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METALS AND CERAMICS DIVISION

CORROSION COUPON STUDIES AT COAL LIQUEFACTION PILOT PLANTS

J. R. Keiser, V. B. Baylor, M. Howell, and J. F. Newsome

NOTICE: This document contains information of a preliminary nature. It is subject to revision or correction and therefore does not represent a final report.

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CONTENTS

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J. R. Keiser, V. B. Baylor, M. Howell, and J. F. Newsome

ABSTRACT

As part of the Fossil Energy Materials Program at Oak Ridge National Laboratory, we have supplied corrosion coupons to coal liquefaction pilot plants for exposure in selected vessels. These vessels were chosen on the basis of previous corrosion experience, anticipated corrosion behavior (especially important when operating conditions were changed), accessibility, and availability. Alloys exposed were selected to give a series with a corrosion resistance ranging from less than to greater than that thought to be needed for each application. Corrosion rates calculated from weight changes of the exposed coupons provide information useful in selecting materials for coal liquefaction plants. The results presented are from coupons exposed in the Wilsonville, Alabama, and Fort Lewis, Washington, Solvent Refined Coal pilot plants; the Catlettsburg, Kentucky, H-Coal Pilot Plant; and the Baytown, Texas, Exxon Coal Liquefaction Pilot Plant.

INTRODUCTION

The development of direct coal liquefaction processes has been pursued with considerable enthusiasm and, not coincidentally, with significant government financial support during recent years. Pilot plants have been built for further evaluating liquefaction methods already tested on the scale of process development units. The Wilsonville, Alabama, and Fort Lewis, Washington, pilot plants began operation in 1974, and pilot plants in Baytown, Texas, and Catlettsburg, Kentucky, commenced operation in 1980. The process type, plant capacity, and other important characteristics for each plant are given in Table 1.

*Research performed for the U.S. Department of Energy, Oak Ridge Operations Solvent Refined Coal Projects Office and AR&TD Fossil Energy Materials Program (B&R AA 15 10 10 0, Work Breakdown Structure Element ORNL-3.4) under contract W-7405-eng-26 with Union Carbide Corporation.

Table 1. Characteristics of coal liquefaction pilot plants included in corrosion coupon studies

Location	Process	Capacity (tons/day)	Dates of operation
Wilsonville, Alabama	SRC-I[a]	6	January 1974 to present
Fort Lewis, Washington	SRC-II or SRC-I	50	October 1974 to July 1981
Baytown, Texas	EDS[b]	250	June 1980 to August 1982
Catlettsburg, Kentucky	H-Coal	600	May 1980 to November 1982

[a]SRC, Solvent Refined Coal.

[b]EDS, Exxon Donor Solvent.

In the direct coal liquefaction processes used at these pilot plants, pulverized coal is slurried in a process-derived solvent. After hydrogen is added, the coal slurry is pumped to the preheater, leaves the preheater, and enters the dissolver (reactor vessel) with a temperature of 400 to 427°C (750–800°F) and a pressure of about 13.8 MPa (2000 psi) [20.7 MPa (3000 psi) in the H-Coal process]. The dissolver vessel effluent passes through a series of vapor-liquid separation steps and pressure letdown valves to provide separation of the gaseous, liquid, and solid components of the effluent. One or two fractionation towers are used to separate the liquid component into several fractions. Flowsheets for the pilot plants (Figs. 1–5) show the specific flow scheme used for each plant and identify the vessels that will be cited in our discussion of coupon exposure sites. Figure 1 shows the flow scheme for the Wilsonville pilot plant as it was until September 1980. Since that time, a hydrotreater unit (Fig. 2) was added to provide further hydrogenation of the heavier oil products. Figure 3 shows the Fort Lewis pilot plant when operation was terminated in the summer of 1981. Figure 4 shows the flow diagram of the Catlettsburg H-Coal Pilot Plant, and Fig. 5 provides the same information for the Baytown Exxon Pilot Plant.

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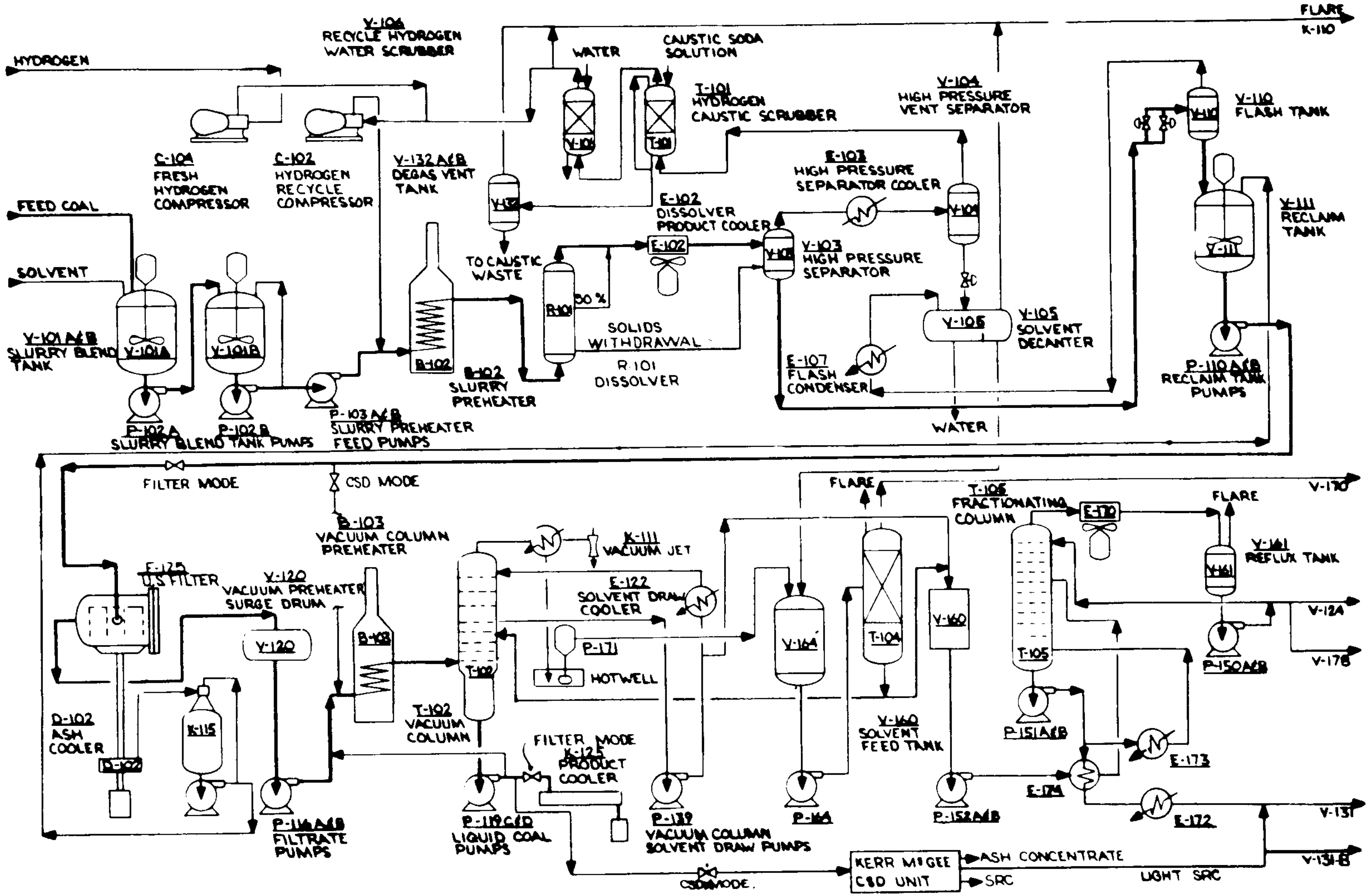

Fig. 1. Flowsheet for the Wilsonville Solvent Refined Coal direct liquefaction pilot plant. Source: Catalytic, Inc., Wilsonville, Ala.

