

bodies, which are not sugars and which may well be present, are included under this heading.

For the isolation of the reducing sugar 5 liters of a fermentation, culture 1-5, were filtered, cleared with basic lead acetate, delead with hydrogen sulfide, and concentrated to a thick sirup under reduced pressure. The osazone was then prepared, purified by successive recrystallizations from pyridine, and its melting point determined. The nitrogen content was also determined by the Dumas combustion method. From these values the sugar was found to be glucose.

ANALYSIS

At 735.7 mm. and 24° C. 0.1014 gram substance gave 14.6 cc. nitrogen and 0.1023 gram substance gave 14.8 cc. nitrogen. Calculated for $C_{15}H_{22}O_4N_4$, 15.64; found, 15.59 per cent. Melting point of glucosazone, 204.5° C.; found, 203.5° C.

Cellobiose could not be demonstrated as there was no soluble osazone, and hydrolysis of the sirup with 7 per cent hydrochloric acid yielded no increase in reducing power. The fact that no cellobiose could be demonstrated might be explained on the basis of the temperatures used. At 55° C. cellobiase

is active, whereas at the temperatures used by Pringsheim only cellulase is active.

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Resistance of Metals to the System Urea-Water-Ammonium Carbamate¹

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WHEN a study of the synthetic urea process (1) was undertaken at this laboratory, it soon became evident that the reacting system would attack the materials used in ordinary plant construction and result in serious operating difficulties. This was especially true for such parts of the apparatus as the discharge valve of the autoclave, which had to be closely machined in order to function properly. This valve was subjected, not only to both corrosion and erosion by the molten reaction mixture consisting of urea, water, and ammonium carbamate, but also to a pressure of 100 atmospheres and a temperature of about 150° C. It was thus apparent that the requirements for the metal used in the apparatus were most exacting. The metal must be resistant to the corrosion and erosion of the melt; it must be capable of being machined; and it must be strong and tough enough to withstand the pressures and temperatures used. This paper will describe the results of an investigation to determine which commercial corrosion-resistant alloys would best meet the exacting demands of actual working conditions.

Thirty-six different metal samples were studied in two tests, one of 4 days and another of 7 days duration. In each case, the samples were subjected to the corrosive action of the molten reaction mixture under pressure and at 142° C. The first test was designed to include a large number of samples whose compositions made them appear suitable. The heat treatment which these samples received in their manufacture was not known. The second and longer run tested the best samples from the 4-day run and also a few metals such as silver, lead, nickel, and tin, which were known to be fairly resistant to corrosion.

Method of Conducting Test

The metal samples tested were in most cases prepared by grinding on an emery wheel so that their total surface approxi-

mated 10 sq. cm. A small hole was drilled in each piece and the samples were weighed and strung on a cotton cord to support them in the molten bath. Knots in the cord prevented contact between specimens. The two ends of the cord were tied to glass supports, the cord forming a loop reaching almost to the bottom of the bath, as shown in Figure 1. The reaction vessel consisted of an 18-inch (46-cm.) tin-lined length of 6-inch (15-cm.) "double extra strong pipe," A, closed at each end by means of a tin-lined heavy steel blind flange, which construction was considered suitable to withstand corrosion by the melt and pressures of 50 to 75 atmospheres, known to be developed by the system in the range 140° to 150° C.

The reaction vessel was heated by boiling xylene in an electrically heated boiler, B, the vapors passing up through the annular space, C, and into a water-cooled condenser, D, from which the condensate returned to the boiler by means of the pipes E and F. The condensate from the vapor bath returned through G and F to the boiler. Thermometers at H

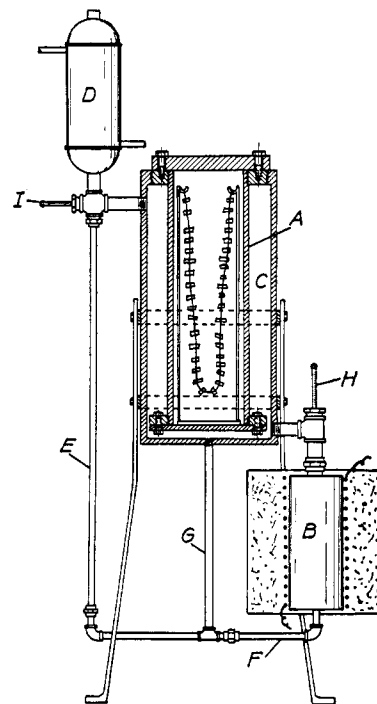


Figure 1—Apparatus for Testing Resistance of Metals to System Urea-Water-Ammonium Carbamate

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Table I—Resistance of Metals to the System Urea-Water-Ammonium Carbamate at 142° C. under Pressure

