

# **CORROSION RESISTANCE OF NICKEL-CONTAINING ALLOYS IN PHOSPHORIC ACID (CEB-4)**

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A PRACTICAL GUIDE TO THE USE  
OF NICKEL-CONTAINING ALLOYS  
N° 415

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# Corrosion Resistance of Nickel-Containing Alloys in Phosphoric Acid

## Part I. LABORATORY STUDIES

PHOSPHORIC ACID is a major chemical product; in the United States the only acids produced in larger tonnage are sulfuric and nitric. It is produced from phosphate rock by either of two methods, the wet process and the electric furnace process. The wet process is responsible for about 68 per cent of American production; its output goes mainly into fertilizers such as triple superphosphate, ammonium phosphate and mixed fertilizers. The purer furnace-process acid finds its way mainly into sodium and potassium phosphates principally used for soap and detergent manufacture; some is used for fertilizers, feed supplements, water conditioning, metal cleaning and coatings, food and beverage products, plasticizers, insecticides, flame-proofing agents and gasoline additives.

Phosphoric acid is generally less corrosive to many metals and alloys than are sulfuric and hydrochloric acids. For several nickel-containing alloys it is only mildly corrosive at temperatures below about 212°F (100°C), independently of concentration. Beyond this temperature its corrosivity becomes substantial toward most metals, regardless of whether they are normally considered resistant to oxidizing or reducing conditions. Its properties are affected quite substantially by its process of manufacture and its impurities. Acid produced by the wet process may contain a significant concentration of sulfate, fluoride and fluosilicate, while furnace-process acid is nearly free of these contaminants. Accordingly corrosion behavior and alloy selection are functions of the manufacturing process as well as of the more familiar variables of concentration, temperature, aeration, impurities and velocity.

In this bulletin concentrations of acid are normally expressed in per cent by weight of orthophos-

phoric acid ( $H_3PO_4$ ) solute, unless otherwise noted. Some concentrations are expressed in per cent by weight of phosphorus pentoxide ( $P_2O_5$ ) equivalent to the phosphoric acid contained; the relation is that 1 per cent  $P_2O_5$  is equivalent to 1.38 per cent  $H_3PO_4$ .

Nominal compositions of nickel alloys which have been reported or used to some extent in phosphoric acid service are shown in Table 1. Most are wrought alloys, used largely in the form of sheet, plate, pipe, tubing, or bar. Those marked (a), (c), or (d) are essentially cast alloys, employed largely for pumps and valves.

### Stainless Steels

Wrought stainless steels are described by type numbers assigned by the American Iron & Steel Institute (AISI); those containing chromium and nickel constitute the 300 series. Cast stainless steels are designated by type numbers of the Alloy Casting Institute (ACI); those containing chromium and nickel and intended for corrosion-resistant service are Types CD to CN.

The austenitic stainless steels of chromium-nickel-molybdenum (and to a lesser extent, chromium-nickel) compositions are commonly used for handling phosphoric acid solutions within limits of concentration, temperature, aeration and purity for which they are suitable. The results of a number of laboratory tests in chemically pure acid are shown in Table 2. These are taken from a number of sources, so that most of them are not closely comparable. Conditions of tests may be found in the original references.

The corrosivity of phosphoric acid is intermediate, such that specimens of stainless steel which

Table 1

## Nominal Compositions of Nickel Alloys in Use or Corrosion-Tested in Phosphoric Acid Solutions

Composition, %	Ni	Fe	Cr	Mo	Cu	C	Si	Mn	Other
<b>Stainless Steels</b>									
AISI Type 304	9.5	70	18	–	–	0.08 max	0.5	1.3	–
AISI Type 304L	10	69	18	–	–	0.03 max	0.5	1.3	–
AISI Type 310	21	52	25	–	–	0.25 max	0.75	1.3	–
AISI Type 316	13	65	17	2.0 min	–	0.08 max	0.5	1.7	–
AISI Type 316L	13	63	17	2.0 mm	–	0.03 max	0.5	1.8	–
AISI Type 317	14	61	19	3.0 min	–	0.08 max	0.4	1.6	–
AISI Type 329	4.5	67	27	1.5	–	0.20 max	–	–	–
AISI Type 347	11	68	18	–	–	0.08 max	0.5	1.3	Cb(+Ta) 0.6
ACI Type CD-4MCu (c)	5.4	62	26	2.0	3.0	0.04 max	1.0 max	1.0 max	–
ACI Type CF-3 (c)	10	68	19	–	–	0.03 max	2.0 max	1.5 max	–
ACI Type CF-8 (c)	9.5	68	19.5	–	–	0.08 max	2.0 max	1.5 max	–
ACI Type CF-3M (c)	11	65	19	2.5	–	0.03 max	1.5 max	1.5 max	–
ACI Type CF-8M (c)	10.5	65	19.5	2.5	–	0.08 max	1.5 max	1.5 max	–
ACI Type CG-8M (c)	11	64	19.5	3.5	–	0.08 max	1.5 max	1.5 max	–
ILLIUM* "P" alloy (c)	8	57	28	2.25	3.25	0.20	0.75 max	1.0 max	–
<b>Iron base-Nickel-Chromium-Copper-Molybdenum Alloys</b>									
WORTHITE* stainless steel (d)	24	48	20	3	1.75	0.07 max	3.25	0.6	–
CARPENTER* Stainless No. 20Cb	29	44	20	2.5	3.3	0.07 max	0.6	0.75	Cb(+Ta) 0.6
ACI Type CN 7M (a)	29	44	20	2.2	3.3	0.07 max	1.5 max	1.5 max	–
CARPENTER* Stainless No. 20Cb-3	34	39	20	2.5	3.3	0.07 max	0.6	0.75	Cb(+Ta)0.6
<b>Nickel base-Iron-Chromium-Molybdenum Alloys</b>									
INCOLOY* alloy 825	41.8	30	21.5	3.0	1.80	0.03	0.35	0.65	A10.15; Ti 0.90
HASTELLOY* alloy G (b)	45	19.5	22.2	6.5	2.0	0.03	0.35	1.3	W0.5; Cb+Ta 2.12
HASTELLOY* alloy F (b)	47	17	22	6.5	–	0.05 max	–	–	Co 2.5 max; W 0.6; Other 5.5
<b>Nickel base-Chromium-Molybdenum Alloys</b>									
ILLIUM* "98" alloy (c)	55	1.0	28	8.5	5.5	0.05	0.7	1.25	–
ILLIUM* "G" alloy (c)	56	6.5	22.5	6.4	6.5	0.20	0.65	1.25	–
INCONEL* alloy 625	61	3	22	9.0	0.1	0.05	0.3	0.15	Cb 4
ILLIUM* "R" alloy	68	1	21	5	3	0.05	0.7	1.25	–
<b>Nickel base-Molybdenum-Chromium Alloy</b>									
HASTELLOY* alloy C (b)	54	5	15.5	16	–	0.08 max	1.0 max	1.0 max	Co 2.5 max; W4;V0.4max
<b>Nickel base-Molybdenum Alloy</b>									
HASTELLOY* alloy B (b)	61	5	1 max	28	–	0.05 max	–	–	Co 2.5max; Other 3
<b>Nickel base-Silicon Alloy</b>									
HASTELLOY* alloy D (c)	82	2 max	1 max	–	3.0	0.12 max	9	–	Co 1.5 max; Other 2
<b>Nickel-Alloyed Cast Irons</b>									
Ni-Resist (Type 1) (c)	15.5	69	2.5	–	6.5	2.8	2.0	1.2	–
Ni-Resist (Type 2) (c)	20	72	2.5	–	–	2.8	2.0	1.0	–
Ni-Resist (Ductile Type D-2) (c)	20	72	2	–	–	3.0 max	2	0.85	P.08 max
<b>Nickel-Copper Alloys</b>									
MONEL* alloy 400	66	1.35	–	–	31.5	0.12	0.15	0.90	–
Copper-Nickel alloy CA 715	31	0.55	–	–	67	–	–	1.0 max	Pb .05max; Zn 1.0 max
<b>Nickel</b>									
Nickel 200	99.5	0.15	–	–	0.05	0.06	0.05	0.25	–
<b>Nickel base-Chromium Alloys</b>									
CORRONEL* alloy 230	55 min	5.0 max	36	–	1.0 max	0.08 max	0.6 max	1.0 max	Ti 1.0 max; Al 0.5 max
INCONEL* alloy 600	76	7.2	15.8	–	0.10	0.04	0.20	0.20	–
<b>Iron-Nickel-Chromium Alloy</b>									
INCOLOY* alloy 800	32	46	20.5	–	0.30	0.04	0.35	0.75	–

\* See inside back cover for registered trademarks

(a) Cast "20" alloys, such as DURIMET\* 20, ALOYCO\* 20, etc.

(b) Composition of wrought alloy; available also in cast form

(c) Available in cast form only

(d) Composition of cast alloy

have been activated before immersion and thereafter exposed to air may remain active when re-immersed or may regain their passivity. In one study by Warren<sup>4</sup>, Type 304 specimens were activated in 15 per cent hydrochloric acid, then exposed in boiling 25 per cent phosphoric acid. During the initial 24-hour exposure they were active, corroding at rates of 7700 to 10,300 mils per year. Those which remained active when re-immersed after re-weighing displayed rates of 8000 to 8500 mils per year, while those which regained passivity had rates of 1 mil per year.

A comprehensive laboratory study of corrosion rates in a rather brief exposure in chemically pure phosphoric acid solution was made by Bünger<sup>7</sup> over a wide range of temperature and concentration. Intervals were 36 degrees Fahrenheit and 10 percentage points concentration. Durations of exposure were seven days at temperatures up to 212°F, 24 hours above that temperature. Results for chromium-nickel stainless steel appear in Fig. 1.

A recently presented laboratory study of furnace-grade acid corrosivity<sup>8</sup> has summarized the behavior of Type 316 stainless steel in Fig. 2, reprinted from this source.

**Table 2**  
**Laboratory Tests of Stainless Steels in Pure Phosphoric Acid**

H <sub>3</sub> PO <sub>4</sub> Conc., % by wt.	Temp, °F	Dura- tion, days	Corrosion Rate, mils per year			Ref
			Type 304		Type 316	
			Not aerated	Not aerated	Aerated	
10	200	≥16	-	0.1	0.2	1
10 (a)	(214) BP	0.08	Nil	-	-	2
10	214	2+2	< 1	<1	-	3
25	(216) BP	5	1	-	-	4
50	230	2 + 2	8	6	-	3
60	(240) BP	0.83	-	107	-	5
70	(259) BP	0.83	-	212	-	5
76	185	16	-	0.05	0.00	6
76	212	16	-	1.4	0.44	6
76	239	16	-	6.7	0.71	6
78-85	Room	≥16	< 0.1	<0.1	-	1
78-85	220	≥16	230	5	-	1
78-85	240	≥16	-	-	9.2	1
80	(294) BP	0.83	-	793	-	5
85	185	16	-	0.14	0.17	6
85	212	16	-	1.19	0.38	6
85	239	16	-	5.7	0.67	6
86	208	2 + 2	> 1000	16	-	3

(a) Specimens activated before exposure  
BP Boiling point  
≥ Greater than or equal to

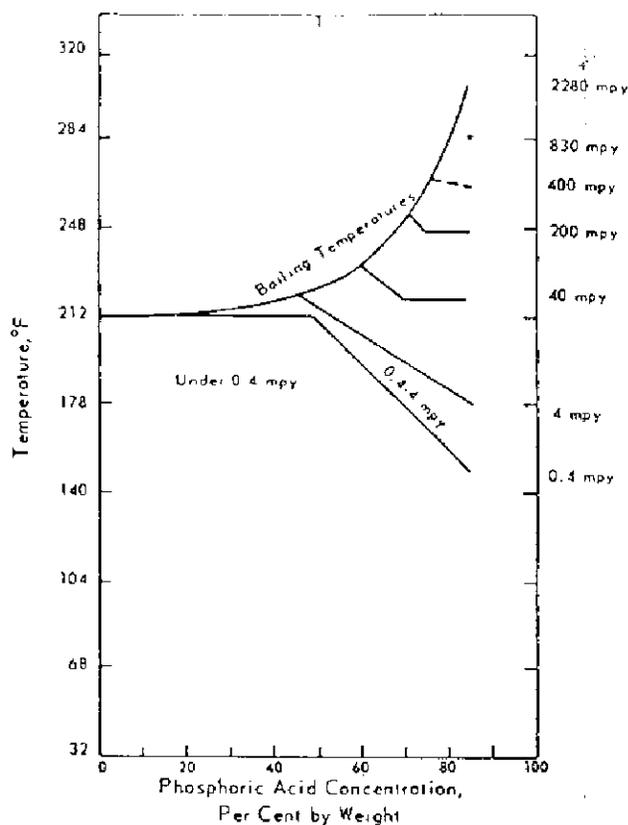


Fig. 1—Corrosion of wrought 18-9 stainless steel in phosphoric acid.

In phosphoric acid concentrations over about 80 to 95 per cent the corrosivity toward stainless steels decreases. Illustrations of this behavior, similar to that of sulfuric acid, are Fig. 2 and in addition, Fig. 3, presented by Kemp and Zeh<sup>9</sup> for Type 317 stainless steel in phosphoric acid of unspecified purity. The acid concentration range covered is from 85 to 115 per cent. Concentrations over 100 per cent represent orthophosphoric acid equivalent of solutions which contain some pyrophosphoric acid.

Some information on corrosion of Type 316 stainless steel is found in studies of the behavior of higher alloyed materials, where it serves as a standard for comparison. This information will be found in the subsequent sections which are devoted to these alloys.

A proprietary stainless steel alloy, ILLIUM "P", has been designed for resistance to combined corrosion and abrasion in phosphoric acid solutions containing suspended matter.

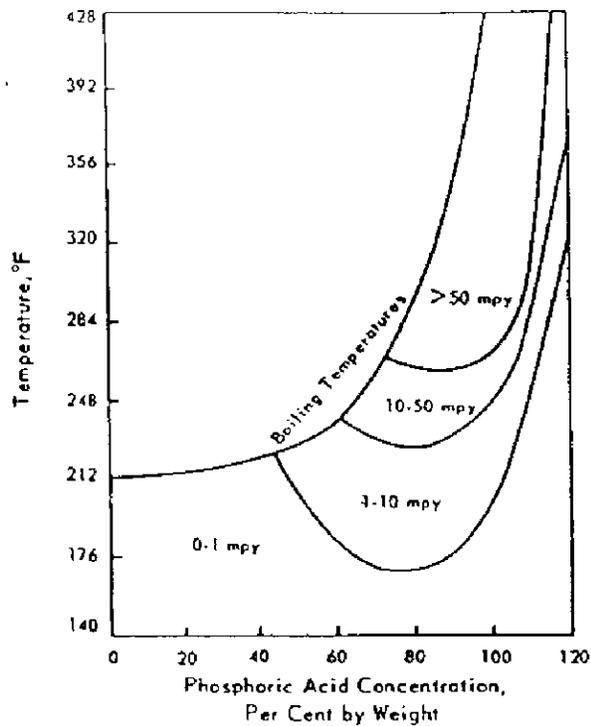


Fig. 2 - Isocorrosion chart for Type 316 stainless steel in furnace-grade phosphoric acid under mildly agitated conditions.

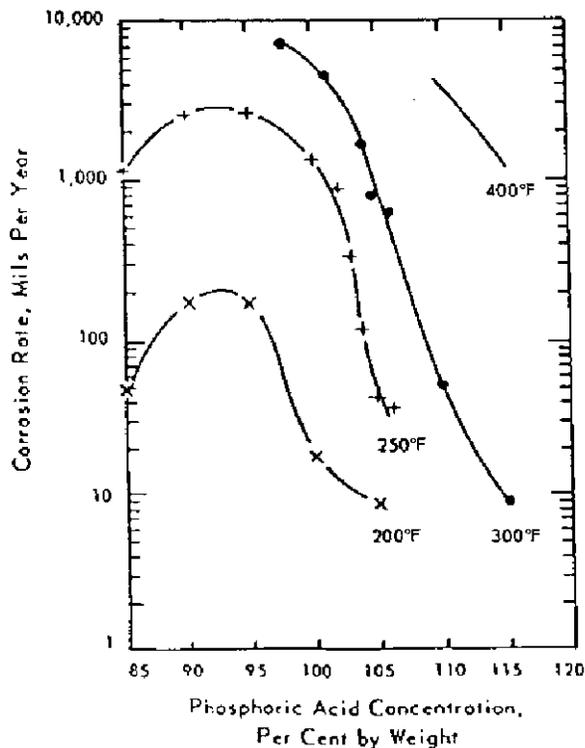


Fig. 3—Corrosion of Type 317 stainless steel in phosphoric acid.

### Oxidizing Ions in Solution

It has been found that cupric<sup>9,10</sup> and ferric ions<sup>11</sup> in solution inhibit the corrosion of stainless steels in phosphoric acid. The cupric ion may be provided by initial corrosion of a copper-containing alloy, in which situation the corrosion rate decreases as the cupric ion accumulates.<sup>10</sup> A correlation between copper content of various heats of Type 316 stainless steel and their corrosion rates in boiling 85 per cent phosphoric acid has been shown by Ebling and Scheil<sup>10</sup>; this is reproduced as Fig. 4.

The effect of copper appears in the improved corrosion resistance of the highly alloyed stainless steels of the "20" series. These are described on page 8.

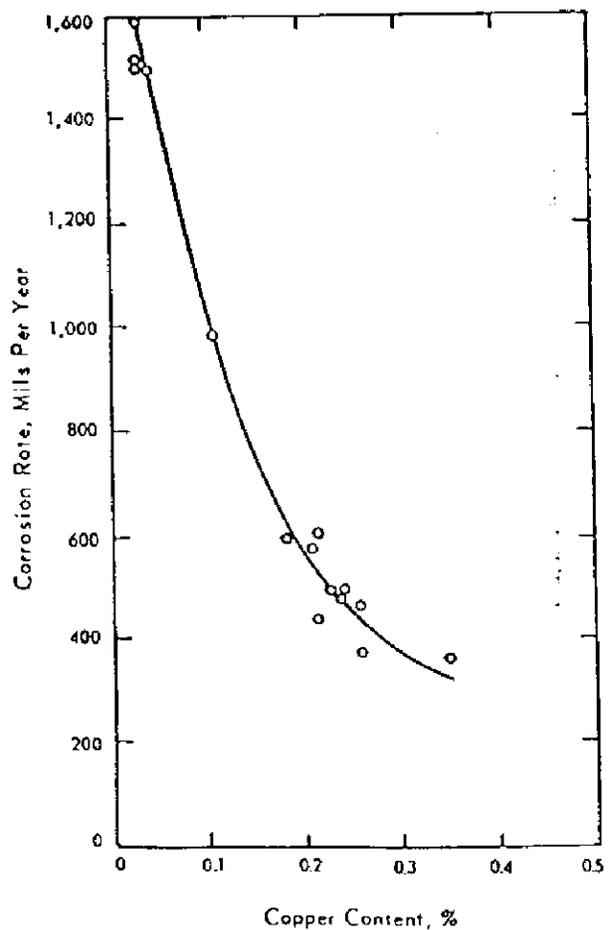


Fig. 4—Effect of copper content in Type 316L stainless steel on resistance to corrosion in boiling 85% phosphoric acid.

## Halide Ions in Solution

Addition of chloride or fluoride ions to phosphoric acid in effect produces mixtures of phosphoric and hydrochloric or hydrofluoric acids. Such mixtures are more effective than is halide-free phosphoric acid in breaking down the film on passive metals, and are consequently more corrosive.

Following a report<sup>12</sup> of corrosion of a Type 304 stainless steel storage tank used for 75 per cent plant phosphoric acid, electric furnace grade contaminated with 356 ppm chloride, a laboratory study showed a positive correlation between concentration of added chloride and higher corrosion rate of Type 304, and indicated the superiority of Type 316. The results are shown in Tables 3 and 4.

**Table 3**  
**Effect of Contamination on Corrosivity of Phosphoric Acid**

Laboratory test in 75% phosphoric acid, ACS specification grade vs. contaminated electric furnace acid. Specimens exposed in Erlenmeyer flasks.

Temperature: 100°F (38°C). No aeration or agitation.

Duration: 168 hours or 7.0 days (but interrupted for weighing at 48 and 120 hours).

Acid Grade	Contaminants, ppm		Corrosion Rate, mils per year	
	Cl <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	Type 304	Type 316
ACS reagent	<5	Unspecified	<0.1	<0.1
Plant	356	250	213	1.4

**Table 4**

### Effect of Chloride on Corrosion by Phosphoric Acid

Laboratory test in 75% reagent grade phosphoric acid containing specified concentrations of added chloride. Specimens submerged in glass flasks.