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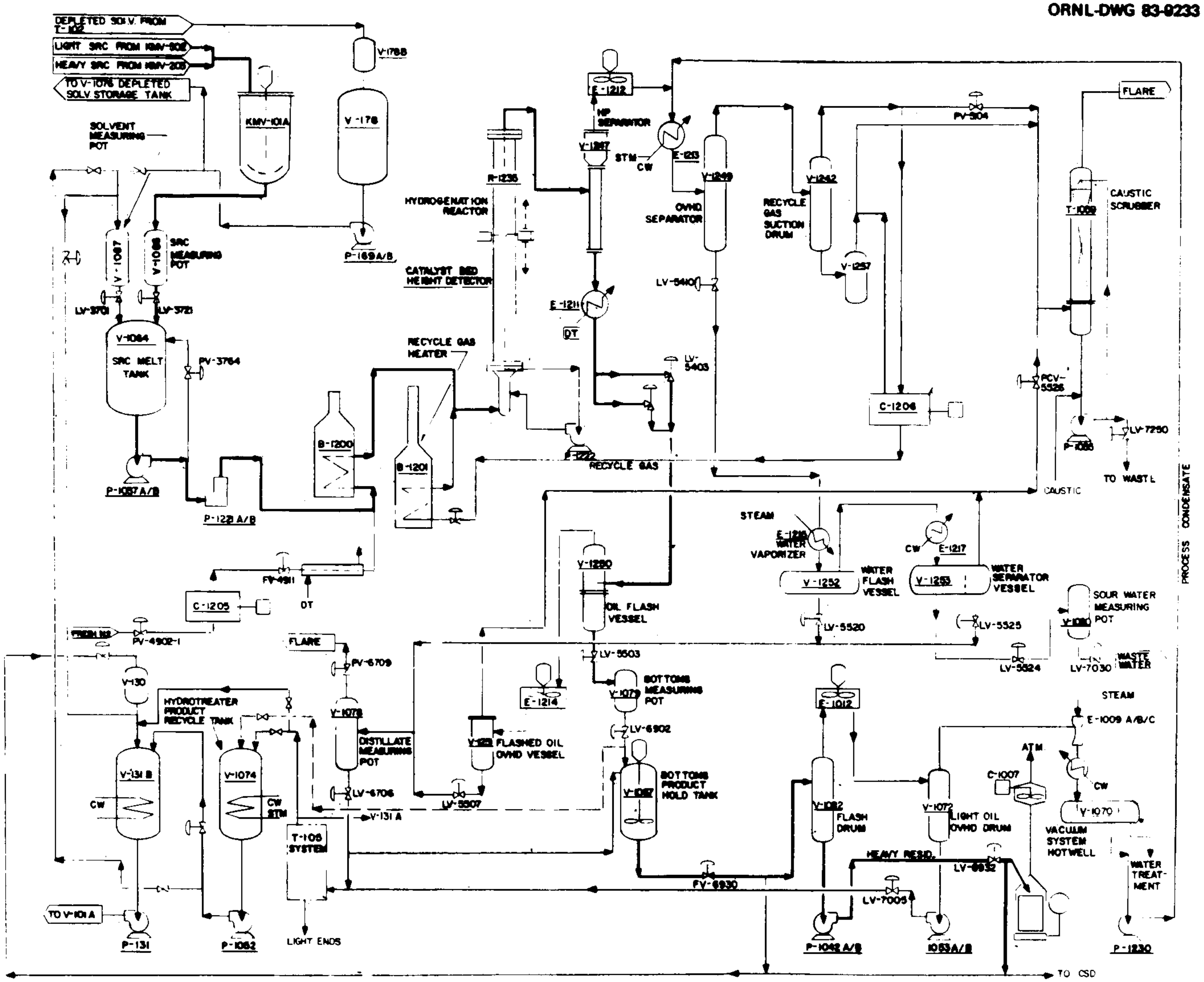

Fig. 2. Flowsheet for the hydrotreater unit at the Wilsonville coal liquefaction pilot plant. Source: Catalytic, Inc., Wilsonville, Ala.

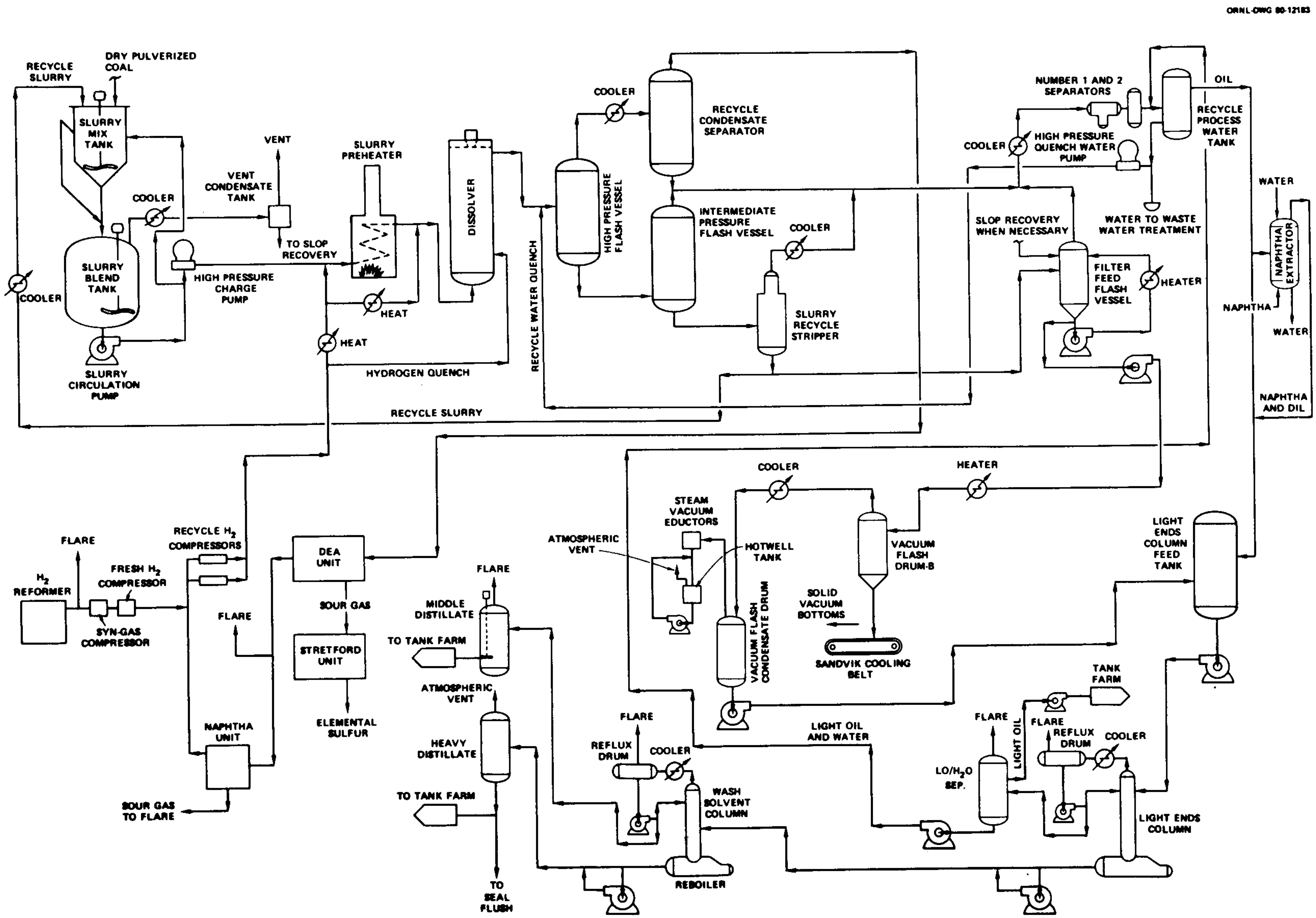

Fig. 3. Flowsheet for the Fort Lewis Solvent Refined Coal direct liquefaction pilot plant.

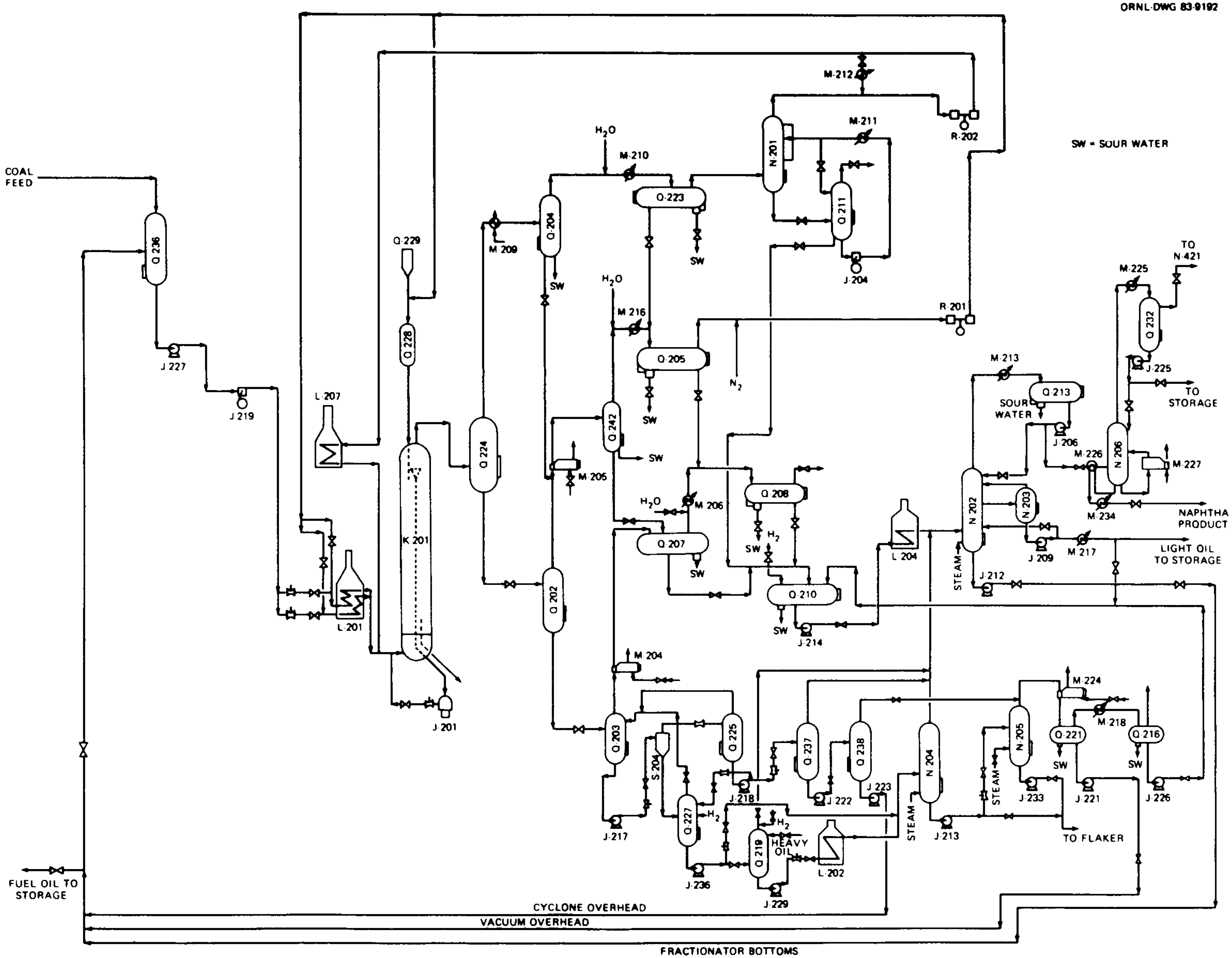

Fig. 4. Flowsheet for the Catlettsburg H-Coal direct liquefaction pilot plant.

