeyer flask. Add 100 mL water, and swirl to dissolve the sample. Add 5 drops of methyl orange indicator or modified methyl orange indicator solution. Titrate with standard 1 N sulfuric acid solution from a 100-mL buret to the pink-color end point of methyl orange indicator. If modified methyl orange indicator is used, titrate to a gray end point. The same indicator and shade of color as used for the end point in the standardization of the acid solution should be used for the sample titration. Record the volume of titrant used and measure the temperature of the sulfuric acid solution. Correct the volume of titrant to 20°C using Table 9-1. Use the corrected-volume value for calculation.

$$\frac{\text{mL H}_2\text{SO}_4 \text{ at } 20^\circ\text{C} \times \text{N} \times 5.299}{\text{Weight of sample}} = \% \text{ Na}_2\text{CO}_3$$

2. Sodium Chloride Reagents

- Benzyl alcohol
- Iron solution indicator
- Nitric acid, concentrated
- Potassium thiocyanate, 0.05 N
- Silver nitrate, 0.05 N
- Sodium chloride, 0.05 N

Sample Preparation

- For samples having more than 0.1 % NaCl: Weigh 10 g, to the nearest 0.01 g, of a well-mixed sample and transfer it to a 300-mL Erlenmeyer flask. Add 25 to 50 mL of water to dissolve the sample, and carefully add 15 mL concentrated nitric acid.
- For samples having less than 0.1 % NaCl: Weigh 20 g, to the nearest 0.01 g, of a well-mixed sample, and transfer to a 500-mL Erlenmeyer flask. Add 100 mL water to dissolve the sample, and carefully add 30 mL concentrated nitric acid.

Procedure

Add 5 mL of iron solution indicator to the prepared sample solution. Then add 1.0 mL of 0.05 N potassium thiocyanate solution from a 50-mL Class A buret, and swirl to mix. Do not rezero this buret. Using another 50-mL Class A buret, titrate with standard 0.05 N silver nitrate solution while vigorously swirling the flask until the red color is completely discharged. Then add 2 mL in excess. Record the volume of silver nitrate solution added. Add 10 drops of benzyl alcohol. Shake the flask vigorously for 30 seconds, and rinse down the inside wall of the flask with water. Back-titrate with 0.05 N potassium thiocyanate solution slowly and with constant swirling until a faint red color persists. (Note 1)

$$\frac{[\text{mL AgNO}_3 - (\text{mL KSCN}) (F^*)] \times \text{N of AgNO}_3 \times 5.84}{\text{Weight of sample}} = \% \text{ NaCl}$$

* See last portion of this section for the determination of factor F.

Analytical Procedures

Remarks: A back-filtration volume of at least 0.5 mL of potassium thiocyanate solution is needed to ensure a sufficient excess of silver nitrate solution to precipitate all the chloride. If a back-titration of less than 0.5 mL of potassium thiocyanate solution is encountered, add 5.0 mL of the standard 0.05 N silver nitrate solution and 10 drops of benzyl alcohol. Shake the contents vigorously and back-titrate as before. Use the total volume of silver nitrate and potassium thiocyanate delivered in the calculation.

3. Sodium Sulfate Reagents

- Barium chloride solution, 100 g/L
- Hydrochloric acid, concentrated
- Methyl orange indicator
- Silver nitrate solution, 50 g/L

Procedure

Weigh 50 g of well-mixed sample to the nearest 0.1 g, and transfer to a 600-mL beaker containing about 200 to 300 mL of water. Add 2 to 4 drops of methyl orange indicator solution, neutralize carefully with concentrated hydrochloric acid, and add 1 mL in excess. If the neutralized solution contains any insoluble material, filter the solution through Whatman No. 42 filter paper or equivalent. Wash the paper once with water. Heat the solution, or filtrate, to boiling. Then add dropwise 10 mL of barium chloride solution to the boiling solution and continue boiling for 3 minutes. Allow the precipitate to settle in a warm place for 4 hours, preferably overnight.