Temperature range: 85-100°F (29-38°C).

Duration: 23 hours, no aeration or agitation.

Stainless Steel Type	ppm Cl <sup>-</sup> added	Corrosion Rate, mils per year
304	0	nil
304	8	1.4
304	15	11
304	23	508 (a)
304	39	910 (a)
304	117	973 (a)
304	156	903 (a)
304	196	1081 (a)
304	274	733 (a)
304	391	912 (a)
316	196	0.6

(a) Hydrogen evolved and acid colored green

In view of this experience and these data the firm involved standardized on Type 316 stainless steel for tanks when the phosphoric acid had a chloride content over 15 parts per million. A few tests in processes involving chloride-containing wet process acid have shown unusually high rates; results of tests appear subsequently among data for industrial applications of the acid.

The effect of chloride contamination in 85% reagent acid has also been studied in detail in a recent publication.<sup>8</sup> Chloride significantly increased acid corrosivity to Type 316 stainless steel at about 110 ppm, and to Type 304 at 30 ppm, both at 212°F (100°C).

Fluoride ion and the related complex fluosilicate ion are more commonly encountered halide contaminants because of their occurrence in wet-process acid. Directly comparable laboratory data are unavailable. The most nearly consistent information available for Type 316 stainless steel is for exposure in boiling 25 per cent solution, presumably un-aerated. This is shown in Table 5.

**Table 5**  
**Laboratory Corrosion Tests on Fluoride Impurity in Boiling 25% Phosphoric Acid**

Hf Content, %	Corrosion Rate of Stainless Type 316, mils per year	Ref
0	0.7	7
0	1.8	13
2	41	14

Further evidence is found in a series of pilot plant tests in hot phosphoric acid containing hydrofluosilic acid with controlled contents of hydrofluoric and sulfuric acids, simulating wet-process acid. These three additions increase the corrosivity of the hot solution, as shown in Fig. 5, taken from Reference 15.

## Effect of Metallurgical Factors

When the structure of stainless steel has been affected by welding or heat-treating, phosphoric acid has been found to corrode the steel intergranularly in some instances. In laboratory studies<sup>6</sup> of sensitized Type 316 stainless steel, intergranular corrosion did not occur in 75 and 85 per cent elec-

tric furnace process acid at 185 to 240°F (85 to 116°C), but field tests in 75 per cent furnace acid showed no attack at 90°F (32°C), mild at 140°F (60°C) and severe at 203°F (95°C). Significant intergranular attack of sensitized Type 316 by boiling impure wet-process acid has been seen in evaporators producing concentrations above 95 per cent, but it has not occurred in all cases. At storage temperatures near 125°F (52°C), standard welded Type 316 stainless has been used successfully. According to one unpublished source, however, Type 316L is definitely preferable to Type 316 at temperatures over 100°F (38°C). Until the limiting conditions are more fully defined, it seems necessary to conclude that phosphoric acid is capable under some conditions of causing intergranular corrosion to sensitized stainless steels. If there is any doubt about the severity of the exposure, it would be prudent to use low-carbon or stabilized alloys for welded equipment.

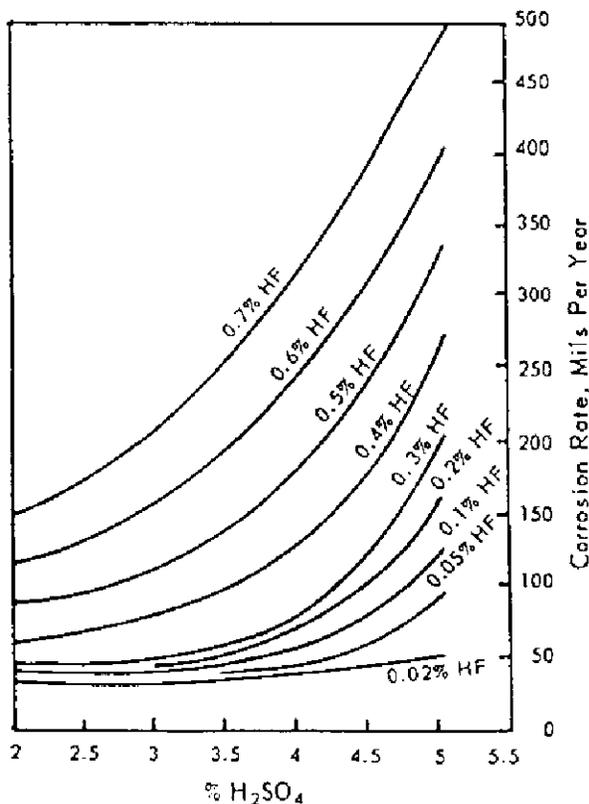


Fig. 5—Corrosion rate of Type 316 stainless steel in 35% phosphoric acid (25% P<sub>2</sub>O<sub>5</sub>) with 1.5% F as H<sub>2</sub>SiF<sub>4</sub> at 190°F (88°C).

### Alloys of Nickel, Iron and Chromium with Molybdenum and Copper

Several commercial alloys of nickel, iron and chromium containing copper and molybdenum are shown in Table 1. These include the highly alloyed stainless steels: WORTHITE, DURIMET 20 and ALOYCO 20, which are cast steels meeting the Alloy Casting Institute specification CN-7M, and wrought CARPENTER Stainless No. 20Cb and No. 20Cb-3. They also include the nickel-base materials INCOLOY alloy 825 and HASTELLOY alloys F and G.

These materials are widely useful in pumps and valves, and show resistance superior to that of the conventional stainless steels at higher temperatures and in the more severe concentrations of acid. The wrought alloys are available as sheet, pipe, tubing, hot-rolled rods and other forms; all are stabilized to avoid intergranular corrosion. To provide maximum resistance to intergranular corrosion, castings have been heat-treated at the foundry. If they are welded afterwards, they will again require a solution-annealing heat treatment and a rapid quench in water. The annealing temperature is in the range 1850 to 2150°F (1010 to 1177°C) depending on the alloy; specific information may be obtained from publications of the producers.

The results of a number of laboratory corrosion tests of these series of alloys in pure phosphoric acid solutions are shown in Tables 6, 7 and 8, and in Figure 6<sup>7</sup>. Direct cross-comparisons cannot be made between tables because conditions are not equivalent. Table 8 again shows the reduction in corrosivity of high concentrations of acid, over 100 per cent for example.

**Table 6**  
Laboratory Corrosion Tests of Nickel-Iron-Chromium-Molybdenum Alloys in Phosphoric Acid<sup>8</sup>

Temperature: Boiling.  
Duration: 20 hr.

H <sub>3</sub> PO <sub>4</sub> Conc., % by wt	Corrosion Rate, mils per year			
	CARPENTER Stainless No. 20Cb	HASTELLOY alloy F	INCOLOY alloy 825	Stainless Steel Type 316L
60	9	97	8	107
70	11	73	7	212
85	38	1200	51	793

**Table 7**  
**Laboratory Corrosion Tests of Stainless Steels in Phosphoric Acid<sup>3</sup>**

Duration: 96 hr.

H <sub>3</sub> PO <sub>4</sub> , Conc., % by wt	Temp., °F	Corrosion Rate, mils per year	
		CARPENTER	
		Stainless No. 20Cb	Stainless Steel Type 316
10	214	<1	<1
50	230	17	6
86	208	2	16

Additional graphical information on HASTELLOY alloy F is presented under nickel-molybdenum alloys, where it is grouped with other information from the manufacturer.

### Alloys of Nickel, Chromium and Molybdenum

Nickel-base alloys of low iron content are shown in Table 1. Among alloys named are those containing intermediate molybdenum levels: ILLIUM alloys "98", "G" and "R" and INCONEL alloy 625. There are also the high-molybdenum alloys typified by HASTELLOY alloy C. These materials are resistant to a wide range of concentration and temperature.

ILLIUM "G", a cast material, is usefully resistant to all concentrations of phosphoric acid at tempera-

tures up to 190°F (88°C). Data provided by the producer indicate a rate below 1.7 mils per year

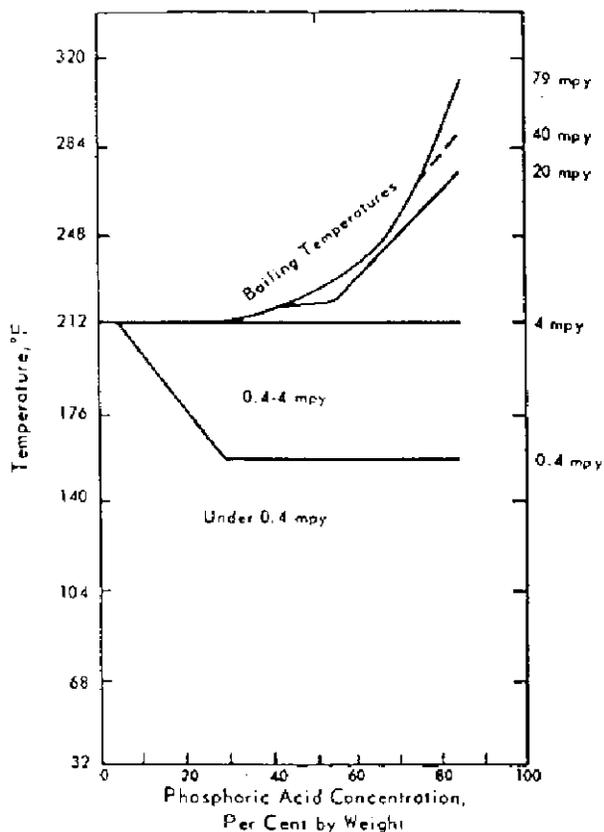


Fig. 6—Corrosion of cast 18 Cr-25 Ni-4 Mo-2 Cu stainless steel in phosphoric acid.

**Table 8**  
**Laboratory Tests of Nickel-Iron-Chromium-Molybdenum Alloys in Phosphoric Acid**

H <sub>3</sub> PO <sub>4</sub> Conc., % by wt	Grade	Temp, °F	Duration	Corrosion Rate, mils per year						Stain- less Type 316	Ref
				CARPENTER Stainless No. 20Cb	DURIMET 20	HASTELLOY Alloy F	G	INCOLOY alloy 825	WORTHITE		
30	unspeci- fied	215	Unspeci- fied	-	-	-	4	-	-	-	16
78-85	unspeci- fied	220	Unspeci- fied	-	-	-	-	10, pitted	-	5	31
78-85	unspeci- fied	240	unspeci- fied	-	9.2	-	-	-	-	-	31
85	CP	320	3 x 24 hr	54	-	287	-	-	129	445(a) 1660(b)	10
85	unspeci- fied	316	unspeci- fied	-	-	-	20	-	-	-	16
104-106 (c)	unspeci- fied	220	unspeci- fied	-	0.1	-	-	1.1	-	0.5	31

(a) Copper content 0.32%

(b) Copper content 0.04 %

(c) P<sub>2</sub>O<sub>5</sub> equivalent 75-77%

in 85 per cent acid at 190°F (88°C) and below 15 mils per year in boiling acid up through 30 per cent. A test in boiling 85 per cent acid at 320°F (160°C)<sup>10</sup> produced a rate of 80 mils per year. Laboratory tests in 117 per cent acid<sup>1</sup> (85% P<sub>2</sub>O<sub>5</sub>) showed 2.4 mils per year aerated at 140°F (60°C), and 2.2 mils per year without aeration at 356°F (180°C).

INCONEL alloy 625 is a wrought material with phosphoric acid resistance superior to that of conventional stainless steels. In freely circulating pure acid at boiling temperatures its manufacturer reports 15 mils per year in 60 per cent, 94 mils per year in 80 per cent, and 400 mils per year in 85 per cent acid. It maintains good resistance in mixtures containing hydrofluoric acid; specifically its corrosion rate in one test<sup>14</sup> in boiling 25 per cent phosphoric acid – 2 per cent hydrofluoric acid was 2 mils per year.

ILLIUM "R" is a wrought form substantially equivalent to ILLIUM "G" in its resistance. At boiling temperatures, its manufacturer reports a rate of 2 mils per year in 40 per cent, 7 mils per year in 75 per cent, and 18 mils per year in 80 per cent chemically pure acid.

The nickel-base high molybdenum-chromium alloys are comparatively similar to the above in corrosion behavior; HASTELLOY alloy C has excellent resistance up to about 150°F (66°C) and good resistance above that temperature except at high concentrations. HASTELLOY alloy C and alloys of similar compositions have excellent resistance in mixtures of phosphoric acid with other components such as hydrofluoric acid or oxidizing salts.

Laboratory data on HASTELLOY alloy C in chemically pure acid determined by the producer<sup>17</sup> are quoted in Table 9.

**Table 9**  
**Laboratory Corrosion Rates of HASTELLOY alloy C in Chemically Pure Phosphoric Acid**

Phosphoric Acid Concentration, % by wt	Corrosion Rate*, mils per year		
	Temperature		
	Room	150°F	Boiling Point
10	0.1	0.2	0.6
30	0.1	0.1	4.0
50	0.1	0.3	4.0
85	Nil	0.3	45.0

\*Steady-state data calculated from a minimum of five 24-hour test periods.

Laboratory data for HASTELLOY alloy C were reported by Bishop<sup>13</sup>, from whose paper selected graphs are reproduced as Figures 7 and 8. Ebling and Scheil<sup>10</sup> found the rate in 85 per cent acid, boiling at 320°F (160°C), to be 133 to 160 mils per year. Laboratory information was also determined by Bünger.<sup>7</sup>

### Fluoride Impurity

The foregoing corrosion rates represent fluoride-free acid, and although directly comparable data for acids containing fluorides are not available, some indication of the general behavior of nickel-molybdenum-chromium alloys in fluoride-containing acid can be gathered from Table 10.

**Table 10**  
**Laboratory Corrosion Tests of Ni-Mo-Cr Alloy in Boiling 25% Phosphoric Acid: Effect of Fluoride Impurity**

HF Content, %	Corrosion Rate, Mils Per Year	Ref
0	1.1	7
0	2 (a)	13
2	6.5 (a)	14

(a) HASTELLOY alloy C

### Alloys of Nickel and Molybdenum

The nickel-base alloys containing primarily molybdenum as an alloying element are typified by HASTELLOY alloy B. This material is the most resistant of all the nickel-containing alloys in hot concentrated pure phosphoric acid.

Laboratory data for HASTELLOY alloy B in chemically pure acid have been reported by the producer.<sup>17</sup> They are shown in Table 11.

**Table 11**  
**Laboratory Corrosion Rates of HASTELLOY alloy B in Phosphoric Acid**

Phosphoric Acid Concentration, % by wt	Corrosion Rate*, mils per year		
	Temperature		
	Room	150°F	Boiling Point
10	0.3	2.0	1.0
30	0.3	0.8	3.0
50	0.1	0.3	3.0
85	Nil	0.4	28.0

\*Steady state data calculated from a minimum of five 24-hour test periods.

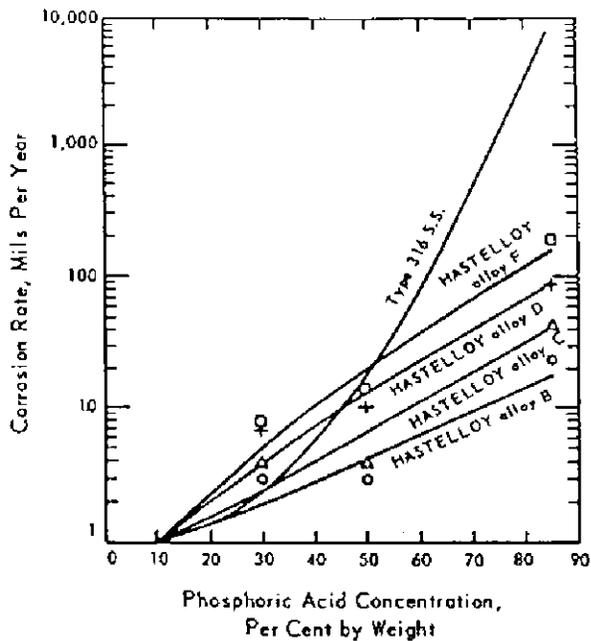


Fig. 7—Corrosion tests on nickel-base alloys and Type 316 stainless steel in boiling phosphoric acid.

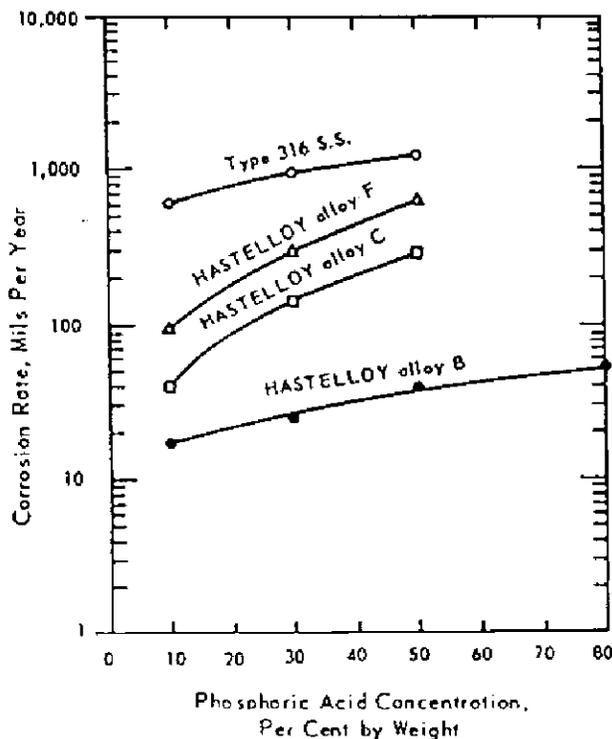


Fig. 8—Corrosion tests on nickel-base alloys and Type 316 stainless steel in phosphoric acid at 374°F (190°C).

Additional laboratory data in pure acid have been reported by Bishop<sup>13</sup> and Büniger<sup>7</sup>. Information from the producer and from Bishop<sup>13</sup> has provided corrosion rate graphs in Figures 7 and 8. Curves are included not only for HASTELLOY alloy B but for the other HASTELLOY alloys C, D and F, and, for comparison, Type 316 stainless steel. Figure 7 shows the corrosion rates in pure phosphoric acid solutions at the boiling point for three 48-hour periods (five 24-hour periods for HASTELLOY alloy D) and Figure 8 at 374°F (190°C) for one 65-hour period using sealed tubes above the boiling point. Ebling and Scheil<sup>10</sup> reported 4 to 51 mils per year in 85 per cent pure acid, boiling at 320°F (160°C).

### Copper Ions in Solutions

The reduction of corrosivity of hot strong phosphoric acid containing cupric ion, previously noted for Type 316 stainless steel, is also evident with HASTELLOY alloy B though the benefits are less consistent.<sup>18</sup> As the cupric ion concentration is raised from zero to ten ppm there is an abrupt drop in acid corrosivity, but further increase can bring a slow rise, as shown in Table 12 at 320°F (160°C) for both 70 and 85 per cent acid. In 70 per cent acid at 265°F (130°C), however, only the rise is noted, and in 100 per cent acid at 450°F (235°C), only the decrease.

Table 12  
Effect of Cupric Ion Added to Phosphoric Acid on Corrosion of HASTELLOY alloy B

Parts Cu** per million parts H <sub>3</sub> PO <sub>4</sub>	Corrosion Rate, mils per year			
	Concentration, %			
	70	70	85	100
	Temperature, °F			
	265	320	320	450
0	2.6	50	35-50	14.4
2	—	—	21	—
5	4.6	27	15	9.8
10	5.9	17	15-19	7.3
20	5.9	19	34	3.8
50	11.4	21	—	4.8
100	—	26	45-48	—
500	—	50	—	—
1000	—	—	200	—

## Nickel-Base Alloy with Silicon

HASTELLOY alloy D is the only commercial material of this type. In pure phosphoric acid its performance is nearly comparable with the nickel-molybdenum-chromium alloys. Data from Reference 17 are included in Figure 7. In pure 85 per cent acid, boiling at 320°F (160°C) Ebling and Scheil<sup>10</sup> reported a corrosion rate of 175 mils per year.

## Ni-Resist Alloys

Austenitic cast irons of the Ni-Resist series have found limited application in phosphoric acid solutions. They are used chiefly with dilute unaerated solutions at atmospheric temperature, and they are valuable in superphosphate production. Results of laboratory corrosion tests<sup>5</sup> are shown in Table 13; data were obtained on two heats of Ni-Resist.