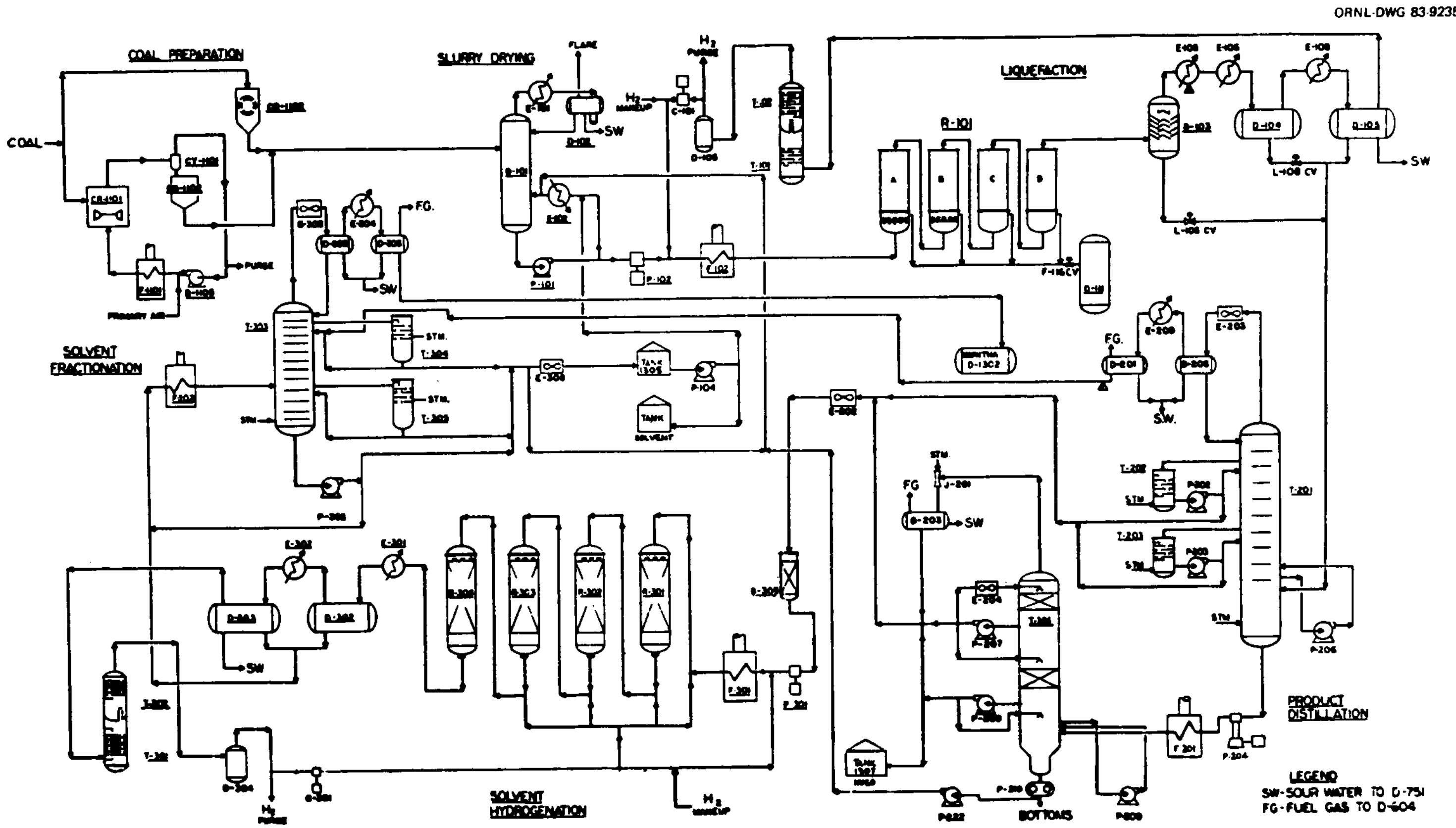

Fig. 5. Flowsheet for the Baytown Exxon Coal Liquefaction Pilot Plant. Source: Exxon Research and Engineering, Florham Park, N.J.

Methods used to monitor corrosion in these pilot plants included surveillance coupons, corrosion probes, and ultrasonic examination. Provision and examination of corrosion coupons was primarily the responsibility of an associated firm or contractor. For example, Gulf Science and Technology provided this service for the Fort Lewis pilot plant, and Catalytic, Inc., and Exxon Research and Engineering served the Wilsonville and Baytown plants, respectively. A contract between Ashland Synthetic Fuels, Inc., operators of the Catlettsburg pilot plant, and the Institute for Mining and Minerals Research (IMMR) provided for the latter to supply corrosion samples for that pilot plant. Since 1978 the Materials Compatibility group of the Oak Ridge National Laboratory (ORNL) Metals and Ceramics Division has supplemented, and in one case assumed total responsibility for, the supply of corrosion coupons to the pilot plants. The purpose of this report is to compile the results from the examinations of the ORNL-supplied corrosion coupons. This report does not present a thorough interpretation of the coupon results, nor does it address the stress corrosion cracking studies. It is intended only to collect the results of corrosion rate calculations for ORNL-supplied samples. Results of ORNL U-bend stress corrosion cracking studies have been reported.[1-2]

EXPERIMENTAL METHOD

Most of the corrosion coupons are squares that are about 50 mm on each side and range in thickness from 1 to 9 mm. In a few cases, smaller round coupons were used because of the restricted access ports through which they were inserted. The coupons were mounted on alumina spacers to provide electrical insulation and thus to prevent galvanic corrosion. Racks of the coupons were mounted either on a rod threaded on one end to permit screwing into a nut welded to the vessel or on a rod attached on each end to brackets that were fastened to the vessel. Compositions of alloys tested are given in Table 2. Examples of racks constructed for the two mounting methods are shown in Figs. 6 and 7.

Evaluation of exposed coupons typically consisted of cleaning and weighing and, on occasion, metallographic examination. Cleaning techniques