SAMPLE	NON-FERROUS COMPOSITION					4-DAY TEST Loss <i>Mg./sq. cm./day</i>	7-DAY TEST		
	Carbon	Chromium	Nickel	Silicon	Other elements		Loss <i>Mg./sq. cm./day</i>	Penetration per year <i>Inches</i> <i>Mm.</i>	
1 (Duriron)	14.5		0.024	0.012	0.00021	0.00533
2 (Silver)	Ag 99.9	..	0.026	0.00035	0.00889
3 (Illum G)	0.5	21.5	58.5	1.4	Cu, 8; Mo, 3; W, 2	0.069	0.066	0.0012	0.0305
4 (Chromium)	0.14 (gain)	0.084	0.0017	0.0432
5 (Lead)	0.11	0.0014	0.0356
6 (Nickel strip)	0.15	0.0025	0.0635
7 (Pioneer)	..	20	38	4	Mo, 3; some C, Cu, Mn	0.029	0.17	0.0031	0.0787
8 (Corrosiron)	12	..	0.68	0.175	0.0037	0.0940
9 (Rezistal 4)	0.23	18	22.5	2.9	..	0.90	0.95	0.017	0.432
10 (Stellite)	2 (approx.)	15-33	Co, 40-75; W, 10-17	..	1.1	0.019	0.483
11 (Invar)	36	1.49	0.027	0.686
12 (Cimet 1)	0.7	29	1	0.77	36	0.87	22.10
13 (Cimet 2)	1	20	60	1.4	..	0.97	0.10 (gain)
14 (Rezistal 5)	0.31	11	36	2	..	1.4
15 (Hybnickel S)	0.15	17	21	1.7
16 (Cyclops 17-B)	0.20	6.5	2	1	..	1.7
17 (Block tin)	2.68	0.053	1.346
18 (Ferro-Molyb. A 111)	Mo, 5.75	..	57	1.07	27.18
19 (Delhi cast)	0.45	17	3	4.4
20 (Bethlehem chrome steel)	0.35	15.5	0.3	4.5-6.9 ^a
21 (Silchrome)	..	18.5	0.4	3.0	W, 3.5	5.7
22 (Delhi rolled)	0.09	20.0	6.5
23 (Enduro A)	0.09	17.0	0.35	14.0
24 (Duraloy)	0.32	23.5	1.1	17.0
25 (Chrome iron)	0.13	14.8	V, 1.2	17.0
26 (Uniloy 2825)	0.30	28.3	19.0
27 (Chrome iron)	0.13	14.0	34.0
28 (Electrolytic iron)	0.02	0.01	..	43.0
29 (Chrome iron)	0.20	25.0	59
30 (Uniloy 1409)	0.09	12.4	0.5	84
31 (Vanadium alloy steel)	..	13.6	V, 0.5	97
32 (Rezistal 7)	0.9	5.5	0.3	101
33 (Carpaloy 1)	0.1	14.0	109
34 (Ascocol 33)	0.09	10.5	143
35 (Carpaloy 3)	0.27	20.0	V, 0.7	174
36 (Uniloy 1809)	0.10	18.8	0.5	179

^a Range of triplicate samples.

and *I* gave the temperature of the vapor. The boiler *B*, which was a 3.5-inch (8.9-cm.) standard pipe, 10 inches (25.4 cm.) long, with welded ends, was filled almost to the top with xylene. The electric heater, vapor pipe, and vapor bath were suitably lagged. It required about 7 amperes at 110 volts to heat the apparatus continuously.

The container was loaded with crystalline ammonium carbamate, which was carefully rammed in about the glass sample supports, to which the cord carrying the samples had been attached. The head was then replaced and heating started. After running continuously at 142° C., the boiling point of xylene, for 4 days, the bath was cooled, the pressure container opened, and the solid crystalline mass which had been partly converted to urea dissolved out by a stream of water. The metal samples were carefully washed with distilled water, dried, and weighed.

Those samples which had shown the best results in the 4-day test, together with a few additional samples, were included in the second test, which was conducted over a period of 7 days.

Results

After each run the carbamate-urea cake had a decided reddish color due to the solution of chromium and other constituents of the metallic samples. All the samples were visibly affected by the exposure, the effects ranging from a slight tarnishing of the surface to an almost complete disappearance of the sample. The results of the two tests are listed in Table I.

In the 4-day run triplicate samples of chrome steel, in different positions in the bomb, lost between 4.5 and 6.9 mg. per sq. cm. per day. This agreement is regarded as evidence of an ability to duplicate results, and practically eliminates the possibility of the rate of attack being influenced by the position of the sample in the bomb. In this run approximately half the samples showed either excessive corrosion or pitting. Because of the excessive corrosion

and the possible influence of some of the products of corrosion upon the other samples, it was not considered worth while to calculate the results of this test in terms of penetration per year. The more promising metals were therefore more carefully studied in a second test.

The 7-day test may be assumed to indicate the results which would be obtained in actual service. Of the selected metals included in this test, eight show less than 0.01 inch (0.25 mm.) penetration per year, with three others showing from 0.02 to 0.03 inch (0.5 to 0.76 mm.). There is no obvious connection between the composition of these samples and their resistance to this corrosion. This is especially true of the steel samples. It seems quite probable that heat treatment may have an important bearing on their corrosion resistance.

The eleven samples showing less than 0.03 inch (0.76 mm.) penetration per year cover a wide range of mechanical properties. Of the metals tested, Rezistal 4 (sample 9), an austenitic chrome-nickel steel, is probably the best all-round metal for the construction of bombs, heads, etc. Duriron and Corrosiron (samples 1 and 8, respectively) are resistant to corrosion, but are brittle and almost unmachinable. Stellite (sample 10) is very hard and is unmachinable, but is tough and might be used for valve parts, finished by grinding. Illum G and Pioneer (samples 3 and 7, respectively) are both promising, particularly for valve parts and fittings. Both are reasonably hard but are machinable. Both are special alloys and probably too expensive for bulky construction. Lining the interior of the bomb with silver, lead, or nickel would be preferable to a tin lining. Chromium plating would be especially applicable to moving valve parts or in places where extreme hardness is desired. Invar (sample 11), a high-nickel steel, has rather a high rate of penetration.

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