**Table 13**  
**Laboratory Tests of Ni-Resist Austenitic Cast Iron in Phosphoric Acid**

Acid Conc., % by wt	Temp, °F	Duration, days	Type	Corrosion Rate, mils per year	
				Not aerated	Aerated
5	122	6	2	956	1810
5	122	6	D2	154	1260
5	86	1	1	66	90
5	190	0.88	1	275	—
15	86	0.83	1	—	52
15	190	0.83	1	319	—
25	86	1	1	—	43
25	190	0.83	1	510	757
85	86	12	2	—	87
85	86	12	D2	—	240
85	86	1	2	199	204
85	112	1	2	—	863
85	176	0.25	2	—	2660

## Alloys of Nickel and Copper

Only two alloys of nickel and copper have established significant use in handling phosphoric acid: the high-nickel Alloy 400 and the high-copper (70/30) Alloy 715.

### Nickel-Copper Alloy 400

Alloy 400 shows fairly good resistance to pure unaerated phosphoric acid over a wide range of concentrations. Table 14 shows laboratory corrosion rates under two mils per year in all concentra-

tions up to 120°F (49°C) and rates of ten mils per year or less up to 220°F (104°C). Local corrosion of welds has been reported in 80 per cent acid at 220°F (104°C). Aeration and oxidizing agents accelerate corrosion of Alloy 400.

**Table 14**  
**Laboratory Tests of MONEL alloy 400 in Phosphoric Acid Solutions**

Acid Conc., % by wt	Temp, °F	Duration, days	Corroaon Rate, mils per year	Ref
3.2	77	49	1.6	5
3.2	212	0.17	4.9	5
10	214	2 + 2	10	3
40	80	5	1.0	5
50	230	2 + 2	4	3
78-85	77 (est)	unspecified	0.1	31
78-85	120	unspecified	1.0	31
78-85	220	unspecified	8.9	31
90	208	2 + 2	1	3
85	255	≥6 not aerated	10	1
85	255	aerated	440	1
85	320	1	4525	9
117	140	≥6 aerated	<10	1
117	356	≥6 not aerated	3.3	1
117	480-490	unspecified	excessive	1

## Copper-Nickel Alloys

Very little application has been made of the high-copper, low-nickel alloys in phosphoric acid exposure. Laboratory information is available only at 8.4 and 116 per cent (6.1 and 84% P<sub>2</sub>O<sub>5</sub>). The first concentration was used in five 8-hour exposures in glass bottles; it is apparent that the alloy compositions are superior to either pure metal. The rates, obtained presumably at room temperature, are shown in Figure 9.<sup>19</sup>

The high-concentration acid, actually tetraphosphoric acid, produces a similar relationship according to available data. Results obtained in a 48-hour laboratory test at three temperatures are shown in Table 15.<sup>20</sup> As with Alloy 400, oxidants raise the rates.

## Nickel

Laboratory information is rather sparse on the resistance of wrought Nickel 200 in phosphoric acid.

Performance appears to be limited more by temperature than by acid concentration. Good resistance has been shown at room temperature, the rate increasing near 120°F (49°C), accompanied by local corrosion.

Data are shown in Tables 15 and 16.

**Table 15**  
**Laboratory Tests of Copper-Nickel and Nickel 200 in 116% Phosphoric Acid (84% P<sub>2</sub>O<sub>5</sub>)**

Temp, °F	Corrosion Rate, mils per year		
	Copper-Nickel alloy 70/30		Nickel 200
	Not aerated	Aerated	Not aerated
140	0.1	1.0	13
248	2.0	11.4	48
356	26.2	79.5	—

**Table 16**  
**Laboratory Tests of Nickel 200 in Phosphoric Acid Solutions**

Acid Conc., % by wt	Temp, °F	Duration, days	Corrosion Rate, mils per year	Ref
40	80	5	1.0	5
78-85	77 (est)	unspecified	0.3	31
78-85	120	unspecified	4.2, local corrosion	31
78-85	220	unspecified	15 <sub>0</sub>	31
117	48-490	unspecified	excessive	1

### Nickel-Chromium and Iron-Nickel-Chromium Alloys

These materials are not normally used in phosphoric acid solutions, since other alloys are more resistant. The few available test results in pure acid appear in Table 17. INCOLOY iron-nickel-chromium alloy 800 has been exposed only in wet-process acid, where it showed better resistance than INCONEL nickel-chromium alloy 600. These results are found in Tables 20 and 48.

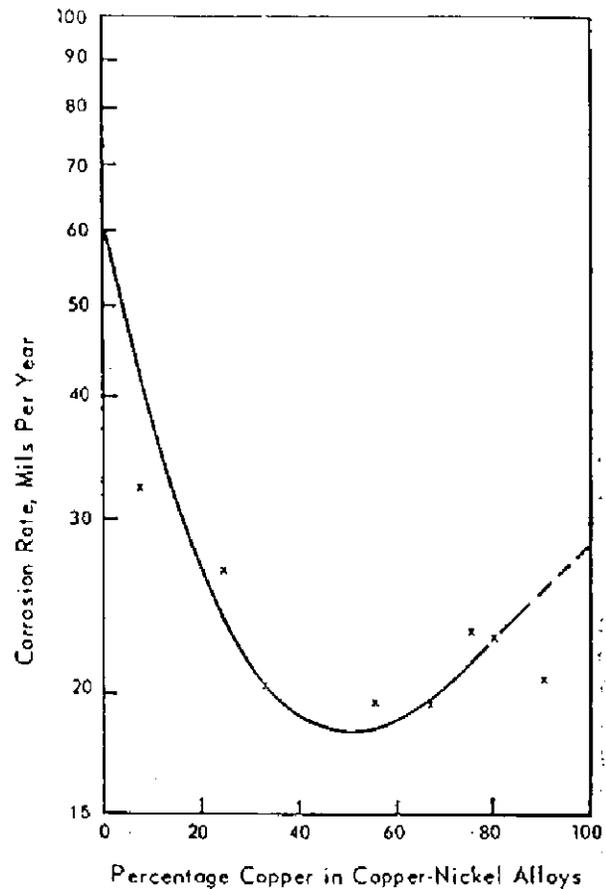


Fig. 9—Corrosion rates of copper-nickel alloys in 8.4% phosphoric acid.

**Table 17**  
**Corrosion Data on INCONEL Nickel-Chromium Alloy 600 and Iron-Nickel-Chromium Alloy in Pure Phosphoric Acid Solutions**

Acid Conc., % by wt	Temp, °F	Dura - tion, days	Corrosion mils per Rate, year		Ref
			INCONEL Alloy 600	Fe-Ni-Cr Alloy	
10 laboratory	214	2 + 2	<1	—	3
50 laboratory	230	2 + 2	176	—	3
86 laboratory	208	2 + 2	46	—	3
75 storage tank	122 av.	60	5.9	—	5
75 in tank (a)	158	39	27	—	5
85 laboratory	(320) BP	1	14,600	—	10
75 laboratory (b)	167	2	52 (c)	205 (d)	5

(a) Solution circulated  
(b) Solution aerated  
(c) Composition similar to INCONEL alloy 610 casting  
(d) Casting composition 42 Fe. 37 Ni. 19 Cr  
BP Boiling point

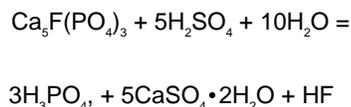
## Part II. PLANT TESTS

WHEN SELECTING materials to handle unmixed phosphoric acid solutions, the choice can often be based on data determined in laboratory tests in pure solutions. It is less satisfactory to use laboratory tests to predict the behavior of materials in impure phosphoric acid produced by the wet process, or in reactions in which phosphoric acid is mixed with other chemicals under varying conditions of temperature, aeration and agitation. In such cases it is usually advisable to make tests or refer to data developed from corrosion tests made in plant or pilot-plant equipment under actual operating conditions.

The results of numerous plant corrosion tests are presented in the following pages to show the comparison in performance among the various nickel alloys and other materials. The testing device used in most of these tests was the spool-type specimen holder described by Searle and LaQue.<sup>21</sup> This method of testing is substantially in accord with the ASTM Recommended Practice for Conducting Plant Corrosion Tests, A 224-46.<sup>22</sup> Briefly, the assembly consists of previously cleaned and weighed specimens, in duplicate, of the several metals and alloys to be tested, mounted on a spool-type holder, with non-metallic parts of TEFLON, BAKELITE or porcelain to separate and insulate the specimens from each other, from the metal strengthening members of the device, and from the equipment in which the test is made. Details are shown in Figures 10 and 11. The completed assemblies are installed in the desired locations, and allowed to remain for a sufficient length of time to give reliable indications of corrosion behavior. Upon completion of a test, the assembly is removed, dismantled, and the specimens examined, cleaned of all adhering corrosion product, and reweighed. From the known weight losses, areas of specimens, and duration of tests, the corrosion rates are calculated. In addition, the specimens are examined for pitting or other local attack.

### Materials of Construction for Wet Process Phosphoric Acid Equipment

The wet process of manufacture of phosphoric acid consists essentially of the reaction of calcium phosphate rock with sulfuric acid, and filtration to separate the product from calcium sulfate. The equation is:



Several other operations which are part of the process are shown in a flow sheet, Fig. 12. These consist of calcination and size reduction of phosphate rock to provide adequate contact area and speed of reaction, scrubbing of fluoride by-products from the reaction products, washing of acid from the filter cake, disposal of the washed cake, and concentration of the product acid.

For general information on wet process phosphoric acid manufacture, the reader is referred to the published literature. Among good sources is Chapter 9 of the book "Chemistry and Technology of Fertilizers."<sup>23</sup>

Phosphate rock is non-corrosive, but every other process material is more or less aggressive, requiring some attention to corrosion protection. Examples will be cited of the alloys found useful in the manufacturing process, as well as of test results which have been obtained in operations numbered in Fig. 12.

### Dilution of Sulfuric Acid

Before acidulation of ground phosphate rock, entering concentrated sulfuric acid may be mixed with recirculated phosphoric acid of intermediate concentration, a dilution operation. A set of data obtained at this step is shown as Table 18.

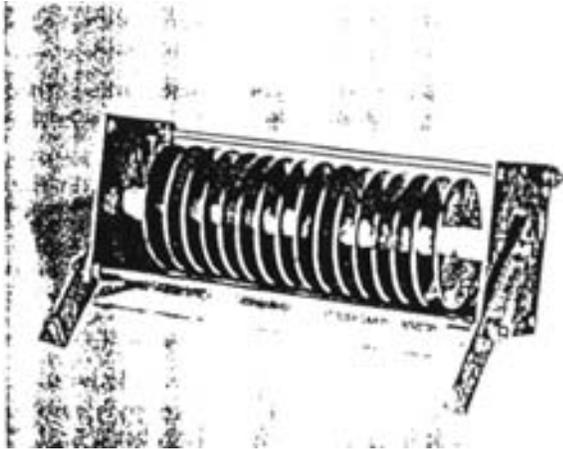


Fig. 10—Spool-type specimen holder.



Fig. 11—Pipe spool-type specimen holder.

**Table 18**  
**Plant Test in Sulfuric Acid Dilution with Recirculated Phosphoric Acid**

Phosphoric acid (wet-process) 28% (20%  $P_2O_5$ ), sulfuric acid 20-22%, fluoride approx. 1-1.5%. probably as hydrofluosilicic acid. Continuous dilution of concentrated sulfuric acid with recirculated phosphoric acid. Specimens exposed at bottom of dilution tank.

Temperature: 180-230°F (82-110°C), average 200°F (93°C).

Duration of test: 42 days; moderate aeration, agitation by convection only.

All specimens were badly scaled.

Material	Corrosion Rate, mils per year	Concentration Cell Depth, mils
CARPENTER Stainless No. 20Cb	1.1	-
ALOYCO 20	1.5	-
INCOLOY alloy 825	2.7	5
HASTELLOY alloy C	2.8	-
ILLIUM "G"	3.0	-
INCONEL alloy 718 (a)	3.2 (b)	-
WORTHITE	4.3	3
INCOLOY alloy 901 (c)	6.1	5
ILLIUM "R"	6.9, 14.3	-
MONEL alloy K-500 (d)	31	(e)
MONEL alloy 400	38	-
HASTELLOY alloy B	78	-
Stainless Type 317	> 150 (s)	-
Stainless Type 316	> 180 (s)	-

(a) Composition 52.5 Ni, 18.6 Cr, 18.5 Fe, 5.0 Cb, 3.1 Mo

(b) Pitted to a maximum depth of 5 mils

(c) Composition 42.7 Ni, 34.0 Fe, 13.5 Cr, 6.2 Mo, 2.5 Ti

(d) Composition 65.0 Ni, 29.5 Cu, 2.8 Al, 1.0 Fe

(e) Pitted to a maximum depth of 3 mils

(s) Stress corrosion cracking around markings

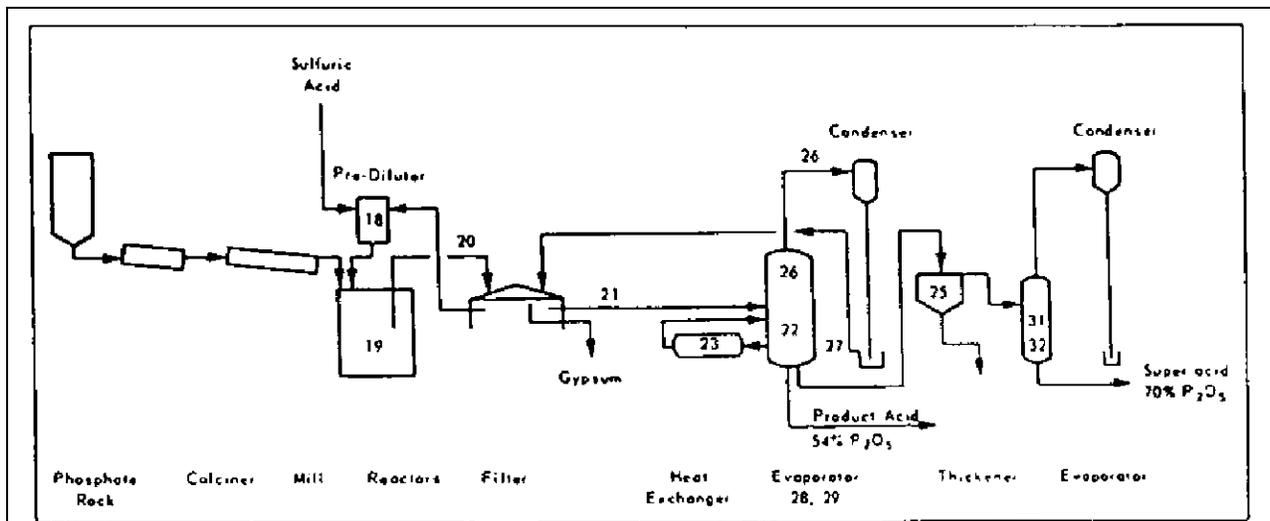


Fig. 12—Wet-process phosphoric acid flow sheet with test spool locations; numbers identify tables of corrosion data.

**Table 19**  
**Plant Test in Phosphoric Acid Reactor**

Environment: Phosphoric acid (wet-process) 39% (28% P<sub>2</sub>O<sub>5</sub>), sulfuric acid 2%, hydrofluosilicic and hydrofluoric acid trace amounts, total fluoride equivalent about 1.2%, suspended gypsum about 20%. Specimens in primary digestion tank.

Temperature: 170-183°F (77-84°C), average 180°F (82°C).

Duration of test: 96 days; moderate aeration, vigorous agitation.

No pitting.

Material	Corrosion Rate, mils per year
INCONEL alloy 625	0.7
HASTELLOY alloy G	1.1
ILLIUM "98"	2.7
Lead, antimonial	3.2
HASTELLOY alloy C	3.4
CARPENTER Stainless No. 20Cb-3	6.5
HASTELLOY alloy F	6.7
WORTHITE	6.8
CARPENTER Stainless No. 20Cb	6.8
INCOLOY alloy 825	7.4
DURIMET 20	11.6
CHLORIMET 3 (a)	12.2
ILLIUM "G"	14.8
Stainless Type 317	22
ILLIUM "P"	27
Stainless Type 316L	41
Stainless Type 316 sensitized	50
Stainless Type CF-8M	>210
HASTELLOY alloy B	>66 (d)
ILLIUM "R"	>91 (d)
CHLORIMET 2 (b)	>310 (d)

(a) Composition 60 Ni, 18 Cr, 18 Mo, 3 Fe, 1 Si

(b) Composition 66 Ni, 31 Mo, 2 Fe

(d) Destroyed

**Table 20**

**Plant Test in Phosphoric Acid-Gypsum Slurry**

Slurry composition: 30% solids, 39% phosphoric acid (29% P<sub>2</sub>O<sub>5</sub>), 1.4% sulfuric acid, 2% fluoride probably combined as hydrofluosilicic acid. Specimens in launder between reactors.

Temperature: 160-195°F (71-91°C), average 180°F (82°C).

Duration: 35 days; extensive aeration; flow rate of slurry 5 feet per second.

No pitting.

Material	Corrosion Rate, mils per year
Stainless Type 309 (a)	0.6
Stainless Type 317	1.6
Stainless Type 316	4.4
Stainless Type 304	13.5
INCOLOY alloy 800	14.6
Lead, chemical	17.1
MONEL alloy 400	45
INCONEL alloy 600	>73(d)

(a) Composition 61 Fe, 23 Cr, 13.5 Ni, 1.3 Mn, 0.6 Si, 0.20 max C  
(d) Destroyed

**Reaction (Extraction)**

The reactors themselves, also called digesters, extractors, or attack vessels, are usually lined with rubber or brick or both, exposing metal only in the agitators where mechanical strength is the overriding requirement. Table 19 provides data in the slurry within such a reactor, though it may not fully reflect the effect of erosion. Table 20 results were obtained from a launder between reactors, and Table 21 in product acid.

**Table 21**  
**Plant Test in Reaction Product Phosphoric Acid**

	Test 1	Test 2
Phosphoric Acid conc.: H <sub>3</sub> PO <sub>4</sub> , %	33	42
P <sub>2</sub> O <sub>5</sub> , %	24	30.5
Sulfuric acid, %	Some, occasionally	2
Hydrofluosilicic acid, %	3	Present
Hydrofluoric acid, %	Traces	Present
Gypsum, suspended, %	Present	2
Locations	Thickener trough outlet following filtration	Acid recycle diversion tank
Temperature: minimum, °F (°C)	160 (71)	135 (57)
maximum	180 (82)	145 (63)
average	170 (77)	
Duration: days	33	72
Aeration	None reported	None
Agitation Flow Rate	1.5 ft/sec	20-30 gal/min
Material	Corrosion Rate, mils per year	
Lead, chemical	Nil (increase)	-
DURIMET 20	-	0.2
WORTHITE	-	0.3
CARPENTER Stainless No. 20Cb	-	0.3
Stainless Type 329	-	0.3 (a)
Stainless Type 317	0.9	0.4
Stainless Type 316	1.6	0.5 (L)
HASTELLOY alloy F	-	0.6
INCOLOY alloy 825	-	1.5
HASTELLOY alloy C	-	3.1
Stainless Type 304	6.8 (p)	-
Iron-silicon alloy (14.5%)	-	106, 320
INCONEL alloy 600	>142 (od)	-
Nickel 200	>166 (d)	-
MONEL alloy 400	>169 (d)	>82 (d)
HASTELLOY alloy 8	-	>95 (d)
Carbon Steel	>340 (d)	-
Ni-Resist Type 1	>590 (od)	-
Cast Iron	>884 (d)	-

(a) Data for one specimen. Part of other specimen broke off during test; attack apparently intergranular.

(L) Type 316L

(p) Pitting to 13 mils max

(d) Destroyed

(od) Data for one specimen; other specimen destroyed

## Filtration

The filtration step yields three concentrations of acid: strong or product acid with a concentration of about 35 to 44 per cent  $H_3PO_4$  (25 to 32 %  $P_2O_5$ ), and two weaker solutions resulting from washing of filter cake. These have acid contents of 25 to 33 and 3 to 7 per cent (18 to 24 and 2 to 5%  $P_2O_5$ ). The latter, weak wash liquor, is recycled to the filtration operation; the former, weak acid, is recycled to the filtration operation or to the reaction system, depending on the process. Pumps are likely to be of ACI Type CF-7M alloy, and filters have been constructed of Types 316L and 317L stainless steel. One such filter, used in wet-process acid filtration, is shown in Fig. 14.



Fig. 13—Submerged pump handling 32% phosphoric acid–gypsum slurry at 170°F, from digester to filter. Submerged cast parts of ILLIUM "P"; shaft, bolts, nuts and studs are wrought Alloy 20.

*Courtesy of Chas. S. Lewis & Co., Inc*

### Concentration of Product Acid

Some of the fertilizer uses of phosphoric acid require concentrations in the range of 58 to 75 per cent (42 to 54%  $P_2O_5$ , which are not obtained economically by direct reaction. Product 42 percent (30%  $P_2O_5$ ) acid is readily concentrated, however, by vacuum evaporation or by submerged combustion.