Table 2. Composition of materials used for corrosion coupons

Alloy	Content (wt %)												
	Fe	Ni	Cr	Mo	C	Si	Mn	P	S	Ti	Cu	Co	Others
Carbon steel[a]	Bal				0.28 max		0.90 max	0.035 max	0.040 max				
2 1/4 Cr-1 Mo[b]	Bal		2.40	1.01	0.09	0.35	0.39	0.009	0.009				
5 Cr-1 Mo (heat 5)[b]	Bal	0.16	5.18	1.02	0.10	0.14	0.32	0.008	0.008	0.02	0.03	0.015	Nb, 0.13
5 Cr-1 Mo (heat 6)[b]	Bal	0.06	5.01	1.01	0.087	0.11	0.34	0.008	0.009	0.01	0.04	0.016	Nb, 0.38
7 Cr-1 Mo (heat 7)[b]	Bal	0.05	7.05	0.98	0.089	0.17	0.34	0.007	0.007	0.02	0.04	0.019	Nb, 0.14
7 Cr-1 Mo (heat 8)[b]	Bal	0.05	7.00	1.02	0.088	0.14	0.34	0.008	0.008	0.02	0.04	0.019	Nb, 0.37
9 Cr-1 Mo[b]	Bal		9.03	0.98	0.070	0.60	0.47	0.015	0.005				
9 Cr-1 Mo modified[b]	Bal	0.07	8.81	0.94	0.10	0.30	0.38	0.009	0.009				Nb, 0.06
Type 409 SS[a]	Bal		10.5–11.75		0.08 max	1.0 max	1.0 max	0.045 max	0.045 max	6×C; 0.75 max			
Type 410 SS[a]	Bal		11.5–13.5		0.15 max	1.0 max	1.0 max	0.040 max	0.030 max				
18 Cr-2 Mo[a]	Bal		18–20	1.75–2.25	0.04 max (C+N)	1.0 max	1.0 max	0.04 max	0.03 max	5×(C+N) min			Nb, 9×(C+N) min
26 Cr-1 Mo[a]	Bal	0.50 max	25.0–27.5	0.75–1.50	0.010 max	0.40 max	0.40 max	0.02 max	0.02 max		0.20 max		Nb, 0.05–0.20; N, 0.015 max
26 Cr-1 Mo stabilized[a]	Bal	0.50 max	25.0–27.0	0.75–1.50	0.06 max	0.75 max	0.75 max			0.20–1.00; 7×(C+N) min	0.20 max		N, 0.04 max
29 Cr-4 Mo[a]	Bal	0.15 max	28.0–30.0	3.5–4.2	0.010 max	0.20 max	0.30 max	0.025 max	0.020 max		0.15 max		C+N, 0.025 max
SC-1[a]	Bal	2.5	26.0	3.0	0.025	0.30	0.30			0.50			N, 0.025
MONIT[a]	Bal	3.5–4.5	24.5–26.0	3.8–4.3	0.025 max	0.50 max	0.50 max			Ti+Nb 0.3–0.6			N, 0.025 max
Ferralium alloy 255[b]	Bal	5.65	25.81	3.16	0.020	0.32	0.77	0.021	0.005		1.74		N, 0.19
SAF 2205[b]	Bal	5.55	22.04	2.96	0.017	0.43	1.53	0.022	0.003				N, 0.14
Type 304 SS[a]	Bal	8–10.5	18–20		0.08 max	1.0 max	2.0 max	0.045 max	0.030 max				
Type 304L SS[a]	Bal	8–12	18–20		0.03 max	1.0 max	2.0 max	0.045 max	0.030 max				
Type 310S SS[a]	Bal	19–22	24–26		0.08 max	1.5 max	2.0 max	0.045 max	0.030 max				
Type 316 SS[a]	Bal	10–14	16–18	2.0–3.0	0.08 max	1.0 max	2.0 max	0.045 max	0.030 max				
Type 316L SS[a]	Bal	10–14	16–18	2.0–3.0	0.03 max	1.0 max	2.0 max	0.045 max	0.030 max				
Type 317 SS[a]	Bal	11–15	18–20	3.0–4.0	0.08 max	1.0 max	2.0 max	0.045 max	0.030 max				
Type 317L SS[a]	Bal	11–15	18–20	3.0–4.0	0.03 max	1.0 max	2.0 max	0.045 max	0.030 max				
Type 317LM SS[a]	Bal	12–16	18–20	4.0–4.5	0.03 max	1.0 max	2.0 max	0.045 max	0.030 max				
Type 321 SS[a]	Bal	9–12	17–19		0.08 max	1.0 max	2.0 max	0.045 max	0.030 max	5×C min			
Type 347 SS[a]	Bal	9–13	17–19		0.08 max	1.0 max	2.0 max	0.045 max	0.030 max				Nb+Ta, (10×C) min
Crutemp 25[a]	Bal	25.0	25.0		0.06	0.50	1.50	0.020	0.015				
Incoloy 800[a]	39.5 min	30.0–35.0	19.0–23.0		0.10 max	1.0 max	1.50 max		0.015 max	0.15–0.60	0.75 max		Al, 0.15–0.60
Incoloy 801[a]	Bal	30.0–34.0	19.0–22.0		0.10 max	1.0 max	1.5 max		0.015 max	0.75–1.50	0.5 max		

Table 2. (continued)

Alloy	Content (wt %)												
	Fe	Ni	Cr	Mo	C	Si	Mn	P	S	Ti	Cu	Co	Others
Incoloy 825[a]	22.0 min	38.0–46.0	19.5–23.5	2.5–3.5	0.05 max	0.5 max	1.0 max		0.03 max	0.6–1.2	1.5–3.0		Al, 0.2 max
Nitronic 50[a]	Bal	11.50–13.5	20.50–23.50	1.50–3.00	0.06 max	1.00 max	4.00–6.00	0.040 max	0.030 max				N, 0.20–0.40; Nb, 0.10–0.30; V, 0.10–0.30
Carpenter 20Cb-3[a]	Bal	32.50–35.00	19.00–21.00	2.00–3.00	0.06 max	1.00 max	2.00 max	0.035 max	0.035 max		3.00–4.00		Nb+Ta, (8×C) min; 1.00 max
Sandvik 2RE69[b]	Bal	21.98	25.50	2.18	0.014	0.14	1.68	0.010	0.005				N, 0.14
Haynes 20 modified[a]	Bal	25.0–27.0	21.0–23.0	4.0–6.0	0.05 max	1.0 max	2.5 max	0.04 max	0.03 max	4×C min			
Alloy 904L[a]	45.0	25.5	21.0	4.7	0.02						1.5		
RA 333[a]	Bal	44.00–47.00	24.00–27.00	2.50–4.00	0.08 max	0.75–1.50	2.00 max	0.030 max	0.030 max			2.50–4.00	W, 2.50–4.00
Crucible 6M[b]	Bal	24.81	20.43	6.18	0.016	0.036	1.72	0.004	0.012				
Monel 400[a]	2.5 max	Ni+Co 63.0 min			0.3 max	0.5 max	2.0 max		0.024 max		28.0–34.0		
Inconel 600[a]	6.00–10.00	72.0 min	14.0–17.0		0.15 max	0.50 max	1.00 max		0.015 max		0.50 max		
Inconel 601[a]	Bal	58.0–63.0	21.0–25.0		0.10 max	0.50 max	1.0 max		0.015 max		1.0 max		Al, 1.0–1.7
Inconel 625[a]	5.0 max	58.0 min	20.0–23.0	8.0–10.0	0.10 max	0.50 max	0.50 max	0.015 max	0.015 max	0.40 max		1.0 max	Al, 0.40 max; Nb+Ta, 3.15–4.15
Inconel 671[b]	0.32	52.92	46.02		0.05	0.21	0.07		0.21	0.41			
Inconel X-750[a]	5.00–9.00	70.00 min	14.0–17.0		0.08 max	0.50 max	1.00 max		0.010 max	2.25–2.75	0.50 max	1.0 max	
Udimet 720[a]		Bal	18.0	3.0	0.03					5.0		15.0	Al, 2.5; W, 1.5; B, 0.02
Haynes 263[b]	0.14	Bal	19.71	5.82	0.080	0.28	0.41	0.003	<0.002	2.15	<0.01	19.94	Al, 0.43
Hastelloy B-2[b]	1.45	Bal	0.18	28.24	<0.002	0.02	0.23	<0.005	<0.002			<0.10	
Hastelloy C-4[b]	0.54	Bal	15.44	16.19	0.005	0.02	0.14	<0.005	<0.002	0.24		<0.10	
Hastelloy C-276[a]	4.00–7.00	Bal	14.50–16.50	15.00–17.00	0.02 max	0.08 max	1.00 max	0.04 max	0.03 max			2.50 max	W, 3.00–4.50; V, 0.35 max
Hastelloy G[b]	18.89	Bal	21.80	6.19	0.008	0.30	1.38	0.010	<0.002		1.87	1.05	Nb+Ta, 2.14; W, 0.29
Hastelloy G-3[b]	18.26	Bal	22.84	6.98	0.008	0.37	0.82	0.014	<0.005		1.84	3.53	Nb+Ta, 0.25; W, 0.94
Hastelloy N[a]	5.00 max	Bal	6.00–8.00	15.00–18.00	0.04–0.08	1.00 max	0.8 max	0.015 max	0.020 max	Al+Ti, 0.50 max	0.35 max	0.20 max	W, 0.50 max; B, 0.010 max
Titanium (grade 2)[a]	0.30 max				0.10 max					Bal			H, 0.015 max; N, 0.03 max; O, 0.25 max
Nickel 200[a]	0.40 max	99.0 min			0.15 max	0.35 max	0.35 max		0.01 max		0.25 max		
Aluminum[a]							1.2				0.12		Al, Bal
Molybdenum[a]				Total									
Zirconium 702[a]	Fe+Cr, 0.20 max				0.05 max								Zr+Hf, 99.2 min; Hf, 4.5 max; H, 0.005 max; N, 0.025 max; O, 0.16 max

[a]Nominal composition.

[b]Composition from analysis.

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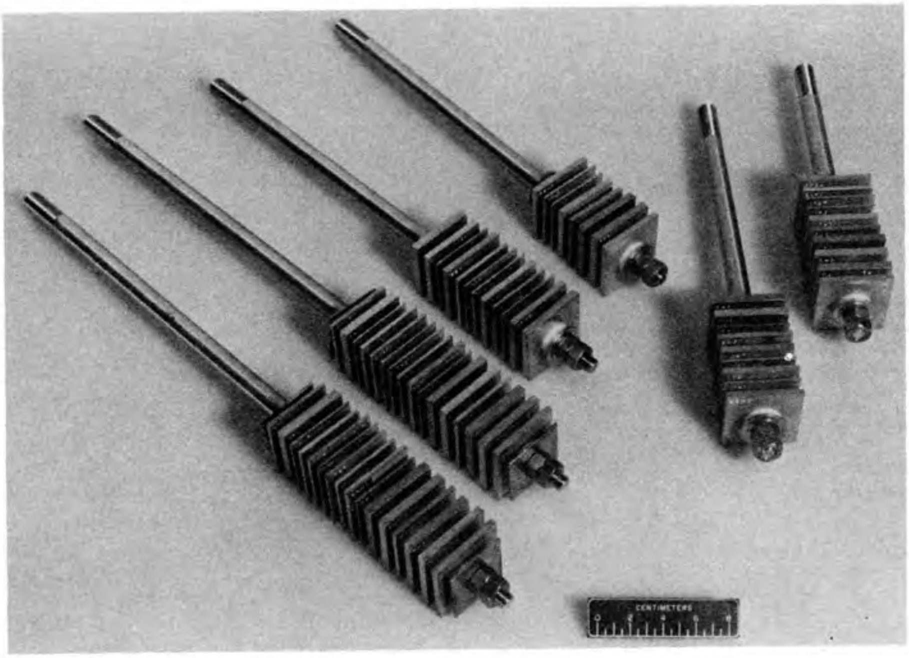

Fig. 6. Example of racks of corrosion coupons prepared for exposure at the Wilsonville Solvent Refined Coal Pilot Plant.