Quantitatively transfer the precipitate to Whatman No. 42 filter paper or equivalent with a fine stream of hot water from a wash bottle. Filter and wash the precipitate with small portions of hot water until the washings are free of chloride. Test for chloride with 3 to 4 drops of silver nitrate solution. Fold the washed filter paper containing the precipitate and place it in an ignited porcelain or platinum crucible, tared to the nearest 0.0001 g. (If desired, the folded, washed filter paper containing the precipitate can be placed in a clean, unglazed fireclay crucible.) Dry and carefully char without flaming. Then ignite at 850 to 900°C for

at least 30 minutes. Partially cool, place in a dessicator and cool to room temperature. If a platinum or porcelain crucible was used, reweigh to the nearest 0.0001 g. Determine the weight of the BaSO₄ precipitate from the difference of the weights. If an unglazed crucible was used, brush the precipitate into a tared watch glass and weigh to the nearest 0.0001 g.

$$\frac{\text{grams BaSO}_4 \times 60.86}{\text{Weight of sample}} = \% \text{ Na}_2\text{SO}_4$$

4. Loss on Heating

Place 10 g of sample in a tared platinum or glazed porcelain dish that has been dried at 250 to 285°C. Weigh accurately and heat in an oven for 2 hours at 265 to 285°C or 4 hours at 250 to 265°C. Cool in a dessicator and weigh accurately and quickly.

$$\frac{\text{Loss in weight in grams} \times 100}{\text{Weight of sample}} = \% \text{ Loss on heating.}$$

5. Iron (Fe) Reagents

- Ammonium acetate solution, 50%
- Ammonium hydroxide solution, 1:1
- Hydrochloric acid, concentrated
- Hydrochloric acid, approximately 10 N
- Hydrochloric acid solution, 1.1
- Hydroxylamine hydrochloride solution, 100 g/L
- Iron solution, standard, one mL = 0.100 mg Fe
- Iron solution, working standard, one mL = 10 (g Fe
- Nitric acid, concentrated
- Orthophenanthroline solution, 3 g/L
- Congo red indicator paper

Analytical Procedures

Special Apparatus

Spectrophotometer (a filter photometer may be substituted, if no spectrophotometer is available) and absorption cells (10 mm and 50 mm)

Preparation of Sample

Weigh 100 g of well-mixed sample to the nearest 0.1 g. Transfer the sample to a 1000-mL Erlenmeyer flask. Slurry the sample with water, keeping the volume below 200 mL. Carefully acidify the sample with 200 mL of 10 N hydrochloric acid. Add the acid in small increments, swirling the sample solution between additions. Allow CO₂ gas to escape before adding the next increment. Heat to boiling on a hot plate, and boil for 1 minute to expel CO₂. Remove from the heat and allow to cool. **If the cooled solution is free of scale**, quantitatively transfer to a 500-mL volumetric flask. Dilute to the mark with water and mix thoroughly.

If scale or insoluble material is present, decant the clear solution into a 500-mL volumetric flask. Add 5 mL of concentrated hydrochloric acid and 2 mL of concentrated nitric acid to the Erlenmeyer flask. Heat to boiling in a hood, and evaporate to near dryness. Cool and quantitatively transfer the residual solution to the 500-mL volumetric flask. Dilute to the mark with water and mix thoroughly.

6. Preparation of Blank

Evaporate 20.0 mL of 10 N hydrochloric acid to near dryness in a 250-mL beaker. If acid treatment of any scale is used in the sample preparation, evaporate a mixture of 20 mL of 10 N hydrochloric acid and 0.2 mL of concentrated nitric acid to near dryness in a 250-mL beaker. Remove from the heat and allow to cool. Transfer the evaporated residual solution to a 100-mL volumetric flask, and dilute to about 50 mL with water.

Procedure

Transfer a 50-mL aliquot of the sample as prepared above to a 100-mL volumetric flask. Carry both the sample and blank solutions through the procedure. Add 4 mL of 1:1 hydrochloric acid solution to each flask, and swirl to mix. Add 5 mL of hydroxylamine hydrochloride solution, shake well and let stand for about 5 minutes to reduce the iron to the ferrous state. Add 5 mL of orthophenanthroline solution, a small piece of Congo red indicator paper, 5 mL of ammonium acetate solution and mix. The indicator paper should now be red.