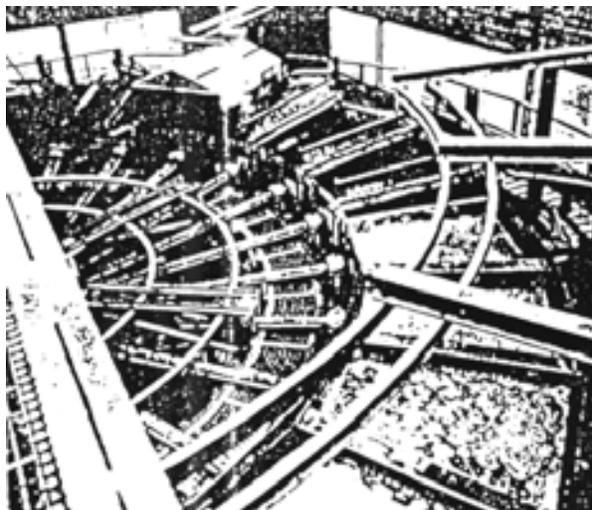


Fig. 14—Type 316L stainless steel Bird-Prayon tilting-pan filler used to separate gypsum from wet-process phosphoric acid.

*Courtesy of Bird Machine Company*

## Vacuum Evaporation

In vacuum evaporation, the physical properties and scale-forming tendency of the acid dictate the use of forced circulation through an external heat exchanger. The flash-chamber body is often constructed with rubber-lined steel surfaces while heat-exchanger tubes have been made of metal or graphite. Both types of material acquire scale deposits, largely gypsum, necessitating provision for cleaning in the operating schedule.

Graphite tubes are susceptible to mechanical breakage during cleaning. In alloy tubes, scale cracking has been known to permit localized corrosion at the cracks, particularly when a low silica content in the phosphate rock has been inadequate to complex all the hydrofluoric acid as hydrofluosilicic acid. Table 22 records results of tests in 53 per cent acid in the evaporator body itself. Table 23 shows tests in 65 per cent acid in the external heat exchanger used with a forced-circulation system. For each of the five alloys common to both tests the 65 per cent acid environment was more severe despite lower bulk temperature of the liquid. It should be mentioned that corrosion rates of heat-transfer tube surfaces are ordinarily higher than those calculated from tests of disk specimens, because the tubes are exposed to a film of liquid hotter than the bulk of the liquid. This effect may be seen in laboratory data reported<sup>3</sup> for pure acid.

**Table 22**  
**Plant Test in Phosphoric Acid Evaporator**

Exposure to 53% phosphoric acid (38% P<sub>2</sub>O<sub>5</sub>) containing 1-2% sulfuric acid and 1.2-1.5% fluoride. Specimens submerged in liquid in evaporator.

Temperature: 250°F (121°C).

Duration: 42 days. No information on aeration or agitation.

No pitting.

Specimens completely covered with scale after 12 days.

Material	Corrosion Rate, mils per year
CARPENTER Stainless No. 20Cb	4.7
HASTELLOY alloy C	5.0
INCOLOY alloy 825	6.2
Stainless Type 317	10.4
MONEL alloy 400	25
Stainless Type 316	44
INCONEL alloy 600	> 1300 (d)
HASTELLOY alloy B	> 1500 (d)

(d) Destroyed

**Table 23**  
**Plant Test in Heat Exchanger**

Exposure to 65%, phosphoric acid (47% P<sub>2</sub>O<sub>5</sub>), 3 to 4% sulfuric acid, traces hydrofluosilicic and hydrofluoric acid, 5 to 7% suspended solids. Specimens in forced-circulation heat exchanger connected to a multiple-effect evaporator.

Average temperature: 180°F (82°C).

Duration 4.1 days, moderate aeration and considerable agitation.

Material	Corrosion Rate, mils per year
Lead, chemical	1.3
ILLIUM "G"	8.8
Stainless Type 329	10.0
INCOLOY alloy 825	15.5
HASTELLOY alloy F	18.7
CARPENTER Stainless No. 20Cb	20.2
Stainless Type 317	28
HASTELLOY alloy C	35
Stainless Type 316, 316L	350, 870
Stainless Type 316 sensitized	>2100(d)

(d) Destroyed

Two series of tests run under laboratory conditions in a boiling simulated wet process acid made from reagent grade materials exposed most of the chromium-nickel-molybdenum alloys, with and without iron, that are of likely interest. The results, in Table 24, clearly indicate the effectiveness of the molybdenum- and copper-containing types for resistance to this phosphoric acid mixture.

The concentrated acid may be cleared of sludge and scale by settling or filtration. Equipment for this step is subject to less corrosive service than that in evaporation because of lower temperature and absence of heat transfer. Table 25 provides data obtained in a thickener.

The vapor phase produced in the evaporation of wet-process acid carries suspended fumes and mists. These phases contain phosphoric, sulfuric, hydrofluoric and hydrofluosilicic acids in low but corrosive concentrations. Table 26 presents results of

**Table 24**  
**Laboratory Test in Simulated Wet Process Acid**

Solution of 55% phosphoric acid, reagent grade (40% P<sub>2</sub>O<sub>5</sub>), 0.8% hydrofluoric acid.

Temperature: 228°F (109°C).

Duration: 2.0 days. No aeration; agitation by boiling.

Material	Corrosion Rate, mils per year	
	Test 1	Test 2
ILLIUM "R"	6.4	—
ILLIUM "G"	7.7	—
HASTELLOY alloy G	—	9.0
CARPENTER Stainless No. 20Cb-3	—	13.2
ILLIUM "98"	—	15.8
INCOLOY alloy 825	13.4	16.2
INCONEL alloy 625	16.6	—
HASTELLOY alloy N (a)	24.8	—
HASTELLOY alloy C	28.7	—
HASTELLOY alloy B	29.6	—
HASTELLOY alloy F	91	—
CARPENTER Stainless No. 20Cb	101	—
Stainless Type 317	201	—

(a) Composition 71 Ni, 16 Mo, 7 Cr, 4 Fe

**Table 25**  
**Plant Test in Phosphoric Acid Thickener**

Exposure in 53.8% phosphoric acid (39% P<sub>2</sub>O<sub>5</sub>) containing 2.05-2.15% hydrofluosilicic acid, 1.5-2.5% sulfuric acid, 2% calcium sulfate. Specimens submerged 18 inches below the liquid surface of a thickener.

Temperature: 122-150°F (50-65°C). average 130°F (55°C).

Duration: 51 days: no aeration, slight agitation.

Material	Corrosion Rate, mils per year
Lead, chemical	0.4
INCOLOY alloy 825	0.5
CARPENTER Stainless No. 20Cb	0.9
HASTELLOY alloy C	1.7
Stainless Type 316	20

exposure tests in the vapor head of an evaporator, in a vapor line from such a head, and in the liquid in the recycle tank of an evaporator fume scrubber.

Condenser water from these evaporators contains low concentrations of hydrofluosilicic and phosphoric acids picked up from the vapors. It has oxidizing properties strong enough to corrode numerous alloys, but can be handled by annealed austenitic chromium-nickel and by chromium-nickel-molybdenum stainless steels. A set of data in this condenser water is shown in Table 27.

**Table 26**

**Plant Test in Evaporator Vapor and Fumes**

Plant spool tests in fumes and mists produced in evaporation of wet-process phosphoric acid. Contains unspecified concentrations of phosphoric acid, hydrofluosilicic acid, hydrofluoric acid, sulfuric acid.

Test 1: Exposure in vapor head of evaporator.

Temperature 80-150°F (25-65°C), average 120°F (50°C).

Duration: 41.5 days. No aeration, gas flow 4 feet per second.

Test 2: Exposure in vapor line from evaporator head.

Temperature 140-160°F (60-71°C), average 150°F (65°C).

Duration: 59 days. No aeration, rapid gas flow.

Test 3: Exposure in evaporator fume scrubber recycle tank 15% phosphoric acid (11% P<sub>2</sub>O<sub>5</sub>), 20% hydrofluosilicic acid, 1% sulfuric acid.

Temperature 165-185°F (75-85°C), average 175°F (79°C).

Duration: 16 days. Extensive aeration, slow agitation

Material	Corrosion Rate, mils per year		
	Test 1	Test 2	Test 3
HASTELLOY alloy C	0.4	1.9	2.0
ILLIUM "G"	—	2.9	9.4
ILLIUM "R"	—	3.1	—
HASTELLOY alloy F	—	3.2	5.8
INCOLOY alloy 825	0.3	4.2	1.0 4.6
CORRONEL alloy 230	—	4.2	—
CARPENTER Stainless No. 20Cb	0.4	5.0	0.7
ALOYCO 20	—	6.5	—
HASTELLOY alloy B	14	6.6 (a)	—
WORTHITE	—	8.1	—
Stainless Type 329	—	8.3	0.3
Stainless Type 317	0.48	10.3	2.9 14.8
Stainless Type 316	2.9	10.7	21 (L)
Stainless Type 316 sensitized	—	27	—
INCONEL alloy 600	15	—	—
MONEL alloy 400	30	—	—

(a) Crevice corrosion 4 mils

(L) Type 316L

**Table 27**

**Plant Test in Condenser Effluent**

Plant test in 0.1% hydrofluosilicic acid, 0.08% phosphoric acid (0.05% P<sub>2</sub>O<sub>5</sub>) with trace of hydrogen sulfide; pH 2.0. Specimens immersed in condenser effluent, used for filter wash water.

Temperature: 80-175°F (27-80°C), average 150°F (65°C).

Duration: 76 days; moderate aeration, flow rate about two feet per second.

Material	Corrosion Rate, mils per year
INCOLOY alloys 804 (a) and 825	<0.1
Stainless Types 304, 309 (b), 316, 317	<0.1
Stainless Type 304 sensitized	0.3 pitted to 12 mils & intergranularly corroded
Stainless Type 316 sensitized	<0.1
CARPENTER Stainless No. 20Cb	<0.1
DURIMET 20	<0.1
ILLIUM "R" and "G"	<0.1
HASTELLOY alloys C and F	<0.1
INCONEL alloy 600	29 perf. in crevice
HASTELLOY alloy B	38
MONEL alloy 400	49
Copper-nickel CA 715 (70/30)	53
Copper	63
Ni-Resist Type 1	13 <sub>0</sub> pitted to 23 mils
Ni-Resist Type 2	23 <sub>0</sub>
Nickel 200	>12 <sub>0</sub> virtually destroyed

(a) Composition 42.6 Ni, 29.3 Cr, 25.4 Fe

(b) See Table 20

**Evaporation by Direct Contact**

An alternate method of concentration, submerged combustion, provides direct contact between hot combustion gases and solution, avoiding a heat-transfer surface. While scaling and corrosion of such a surface are eliminated, other problems are created, namely high corrosivity of vapor-gas mixtures which require fume and mist separators, either wet or electrostatic. The severity of these problems is indicated by the data in Tables 28 and 29. Table 28 presents results of an exposure in the gas line between a submerged-combustion evaporator and the scrubber. Table 29 was obtained by exposure in the entrainment separator following a drum concentrator, a form of submerged-combustion evaporator.

**Defluorination**

Although most wet-process phosphoric acid is used for fertilizer manufacture, some is defluorinated for use in the preparation of animal feed supplements. One such process consists of steam-sparging the

acid. A study of the resistance of construction materials for the steam-sparger and for the fume hood showed both environments to be very corrosive; none of the alloys tested was wholly satisfactory. Results are shown in Table 30.

**Table 28**  
**Plant Test in Submerged Combustion Evaporator Gas Line**

Exposure In phosphoric acid gases evolved by submerged combustion evaporator. Reported composition (incomplete): 0.7-1.4% phosphoric acid (0.5-1.0%  $P_2O_5$ ), 0.5-1.0% combined fluorine, both by weight, water vapor 40-50% by volume. Specimens exposed in gas line between evaporator and scrubber.

Temperature: 190°F (88°C).

Duration: 14 days. Some air in moving gas stream.

Material	Corrosion Rate, mils per year
HASTELLOY alloy C	50
HASTELLOY alloy B	84
INCOLOY alloy 825	199
CARPENTER Stainless No. 20Cb	210
INCONEL alloy 600	214
MONEL alloy 400	268
Stainless Type 317	383
Stainless Type 316	502

**Table 29**  
**Plant Test in Drum Concentrator Entrainment Separator**

Exposure in wet vapors produced by concentration of 50-55% phosphoric acid (36-40%  $P_2O_5$ ) containing hydrogen fluoride. Specimens exposed In wet separator on top of concentrating drum.

Temperature: 200-300°F (93-149°C), average 225°F (107°C).

Duration: 21 days. Moderate aeration and agitation.

Material	Corrosion Rate, mils per year
HASTELLOY alloy F	20
HASTELLOY alloy C	24
INCOLOY alloy 825	29
CARPENTER Stainless No. 20Cb	33
Stainless Type 316	56
MONEL alloy 400	>136 (a) (b)
HASTELLOY alloy B	194 (b)
Ni-Resist Type 1	365 (b)

(a) One specimen presumably destroyed

(b) One specimen perforated

**Table 30**  
**Spool Tests in Phosphoric Acid Defluorination Service**

Environment: 75% phosphoric acid (54%  $P_2O_5$ ). 1% sulfuric acid, HF present.  $SiF_4$  present in vapor hood.

Specimens in Test 1 submerged in acid at temperature of 250-315°F (121-157°C), average 300°F (150°C), for 7.8 days. Specimens in Test 2 located in fume hood above defluorinator, at temperature of 225-300°F (107-150°C), average 260°F (127°C), for 10 days.

Specimens in Test 3 (a later test) located in the gas flow, at temperature of 160-360°F (71-182°C), average 320°F (160°C), for 15 days.

No aeration and violent agitation in all three exposures.

Material	Corrosion Rate, mils per year		
	Test 1	Test 2	Test 3
	Liquid	Fume Hood	Gas Flow
HASTELLOY alloy F	60	-	62
CARPENTER Stainless No. 20Cb	87	22	59
INCOLOY alloy 825	117	26	56
ILLIUM "G"	-	-	109
Stainless Type 317	119	-	162
HASTELLOY alloy C	131	25	54
Stainless Type 316	153	46	240
HASTELLOY alloy B	>1100 (d)	55	-
MONEL alloy 400	-	62	-
Nickel 200	-	66	-
Copper-nickel CA 715 (70/30)	-	95	-
Copper	-	104	-

(d) Destroyed

### Evaporation to Superphosphoric Acid

A practice has been recently introduced producing "superphosphoric" acid having a concentration of 97 to 99 per cent (70 to 72%  $P_2O_5$ ) by a further evaporation step. In addition to the advantage of more economical transportation resulting from the reduced water content, the complex polyphosphate content has metal-ion-sequestering properties not possessed by orthophosphate, permitting preparation and handling of liquid fertilizer products of high concentration. Feed acid which has been partially freed of fluorine compounds in a prior evaporation has reduced corrosivity so that metallic construction has been satisfactory. Evaporator heaters of alloys of nickel, iron, chromium, copper and molybdenum are being used in connection with the production of superphosphoric acid in the temperature ranges indicated. Such an evaporator is illustrated, in part, in Figure 15. Results of corrosion tests in such secondary evaporation are shown in Tables 31 and 32.

**Table 31**

**Pilot Tests in Superphosphoric Acid Evaporators**

Spool tests in Superphosphoric acid evaporators using feed acid derived from Florida phosphate rock.

Feed 70% phosphoric acid (52% P<sub>2</sub>O<sub>5</sub>); composition of liquid phase in evaporator approximately 97% phosphoric acid (70% P<sub>2</sub>O<sub>5</sub>).

Contaminants reported in Test 1 – 5% sulfuric acid, 0.5% hydrogen fluoride plus other contaminants; in Test 3 – gypsum, iron and aluminum phosphates, hydrofluosilicic acid, hydrofluoric acid.

Vapor phases largely water vapor, silicon tetrafluoride, trace hydrogen fluoride vapor, mist containing phosphoric acid and hydrofluosilicic acid.

All columns from pilot-scale tests; hours at operating temperature and at lower temperatures (between day shifts) shown separately; total time used for rate calculations.

All test media were unaerated. Media in Tests 1, 2 and 4 considerably agitated; Test 3, submerged in acid pool in vapor head, agitation unspecified.

	Test 1	Test 2	Test 3	Test 4
Phase Location	Liquid Liquor	Vapor Head	Liquid (See above)	Vapor Head
Temperature: minimum °F (°C)	320 (160)		85 (29)	
maximum	400 (204)		370 (188)	
average	370 (188)		350 (176)	
Duration: days	38.5		76	
hr at described conditions	834		475	
hr at different conditions	90 (a)		1350 (b)	
	Corrosion Rate, mils per year			
Material	Test 1	Test 1	Test 3	Test 4
ILLIUM "G"	20.5	20.4	5.5	7.1
ILLIUM "R"	21.0	20.7	–	–
HASTELLOY alloy C	28	3.8	5.7	3.1
HASTELLOY alloy F	29(c)12	22.9	6.3	4.0
INCOLOY alloy 825	46	20.2	9.1	8.4
CARPENTER Stainless No. 20Cb	48	22.6	10.3	7.1 (p) 5
ALOYCO 20	49	22.2	–	–
Stainless Type 329	59	31 (r) 6	–	–
WORTHITE	67	28	–	–
Stainless Type 317	71	30	18	9.2 (p) 5
Stainless Type 316	80	37 (r) 3	21 (L)	14 (L)(p) 12
Stainless Type 316 sensitized	83	39(r)5	21	18
HASTELLOY alloy B	86(m)	18.4	21 (c) 6	22
MONEL alloy 400	–	–	27	39
Stainless Type 304	–	–	38	29

- (a) Environment 80-86% H<sub>3</sub>PO<sub>4</sub> (58-62% P<sub>2</sub>O<sub>5</sub>)
- (b) 1326 hr cooling from 350°F (177°C) to 85°F (29°C). 24 hr in water at 212°F(100°C)
- (c) Crevice corrosion, maximum depth in mils shown
- (L) Type 316L
- (m)Crevice corrosion, metal Ion type. 4 mils maximum depth
- (p) Pits, maximum depth in mils shown
- (r) Corroded ring developed outside spacer, apparently in retained liquid; maximum depth In mils shown

**Table 32**

**Pilot and Field Tests in Superphosphoric Acid Evaporators**

Spool tests in superphosphoric acid evaporators using feed acid derived from western phosphate rock.

Feed 72% phosphoric acid (52% P<sub>2</sub>O<sub>5</sub>); composition of liquid phase in evaporator 97% phosphoric acid (70% P<sub>2</sub>O<sub>5</sub>).

Gas phase largely water vapor, silicon tetrafluoride, trace hydrogen fluoride, mist containing phosphoric acid and hydrofluosilicic acid.

Tests 1 and 2 from pilot plant exposure; Tests 3 and 4 from plant scale exposure not related to Tests 1 and 2. All test media were unaerated and considerably agitated.

	Test 1	Test 2	Test 3	Test 4
Phase	Liquid	Vapor	Liquid (a)	Vapor
Temperature: minimum °F (°C)	350 (177)	350 (177)	355 (180)	80(27)
maximum	400 (205)	400 (205)	395 (202)	150(65)
average	380 (193)	380 (193)	370 (188)	120(49)
Sulfuric acid content, %	5	-	Not specified	
Hydrofluoric acid content, %	0.5	-	Not specified	Trace
Duration: days	25	25	41.5	41.5
Scale of Operation	Pilot	Pilot	Production	Production
Material	Corrosion Rate, mils per year			
CORRONEL alloy 230	-	-	4.8 (c) 7	4.2
HASTELLOY alloy C	2.6	4.1	6.4	1.9
HASTELLOY alloy F	4.0	7.4	6.6 (c) 5	3.2
ILLIUM "G"	3.4	7.2	7.1	2.9
ILLIUM "R"	3.9	7.1	7.3 (c) 4	3.1
CARPENTER Stainless No. 20Cb	4.7	9.3	8.6 (c) 3	5.0
INCOLOY alloy 825	4.2	8.2	8.7	4.2
Stainless Type 329	8.4	15	10.8	8.3
ALOYCO 20	4.6	9.0	11.8	6.5
WORTHITE	5.8	12.7	12.0 (c) 3	8.1
Stainless Type 317	8.3	17	15.5 (c) 3	10.3 (c) 3
Stainless Type 316	14	23	18	11
Stainless Type 316 sensitized	14	22	45	27
HASTELLOY alloy B	18	21 (s) >170		6.6 (r)

- (a) Specimens submerged in recycle pipe line
- (c) Crevice corrosion, maximum depth in mils shown
- (r) Crevice corrosion outside spacer, maximum depth 4 mils
- (s) Stress-corrosion cracking developed around stamped Identification marks

**Effect of Hydrochloric Acid on Wet Process Equipment Materials of Construction**

The effect of incidental chloride impurity in pure phosphoric acid has been discussed previously. Higher concentrations occur in wet processes in which hydrochloric acid is used in addition to or instead of sulfuric acid. One test has been made

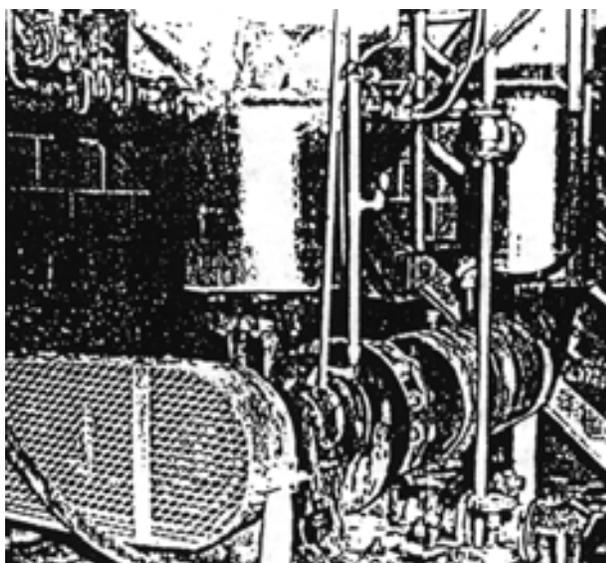


Fig. 15—Evaporator for production of superphosphoric acid from wet-process acid. Photo shows bottom of heater and flash chamber—both of INCOLOY alloy 825, and circulating pump and valves of ACI Type CN-7M (cast alloy 20).