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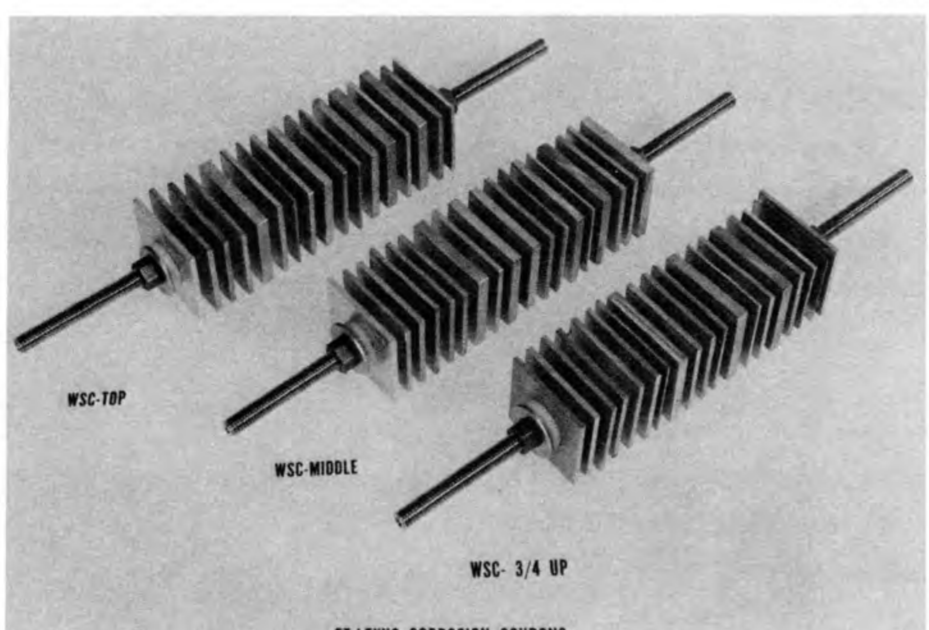

Fig. 7. Example of racks of corrosion coupons prepared for exposure in the Fort Lewis wash solvent column. Samples are 50.8-mm (2-in.) squares.

always included ultrasonic cleaning in either a detergent solution or an organic solvent. For the first few sets of samples returned, gentle wire brushing was used to remove adherent deposits or scale. Low-pressure sand blasting was used later to remove any adherent material. We determined by testing that the amount of material removed from an unexposed coupon by this sandblasting was negligible. In a few cases racks of coupons were examined during an on-site visit to the Wilsonville pilot plant; cleaning could be done only by using solvent, a wire brush, and rags.

Corrosion rates were calculated from measured weight changes by using the equation

$$\text{corrosion rate (mm/year)} = \frac{(\Delta W) \times 8766 \times 10}{(A) \times \rho \times t}$$

where

ΔW = measured weight change (g),
A = calculated initial exposed area of coupon (cm^2),
ρ = published value of alloy's density (g/cm^3),
t = exposure time as measured by plant engineer (h).

In making this calculation, we assumed that material was lost from the surface in a uniform manner and that the amount lost was small enough that sample dimension changes were insignificant. Deviations from these assumptions introduce errors into the calculations.

EXPERIMENTAL RESULTS

The results in this chapter are grouped by the pilot plant in which the coupons were exposed. Within the section for each pilot plant, results are reported by functional areas: fractionation area, separation area, and so forth.

WILSONVILLE, ALABAMA, SRC-I PILOT PLANT

The Solvent Refined Coal (SRC)-I pilot plant at Wilsonville was the first plant to which ORNL supplied coupons. Coupons were initially supplied for six vessels; subsequently, racks of coupons were added to other vessels as the need arose. The vessels in which coupons have been or are being exposed are listed in Table 3. Some racks of these coupons have never been removed for examination, but others have been checked on several occasions.

Table 3. Exposure sites of ORNL-supplied corrosion coupons at the Wilsonville Solvent Refined Coal Pilot Plant

Vessel	Identification	Rack location
T-105 T-105B	Fractionation column	In vapor at top manway cover flange In vapor at middle manway cover flange In vapor at lower manway cover flange
T-102	Vacuum column	In vapor above tray 1 In vapor below tray 8
V-103	High-pressure separator	In vapor
V-105	Solvent decanter	In oil
V-164	Light organics column feed tank	In oil
V-170	Light oil tank	In oil
V-178	T-105 overhead storage	In oil
V-11064	SRC melt tank	In oil
V-11074	Hydrotreater product recycle tank	In oil
R-1235	Hydrotreater reactor	In vapor

In the 20-tray T-105 fractionation column, coupons were exposed at the top manway (above tray 1), middle manway (between trays 10 and 11), and bottom manway (below tray 20), as shown in Fig. 8. The coupons exposed at the middle manway were examined in September 1979, those exposed at all three manways were examined in December 1979, and those from the three locations were checked in March 1980. Corrosion rates in Table 4 were calculated from weight change measurements from the first two examinations, and those in Table 5, from the third examination. Average

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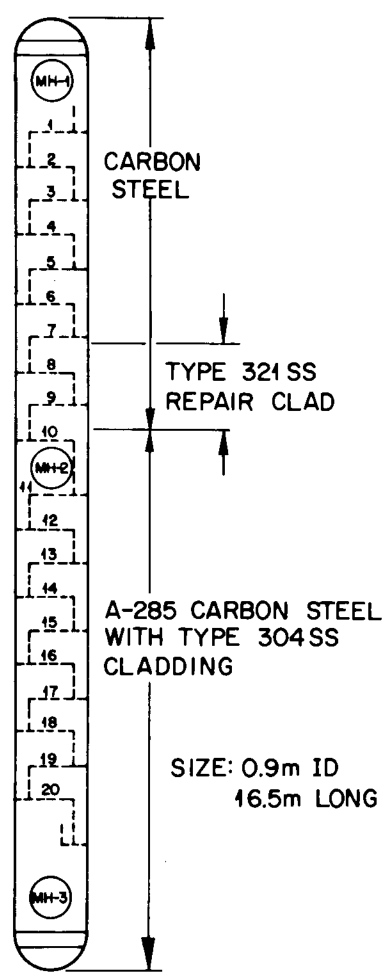

Fig. 8. Schematic of the Wilsonville T-105 fractionation column.

temperatures at the upper, middle, and lower manways were about 200, 232, and 288°C (392, 450, and 550°F), respectively. The results show that corrosion was far more severe at the middle manway than at the upper and lower manways. It is also apparent that nickel-base alloys were the most corrosion resistant and that the ferritic and martensitic steels were the least resistant.

Since July 1979, varying amounts of sodium carbonate were added to the feed coal to control corrosion. During periods of sodium carbonate addition, corrosion rates (as indicated by corrosion probes) were low.

Table 4. Corrosion rates of coupons exposed in the top, middle, and bottom manways of the Wilsonville fractionation column

Alloy	Corrosion rate[a] [mm/year (mils/year)]			
	Period 1[b]	Period 2[c]		
	Middle manway	Top manway	Middle manway	Bottom manway
Hastelloy C-276	<0.003 (<0.1)	<0.003 (<0.1)	0.003 (0.1)	*e*
Incoloy 825[d]			0.014 (0.6)	
Hastelloy G-3	0.044 (1.7)	<0.003 (<0.1)	0.018 (0.7)	*e*
Hastelloy G	0.042 (1.6)	<0.003 (<0.1)	0.020 (0.8)	*e*
Titanium	0.22 (8.8)	*e*	0.072 (2.8)	*e*
Haynes 20 modified	0.17 (6.6)	<0.003 (<0.1)	0.16 (6.4)	
Type 321 SS[d]			0.20 (7.7)	
Type 317LM SS[d]			0.34 (15)	
Type 317 SS	0.85 (34)	0.003 (0.1)	0.46 (18)	<0.003 (<0.1)
2RE69	0.56 (22)	*e*	0.52 (21)	0.08 (3)
Type 304 SS	1.23 (49)	<0.003 (<0.1)	0.70 (28)	<0.003 (<0.1)
Nitronic 50	Not weighed	<0.003 (<0.1)	0.73 (29)	0.012 (0.5)
Type 410 SS	2.02 (80)	*e*	1.06 (42)	0.64 (25)
MONIT	2.98 (117)	<0.003 (<0.1)	1.16 (46)	*e*
SC-1	4.11 (162)	*e*	*f*	*e*
Carbon steel	4.58 (180)	0.012 (0.5)	1.97 (77)	0.52 (20)
18 Cr-2 Mo	5.01 (197)	<0.003 (<0.1)	*f*	*e*
26 Cr-1 Mo stabilized	6.23 (245)	<0.003 (<0.1)	*f*	0.005 (0.2)

[a] Calculated from weight change, assuming uniform material removal.