Cool, make up to the mark with water, mix well, and allow the solution to stand for 15 minutes for complete color development.

Adjust the spectrophotometer to zero absorbance at 510 nm with water in a 50-mm absorption cell (use a 10-mm cell, if the sample contains more than 80 (g Fe). Measure and record the absorbances of the blank and sample solutions. Subtract the blank absorbance from the sample absorbance to obtain net absorbance. Determine the micrograms of iron from the prepared standard curve.

$$\frac{\text{Micrograms of Fe}}{\text{g of sample in aliquot}} = \text{ppm Fe}$$

7. Preparation of Standard Curve

Add 20 mL of water to each of 12 100-mL volumetric flasks. Add, respectively, 0, 0.1, 0.5, 1, 2, 4, 6, 8, 10, 20, 30 and 40 mL of working standard iron solution (1 mL = 10 (g Fe). These volumes are equivalent to 0, 1, 5, 10, 20, 40, 60, 80, 100, 200, 300 and 400 micrograms of Fe. Add water, where necessary, to dilute the solution in each flask to 60 mL and mix thoroughly.

Continue as directed in the procedure with the addition of 5 mL of hydroxylamine hydrochloride solution. Adjust the spectrophotometer to zero absorbance at 510 nm as above, using water in a 50-mm absorption

Analytical Procedures

cell. Using 50-mm cells, measure and record the absorbance of the standard containing no added iron and then the absorbances of the remaining standards. Subtract the absorbance of the zero standard from each of the standard absorbances and plot the net absorbances against the corresponding micrograms of Fe on linear graph paper. Repeat using 10-mm absorption cells and the 0 to 400 (g Fe standards plotting the net absorbances on a separate sheet of linear graph paper.

Remarks: Before each measurement, check the instrument zero with the reference cell. Recheck the standard curve at least every 6 months by running a standard of about the same Fe content as the sample along with the sample analysis.

8. Reagents, Indicators and Standard Solutions

Preparation methods for the reagents, indicators and standard solutions required by the above procedures follow. Use reagent grade chemicals, unless otherwise specified, and distilled or deionized water.

Reagents

- **Acid, Hydrochloric, Concentrated**, 36-38% HCl.
- **Acid, Hydrochloric**, approximately 10 N — Dilute 850 mL of concentrated hydrochloric acid to the mark in a 1000-mL volumetric flask with water.
- **Acid, Hydrochloric, Solution**, 1:1 — To 25 mL of water add 25 mL of reagent grade concentrated hydrochloric acid (36-38% HCl). Store in a dropping bottle.
- **Acid, Nitric, Concentrated**, 69-71% HNO₃.
- **Acid, Sulfuric, Concentrated**, 96-98% H₂SO₄.
- **Ammonium Acetate**, 50% — Dissolve 500 g of reagent grade ammonium acetate crystals (CH₃COONH₄) in 500 mL of water in a 1500-mL beaker. Mix thoroughly and store in a polyethylene bottle.

- **Ammonium Hydroxide, Concentrated**, 28% NH₃.

• **Ammonium Hydroxide Solution**, 1:1 — To 25 mL of water add 25 mL of reagent grade ammonium hydroxide (28-30% NH₄OH). Store in a polyethylene dropping bottle.

• **Barium Chloride Solution**, 100 g/L — Dissolve 100 grams of barium chloride (BaCl₂·2H₂O) in sufficient water to make 1000 mL. Filter if turbid.

• **Benzyl Alcohol**, C₆H₅CH₂OH — Dispense from an amber-glass dropping bottle.

• **Hydroxylamine Hydrochloride Solution**, 100 g/L — Dissolve 100 g of reagent grade hydroxylamine hydrochloride crystals (NH₂OH·HCl) in about 600 mL of water in a 1000-mL beaker. Transfer to a 1000-mL volumetric flask, dilute to the mark with water, and mix thoroughly. Store in a polyethylene bottle.