*Courtesy of Swenson Evaporator Company*

in the evaporation of phosphoric acid containing both hydrochloric acid and sulfuric acids, a severe environment which caused quite high corrosion rates in most alloys exposed. Only HASTELLOY alloy C was resistant. The results in Table 33 may be compared with those in Tables 22 and 23.

A process has been described<sup>24</sup> in which phosphate rock is reacted with hydrochloric acid rather than sulfuric acid, clarified, and treated with butyl and amyl alcohols to extract the phosphoric acid content. The solution is washed with water to recover the solvent, and high-purity phosphoric acid is concentrated by evaporation. Few metals are sufficiently resistant to the very corrosive nature of the hydrochloric acid-containing solutions. HASTELLOY alloy C is used for metal parts of rakes contacting the solution.

One set of corrosion test results is available for a wet process based on hydrogen chloride gas rather than on sulfuric acid. The very high corrosion rates suffered by all materials in Table 34 are an indication of the limitations in metallic construction.

A modification of the hydrochloric acid reaction produces dicalcium phosphate suitable for fertilizer or for animal feed. Corrosion problems are similar to those indicated in the parent reaction.

**Table 33**

**Plant Test in Evaporation of Chloride-Containing Wet Process Phosphoric Acid**

Plant test in wet-process phosphoric acid, normally 58% (42% P<sub>2</sub>O<sub>5</sub>) but 75% (54% P<sub>2</sub>O<sub>5</sub>) part of the time, containing 5% hydrochloric acid, 3% sulfuric acid, some hydrofluoric acid, and traces of sodium and potassium fluosilicates, with suspended gypsum. Specimens installed in dome of shell-and-tube heat exchanger but found in bottom of cone downstream.

Temperature: 180 to 200°F (82 to 93°C), average 180-185°F (82-85°C).

Duration: 10 days, no aeration, violent agitation.

Material	Corrosion Rate, mils per year
HASTELLOY alloy C	11
HASTELLOY alloy D	98
WORTHITE	116
MONEL alloy 505 (a)	119
HASTELLOY alloy B	167 (r) 10
MONEL alloy 400	178 (r) 3
Ni-Resist Type 1	184
Ni-Resist Ductile Type D-4 (b)	199
CARPENTER Stainless No. 20Cb	212 (r) 16, (s)
INCOLOY alloy 825	>220(r) 30, (s)
HASTELLOY alloy F	>220 (r) 30
Ni-Resist Ductile Type D-2	227
INCONEL alloy 600	>240(r)30
Ni-Resist Type 2	253
Stainless Type 316	>260 (r) 19
Stainless Type 317	>280 (r) 20
81 Iron-14.5 Silicon-3 Molybdenum alloy	402 (p) 11
Stainless Type 446	>610

(a) Cast equivalent of MONEL nickel-copper alloy 400 modified with 4% silicon

(b) Composition 55 Fe, 31Ni, 5.5 Si, 5 Cr, 2 C

(p) Specimen pitted

(r) Corroded ring developed outside spacer, apparently in retained liquid: maximum depth in mils shown

(s) Stress-corrosion cracking pattern developed around stamped identification marks

**Table 34**

**Field Test in Hydrogen Chloride Reaction with Phosphate Rock**

Exposure in a mixture of phosphoric, hydrochloric and hydrofluosilicic acids containing metallic chlorides and phosphates: concentrations unspecified. Spool located in absorption tower for hydrogen chloride – phosphate rock reaction.

Temperature: 176°F (80°C) maximum.

Duration: 3.78 days. Aeration and agitation present.

No pitting observed.

Material	Corrosion Rate, mils per year
MONEL alloy 400	55
HASTELLOY alloy C	65
Stainless Type 316	97 (c) 6
INCONEL alloy 600	124
Nickel 200	128
Ni Resist Type 1	228
Stainless Type 304	446 (c) 12

(C) Crevice Corrosion, maximum depth in mils shown

## Materials of Construction for Electric Furnace Process Phosphoric Acid Equipment

The electric furnace process produces phosphoric acid of much higher purity than the wet process. It consists essentially of the reduction of phosphate rock to elemental phosphorus, and burning of this to phosphorus pentoxide which is hydrated to phosphoric acid. Other operations which are part of the process are shown in a flow sheet, Fig. 16. These are agglomeration of undersize phosphate rock, distillation and condensation of phosphorus, and collection of acid in an electrostatic precipitator.

The operations in which corrosion data have been obtained are shown by table numbers in Fig. 16.

Further descriptions of the process can be found in Reference 25, covering both steps, in an article on accepted industrial practice<sup>26</sup>, and in two articles<sup>27,28</sup> from a major plant designer.

### Pretreatment

The first step of the electric furnace process agglomerates undersize phosphate rock at 2200 to 2700°F (1200 to 1500°C) in a rotary kiln lined with firebrick. Corrosion data have been gathered in equipment associated with the nodulizing kiln, in environments found in the scrubbing of kiln gas,

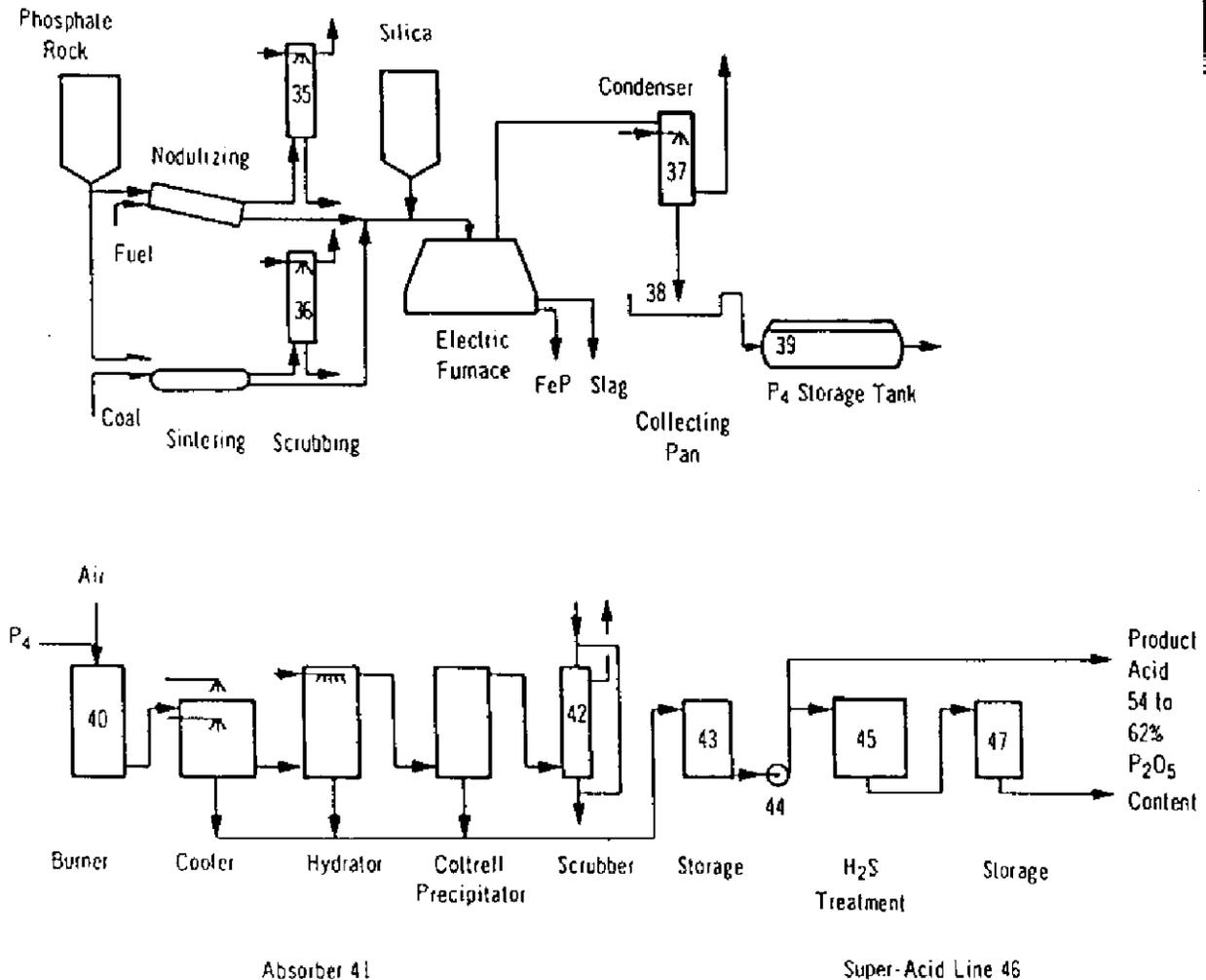


Fig. 16—Electric furnace-process phosphoric acid flow sheet with test spool locations; numbers identify tables of corrosion data.

specifically the stream of slurry produced, a gas stream in the direct path of the slurry spray, and the gas stream following the demister. All are oxidizing acid environments. Results are shown in Table 35.

**Table 35**  
**Pilot Plant and Plant Tests in Nodulizing Kiln Gas Scrubber**

Flue gas contains low concentrations of compounds of sulfur, fluorine, phosphorus and chlorine, 16% oxygen, 5% carbon dioxide.

Liquid contains about 1% fluoride and 0.2% solids, pH near 3.

Specimen location of Test 1 in slurry stream. Test 2 in gas stream in direct path of slurry spray, Test 3 in gas stream two feet above demister. Extensive aeration and agitation.

	Test 1	Test 2	Test 3
Temperature: minimum, °F (°C)	72 (22)	72 (22)	122 (50)
maximum	151 (66)	572 (300)	140 (60)
average	140 (60)	144 (62)	131 (55)
Duration: days	47	47	58
Material	Corrosion Rate, mils per year		
DURIMET 20	<0.1	0.1	<0.1
CARPENTER Stainless No. 20Cb	0.1	1.0	<0.1
INCOLOY alloy 825	-	-	<0.1
HASTELLOY alloy F	-	-	<0.1
Stainless Type 316	0.1	1.6	<0.1
Stainless Type 304	<0.1	29	<0.1
MONEL alloy 400	9.0	28	20
Copper-nickel CA 715 (70/30)	12.0	38	-
CHLORIMET 2 (a)	-	-	30
HASTELLOY alloy B	9.3	39	37
INCONEL alloy 600	5.9 (p)	40	-
Ni-Resist Type 1	-	-	46
Nickel 200	13.5	65	-

(a) Composition 66 Ni, 31 Mo, 2 Fe

(p) Pitted, maximum depth 10 mils; also corroded at crevice

Sintering is also used as a means of agglomerating undersize rock. The particles of rock are blended with coal and fired, the heat fusing the rock and forming a coherent mass which is crushed and screened. Corrosion tests have been run in the scrubbing of the flue gas evolved. Table 36 indicates that severe conditions exist at the middle of the scrubbing tower, less severe conditions in the pan at the base and in the moist gas duct leaving the tower.

**Table 36**  
**Plant Tests in Sinter Flue Gas Scrubbing Tower**

Flue gases contain low concentrations of hydrogen fluoride, sulfur dioxide and silicon tetrafluoride: water contains about 0.25% hydrofluosilicic acid.

Test 1: In water at base, no aeration or agitation.

Test 2: In middle of tower, slight aeration, good agitation.

Test 3: In unspecified gas duct, slight aeration, good agitation.

Duration, 22 days.

	Test 1	Test 2	Test 3
Temperature: minimum, °F (°C)	-	86 (30)	86 (30)
maximum	-	140 (60)	140 (60)
average	86 (30)	122 (50)	122 (50)
Material	Corrosion Rate, mils per year		
HASTELLOY alloy C	0.2	0.2	0.6
CARPENTER Stainless No. 20Cb	1.4	2.1	6.2
Stainless Type 317	1.8	3.0	28
Stainless Type 316	3.0	5.3	66
MONEL alloy 400	25	45	12
Stainless Types 302 and 304	-	-	18
INCONEL alloy 600	28	44	39
Ni-Resist Type 4 (a)	59	135	-
Nickel 200	74 (p)	123	43
Carbon steel	220 (pf)	412	198

(a) Composition 57 Fe, 30 Ni, 5.5 Si, 5 Cr, 2 C

(p) Pitted. maximum depth 18 mils

(pf) Perforated

## Reaction

Silica and coke are reacted with phosphate rock to produce phosphorus approximately according to the following equation:



This equation is calculated from the average composition for the slag produced, according to Reference 25. A small proportion of the phosphorus reacts with reduced iron from iron compounds in phosphate rock, silica and coke, forming ferrophosphorus (an iron-phosphorus alloy), which is drawn off as a liquid slag. Because of temperatures encountered, non-metallics are usually used as the materials of construction for the reaction furnace.

## Condensation

The gaseous product containing phosphorus vapor, carbon monoxide, silicon tetrafluoride, and dust, passes to electrostatic precipitators to remove dust, then to condensers operating just above the melting point of phosphorus. Tests run in the gas stream in air-cooled condensers are shown in Table 37, and indicate a comparatively mild exposure.

**Table 37**

### Plant Tests in Gas Phase in Phosphorus Condenser

Exposure in gas stream of composition 83% carbon monoxide, 8% phosphorus, 7% nitrogen, 2% carbon dioxide (all by volume).

Location of Test 1, in the first vertical 24-inch pipe of the air-cooled condenser; Test 2, in the last.

Aeration absent. Gas flow at moderate velocity, 0.6 to 0.9 feet per second.

	Test 1	Test 2
Temperature: minimum, °F (°C)	275 (135)	150 (65)
maximum	310 (155)	175 (80)
Duration: days	50	56
Material	Corrosion Rate, mils per year	
Silicon bronze CA 655	5.8	0.4
Red brass	4.0	0.5
Nickel 200	0.5	0.9
MONEL alloy 400	0.8	1.3
Stainless Type 316	0.1	2.3
INCONEL alloy 600	0.4	2.8
Ni-Resist Type 2	4.0	3.1
Ni-Resist Type 3 (a)	1.1	3.9
NI-Resist Type 1	26	5.4
Stainless Type 304	<0.1	21
Cast Iron	11	205

(a) Composition 63 Fe. 30 Ni. 2.5 C. 1.5 Si

A small proportion of suspended phosphorus and dissolved phosphoric acid and hydrofluosilicic acid are removed from the condensers by recirculated water ("phossey water"). The acidity may be controlled by soda ash additions to reduce the corrosivity. The suspended impurities, largely hydrated silica, fluosilicates and finely divided phosphorus, are called "mud."

Several tests run in various wet phases in phosphorus condensers are shown in Table 38.

It has also been shown<sup>20</sup> that "phossey water" and mud can cause slight intergranular corrosion of sensitized chromium-nickel-molybdenum stainless steel.

**Table 38**

### Plant Tests in Wet Phases in Phosphorus Condensers

All environments ("phossey water") contained phosphoric and hydrofluosilicic acids.

Test 1: Water layer of collecting pan of air-cooled condenser, containing finely divided phosphorus, coke and phosphate rock.

Test 2: Mud layer of collecting pan of air-cooled condenser, containing finely divided phosphorus, coke and phosphate rock.

Test 3: Liquid level of entrained solids trap, No. 1 leg of furnace.

Test 4: In scrubber-condenser below inlet of duct from vapor head.

	Test 1	Test 2	Test 3	Test 4
Phosphoric acid, %	3	3	3.86	(a)
Hydrofluosilicic acid, %	-	-	0.15	-
Fluorine equivalent, %	1	1	0.38	-
pH	-	-	<1.2	3-6
Temperature:				
minimum, °F (°C)	113 (45)	113 (45)	122(50)	140(60)
maximum	122(50)	122(50)	167(75)	194(90)
average	-	-	-	176(80)
Duration: days	238	238	64	112
Agitation	None	None	None	by water spray
Aeration	None	None	None	Trace
Material	Corrosion Rate, mils per year			
INCOLOY alloy 825	-	-	-	2.3
Stainless Type 317	-	-	-	2.6
DURIMET 20	-	-	-	2.8
Stainless Type 316	0.1	0.1	2.1(p)23	2.8
Stainless Type 304	<0.1	0.2	(b)29(p)21	3.2
INCONEL alloy 600	<0.1	<0.1	7.7	5.5
MONEL alloy 400	0.6	0.2	32	5.0
Nickel 200	1.3	0.2	24	-
Silicon bronze CA 655	0.2	0.2	32	26.8
Red brass	<0.1	0.3	46 (c)	-
Ni-Resist Type 3 (e)	-	-	26	-
Ni-Resist Type 1	6.2	3.0	26	33.8
Ni-Resist Type 2	-	-	31	-
Cast Iron	>123 (d)	>121 (d)	135	-

(a) Phosphoric acid not specified, but present, partly neutralized by soda ash; phosphorus vapor and liquid: traces hydrogen, hydrocarbons, carbon monoxide, carbon dioxide, phosphine

(b) Type 302

(c) Both specimens non-uniformly corroded to perforation

(d) Destroyed

(e) See Table 37

(p) Pitting, maximum depth in mils shown

## Storage

Liquid white phosphorus from the condensers is stored under water. Corrosion tests reported in Table 39 indicate that equipment is subjected to an extremely mild environment.

**Table 39**

### Plant Tests in Handling and Storage of Phosphorus

Tests 1 and 2: Exposures in transfer of liquid phosphorus from storage tanks into drums, respectively in liquid phosphorus and in buffered phosphy water phases. Aeration was moderate, agitation was absent.

Test 3: Data previously published<sup>1</sup> with no details beyond "elemental phosphorus in storage."

	Test 1	Test 2	Test 3
Phase	P <sub>4</sub>	H <sub>2</sub> O	P <sub>4</sub>
Temperature: minimum, °F (°C)	136(58)	136(58)	149(65)
maximum	144(62)	144(62)	158(70)
average	140(60)	140(60)	-
PH	-	9	-
Duration: days	185	185	Unspecified
Material	Corrosion Rate, mils per year		
Stainless Type 446	<0.1	nil	-
Stainless Type 316	<0.1	<0.1	0.1
Stainless Types 302, 304	<0.1	<0.1	0.1
Stainless Type 430	<0.1	<0.1	-
Stainless Type 410	<0.1	<0.1	-
INCOLOY alloy 800	<0.1	<0.1	-
INCONEL alloy 600	<0.1	<0.1	-
HASTELLOY alloy C	-	-	0.2
HASTELLOY alloy D	-	-	0.3
Ni-Resist Type 2	-	-	0.3
Ni-Resist Type 1	-	-	0.4
MONEL alloy 400	0.5	0.1	0.8
Nickel 200	0.5	0.1	1
Silicon bronze CA 655	-	-	1.2
Red brass	-	-	1.4
Carbon steel	1.1	0.4	4
Ductile cast Iron	1.4	0.7	-
Cast Iron	1.9	0.6	7

## Burning

Liquid phosphorus is converted to phosphorus pentoxide in a combustion process step. It is atomized with an air stream in a nozzle and burns to phosphorus pentoxide in a combustion chamber composed of graphite bricks or stainless Type 316L externally cooled by running water. Combustion, hydration and absorption equipment constructed of Type 316L is shown in Fig. 17. Corrosion rate results are available from a test in a phosphorus burner; these are shown in Table 40.