[b] July to September 1979; 1128 h.

[c] July to December 1979; 3552 h.

[d] Specimen inserted on middle rack after 1128 h of operation; exposed 2424 h, September to December 1979.

[e] Weight gain, indicating formation of a tenacious film not removed by wire brushing and ultrasonic cleaning.

[f] Specimen removed from middle rack after 1128 h of operation.

Additions were usually about 1% of the total coal feed, but on a few occasions they were reduced or eliminated. This variation in carbonate addition resulted in significant effects on the corrosivity of the oils in the fractionation column. The weight losses for the coupons reported in Tables 4 and 5 almost certainly occurred during periods when sodium carbonate additions were reduced or eliminated, but the calculated corrosion rates are based on the total exposure time. Consequently, the rates shown in these tables are considerably lower than they would be if the calculations had reflected only exposure time to the corrosive oil. A more thorough analysis of the coupons exposed in the fractionation columns at the SRC pilot plants is given elsewhere.[3]

Table 5. Corrosion rates of coupons exposed from December 1979 to March 1980 in the top, middle, and bottom manways of the Wilsonville fractionation column

Manway	Alloy	Corrosion rate[a] [mm/year (mils/year)]
Top	Alloy 625	<0.003 (<0.1)
	Alloy 904L	<0.003 (<0.1)
	Alloy 825	<0.003 (<0.1)
	Titanium	<0.003 (<0.1)
	2RE69	<0.003 (<0.1)
	Type 321 SS	<0.003 (<0.1)
	SC-1	<0.003 (<0.1)
	Hastelloy C-276	<0.003 (<0.1)
	Hastelloy G	<0.003 (<0.1)
	26 Cr-1 Mo stabilized	<0.003 (<0.1)
	Haynes 20 modified	<0.003 (<0.1)
	Hastelloy G-3	<0.003 (<0.1)
	Nyby MONIT	<0.003 (<0.1)
	Type 304 SS	<0.003 (<0.1)
	18 Cr-2 Mo	<0.003 (<0.1)
	Type 317 SS	0.005 (0.2)
Middle	Hastelloy C-276	<0.003 (<0.1)
	Alloy 625	0.006 (0.2)
	Hastelloy G-3	0.007 (0.3)
	Hastelloy G	0.008 (0.3)
	Alloy 825	0.031 (1.2)
	Carpenter 20Cb-3	0.043 (1.7)
	Haynes 20 modified	0.043 (1.7)
	Monel 400	0.065 (2.5)
	Alloy 904L	0.066 (2.6)
	Type 321 SS	0.169 (6.7)
	Type 317 SS	0.275 (10.8)
	Type 317LM SS	0.297 (11.7)
	2RE69	0.319 (12.6)
	Type 304 SS	0.337 (13.3)
	Type 310 SS	0.454 (17.9)
Bottom	26 Cr-1 Mo	<0.003 (<0.1)
	18 Cr-2 Mo	<0.003 (<0.1)
	Type 317 SS	<0.003 (<0.1)
	Alloy 825	<0.003 (<0.1)
	SC-1	<0.003 (<0.1)
	Nyby MONIT	<0.003 (<0.1)
	Alloy 625	<0.003 (<0.1)
	Haynes 20 modified	<0.003 (<0.1)
	Hastelloy G-3	<0.003 (<0.1)
	Titanium	<0.003 (<0.1)
	Hastelloy G	<0.003 (<0.1)
	Hastelloy C-276	<0.003 (<0.1)
	Type 304 SS	0.006 (0.2)
	2RE69	0.131 (5.2)

[a]Calculated from weight change, assuming uniform material removal.

Racks of corrosion coupons were exposed at two locations in the T-102 vacuum column: below tray 8 (near bottom of column) and above tray 1 (near top of column). Exposure periods and nominal operating conditions for each period are given in Table 6, calculated corrosion rates for coupons exposed in the top of the column are given in Table 7, and rates for coupons exposed below tray 8 are given in Table 8. The data of Table 7 show that all materials exposed at the top of the T-102 vacuum column had acceptable corrosion rates. However, Table 8 shows that the corrosion rate for coupons exposed below tray 8 increased as the amount of chromium in the alloy decreased. Furthermore, carbon steel had an average rate of about 0.68 mm/year (27 mils/year), which is near the limit that can be tolerated.

Other coupons are exposed in the fractionation area in feed tanks or in product collection vessels. Racks of coupons are now being exposed in V-164, light organics column feed tank; in V-170, light oil tank; and in V-178, T-105 overhead storage tank. These coupons have not yet been removed for examination. However, the three tanks are operated at or near ambient temperature, and corrosion rates are expected to be low.

Two racks of coupons were exposed in the pressure letdown gas-liquid separation area. One rack was exposed in the V-103 high pressure separator, and another, in the V-105 solvent decanter. As shown in the coupon exposure

Table 6. Exposure periods and nominal operating conditions for Wilsonville T-102 vacuum column

Installed	Removed	Exposure (h)	Nominal conditions	
November 1979	May 1980	3366	Temperature, °C (°F)	
May 1980	November 1980	3456	Top	<95 (<200)
November 1980	March 1981[a]	1596	Tray 8	170–205 (340–400)
March 1981	September 1981	3526		
September 1981			Pressure, kPa (psia)	3.4 (0.5)

[a]Coupons in top of column not examined at end of this period.

Table 7. Corrosion rates of coupons exposed in the top of the Wilsonville T-102 vacuum column

Sample	Corrosion rate[a] [mm/year (mils/year)]		
	November 1979—May 1980 3366 h	May 1980—November 1980 3456 h	November 1980—September 1981 5122 h
Carbon steel	<0.003 (<0.1)	0.003 (0.1)	0.009 (0.4)
Type 410 SS	[b]	<0.003 (<0.1)	<0.003 (<0.1)
18 Cr-2 Mo	[b]	[b]	<0.003 (<0.1)
29 Cr-4 Mo	[b]	[b]	<0.003 (<0.1)
Type 304 SS	[b]	[b]	<0.003 (<0.1)
Type 321 SS	[b]	[b]	<0.003 (<0.1)
Type 347 SS	[b]	[b]	<0.003 (<0.1)
Type 316 SS	[b]	[b]	<0.003 (<0.1)
Type 317 SS	[b]	<0.003 (<0.1)	<0.003 (<0.1)
2RE69	[b]	0.0 (0.0)	<0.003 (<0.1)
Incoloy 800	[b]	[b]	<0.003 (<0.1)
Incoloy 825	[b]	[b]	<0.003 (<0.1)
Inconel 600	[b]	[b]	<0.003 (<0.1)
Hastelloy G	[b]	<0.003 (<0.1)	<0.003 (<0.1)
Hastelloy C-276	[b]	<0.003 (<0.1)	<0.003 (<0.1)
Titanium	[b]	[b]	<0.003 (<0.1)

[a]Corrosion rates calculated from weight change, assuming uniform removal of material.

[b]Weight gain indicating presence of a tightly adherent scale, which was not removed by wire brushing or ultrasonic cleaning in an organic solvent.

Table 8. Corrosion rates of coupons exposed below tray 8 of the Wilsonville T-102 vacuum tower

Sample	Corrosion rate[a] [mm/year (mils/year)]			
	November 1979—May 1980 3366 h	May 1980—November 1980 3456 h	November 1980—March 1981 1596 h	March 1981—September 1981 3526 h
Carbon steel	0.69 (27)	0.35 (14)	1.25 (49)	0.406 (16)
5 Cr-1 Mo (0.13 Nb)				0.035 (1.4)
5 Cr-1 Mo (0.38 Nb)				0.047 (1.9)
7 Cr-1 Mo (0.14 Nb)		0.46 (1.8)	0.111 (4.4)	0.025 (1.0)
7 Cr-1 Mo (0.37 Nb)				0.034 (1.3)
9 Cr-1 Mo		0.21 (0.8)	0.055 (2.1)	0.022 (0.9)
9 Cr-1 Mo modified				0.036 (1.4)
Type 409 SS				<0.003 (<0.1)
Type 410 SS	<0.003 (<0.1)	<0.003 (<0.1)	0.006 (0.2)	0.003 (0.1)
18 Cr-2 Mo	*b*	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
26 Cr-1 Mo stabilized				<0.003 (<0.1)
29 Cr-4 Mo	*b*	<0.003 (<0.1)	0.004 (0.2)	
Type 304 SS	*b*	<0.003 (<0.1)	<0.003 (<0.1)	
Aluminized 304 SS		0.004 (0.2)	0.012 (0.5)	
Type 304L SS				<0.003 (<0.1)
Type 321 SS	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Type 347 SS	*b*	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Type 316 SS	*b*			
Type 317 SS	*b*	<0.003 (<0.1)	<0.003 (<0.1)	
Crutemp 25		<0.003 (<0.1)	<0.003 (<0.1)	
2RE69	*b*	<0.003 (<0.1)	<0.003 (<0.1)	
Incoloy 800	*b*			
Incoloy 825	*b*	<0.003 (<0.1)	<0.003 (<0.1)	
Inconel 600	<0.003 (<0.1)	<0.003 (<0.1)	0.005 (0.2)	
Haynes 263		<0.003 (<0.1)	<0.003 (<0.1)	
Hastelloy G	*b*	<0.003 (<0.1)	<0.003 (<0.1)	
Hastelloy C-276	*b*			
Titanium	*b*	<0.003 (<0.1)	<0.003 (<0.1)	

[a]Corrosion rates calculated from weight changes, assuming uniform removal of material.