• **Orthophenanthroline Solution**, 3 g/L — Warm about 500 mL of water in a 1000-mL beaker to 60-65°C. Add 3.0 g of reagent grade 1,10-phenanthroline monohydrate crystals (C₁₂H₈N₂·H₂O) and stir to dissolve. Cool the solution to room temperature. Add 1 mL of reagent grade concentrated hydrochloric acid and mix. Transfer the solution to a 1000-mL volumetric flask, dilute to the mark with water and mix well. Store the solution in a polyethylene bottle.

• **Silver Nitrate Solution**, 50 g/L — Dissolve 5 g of silver nitrate (AgNO₃) in water and dilute to 100 mL.

Indicators

• **Iron Solution Indicator** — To a 1500-mL beaker add 62 grams of ferric ammonium sulfate [Fe₂(SO₄)₃·(NH₄)₂SO₄·24H₂O] and 500 mL of water. Stir until the crystals are dissolved. Add 440 mL of concentrated nitric acid and mix. Store the solution in a polyethylene bottle.

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•**Methyl Orange Indicator** — Dissolve 0.1 g of methyl orange in 100 mL of water. Filter the solution, if necessary.

•**Methyl Orange Indicator, Modified** — Dissolve 0.1 g of methyl orange and 0.14 g of xylene cyanole FF dye, technical grade, in 100 mL of water. Filter, if necessary.

Standard Solutions

For precise analytical work, prepare standard solutions with distilled water at 20°C. Perform all titrations at this temperature or apply corrections as given in the temperature correction method given below.

•**Acid, Sulfuric, Standard, 0.05-1 N.**

Measure the required volume* of concentrated sulfuric acid in a graduated cylinder, and pour carefully into a volume of water equal to one half the final desired volume of solution. Rinse the cylinder into the mixture with water. Mix thoroughly, allow to cool, and dilute to the final desired volume with water. Mix well again and store in a tightly closed glass container.

* The required quantity of concentrated sulfuric acid can be approximated as:

$$\frac{\text{Liters of final solution desired}}{\text{x Normality desired} \times 30.0} = \frac{\text{mL of concentrated}}{\text{sulfuric acid to use.}}$$

Transfer 16 g of primary standard grade anhydrous sodium carbonate to a Pyrex weighing bottle and dry in an oven at 265 to 285°C for 2 hours or at 250 to 265°C for 4 hours. Place the cover on the weighing bottle loosely and cool in a desiccator. Weigh, to the nearest 0.0001 g, three portions of the required weight of dried sodium carbonate into three 500-mL Erlenmeyer flasks. The weights of dried sodium carbonate required for the corresponding normalities of sulfuric acid solution are shown in Table 9-1.

Normality of H ₂ SO ₄ Solution	Weight of Dried Na ₂ CO ₃ , Required, g
0.05	0.25-0.26
0.10	0.50-0.52
0.20	1.00-1.04
0.50	2.5-2.6
1.0	5.0-5.2

Table 9-1: Normalizing Sulfuric Acid with Dry Soda Ash

Add 100 mL of water to each flask, and swirl to dissolve the solid. Add 5 drops of methyl orange indicator solution or modified methyl orange indicator solution. Titrate with the sulfuric acid solution from a 100-mL buret to the pink-color end point of methyl orange indicator. If modified methyl orange indicator is used, titrate to a gray end point. Record the volume of sulfuric acid used for each titration. Correct the volume delivered to 20°C as given in the temperature correction method below. Restandardize monthly. Average the triplicate results.

$$\frac{\text{g Na}_2\text{CO}_3 \text{ used} \times 18.870}{\text{mL of H}_2\text{SO}_4 \text{ corrected to } 20^\circ\text{C}} = \text{Normality of H}_2\text{SO}_4$$

•**Iron Solution, Standard, 1 mL = 0.100 mg Fe.**

Weigh exactly 0.7022 g of reagent grade ferrous ammonium sulfate hexahydrate [Fe(NH₄)₂(SO₄)₂·6H₂O], and transfer it to a 1000-mL volumetric flask containing about 500 mL of water and 20 mL of concentrated reagent grade sulfuric acid (H₂SO₄). Swirl to dissolve the crystals, and dilute to the mark with water. Mix thoroughly. Discard after one month.