**Table 40**

### Plant Test in Phosphorus Burner

Environment: Phosphorus pentoxide, condensed phosphoric acid, with combustion air. Specimens supported about one foot from atomizing nozzle.

Temperature: 480-1470°F (250-800°C; average 930°F (500°C).

Duration: 49 days; aeration present, agitation extensive (12 feet per second).

No pitting, but high-velocity effects were found.

Material	Corrosion Rate, mils per year	
CARPENTER Stainless No. 20Cb	6.8	
Stainless Type 310	7.6	
Stainless Type 317	6.5	14
Stainless Type 316	6.4	15
HASTELLOY alloy B	63	
Graphite	Oxidized within 28 hr	

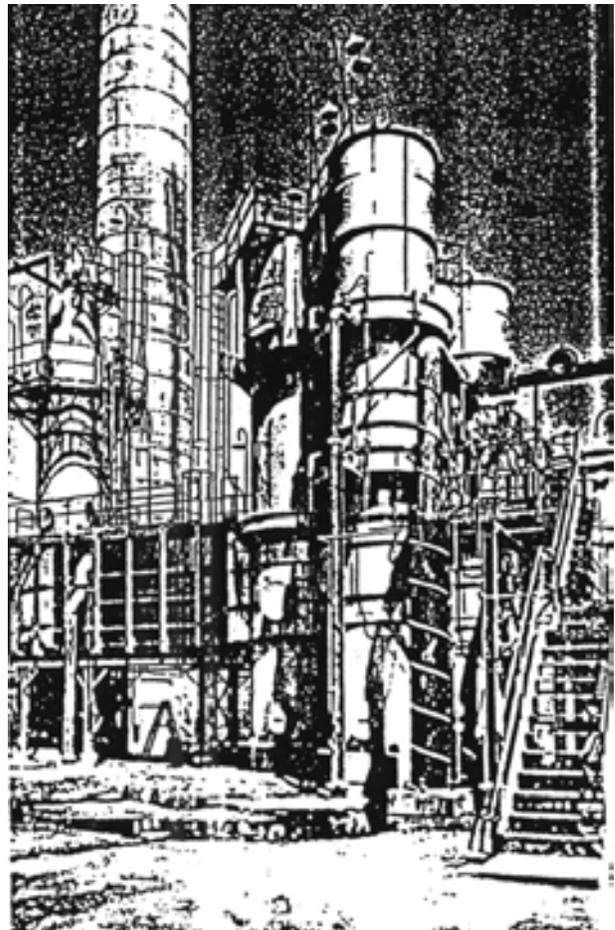


Fig. 17—Electric furnace-process phosphoric acid plant, showing equipment constructed of Type 316L stainless steel, jacketed with Type 304. Units from right to left: two combustion chambers (one partly hidden), hydrator, Venturi scrubber, entrainment separator.

*Courtesy of Tennessee Valley Authority*

Gas from the combustion chamber is absorbed in a gas cooler, also externally water-cooled, equipped with internal sprays to prevent accumulation of glassy phosphoric acid. The gas passes through water sprays in a carbon-lined or stainless Type 316 hydrator, forming a mist of 75 to 85 per cent phosphoric acid. One set of test results (Table 41) is available from an acid spray absorber; it shows several common stainless steels to be highly resistant to the low-temperature exposure.

**Table 41**

**Plant Test in Phosphoric Acid Absorber**

Plant corrosion test in a spray of 75% electric furnace process phosphoric acid (54% P<sub>2</sub>O<sub>5</sub>). Specimens exposed near top of absorber.

Temperature: 150-160°F (65-70°C), average 153°F (67°C).

Duration: 328 days; aeration extensive, agitation moderate.

Material	Corrosion Rate, mils per year
Stainless Type 304	<0.1
DURIMET 20	<0.1
Stainless Type 317	<0.1
Stainless Type 316	<0.1
MONEL alloy 400 (a)	22

(a) Specimens exposed 189 days

**Table 42**

**Plant Test in Exhaust Gas**

Plant spool test in duct carrying exhaust gas, water-saturated, of composition (on dry basis) 84% nitrogen, 15% oxygen, rphosphoric acid (anhydrous basis) 0.16 to 0.27 pounds per 1000 cubic feet of gas, hydrofluosilicic and hydrofluoric acids equivalent to .004 pound fluorine per 1000 cubic feet of gas. Composition of condensed liquid 0.7% H<sub>3</sub>PO<sub>4</sub>, fluorine compounds equivalent to .026% F.

Temperature: 190-220°F (88-104°C), average 204°F (96°C).

Duration: 5 days, aerated, gas velocity 35 feet/second.

No pitting.

Material	Corrosion Rate, mils per year
CARPENTER Stainless No. 20Cb	5.7
Stainless Type 316	5.7
HASTELLOY alloy B	12.0
Stainless Types 302, 304	37, 107
MONEL alloy 400	132
Nickel 200	211
INCONEL alloy 600	342
Ni-Resist Type 1	596
Carbon steel	>2440(d)
Cast iron	>5480(d)

(d) Destroyed

**Exhaust Gas Absorber**

The part of the phosphoric acid not collected in the hydrator remains in the form of an acid mist which is extracted by electrostatic precipitation on stainless steel wires in a carbon-lined or stainless steel chamber. The exhaust gas after precipitation is scrubbed and discharged to the atmosphere. A test in the gas is reported in Table 42; the scrubbing solution, composed of sodium carbonate and disodium phosphate, is not corrosive.

**Product Acid**

The resistance of a number of alloys to corrosion by product acid resulting from the electric furnace process<sup>1</sup> is reported in Tables 43 and 44.

The normal acid concentration is 75 per cent; plant equipment is usually made of Type 316 stainless steel. The data in Table 44 correlate quite closely with those published by Morgans<sup>30</sup> for plan exposures of Type 316L and Type 317 stainless steel over a temperature range of 170 to 217°F , (77 to 103°C). The same study includes laboratory and pilot scale tests of wrought, welded and cast materials in a variety of conditions; these were less severe environments than was the plant.

**Table 43**

**Plant Test in Phosphoric Acid Containing Lower Oxides of Phosphorus**

Corrosion test exposure in 82-87% phosphoric acid (59-63% P<sub>2</sub>O<sub>5</sub>) containing lower oxides of phosphorus. Specimens exposed in brick-lined acid storage sump six feet from air port.

Temperature: 190-215°F (88-102°C), average 209°F (98°C).

Duration: 5 days; agitation by means of aeration.

Material	Corrosion Rate, mils per year
HASTELLOY alloy B	1.6
CARPENTER Stainless No. 20Cb	4.9
Stainless Type 316	22
MONEL alloy 400	22
Nickel 200	119
INCONEL alloy 600	201
Ni-Resist Type 1	209
Stainless Type 302	576
Stainless Type 304	(d)
Carbon steel	(d)
Cast iron	(d)

(d) Destroyed

**Table 44**  
**Plant Test in Phosphoric Acid Containing Phosphorus**

Corrosion test exposure in 75% phosphoric acid (54% P<sub>2</sub>O<sub>5</sub>) occasionally containing elemental phosphorus. Specimens exposed in circulating acid pump tank.

Average temperature: 160°F (70°C).

Duration: 39 days except as noted; aeration and agitation present.

Material	Corrosion Rate, mils per year
CARPENTER Stainless No. 20Cb	<0.1(a)
WORTHITE	0.1
Stainless Type 317	0.2
Stainless Type 316	0.5
ILLIUM "R"	0.6
HASTELLOY alloy B	5.8
MONEL alloy 400	8.5
Copper-nickel CA 715 (70/30)	23 (b)
Copper-nickel CA 706 (90/10) (c)	24 (b)
Brass, red	27 (b)
INCONEL alloy 600	27
Brass, yellow	34 (b)
Nickel 200	85
Lead, chemical	145
Stainless Type 347	307 (d)
Stainless Type 304	596 (e)

(a) Duration 45 days

(b) Duration 10 days

(c) Composition 88 Cu, 10 Ni, 1.5 Fe

(d) Duration 12.4 days

(e) Duration 23 days

### Purification, Food Grade Acid

When phosphoric acid is to be purified to food-grade specification, it is saturated with hydrogen sulfide to precipitate sulfides of lead and arsenic, elements which were originally present in the ore. The precipitate is removed by filtration. The dissolved hydrogen sulfide increases the corrosivity of the acid, as shown in Table 45.

Super-acid is made in the electric furnace process by limiting the water feed to the hydrator, thereby raising the acid concentration to 105 per cent (76% P<sub>2</sub>O<sub>5</sub>). Additional cooling is provided in the combustion chamber, the hydrator, and by a recycle line from and to the hydrator. Dilute acid produced in the electrostatic precipitator may also be recycled to the hydrator. These coils may be Type 316L or 317L stainless steel, choices supported by the results of the plant-scale tests run in super-acid and shown as Table 46.<sup>31</sup>

**Table 45**  
**Field Spool Tests in Hydrogen Sulfide Treatment of 75% Phosphoric Acid**

Specimens exposed in acid containing 100 ppm H<sub>2</sub>S in treating tank (Test 1), and in the vapor area above the acid containing an unspecified concentration of H<sub>2</sub>S in storage tank where they were exposed to occasional acid splash (Test 2).

Phase	Test 1	Test 2
	Liquid	Gas
Temperature: minimum, °F (°C)	113(45)	104(40)
maximum	149(65)	149 (65)
average	131(55)	122 (50)
Duration: days	60	105
Aeration	None	Moderate
Agitation	Mild	Occasional
Material	Corrosion Rate, mils per year	
Stainless Type 317	0.3	5.0(d)(r)
CARPENTER Stainless No. 20Cb	0.5	6.8 (d) (r)
Stainless Type 316	0.9 (a)	5.7 (b) (d) (r)
HASTELLOY alloy B	2.4	2.3
INCONEL alloy 600	30	11.5 (d)
MONEL alloy 400	33	15.4
Stainless Type 347	61	11.1 (d) (r)

(a) Type 316 tank has pitted and rusted in vapor areas; severe intergranular corrosion in heat-affected zone near weld

(b) Type 316 tank severely rusted and pitted

(d) Specimens covered with heavy rust deposit

(r) Specimen surface severely roughened; numerous small pits formed

**Table 46**  
**Plant Tests in Superphosphoric Acid**

Plant corrosion tests in 105% electric furnace process acid (76% P<sub>2</sub>O<sub>5</sub>). Specimens in hot acid line between circulating pumps and heat exchanger.

Temperature: 250°F (120°C) and 275°F (135°C).

Duration: 4.5 and 5.0 days.

Material	Corrosion Rate, mils per year	
	250 F	275 F
Stainless Types 316, 316L	1.3	4.3
Stainless Types 317, 317L	1.0	3.4

**Transportation and Storage of Phosphoric Acid**  
Transportation and storage of phosphoric acid can be handled in Type 316 (or, if welded, Type 316L) stainless steel equipment. Under practical conditions molybdenum-free Type 304 is a borderline

material. At ambient temperature, electric furnace-process acid free of chlorides corrodes Type 304 at a rate less than 1 mil per year, but chloride contents of 25 to 400 ppm raise this rate to excessive values. In Lowe's study of chloride contamination



Fig. 18—Tank car for transportation of superphosphoric acid. Inner tank Type 316L stainless steel, covered with polyurethane insulation.

*Courtesy of Union Tank Car Co.*

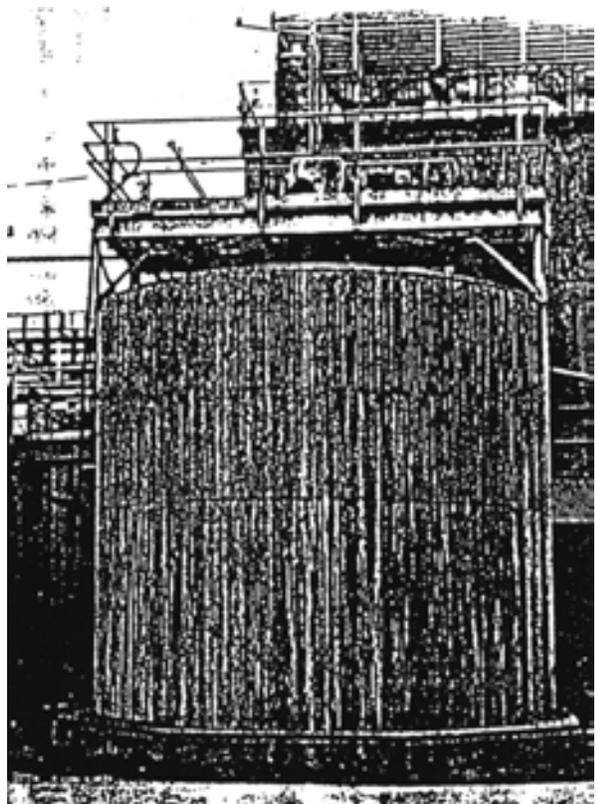


Fig. 19—Type 316L stainless steel phosphoric acid storage tank.

*Courtesy of Tennessee Valley Authority*

effect<sup>12</sup>, seven-day exposure at 100°F in 75 per cent furnace acid contaminated with 335 to 355 ppm of chloride corroded Type 304 at a rate of 213 mils per year and Type 316 at only 1.4 mils per year. Laboratory tests with controlled chloride additions to reagent grade acid, shown in Table 4, produced an even sharper contrast. The plant exposure results shown in Test 1 of Table 47 present a clear difference between Type 316 and molybdenum-free Type 347; the latter exhibited both a higher rate of weight loss and development of pits.

**Table 47**

**Plant Test in Phosphoric Acid Storage Tanks**

Environment: Electric-furnace acid. Aeration was present, agitation absent except during transfers to and from tanks.

	Test 1	Test 2
Phosphoric acid concentration, %	75	70
Temperature: minimum, °F (°C)	104(40)	5(-15)
maximum	140(60)	130(54)
average	122(50)	75(24)
Duration: days	60	310
Material	Corrosion Rate, mils per year	
CARPENTER Stainless No. 20Cb	0.1	—
Stainless Type 317	0.1	—
Stainless Type 316	0.5	<0.1
Stainless Types 304. 304L	—	<0.1
Stainless Type 304 sensitized	—	15
HASTELLOY alloy C	—	<0.1
HASTELLOY alloy B	1.0	—
HASTELLOY alloy F	—	0.1
Stainless Type 347	3.0 (p)	—
INCONEL alloy 600	5.9	0.2
MONEL alloy 400	6.3	—

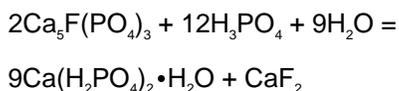
(p) Pitted; depth not stated

The preference for a low carbon content, mentioned under Metallurgical Factors, is supported by experience with both molybdenum-free and molybdenum-containing stainless steels. Test 1, discussed above, was conducted following an experience with intergranular corrosion in welded zones of a standard-carbon Type 316 storage tank for 75 per cent furnace acid. The results in Test 2 are from a different storage tank holding 70 per cent furnace acid. Here it is obvious that the sensitized Type 304 specimen underwent greatly accelerated corrosion.

## Materials of Construction for Producing Phosphate Fertilizers

### Triple Superphosphate

Triple superphosphate is manufactured from phosphate rock and wet-process phosphoric acid of 66 to 69 per cent concentration (48 to 50%  $P_2O_5$ ). The reaction has been presented as follows<sup>32</sup>:



The acid is mixed with ground phosphate rock in a reactor, then discharged into a den or chamber or onto a belt, where the reaction continues and the fluid mix becomes solid within a few minutes. The material may then “cure” in a pile for a period of days to weeks. Alternatively the mixed slurry may be fed from the reactor to a blade mixer such as a blunger where it is coated onto a fine previously dried product, thus building up to granules. These are fed to a continuous rotary dryer, sized by screening, then stored.

Corrosion-resistant materials of construction, for example Ni-Resist or Type 316 stainless steel, are required for the reactor and associated equipment in the production line to the stage where the progress of the reaction has reduced the acidity. Corrosion problems also arise from the fluoride-containing gas, principally silicon tetrafluoride, that is evolved rapidly from the mixer and den or belt, and slowly from the storage pile. Gas and air from these sections of the plant are gathered and scrubbed in equipment that must be constructed from corrosion-resistant materials.



Fig. 20—Type 1 Ni-Resist mixer-reactor vessel for superphosphate manufacture.



Fig. 21—Ductile Type D-2 Ni-Resist paddles used with mixer-reactor vessel for superphosphate manufacture.

Numerous uses of Ni-Resist and of Type D-2 ductile Ni-Resist in the production of normal superphosphates in Broadfield dens in New Zealand fertilizer plants are described in Reference 33. The Broadfield den consists of a rotary mixer with semi-circular bottom and vertical sides in which sulfuric acid and ground phosphate rock are partially reacted and the slurry discharged onto a den floor conveyor, where it hardens into a cake. The conveyor travels with the “cake” of superphosphate to the end of the chamber between the walls of the den, the rear sections of which move with a reciprocating motion to prevent sticking. The cake is fed to a rotary cutter from which it is discharged to a rubber conveyor belt and transported to storage for maturing.

Type 1 Ni-Resist is used for lining the mixer walls and in some cases for the complete mixer body. Type D-2 ductile Ni-Resist is used for mixer shaft sleeves, for mixer paddles, for bolts and nuts which fasten the paddles to the shaft, and for an adjustable discharge weir. In the conveyor section of the den, Type D-2 ductile Ni-Resist is used for cutter blades, and in some cases for reciprocating den sides.

In some plants, Type 1 Ni-Resist is also used for acid entrance headers and splash pots feeding sulfuric acid to the den mixer.

Several corrosion tests have been run in triple superphosphate processes. Table 48 covers an exposure in the wet-process acid used. Table 49 includes two tests in fumes in dens (used for the manufacture of normal rather than triple superphosphate);

Table 50 provides two tests in fumes in ducts, one ahead of the exhaust fan, one after water sprays. Table 51 covers an exposure in the scrubber solution recycle tank. Table 52 shows the results of exposure in flue gas after its use to dry triple superphosphate.

**Table 48**  
**Plant Test in Fertilizer Acid**

Plant exposure test in 69% wet-process phosphoric acid (50% P<sub>2</sub>O<sub>5</sub>) used in triple superphosphate manufacture; also contains 3% sulfuric acid and 1.85% fluoride. Specimens exposed in belt acid feed surge box.

Temperature: 135-180°F (57-82°C), average 150°F (65°C).

Duration: 41 days; no aeration, agitation very slight.

Material	Corrosion Rate, mils per year
Stainless Type 317	2.3
Stainless Type 309 (a)	2.9
Stainless Type 316	3.9
Lead, chemical	7.0
INCOLOY alloy 800	27
MONEL alloy 400	65
Stainless Type 304	79
HASTELLOY alloy B	> 141 (d)
INCONEL alloy 600	> 142 (d)

(a) See Table 20

(d) Destroyed

**Table 49**  
**Plant Tests in Reaction Dens**

Exposure in fumes containing hydrogen fluoride, silicon tetrafluoride, hydrofluosilicic acid, and in Test 1 a mist of sulfuric and phosphoric acids.

Test 1, Spool location: Attached to ceiling of den.

Temperature: 100-200°F (38-93°C), average 150°F (65°C).

Duration: 45 days, no positive agitation.

Test 2, Spool location: In fumes in den.

Average temperature: 250°F (121°C).

Duration: 30 days.

Material	Corrosion Rate, mils per year	
	Test 1	Test 2
HASTELLOY alloy B	3.2	10
HASTELLOY alloy C	–	11
Stainless Type 317	–	18
Stainless Type 316	2.7	18
Lead, chemical	3.3	17
INCONEL alloy 600	4.3	20
MONEL alloy 400	5.8	23
Stainless Type 304	3.1	26
Nickel 200	7.9	29
Carbon Steel	6.4	45
Ni-Resist Type 1	–	53
Cast Iron	–	218

**Table 50**  
**Plant Tests in Fumes from Superphosphate Manufacture**

Test 1: Fumes described as containing hydrogen fluoride, silicon tetrafluoride, hydrofluosilicic acid, and a mist of sulfuric and phosphoric acids. Specimens exposed in fume duct, ahead of exhaust fan, from normal superphosphate mixing den.

Average temperature: 130°F (54°C).

Duration: 57 days, aeration and agitation present.

Test 2: Fumes described as containing silicon tetrafluoride and other "silicon volatiles," carbon dioxide, water vapor. Specimens exposed in fume scrubber duct from triple superphosphate reaction, after water sprays.

Temperature: 160-220°F (71-105°C), average 175°F (80°C).