[b]Weight gain indicating presence of a tightly adherent scale, which was not removed by wire brushing or ultrasonic cleaning in an organic solvent.

schedule in Table 9, the samples have not been examined as often as have those in the vacuum column. Calculated corrosion rates for the V-103 coupons are given in Table 10, and rates for the V-105 coupons are given in Table 11. From Table 10, it is apparent that the ferritic stainless steels had lower corrosion rates than did the austenitic stainless steels. The variation in corrosion rate among the austenitics can be roughly correlated with the molybdenum content, but data are too limited to substantiate this observation. The coupons exposed in V-105 all experienced a very low corrosion rate during the first exposure period. This is not unexpected, because the coupons were exposed to a fairly low-boiling oil at ambient temperature.

Other coupons are being exposed at Wilsonville in the relatively new hydrotreater area. Three sites are being used for exposing coupons: R-1235 hydrogenation reactor, V-1064 SRC melt tank, and V-1074 hydrotreater product recycle tank. The coupon exposure schedule and nominal operating conditions for these vessels are given in Table 12. From Tables 13 and 14 it is obvious that corrosion rates in V-1064 and V-1074 were low; the highest corrosion rate was for carbon steel in V-1074, with a rate of 0.012 mm/year (0.5 mil/year).

Table 9. Exposure schedule for V-103 and V-105

Installed	Removed	Exposure (h)	Nominal conditions			
			Temperature		Pressure	
			(°C)	(°F)	(MPa)	(psi)
		V-103 high-pressure separator				
November 1979	May 1980	3366	393[a]	740	11.0–15.2	1600–2200
May 1980	February 1981	5052				
February 1981	*b*					
		V-105 solvent decanter				
November 1979	February 1981	8418	Ambient			
February 1981	*b*					

[a]"Hot flash" mode nominal temperature; often operated in 343 to 393°C (650–740°F) range.

[b]Not yet removed.

Table 10. Corrosion rates of samples exposed in the Wilsonville V-103 high-pressure separator

Sample	Corrosion rate[a] [mm/year (mils/year)]			
	November 1979–May 1980 3366 h		May 1980–February 1981 5052 h	
26 Cr-1 Mo	<0.003	(<0.1)	<0.003	(<0.1)
26 Cr-1 Mo stabilized	<0.003	(<0.1)	<0.003	(<0.1)
SC-1	<0.003	(<0.1)	<0.003	(<0.1)
29 Cr-4 Mo	<0.003	(<0.1)	0.005	(0.2)
Type 304 L SS	0.129	(5.1)	0.041	(1.6)
Type 316L SS	0.126	(5.0)	0.044	(1.7)
Type 317L SS	0.066	(2.6)	0.028	(1.1)
Type 317LM SS	0.046	(1.8)	0.023	(0.9)
Type 347 SS	0.117	(4.6)	0.034	(1.3)
2RE69	*b*		0.003	(0.1)
Incoloy 825	0.069	(2.7)	0.029	(1.1)
Hastelloy G-3	0.016	(0.6)	0.012	(0.5)

[a]Corrosion rates calculated from weight change, assuming uniform removal of material.

[b]Weight gain indicating presence of tightly adherent scale, which was not removed by wire brushing or ultrasonic cleaning in an organic solvent.

Table 11. Corrosion rates of samples exposed in the Wilsonville V-105 decanter vessel

Sample	Corrosion rate[a] [mm/year (mil/year)]	
	November 1979–February 1981 8418 h	
Carbon steel	0.003	(0.1)
Type 410 SS	<0.003	(<0.1)
18 Cr-2 Mo	<0.003	(<0.1)
26 Cr-1 Mo	<0.003	(<0.1)
26 Cr-1 Mo stabilized	<0.003	(<0.1)
SC-1	<0.003	(<0.1)
Type 304L SS	<0.003	(<0.1)
Type 316 SS	<0.003	(<0.1)
Titanium	*b*	

[a]Corrosion rates calculated from weight change, assuming uniform removal of material.

[b]Weight gain indicating presence of a tightly adherent scale, which was not removed by wire brushing or ultrasonic cleaning in an organic solvent.

Table 12. Exposure periods and nominal operating conditions for coupons exposed in the Wilsonville hydrotreating area

Installed	Removed	Exposure (h)	Nominal conditions			
			Temperature		Pressure	
			(°C)	(°F)	(MPa)	(psig)
		V-1064 SRC melt tank				
February 1981	March 1982	2901	260	500	0.07	10
March 1982	*a*					
		V-1074 hydrotreater product recycle tank				
February 1981	March 1982	2901	121	250	0.03	5
March 1982	*a*					
		R-1235 hydrotreater reactor				
March 1982	*a*		455	850	20.7	3000

[a]Not yet removed.

Table 13. Corrosion rates of coupons exposed in the Wilsonville V-1064 SRC[a] melt tank

Sample	Corrosion rate [mm/year (mil/year)] February 1981—March 1982 2901 h	
Carbon steel	0.010	(0.4)
2 1/4 Cr-1 Mo	0.010	(0.4)
5 Cr-1 Mo	0.004	(0.2)
7 Cr-1 Mo	0.004	(0.1)
9 Cr-1 Mo modified	0.004	(0.2)
Type 409 SS	<0.003	(<0.1)
Type 410 SS	<0.003	(<0.1)
Type 304 SS	<0.003	(<0.1)
Type 316 SS	<0.003	(<0.1)
Incoloy 825	<0.003	(<0.1)

[a]Name of solid product.

Table 14. Corrosion rate of coupons exposed in the Wilsonville V-1074 hydrotreater product recycle tank

Sample	Corrosion rate [mm/year (mil/year)] February 1981–March 1982 2901 h	
Carbon steel	0.012	(0.5)
2 1/4 Cr-1 Mo	0.004	(0.2)
5 Cr-1 Mo	0.006	(0.2)
7 Cr-1 Mo	0.003	(0.1)
9 Cr-1 Mo modified	<0.003	(<0.1)
Type 409 SS	<0.003	(<0.1)
Type 410 SS	<0.003	(<0.1)
Type 304 SS	<0.003	(<0.1)
Type 316 SS	<0.003	(<0.1)
Incoloy 825	<0.003	(<0.1)

FORT LEWIS, WASHINGTON, SRC-II OR -I PILOT PLANT

Corrosion coupons were first supplied to the Fort Lewis pilot plant for studying the corrosion problem in the middle to upper portion of the wash solvent column (WSC), an atmospheric pressure fractionation column. We later increased the exposure sites to include the bottom of the WSC and the bottom of the light ends column, which is the source of the WSC feed.

As shown in Fig. 9, the Fort Lewis WSC had 21 trays, numbered from the bottom to the top of the column. Racks of coupons can be exposed above and below tray 20, below tray 19, and above tray 12. Three racks (one above tray 20, one below tray 19, and one above tray 12) were exposed from November 1979 to January 1980, and racks were exposed at all four locations from January 1980 to April 1980. A third set of racks inserted in May 1980 was removed in October 1980, and a fourth set was exposed from October 1980 through March 1981.

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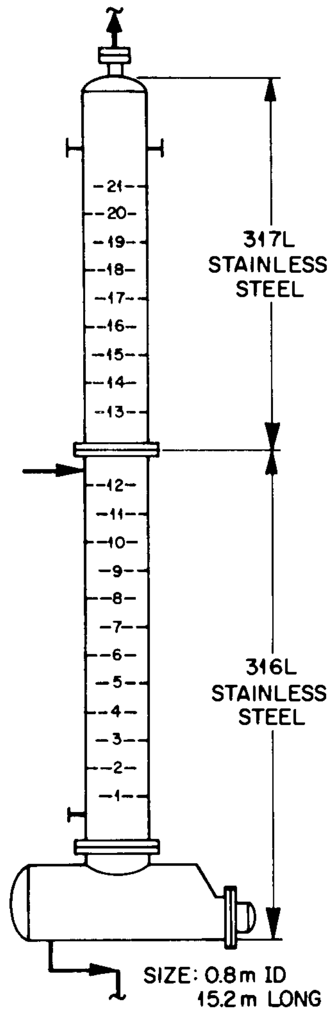

Fig. 9. Schematic of the Fort Lewis wash solvent column.

The results (Table 15) for samples exposed from November 1979 to January 1980 show that corrosion in the column was severe; for example, type 317 stainless steel corroded at a rate greater than 1 mm/year. The pattern noted on the samples exposed at Wilsonville was evident for this set of Fort Lewis samples: titanium and high-nickel alloys had the lowest corrosion rates, austenitic stainless steels had higher corrosion rates, and ferritic steels were the most severely corroded. Of the high-nickel alloys, Hastelloy C-276 had the lowest corrosion rate; of the austenitic stainless steels, type 321 had the lowest corrosion rate.