•**Iron Solution, Working Standard, 1 mL = 10.0 micrograms Fe.**

To a 1000-mL volumetric flask add about 500 mL of water. Pipet exactly 100 mL of the standard Fe solution (1 mL = 0.100 mg Fe) into the flask. Dilute to the mark with water and mix thoroughly. Prepare fresh daily as needed.

Analytical Procedures

•Potassium Thiocyanate, Standard, 0.05 N.

Weigh 4.86 g of potassium thiocyanate (KSCN) to the nearest 0.01 g in a tared polypropylene weighing bottle. Using a wash bottle, quantitatively transfer the crystals through a powder funnel into a 1-liter volumetric flask. Add 200 to 300 mL of water, and swirl to dissolve the crystals. Dilute to the mark with water and mix thoroughly. Store the solution in a polyethylene bottle.

Silver Nitrate, Standard, 0.05N.

Weigh 8.495 g of silver nitrate, (AgNO₃) to the nearest 0.001 g, in a tared polypropylene weighing bottle. Using a water wash bottle, quantitatively transfer the crystals through a powder funnel into a 1-liter volumetric flask. Add 200 to 300 mL of water and swirl to dissolve the crystals. Dilute to the mark with water and mix thoroughly. Store in a tightly stoppered amber-glass bottle. Standardize the solution as follows.

Pipet 25.00 mL of standard 0.05 N sodium chloride solution into each of three 500-mL Erlenmeyer flasks. Carry each flask through the following steps. Add 100 mL of water and 3 mL of iron indicator solution. Swirl to mix. Add 27 mL of the silver nitrate solution being standardized from a 50-mL buret, while swirling vigorously. Add 10 drops of benzyl alcohol and shake the flask for 30 seconds. Rinse down the inside wall of the flask with water. Using a 50-mL buret, back-titrate slowly and with constant swirling, with 0.05 N potassium thiocyanate solution until a faint red color persists. Average the results and restandardize monthly.

$$\frac{\text{mL NaCl solution} \times 0.0500}{[\text{mL AgNO}_3 \text{ solution} - (\text{mL KSCN solution} \times F^*)]} = \text{Normality of AgNO}_3$$

*Determine the factor F as follows: Add about 9 mL of the silver nitrate solution from the 50-mL buret to a 250-mL Erlenmeyer flask, Add 100 mL of water and 3 mL of iron indicator solution. Swirl to

mix. Titrate slowly with the potassium thiocyanate solution, while constantly swirling, until a faint reddish color persists.

$$F = \frac{\text{mL AgNO}_3}{\text{mL KSCN}}$$

Repeat the determination and use the average factor rounded off to the nearest 0.001 mL AgNO₃/mL KSCN. Determine the factor each time a new KSCN or AgNO₃ solution is prepared.

•Sodium Chloride, Standard, 0.05 N.

Dry about 3.1 g of sodium chloride (NaCl) on a large watch glass in an oven at 105 to 110°C for 2 hours. Cool the crystals to room temperature in a dessicator. Weigh 2.9221 g of the dried sodium chloride to the nearest 0.0001 g in a tared polypropylene weighing bottle. Using a water wash bottle, quantitatively transfer the crystals through a powder funnel to a 1-liter volumetric flask. Add 200 to 300 mL of water and swirl to dissolve the crystals. Dilute to the mark with water and mix thoroughly. Store the solution in a polyethylene bottle.

9. Temperature Corrections for Volumetric Solutions

As volumetric solutions are standardized at 20°C, titrations should be made at this temperature or corrections applied to reduce the volume of the solution used in filtration to 20°C. Table 9-2 shows corrections for temperatures from 15 to 30°C. Columns labelled "mL" give the milliliters to be added (≥) or deducted (≤) for each mL used in titration. "Factor" gives the correction factor for converting the quantity of solution to standard volume at 20°C. Corrections for 0.05 N solution apply also to weaker solutions and distilled water.