Duration: 52 days. Aeration extensive, gas velocity 60 feet per second

Material	Corrosion Rate, mils per year	
	Test 1	Test 2
DURIMET 20	–	3.7
Stainless Type 316	13	58(s)
MONEL alloy 400	21	78(s)
HASTELLOY alloy D	–	100
Nickel 200	13	>140 (m)
Bronze 85/5/5/5	13	–
INCONEL alloy 600	16	>141 (m)
Stainless Type 302	28	>158 (m) (t)
Lead, chemical	32	>212 (m)
Ni-Resist Type 3 (a)	33	–
Carbon steel	40	>153 (m)
Bronze 88/10/2	–	280 (c)
Hi-Resist Type I	–	464
Cast iron	197	>556 (m)

(a) See Table 37

(c) Substantial copper deposit on specimens; weight loss is actually greater than indicated

(m) Missing, presumably destroyed

(s) Stress-corrosion cracking through imprints; part of metal cracked off

(t) Type 304

**Table 51**  
**Plant Test in Den Scrubber Solution Recycle Tank**

Exposure in 20% hydrofluosilicic acid.

Temperature: 130-140°F (54-60°C), average 135°F (57°C).

Duration: 28 days; extensive aeration, slow agitation.

Material	Corrosion Rate, mils per year
CARPENTER Stainless No. 20Cb	3.3
ILLIUM "G"	3.4
INCOLOY alloy 825	4.6
Stainless Type 317	4.9
HASTELLOY alloy C	5.1
Stainless Type 316L	5.3
HASTELLOY alloy F	6.4
Stainless Type 329	81

**Table 52****Plant Test in Triple Superphosphate Drier Gases**

Plant spool test in fuel oil burner gases after use to dry triple superphosphate.

Test 1: In steel duct leading to scrubber.

Test 2: Inside top of scrubbing tower.

Composition: flue gas (nitrogen, oxygen, carbon dioxide sulfur dioxide), silicon tetrafluoride, silicon dioxide, sulfuric acid, phosphoric acid equivalent to 0.2% P<sub>2</sub>O<sub>5</sub> and in Test 2 some fine water spray.

Duration: 146 days. Aeration present, flow rate high.

	Test 1	Test 2
Temperature: minimum, °F (°C)	300 (149)	135(57)
maximum	500 (260)	180(82)
average	400 (204)	150(66)
Material	Corrosion Rate, mils per year	
INCOLOY alloy 825	<0.1	0.1
Stainless Type 316	<0.1	0.7
MONEL alloy 400	0.1	318
Carbon steel	2.8	>550 (d)

(d) Destroyed

**Ammonium Phosphate Fertilizer**

Water-soluble solid ammonium phosphate fertilizer is made by reacting phosphoric acid, usually wet-process, about 58 per cent H<sub>3</sub>PO<sub>4</sub> or 42 per cent P<sub>2</sub>O<sub>5</sub> content, with anhydrous ammonia. Sulfuric acid may be added when the product grade requires reducing the P<sub>2</sub>O<sub>5</sub>/NH<sub>3</sub> ratio. As in the case of superphosphates, the reactor uses Type 316 stainless steel in the high-temperature area. Where the product is nearly neutral and comparatively non-corrosive, carbon steel may be used for such equipment as blunger, drier, screens and conveyors.

Liquid ammonium phosphate fertilizer is of commercial interest because of high solubility and consequent ease of handling. Its analysis is typically 10 per cent N, 34 per cent P<sub>2</sub>O<sub>5</sub>. It is prepared from anhydrous ammonia and superphosphoric acid, the latter derived principally from wet-process acid. This super-acid has a P<sub>2</sub>O<sub>5</sub> content of about 72 per cent, of which substantially four-tenths is in a non-ortho complex form. The reactor may be Type 316 stainless steel or equipped with Type 316 stainless steel cooling coils. The product is often transported in stainless steel tank cars or trucks.

There is one process for the manufacture of

diammonium phosphate from anhydrous ammonia and furnace-process phosphoric acid<sup>34</sup>. The raw materials are fed to a vacuum crystallizer; product crystals are centrifuged, dried, and screened. Alternatively the equipment may be a pre-neutralizer, scrubber, TVA ammoniator-granulator, drier, screen, product cooler, and gas scrubber. The crystallizer or pre-neutralizer may be made of a 300-series stainless steel; the screen cloth is a 300-series stainless steel.

Fertilizer-grade diammonium phosphate is produced from coke-oven by-product ammonia and furnace-grade phosphoric acid. Descriptions of the two processes can be found in Reference 35. The ammonia is absorbed from coke-oven gas by dispersal under the liquid surface or by contact with sprayed phosphoric acid solution. In both cases secondary absorption or clean-up is accomplished by sprays. The absorbing acid has a pH of 5.5 to 6.8 to prevent formation of the monoammonium salt while minimizing escape of ammonia in the scrubbed gas. The acid, containing suspended diammonium phosphate, is circulated through a settling tank. Temperature is kept near 122°F (50°C) because of the solubility of the various ammonium phosphates.

Materials of construction in contact with un-neutralized acid include Type 316 stainless steel, and nickel-copper Alloy 400.

**Dicalcium Phosphate for Fertilizer and Feed Purposes**

Dicalcium phosphate (CaHPO<sub>4</sub>•2H<sub>2</sub>O) is formed by reaction of phosphoric acid with lime or limestone<sup>36</sup>. Various processes have been described for defluorination by partial neutralization with soda ash or lime. Using lime and wet-process acid, the product is an impure grade of monocalcium-dicalcium phosphate containing fluorides and is suitable for fertilizer. Other processes are proprietary. Subsequent complete neutralization produces the salt in a fluoride-free grade suitable for animal feed. Actual test data are not available, but it has been stated<sup>30</sup> that Type 316 stainless steel may be used for metallic construction.

Electric furnace-process acid may be used to produce a human food-grade product without need of defluorination. Material requirements are similar.

### Liquid Mixed Fertilizers

Liquid mixed fertilizers, containing nitrogen, phosphate and sometimes potash have become an important product of the fertilizer industry. In some cases these are made by dissolving fertilizer salts such as sodium nitrate, ammonium nitrate, urea and potassium chloride in phosphoric acid. In other cases the phosphoric acid is first neutralized with ammonia followed by addition of supplemental nitrogen and potash, if needed. The mixing or "reactor" tanks and fittings are frequently constructed of Type 316 stainless steel.

The liquid mixes are usually transported in tank trucks or "nurse" tanks and applied to the ground by spraying from applicator tanks. Since they can be corrosive to mild steel, cast iron and aluminum, Type 304 stainless steel is frequently used for tanks and fittings, and Ni-Resist alloys for pumps, valves, couplings and other cast parts. Where unneutralized phosphoric acid is used on alkaline soils, Type 316 stainless steel is usually employed.

The results of laboratory corrosion tests<sup>37</sup> in several liquid-mix fertilizer solutions, are shown in Table 53.

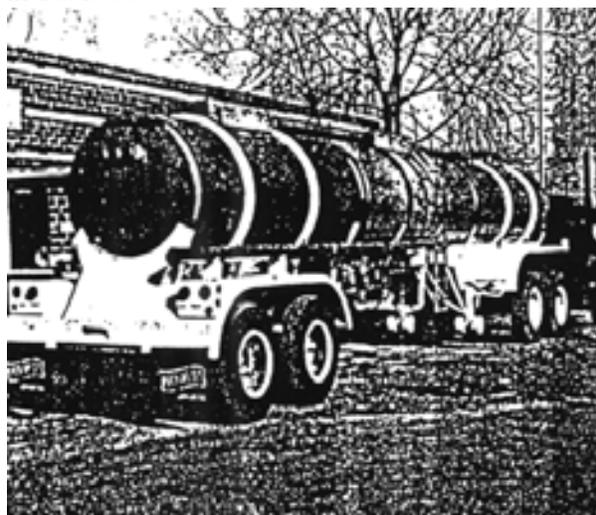


Fig. 22—Type 316 stainless steel tank truck for transportation of phosphoric acid.

Courtesy of Progress Industries

Table 53

#### Laboratory Tests in Liquid Fertilizer Solutions

Specimens partially immersed in stoppered flasks. Temperature 100 to 135°F (38 to 57°C), duration 28 days; some aeration, no agitation. Pit depth tests made in welded metal tanks partially filled with solutions.

Temperature: 135°F (57°C).

Duration: 28 days; some aeration, no agitation.

Test 1: Fertilizer analysis 6-6-6, nitrogen supplied as ammonium nitrate. pH 6.3

Test 2: Fertilizer analysis 6-6-6, nitrogen supplied as urea; pH 6.8

Test 3: Fertilizer analysis 10-10-5, nitrogen supplied as ammonium nitrate; pH 6.2

Test 4: Fertilizer analysis 10-10-5, nitrogen supplied as urea; pH 6.9

Test 5: Fertilizer analysis 3-9-9, nitrogen supplied as ammonium phosphate; pH 6.6

Test 6: Fertilizer analysis 8-24-0, nitrogen supplied as ammonium phosphate; pH 6.6

Material	Corrosion Rate, mils per year					
	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
Stainless Type 304	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Stainless Type 316	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Stainless Type 430	<0.1 (h)	<0.1 (p) 6, (h)	<0.1 (h)	<0.1 (h)	<0.1 (p) 34, (h)	<0.1 (h)
Stainless Type 405	<0.1	<0.1 (perf) 50	<0.1 (p) 8	<0.1 (p) 20	0.4 (perf) 50	<0.1
Stainless Type 502	5.9 (p) 25	0.6 (p) 22	8.2 (p) 42	0.5 (p) 8	14.3 (p) 20	<0.1
Carbon Steel AISI 1020	7.9 (p) 32	3.4 (p) 8	3.7 (p) 36	2.6 (p) 10	3.7 (p) 10	1.7
Aluminum 3003-H14	14.1	8.2	25.9	45.8	24.7	200.
Aluminum 6061-T6	(p) 10	(p) 9	—	—	(p) 4	(p) 48

(h) Cracked in heat-affected zone near welds

(p) Pitted. maximum depth in mils

(perf) Perforated



Fig. 23—Type 304 stainless steel nurse tank and applicator tank used for mixed fertilizer.



Fig. 24—Type 316 stainless steel applicator tank, mounted on tractor. used for 52% phosphoric acid.

### Granular Mixed Fertilizers

Granular mixed fertilizers may be formed from liquid single components and mixes, and from solids. Starting materials are fed at controlled rates to provide desired compositions, mixed, solidified, dried, and cooled. Raw materials include phosphoric acid, sulfuric acid, ammonia, ammonium nitrate, nitrogen solutions, potassium chloride, ammonium phosphate and sulfate. Since there are many different types of granular mixed fertilizers, there is also a wide variation in the design and construction of producing equipment. Type 316 stainless steel is often used for reaction tanks and for phosphoric acid lines, pumps and valves. HASTELLOY alloy C is used for sulfuric acid distributors to resist that acid and the hydrogen chloride formed as a by-product where the acid contacts potassium chloride.

### Mixed Fertilizers from Nitric Acid

Mixed fertilizers can also be produced by treating phosphate rock with nitric rather than sulfuric acid. The by-product of this reaction is calcium nitrate. Being highly soluble it cannot readily be filtered to produce phosphoric acid; being deliquescent it cannot conveniently be left in a mixture which may be bagged and stored. For these reasons, each process includes steps in which calcium nitrate is converted into other materials.

The other reactants are ammonia and one of the following: carbon dioxide, sulfuric or phosphoric acid mixed in small proportion with the nitric acid, potassium chloride, potassium or ammonium sulfate. Calcium and phosphate in the mixture are converted essentially to dicalcium phosphate, and nitrate largely to ammonium nitrate. The product, with much improved handling properties, is used as a mixed fertilizer.

Stainless steels of both Types 304 and 316 have been used for the extractor where the initial reaction occurs; Type 316L is understood to be preferred.<sup>31</sup> Equipment to dry slurries, for example granulators, can be fabricated from carbon steel. Wet scrubbing systems for hot dusty air from driers, however, are constructed of Type 304 stainless steel when required by the high temperature of operation.

## Salt-Suspension Fertilizers

Salt-suspension or slurry fertilizers can be handled as liquids and yet can be made to contain considerably more plant food. They are usually made with wet-process phosphoric acid, frequently using

**Table 54**  
**Corrosion Studies in Production and Storage of Suspension Fertilizers**

Test 1: Plant spool test in reactor.

Environment: nitric acid, phosphoric acid, sulfuric acid, phosphate rock, ammonia, potassium chloride reactants. Product composition quoted in table.

Tests 2 and 3: Plant spool test in storage tanks.

All specimens located one foot from bottom of tank.

	Test 1	Test 2	Test 3
Fertilizer analysis	various	6-12-12	14-7-7
Calcium acid phosphates, as P <sub>2</sub> O <sub>5</sub>	12-14	12	7
Potassium chloride	11.6-30	20	11.6
Ammonium nitrate	13-38	13	38
Water	30	30	30
Inerts, largely Insoluble	12	12	12
pH	low	6-7	3.5-4.5
Temperature: minimum, °F (°C)	10(-12)	10(-12)	20 (-7)
maximum	250(121)	80(27)	170 (77)
average	200(93)	30(-1)	25 (-4)
Duration: Lays	74	107	109
Aeration	moderate	moderate	moderate
Agitation	considerable	slow	slow
Material	Corrosion Rate, mils per year		
INCOLOY alloy 825	2.7 (p) 4	<0.1	<0.1
Stainless Type 317	3.1	<0.1	<0.1
CARPENTER Stainless No. 20Cb-3	3.5	<0.1	<0.1
HASTELLOY alloy C	4.4	<0.1	<0.1
Stainless Type 316L	32	<0.1	<0.1
Stainless Type 316 sensitized	95	<0.1	<0.1
Stainless Type 304	missing	<0.1 (p) 10	<0.1
Stainless Type 304 sensitized	50 (p) 4	<0.1	<0.1
Stainless Type 347	missing	<0.1(p) 6	<0.1(p) 8
HASTELLOY alloy B	>86 (d)	3.1	0.1
MONEL alloy 400	>86 (d)	3.3 (p) 4	0.7(p) 3
Ni-Resist ductile Type D-2	>400 (d)	0.7 (p) 3	2.1
Aluminum 6061	>79 (d)	10.0 (perf)	3.1(perf)
Aluminum 5052	>158 (d)	4.9 (p) 35	3.8(perf)
Aluminum 1100	>151(d)	8.2 (p) 27	4.0(p)28
Carbon steel	>96 (d)	1.0 (p) 15	5.6
Cast Iron	>398 (d)	1.6 (p) 9	6.0

(d) Destroyed  
(p) Pitted, maximum depth in mils  
(perf) Perforated

superphosphoric acid because of its sequestering properties.

Table 54 presents corrosion data gathered in equipment for producing and storing complete mixed suspension fertilizers. The corrosivity of the reaction conditions (Test 1) is clearly shown. In storage the difference between the corrosivity of the two grades may be attributed to the high chloride content of one (Test 2) and the acidity of the other (Test 3).

Table 55 reports a one-year field exposure test in an applicator tank in varied service that included complete mixed fertilizer solutions and suspensions.

**Table 55**  
**Corrosion Test in Fertilizer Applicator Tank**

Field spool test. Specimens on flat head, one foot off bottom of applicator tank, in suspension fertilizers spread on farmland.

Temperature: 30-180°F (-1 to 82°C), average 100°F (38°C).

Duration: 297 days including inactive winter season. Aeration present; agitation moderate.

Material	Corrosion Rate, mils per year
INCOLOY alloy 825	<0.1
HASTELLOY alloy C	<0.1
CARPENTER Stainless No. 20Cb-3	<0.1
Stainless Type 316	<0.1
Stainless Type 316 sensitized	<0.1
Stainless Type 304	<0.1
Stainless Type 304 sensitized	<0.1
Stainless Type 347	<0.1(p) 3
Stainless Type 317	<0.1
Stainless Type 410	0.4 (p) 13, (c) 26
HASTELLOY alloy B	0.4
Ni-Resist, Ductile Type D-2B	0.9
Ni-Resist, Ductile Type D-2	1.4 (p) 4, (c) 5
MONEL alloy 400	1.7 (p) 3
Aluminum 5052	8.1 (p) 32, (cp)
Aluminum 1100	10.0 (p) 36, (cp)
Cast iron, flake graphite	16
Aluminum 6061	>19 (d)
Carbon steel	>20 (pp)
Cast iron, ductile	32

(c) Crevice corrosion. maximum depth in mils  
(cp) Crevice and surrounding area perforated  
(d) Destroyed  
(p) Pitted, maximum depth in mils  
(pp) Profuse perforations. "lacework" appearance of surviving metal

**Table 56**  
**Corrosion Test in Rotary Phosphate Salt Drier**

Field spool test. Specimens suspended in spray of liquor. Temperature: 45-75°F (7-24°C), average 60°F (16°C).

Duration: 59 days:

Disodium dihydrogen pyrophosphate .....	38.6 days
Disodium monohydrogen orthophosphate .....	7.3 days
Monosodium dihydrogen orthophosphate .....	5.7 days
Tripotassium orthophosphate .....	4.3 days
Tetrapotassium pyrophosphate .....	1.8 days
Monopotassium dihydrogen orthophosphate ....	1.0 day

Aeration present: agitation strong.

No pitting.

Material	Corrosion Rate, mils per year
Stainless Type 317	<0.1
Stainless Type 316	<0.1
Stainless Type 304	<0.1
INCONEL alloy 600	<0.1
MONEL alloy 400	2.5
Nickel 200	9.4
Silicon bronze	14
Carbon steel	227

**Table 57**  
**Field Tests in Pickling Solutions**

Spools submerged in pickling tanks.

Test 1: 40% phosphoric acid, 5% sulfuric acid, 0.25% wetting agent, 0.05% inhibitor.

Temperature: 190-205°F (88-96°C), average 200°F (93°C).

Duration: 62 days. No aeration. agitation only by addition or removal of product.

Test 2: 26% phosphoric acid with a wetting agent.

Temperature: 180-200°F (82-93°C). average 195°F (91°C).

Duration: 12.2 and 61 days, no aeration. some agitation.

Material	Corrosion Rate, mils per year		
	Test 1	Test 2	Test 3
Stainless Type 304	<0.1 (a)	0.1	<0.1
Stainless Type 316	0.1 (a)	0.1	0.1
Stainless Type 317	0.2	0.1	0.1
CARPENTER Stainless No. 20Cb	0.4	0.4	0.2
HASTELLOY alloy C	1.3	2.5	1.0
Lead, chemical	14	>518 (d)	—
INCONEL alloy 600	50	>454 (d)	—
MONEL alloy 400	69	205	—
HASTELLOY alloy B	>93 (d)	306	—
Aluminum bronze (10%)	115	315	—

(a) Severe Intergranular corrosion at welds of Types 304 and 316 has been reported in the plant: Types 304L and 316L are required

(d) Destroyed

**Table 58**  
**Laboratory Test in Paint Stripper Solutions**

Laboratory spool exposure in paint stripping solution containing strong phosphoric acid and weaker chromic acid, proportions unspecified. Immersion in 3 gallons solution in tank.

Temperature: 180-220°F (82-105°C), average 220°F (105°C).

Duration: 58 days. Aeration present, agitation by convection.

Material	Corrosion Rate, mils per year
Silicon Iron (14.5%)	0.1
WORTHITE	5.4
HASTELLOY alloy C	12 (si)
Brass, yellow	54 (perf)
Lead, chemical	60 (c) 4
Stainless Type 310	106 (i)
Lead, antimonial	178
HASTELLOY alloy F	>93 (d)
INCOLOY alloy 825	>93 (d)

(c) Crevice corrosion. max depth in mils

(d) Destroyed

(i) Severe Intergranular corrosion with grain-dropping

(perf) Perforated

(si) Shallow Intergranular corrosion with grain-dropping

**Table 59**

**Field Tests in Phosphate-Depositing Solutions**

Exposure in proprietary BONDERITE accelerated zinc phosphate steel-treating solutions. Specimens located in Immersion coating tank.

Test 1: Temperature: 150-160°F (66-71°C).

Duration: 33 days; aeration and agitation considerable.

Tests 2 and 3: Temperature: not reported but hot.

Duration: 30 days. Aeration and agitation not specified.

Material	Corrosion Rate, mils per year		
	Test 1	Test 2	Test 3
HASTELLOY alloy C	<0.1	—	—
Stainless Type 316	0.1	<0.1	<0.1
Stainless Types 302, 304	0.6 (c) (perf)	<0.1	<0.1
Stainless Type 430	—	<0.1	<0.1
Stainless Type 410	—	0.4 (c) 6	0.4 (c) 18
INCONEL alloy 600	1.1 (c) (perf)	—	—
Stainless Type 502	—	16	20
H.S.L.A. (a) steel (B)	—	154	15 (p) 19
H.S.L.A. steel (D)	—	186	22
H.S.L.A. steel (E)	—	186	26
Carbon steel	46 (p) 27	165	44
MONEL alloy 400	77	—	—
Cast iron	77	—	—
Nickel 200	93	—	—
Ni-Resist Type 1	100 (p) 20	—	—

(a) High-strength low-alloy

(B) Composition (nominal) 2 Ni, 1 Cu

(c) Crevice corrosion. max depth in mils

(D) Composition 0.5 Cu, 0.4 Cr, 0.4 Ni, 0.08 P

(E) Composition (nominal) 0.9 Cr, 0.9 Si, 0.4 Cu, 0.14 P

(p) Pitting, max depth in mils

(perf) Perforated

## Materials of Construction for Handling Phosphate Salts

Phosphate salts constitute the major use for electric furnace phosphoric acid. These include sodium and potassium orthophosphate and polyphosphates; the most important by far is sodium triphosphate,  $\text{Na}_3\text{P}_3\text{O}_{10}$ . It has been stated<sup>31</sup> that the reactors for producing phosphates by reacting with soda ash are preferably constructed of Type 316 stainless steel. Drying and dehydration equipment can be fabricated from Type 304 stainless steel where resistance to wet corrosion is required.

Data obtained in exposure in the solution fed to a phosphate salt drier are shown in Table 56.

## Materials of Construction for Handling Metal Treating Solutions

Phosphoric acid finds several applications in the pickling and electropolishing of metals<sup>38</sup>. Acid phosphate solutions are used to apply a phosphate film to ferrous metals. Tables 57 and 58 present results of corrosion tests in sheet steel pickling and in paint stripping equipment. Tables 59 and 60 contain data on corrosion behavior of various solutions used to apply phosphate coatings to steel.

**Table 60**

### Field Test in Phosphate-Depositing Solutions

Exposure in proprietary PARCO phosphate-film-depositing solutions. Specimens located in tank.

Temperature: not reported but hot.

Duration: 30 days; aeration and agitation unspecified.

Test 1: PARCO PARKERIZING Compound

Test 2: PARCO LUBRITE Solution

Material	Corrosion Rate, mils per year	
	Test 1	Test 2
Stainless Type 316	<0.1	0.1
Stainless Types 302, 304	0.1	0.1
Stainless Type 430	<0.1	0.2
Stainless Type 410	0.1	0.3
Stainless Type 502	12	96
H.S.L.A. (a) steel (B)	80	87
Carbon steel	78	139
H.S.L.A. steel (D)	155	148
H.S.L.A. steel (E)	198	148

(a) (B) (D) (E) See Table 59

The first four operating conditions included in these two tables provide corrosion-protective undercoatings; the last forms a coating intended to reduce wear on bearing surfaces. Table 61 contains results of an aluminum bright dip solution and Table 62 solutions used to process aluminum preliminary to painting.

**Table 61**

### Field Tests in Aluminum Bright Dip Solutions

Test 1: Environment: 81% phosphoric acid, 3% nitric acid in open tank, 18" below surface.

Temperature: 160-200°F (71-93°C), average 190°F (88°C).

Duration: 89 days; aeration moderate, no agitation.

Test 2: On top of burner tube in submerged combustion evaporator of solution recovery system, 85% phosphoric acid, 5% nitric acid.

Temperature: 210-300°F (99-149°C), average 255°F (124°C).

Duration: 20.3 days; aeration and agitation extensive.

Material	Corrosion Rate, mils per year	
	Test 1	Test 2
Stainless Type 310	0.7	-
INCOLOY alloy 804 (a)	-	3.0
ILLIUM "R"	-	3.3
NIMONIC alloy 90 (b)	-	4.0
HASTELLOY alloy F	1.1	-
CARPENTER Stainless No. 20Cb	1.1	5.4
Stainless Type 309 (c)	2.0	-
Stainless Type 317	2.4	-
INCOLOY alloy 825	3.2	-
Stainless Type 316, 316L	3.0 4.1	-
Stainless Type 316 sensitized	>99 (nd), (i)	-
Stainless Type 347	3.4	-
Stainless Type 304, 304L	4.1 5.0	9.0
Stainless Type 304 sensitized	>63 (d), (i)	-
Stainless Type 321 (e)	4.7	-
ILLIUM "98"	-	14
CORROHEL alloy 230	-	22
INCONEL alloy 600	32 (i)	-
HASTELLOY alloy C	58 (i)	76
Aluminum 1100	>78 (d)	-
MONEL alloy 400	>90 (d)	-
Nickel 200	> 100 (d)	-
HASTELLOY alloy B	-	>340 (d)

(a) See Table 27

(b) Composition 56 Ni, 20.3 Cr, 17.7 Co, 2.6 Ti, 1.6 Al

(c) See Table 20

(d) Destroyed

(e) Composition 70 Fe, 18 Cr, 10.5 Ni, 1 Mn, 0.4 Si, 0.08 max C, 0.5 Ti

(i) Intergranular corrosion

(nd) Nearly destroyed

**Table 62**  
**Field Tests in Aluminum-Treating Solutions**

Environment: proprietary aluminum surface-treating solutions containing a total of 7% acids: phosphoric, chromic, hydrofluoric. Specimens located in tanks.

Duration: 60 days.

Test 1: Room temperature: no information on aeration or agitation.

Test 2: Temperature range: 75-115°F (24-46°C), no aeration or agitation.

Material	Corrosion Rate, mils per year	
	Test 1	Test 2
Stainless Type 316	<0.1	<0.1
Stainless Type 304	<0.1	<0.1
INCONEL alloy 600	<0.1	0.1 (c) 6
INCOLOY alloy 800	<0.1	0.1 (c) 8
Nickel 200	<0.1	0.3
Stainless Type 430	<0.1	6
MONEL alloy 400	0.1	16
Stainless Type 410	<0.1	31

(c) Crevice corrosion, maximum depth in mils

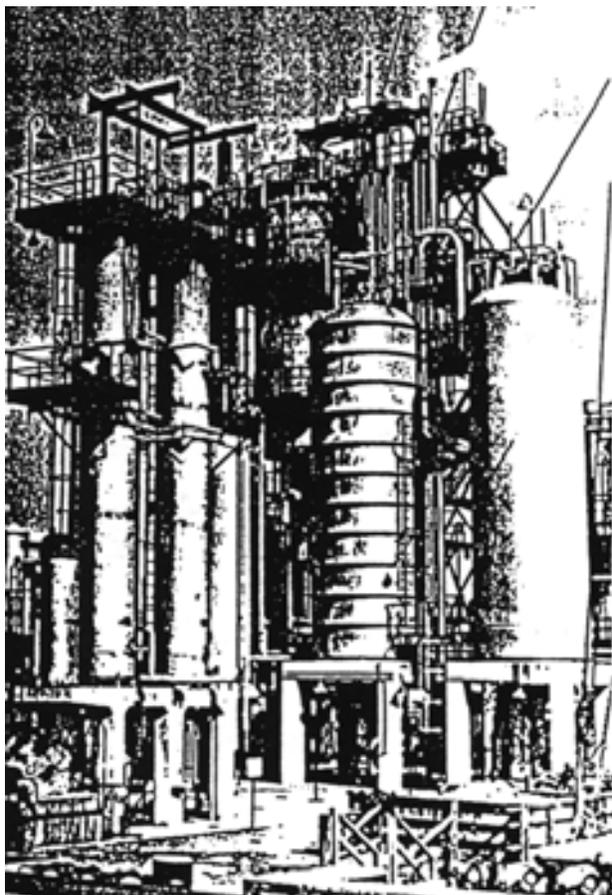


Fig. 25—Type 316L stainless-clad polymerization reactors using supported phosphoric acid catalyst.

*Courtesy of Lukens Steel Company*

## Materials of Construction for Phosphoric Acid-Catalyzed Reactions

Phosphoric acid has had use as a catalyst in organic chemical reactions. Nickel alloys, especially Type 316 stainless steel, find a place in this process, in which corrosive properties vary rather widely. In industrial practice concentrated acid is absorbed by kieselguhr, quartz chips, sand or copper pyrophosphate. The reacting stream passes through this bed of catalyst. Polymerization reactors clad with Type 316L stainless steel are shown in Fig. 25. An inhibitive effect of copper content in either the acid or the stainless steel was described by Ebling and Scheil<sup>10</sup>. Data are presented in several reactions: Table 63 in polymerization to increase the octane number of hydrocarbon fuels, Table 64 in hydrolysis, and Table 65 in an unidentified but severe organic environment.

**Table 63**  
**Corrosion Tests in Polymerization Processes**

Test 1: Plant test in butene polymerization unit. Specimens buried in sand washed with concentrated phosphoric acid.

Temperature: 300°F (149°C).

Duration: 338 days. No aeration, fluid hydrocarbon stream in motion.

Test 2: Plant test in effluent from phosphoric acid polymerization. Specimens located in inlet section of upper heat exchanger shell.

Temperature: 425°F (218°C).

Duration: 502 days. No aeration, fluid stream in motion.

Material	Corrosion Rate, mils per year	
	Test 1	Test 2
HASTELLOY alloy C	0.3	<0.1
HASTELLOY alloy F	—	0.1
CARPENTER Stainless No. 20Cb	—	0.2
HASTELLOY alloy B	—	0.3
Stainless Type 316	0.6	0.3(L)
Stainless Type 304	0.6	—
Stainless Type 310	—	0.4
HASTELLOY alloy D	0.9	—
INCONEL alloy 600	1.7 (p) 5	0.5
MONEL alloy 400	1.1	0.9
Nickel 200	1.7 (p) 9	—
Copper-nickel CA 715 (70/30)	—	2
Carbon steel	—	2.5 (p)16
Ni-Resist Type 1	6.2	—
Stainless Type 329	—	7

(L) Type 316L

(p) Pitting, maximum depth in mils

**Table 64**  
**Corrosion Test in Hydrolysis Reaction**

Plant spool test in hydrolysis of a nitrite using 85% phosphoric acid (62% P<sub>2</sub>O<sub>5</sub>) as catalyst. Specimens submerged in stillpot.

Average temperature: 320°F (160°C).

Duration: 3.0 days. No aeration, agitation present. Rates probably abnormally high because of short test duration.

Material	Corrosion Rate, mils per year
HASTELLOY alloy C	44
HASTELLOY alloy B	98
INCOLOY alloy 825	174
ALOYCO 20	178
HASTELLOY alloy F	180
CARPENTER Stainless No. 20Cb	192
WORTHITE	198
Stainless Type 317	464 (p) 3
Stainless Type 316	2190
Stainless Type 316 sensitized	>2480 (perf)

(p) Pitting, maximum depth in mils

(perf) Non-uniform general corrosion. no pitting, several perforations

**Table 65**  
**Corrosion Test in Organic Reaction**

Plant spool test in unidentified organic reaction with 0.5% phosphoric acid type catalyst containing amine derivatives. Specimens located in reactor on turbulent side of baffle, opposite bottom turbine.

Temperature: 70-610°F (21-320°C), average 525°F (275°C).

Duration: 14 days except where otherwise specified. No aeration; agitation violent.

Considerable disagreement shown between duplicate specimens.

Material	Corrosion Rate, mils per year
Stainless Type 329	5.1(t)
HASTELLOY alloy B	16
Stainless Type 304	17
Stainless Types 316, 316L (a)	18 20
HASTELLOY alloy C	25 (t)
CARPENTER Stainless No. 20Cb	32
HASTELLOY alloy F	43
INCOLOY alloy 825	46
MONEL alloy 400	59
INCOLOY alloy 800	73
INCOLOY alloy 804 (b)	125 (t)
INCONEL alloy 600	126

(a) Type 316L underwent serious pitting in 4½ months as the hemispherical bottom head and severe erosion as the impeller

(b) See Table 27

(t) Duration ten days

## Materials of Construction for Chemical Media Acidified with Phosphoric Acid

Phosphoric acid is used in chemical industry as an acidifier in pH control. Since it is neither a strong oxidant nor reductant, it is comparatively stable, and it does not have the charring effect of strong sulfuric acid. Table 66 provides the results of corrosion tests in acidification processes employing hot mixtures of phosphoric acid. The disagreement between the results in Tests 1a and 1b reflects rather subtle differences in exposure conditions, and illustrates the desirability of basing material selection on data obtained directly under actual operating conditions.

**Table 66**  
**Plant Corrosion Tests in Miscellaneous Acidic Environments**

Test 1: Dehydrated phosphoric acid and acrylic acid, proportions unspecified. Location unspecified.

Average temperature: 325°F (163°C).

Duration: 21 days. Aeration not specified, agitation present.

Test 2: 60% phosphoric acid, 15% sodium dihydrogen phosphate, 15% methyl or ethyl acrylate, 5% methanol, 5% unspecified organic compound. Location unspecified.

Average temperature: 293°F (145°C).

Duration: 69 days. No aeration, slight agitation.

Test 3: 80% phosphoric acid, 19% acrylic acid, 1% unspecified organic compound. Location unspecified.

Temperature: 311-329°F (155-165°C).

Duration: 61 days. Aeration and rapid agitation present.

Material	Corrosion Rate, mils per year			
	Test 1a	Test 1b	Test 2	Test 3
HASTELLOY alloy C	5	1.3	9	0.5, 5
HASTELLOY alloy B	9	6	—	—
CARPENTER Stainless No. 20Cb	18	11	9	44
Stainless Type 317	—	—	12	50
Stainless Type 316	25	13	21	56
MONEL alloy 400	62 (p)	14	—	20 (e)
Stainless Type 347	59	18	—	—
Stainless Type 304	—	42	—	—
Copper	93	45	—	—
Admiralty brass, arsenical	—	—	72	>270 (a)

(a) Almost destroyed

(e) Spotty erosion-corrosion and edge attack

(p) Pitted on edges

## REFERENCES

1. Yates, L. D., Corrosion Tests of Metals and Ceramics, Tennessee Valley Authority Chemical Engineering Report No. 9, 1951, Wilson Dam, Ala., p. 14.
2. Renshaw, W. G., & Lula, R. A., "The Corrosion Properties of Chromium-Nickel-Manganese Austenitic Stainless Steels," *Proceedings, Am. Soc. Testing Materials*, 52, 1956, p. 866.
3. Groves, N. D., Eisenbrown, C. M., & Scharfstein, L. R., "Corrosion by Weak Acids under Heat Transfer Conditions," *Corrosion*, 17, 1961, p. 173t.
4. Warren, D., "Corrosion and Weldability Studies on Chromium-Manganese Austenitic Stainless Steels," *Corrosion*, 16, 1960, p. 119t.
5. International Nickel Company, published and unpublished data.
6. Morgan, A. R., Jr., "Corrosion of Types 316 and 317 Stainless Steel by 75 and 85 Percent Phosphoric Acid," *Corrosion*, 15, 1959, p. 351.
7. Bünger, J., "Die Korrosion durch Phosphorsäure bei legierten Stählen und Nickellegierungen," *Werkstoffe and Korrosion*, 6, 1955, p. 369.
8. Fisher, A. O., "Corrosion by Furnace Grade Phosphoric Acid," *Materials Protection*, 5, No. 3, 1966, p. 29.
9. Kemp, J. D., and Zeh, H. P., U.S.P. 2,653,177, Sept. 22, 1953.
10. Ebling, H. F., & Scheil, M. A., "A Standard Laboratory Corrosion Test for Metals in Phosphoric Acid Service," *Trans. Am. Soc. Mech. Engrs.*, 73, 1951, p. 975.
11. Streicher, M. A., "Corrosion of Stainless Steels in Boiling Acids and its Suppression by Ferric Salts," *Corrosion*, 14, 1958, p. 59t.
12. Lowe, J. B., "Corrosion-Generated Hydrogen Explodes in Phosphoric Acid Tank," *Corrosion*, 17, No. 3, 1961, p. 30.
13. Bishop, C. R., "Corrosion Tests at Elevated Temperatures and Pressures," *Corrosion*, 19, 1963, p. 308t.
14. Private communication, Materials of Construction Group, Research and Development Department, Union Carbide Corporation, Chemicals Division.
15. Pelitti, Enrico, "Corrosion: Materials of Construction for Fertilizer Plants and Phosphoric Acid Service," in Sauchelli, V., "Chemistry and Technology of Fertilizers," ACS Monograph 148, Reinhold, 1960, New York, p. 588.
16. Leonard, R. B., "Developing a New Alloy," *Chemical Engineering*, 71, No. 22, Oct. 26, 1964, p. 150.
17. "Corrosion Resistance of Haynes Alloys," Stellite Division, Union Carbide Corporation, Kokomo, Indiana, September 1961.
18. Piehl, R. L., "Inhibition of HASTELLOY B Corrosion," U.S.P. 2,854,497, Sept. 30, 1958.
19. Claus, W., and Herrmann, L., "Zur Kenntnis der chemischen Beständigkeit der Nickel-Kupfer-Legierungen," *Z. für Metallkunde*, 31, No. 2, 1939, p. 55.
20. Durgin, C. B., Lum, J. H., and Malowan, J. E., "The Chemical and Physical Properties of Strong Phosphoric Acids," *Trans. Am. Inst. Chem. Engrs.*, 33, No. 4, 1937, p. 643.
21. Searle, H. E., and LaQue, F. L., "Corrosion Testing Methods," *Proceedings, Am. Soc. Testing Materials*, 35, 1935, p. 249.
22. Am. Soc. Testing Material Standards, Part 3, 1964, p. 173.
23. Weber, W. C., and Pratt, C. J., "Wet-Process Phosphoric Acid Manufacture," in Sauchelli, V., "Chemistry and Technology of Fertilizers," ACS Monograph 148, Reinhold, New York, 1960, Chapter 9.
24. Baniel, I., Blumberg, R., Alon, A., El-Roy, M., and Goniadski, D., "The I.M.I. Phosphoric Acid Process," *Chem. Eng. Progress*, 58, 1962, No. 11, p. 100.
25. VanWazer, J. R., Encyclopedia of Chemical Technology, Kirk, R. E., and Othmer, D. F., editors, Interscience Encyclopedia, New York, 1953, 10, pp. 446 and 424.
26. Bixler, G. H., Work, J., & Lattig, R. M., "Elemental Phosphorus -Electric Furnace Production," *Industrial & Engineering Chemistry*, 48, 1956, p. 2.
27. Marks, E. C., & Wilson, L. S., "Operation of a 25,000 kw Rotating Phosphorus Furnace," *Journal of Metals*, 17, 1965, p. 306.
28. Barber, J. C., "Corrosion Problems in the Manufacture of Phosphoric Acid from Elemental Phosphorus," *Corrosion*, 14, 1958, p. 357t.
29. Tice, E. A., "Intergranular Corrosion of Chromium-Nickel Stainless Steel - Progress Report No. 1," Bulletin 93, Welding Research Council, New York, 1964, p. 16.
30. Morgan, A. R., Jr., "The Corrosion of Types 316 and 317 Stainless Steel by 75% and 85% Phosphoric Acid," *Corrosion*, 15, 1959, p. 351t.
31. Private communication.
32. Lutz, W. A., and Pratt, C. J., "Manufacture of Triple Superphosphate," in Sauchelli, "Chemistry and Technology of Fertilizers," ACS Monograph 148, Reinhold, New York, 1960, Chapter 8.
33. Chapman, C., "The Utilisation of Austenitic Iron in the New Zealand Fertiliser Industry," paper delivered at Symposium on Austenitic Cast Iron, Melbourne, 1963.
34. Lutz, W. A., and Pratt, C. J., "Manufacture of Concentrated Water-Soluble Fertilizers Based on Ammonium Phosphate," in Sauchelli, "Chemistry and Technology of Fertilizers," ACS Monograph 148, Reinhold, New York, 1960, Chapter 12.
35. Rozian, I. W., "Diammonium Phosphate as Produced at By-Product Coke-Oven Plants," in Sauchelli, "Chemistry and Technology of Fertilizers," ACS Monograph 148, Reinhold, New York, 1960, Chapter 10.
36. Lutz, W. A., and Pratt, C. J., "Miscellaneous Phosphate Fertilizers," in Sauchelli, "Chemistry and Technology of Fertilizers," ACS Monograph 148, Reinhold, New York, 1960, Chapter 13.
37. Vreeland, D. C., and Kalin, S. H., "Corrosion of Metals by Liquid Fertilizer Solutions," *Corrosion*, 12, 1956, p. 569t.
38. Gurklis, J. A., McGraw, L. D., and Faust, C. L., "Electropolishing and Chemical Polishing of High-Strength High-Temperature Metals and Alloys," Defense Materials Information Center Memorandum 98, P.B. 161248, April 12, 1961, Columbus, O.