Analytical Procedures

Temperature °C	mL Correction per mL Used			Factor		
	N/20 or less Solutions	N/10 Solutions	N/1 Solutions	N/20 or less Solutions	N/10 Solutions	N/1 Solutions
15	+0.0008	+0.0008	+0.0011	1.0008	1.0008	1.0011
16	+0.0006	+0.0007	+0.0009	1.0006	1.0007	1.0009
17	+0.0005	+0.0005	+0.0007	1.0005	1.0005	1.0007
18	+0.0003	+0.0004	+0.0005	1.0003	1.0004	1.0005
19	+0.0002	+0.0002	+0.0003	1.0002	1.0002	1.0003
20	0.0000	0.0000	0.0000	1.0000	1.0000	1.0000
21	-0.0002	-0.0002	-0.0003	0.9998	0.9998	0.9997
22	-0.0004	-0.0004	-0.0005	0.9996	0.9996	0.9995
23	-0.0006	-0.0006	-0.0008	0.9994	0.9994	0.9992
24	-0.0008	-0.0009	-0.0012	0.9992	0.9991	0.9988
25	-0.0010	-0.0011	-0.0015	0.9990	0.9989	0.9985
26	-0.0013	-0.0013	-0.0018	0.9987	0.9987	0.9982
27	-0.0015	-0.0016	-0.0022	0.9985	0.9984	0.9978
28	-0.0018	-0.0019	-0.0025	0.9982	0.9981	0.9975
29	-0.0020	-0.0021	-0.0029	0.9980	0.9979	0.9971
30	-0.0023	-0.0024	-0.0033	0.9977	0.9976	0.9967

Table 9-2 *Temperature Corrections for Volumetric Solutions**

*Data in the table are from Bureau of Standards Circular No.19.

10 Precautions in Handling

Soda ash is not classified as highly toxic, but can injure the eyes and irritate the skin upon contact and be harmful if ingested. It has an acute oral LD50 of 2.8 g/kg (rat) and a primary skin irritation index (PSII) of 2.54 (rabbit). As a result, inhalation, ingestion, and exposure to the eyes should be minimized.

Limited exposure to soda ash dust is not ordinarily harmful, because its dust is soluble and does not accumulate in the pulmonary tract. Nevertheless, dust concentrations high enough to impair visibility or that exceed permissible government regulations should be avoided. Some individuals are unusually sensitive to soda ash dust. Although certain minimum standards for soda ash dust must be observed, the use of personal protective equipment may vary depending on individual sensitivity.

Soda ash dust may temporarily irritate the nose and throat. Also, some people are allergic to alkaline materials and may develop dermatitis when working with soda ash. Those with pre-existing skin diseases are particularly susceptible. In warm weather, soda ash dust can dissolve in perspiration and cause some discomfort. This condition is aggravated if lime dust is also present, because the two materials combine to form caustic soda (sodium hydroxide) in the presence of moisture, e.g., perspiration. Caustic soda is a strong and corrosive alkali, so every effort should be made to avoid simultaneous exposure to both materials.

As required, exposed personnel should use a respirator approved by NIOSH for dusts. At a minimum, personnel handling soda ash should wear a hard hat, chemical safety goggles, long-sleeve shirt, trousers and gloves for routine product use (cotton gloves for dry product and impervious gloves for solutions). Personnel should not wear contact lenses.

NIOSH-approved safety belts and lifelines should always be worn by workmen entering bins or silos containing soda ash so it is easier to rescue them if the material should suddenly shift or slide.

Clean up soda ash spills promptly. Correct dusting conditions immediately, and clean up any soda ash dust. Soda ash is not flammable, and its dust is not explosive.

First Aid

Wash skin that comes in contact with soda ash with plenty of water. For eye contact, flush eyes with plenty of water for at least 15 minutes and get medical attention. For ingestion, have victim drink large amounts of water to dilute the material. Do not induce vomiting. Get medical attention for irritation, ingestion or discomfort from inhalation.