The second set of samples examined was removed from the column during the May 1980 shutdown. Of the racks removed, three had been exposed since

Table 15. Corrosion rates of coupons exposed from November 1979 through January 1980 above the middle manway of the Fort Lewis wash solvent column

Alloy	Corrosion rate[a] [mm/year (mils/year)]	
Titanium	0.003	(0.1)
Hastelloy C-276	0.002	(0.1)
Incoloy 825	0.37	(14.4)
Hastelloy G-3	0.49	(19.2)
Hastelloy G	0.65	(25.6)
Type 321 SS	0.86	(33.8)
Type 316 SS	0.93	(36.6)
Type 317LM SS	1.05	(41.4)
Type 317 SS	1.14	(45.0)
Type 304 SS	1.50	(59.0)
Type 410 SS	3.33	(131)
SC-1	5.21	(205)
26 Cr-1 Mo stabilized	9.43	(371)
Carbon steel	10.84	(426)

[a]Corrosion rates calculated from weight change, assuming uniform removal of material.

January 1980, and the other had been installed in November 1979. The temperature profile of the column was changed several times; the rack exposed since November had therefore experienced somewhat different conditions from those experienced by the others (column temperatures were increased during this time).

The corrosion rates calculated from the weight change measurements are shown in Table 16. These results indicate that the corrosion around trays 19 and 20 (top of column) was more severe than that in the middle of the column. Of the alloys exposed for the first time, Haynes 263, a nickel-base alloy containing Co, Cr, Mo, and Ti, performed very well, and aluminized type 304 stainless steel had a corrosion rate about one-half

Table 16. Corrosion rates of coupons exposed in the Fort Lewis wash solvent column from November 1979 to May 1980

Alloy	Corrosion rate[a] [mm/year (mils/year)]							
	Above tray 20 for 2156 h		Below tray 20 for 2156 h		Below tray 19 for 3491 h		Above tray 12 for 2156 h	
Titanium	<0.003	(<0.1)	<0.003	(<0.1)	0.033	(1.3)	<0.003	(<0.1)
Haynes 263	0.018	(0.7)	0.003	(0.1)			0.018	(0.7)
Hastelloy C-276	<0.003	(<0.1)	0.10	(4.0)	<0.003	(<0.1)	<0.003	(<0.1)
Inconel 625	0.18	(6.9)	0.012	(0.5)			<0.003	(<0.1)
Hastelloy G-3					0.224	(8.8)		
Hastelloy G	0.18	(7.2)	0.038	(1.5)	0.250	(9.8)	0.003	(0.1)
Haynes 20 modified	0.14	(5.5)	0.26	(10)			0.010	(0.4)
Inconel 671	0.40	(16)	0.35	(14)			0.004	(0.2)
Monel 400	0.33	(13)	0.28	(11)			0.23	(9.1)
Type 321 SS	0.26	(10)	0.65	(25)	0.45	(18)	0.064	(2.5)
Type 310 SS	0.67	(26)	0.77	(30)			0.076	(3.0)
Incoloy 825	0.49	(19)	0.97	(38)	0.35	(14)	0.17	(6.6)
Aluminized 304 SS	0.53	(21)						
Alloy 904L	0.55	(22)	1.07	(42)			0.08	(3.2)
Type 317LM SS	0.68	(27)	1.07	(42)	0.69	(27)	0.10	(3.7)
Type 317 SS					0.68	(27)		
Type 316 SS	0.76	(30)	1.07	(42)	0.98	(38)	0.20	(8)
Carpenter 20Cb-3	0.54	(21)	1.22	(48)			0.35	(14)
Type 304 SS	0.10	(39)	1.05	(41)	0.92	(36)	0.29	(12)
SC-1					2.02	(79)	0.89	(35)
Type 410 SS	2.13	(84)	1.00	(40)	2.18	(86)		
26 Cr-1 Mo stabilized					3.17	(125)		
Carbon steel					4.74	(187)		

[a]Calculated from weight change, assuming uniform material removal.

that of type 304 stainless steel. From these results, we again note that corrosion is more severe at the top of the column (220–260°C) than in the middle and that the relative corrosion resistance of the nickel-base alloys, austenitic stainless steels, and ferritic steels is the same as that observed for previous Wilsonville and Fort Lewis samples.

A third set of corrosion coupons was removed from the Fort Lewis WSC during the October 1980 shutdown; calculated corrosion rates are given in Table 17. Samples exposed in the upper half of the WSC had the following pattern.

- Some nickel-base alloys such as Hastelloy C-276, Inconel 600, and Haynes alloy 263 had corrosion rates of less than 0.003 mm/year (0.1 mil/year).
- Other nickel-base alloys (like Inconel 625 and 671), iron-nickel-chromium–molybdenum alloys (like Haynes 20 modified, Incoloy 825, and Crucible 6M), and titanium had corrosion rates of less than 0.54 mm/year (20 mils/year).
- Austenitic stainless steel types 304, 316, and 317LM had corrosion rates of 0.45 to 1.06 mm/year (18–42 mils/year), and type 410 stainless steel had a corrosion rate greater than 0.96 mm/year (38 mils/year).

A fourth set of corrosion coupons was exposed in the Fort Lewis WSC for 3190 h from November 1980 to April 1981. Corrosion rates calculated from the weight change of these samples are given in Table 18. Again, we see the familiar pattern of the high-nickel alloys and titanium showing the greatest corrosion resistance, followed by the Ni-Cr-Fe-Mo alloys and austenitic stainless steels with an intermediate level of resistance. The ferritic and martensitic alloys show the least resistance. A more extensive analysis of the results from corrosion coupon exposures in the fractionation columns at the SRC pilot plants was published recently.[3]

Corrosion coupons were also exposed in the reboiler area of both the light ends column and the WSC at the Fort Lewis pilot plant. The exposure schedule for the coupons is given in Table 19; the calculated corrosion rates for the WSC reboiler area coupons, in Table 20; and the rates for the coupons from the reboiler area of the light ends column, in Table 21.

Table 17. Corrosion rates of coupons exposed in the Fort Lewis wash solvent column from May to October 1980

Alloy	Corrosion rate[a] [mm/year (mils/year)]					
	Above tray 12		Below tray 20		Above tray 20	
Hastelloy C-276	[b]		[b]		<0.003	(<0.1)
Hastelloy N	<0.003	(<0.1)	<0.003	(<0.1)		
RA 333			0.003	(0.1)		
Molybdenum			<0.003	(<0.1)		
Inconel 600	0.01	(0.4)	[b]		[b]	
Haynes alloy 263	0.01	(0.4)	0.007	(0.3)		
Titanium	[b]		0.11	(4.2)		
Haynes 20 modified	0.05	(2.0)			0.07	(2.7)
Hastelloy G	0.12	(1.0)			0.11	(4.2)
Inconel 625	0.14	(5.5)	0.11	(4.2)	0.03	(1.0)
Carpenter 20Cb-3	0.07	(2.7)	0.14	(5.7)	0.11	(4.4)
Incoloy 825			0.06	(2.2)	0.21	(8.2)
Crucible 6M			0.17	(6.6)	0.16	(6.3)
Alloy 904L			0.18	(7.0)	0.24	(9.6)
Monel 400					0.23	(9.0)
Crutemp 25	0.47	(18)	0.41	(16)	0.39	(15)
Type 317LM SS	0.47	(18)			0.45	(18)
Inconel 671	0.49	(19)	0.50	(20)		
Type 316 SS	0.75	(30)	0.84	(33)	0.70	(27)
Type 304 SS	1.06	(42)	0.89	(35)	0.92	(36)
Type 410 SS	0.97	(38)	1.42	(56)		
Incoloy 800			1.27	(50)	1.78	(70)
Aluminum	>5.45	(>215)			>5.43	(>214)

[a]Corrosion rates calculated from weight changes, assuming uniform removal of material.

[b]Weight gain indicating presence of a tightly adherent scale, which was not removed by wire brushing or ultrasonic cleaning in an organic solvent.

Table 18. Corrosion rates for coupons exposed in the Fort Lewis wash solvent column for 3190 h from November 1980 to April 1981

Alloy	Corrosion rate[a] [mm/year (mils/year)]					
	Above tray 20		Below tray 20		Above tray 12	
Hastelloy C-276	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)
Hastelloy N	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)
Hastelloy B-2	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)
Inconel 625	0.063	(2.5)	0.06	(2.4)	0.008	(0.3)
Haynes alloy 263	0.073	(2.9)	0.10	(3.9)	0.083	(3.3)
Inconel X750	0.074	(2.9)	0.13	(5.1)	0.078	(3.1)
Titanium	<0.003	(<0.1)	0.009	(0.4)	0.36	(14)
Inconel 601	0.095	(3.7)	0.28	(11)	0.085	(3.4)
Inconel 600	0.20	(7.7)	0.33	(13)	0.14	(5.6)
Alloy 904L	0.58	(23)	0.47	(19)	0.41	(16)
Nickel	0.60	(24)	0.49	(19)	0.61	(24)
Type 321 SS	0.55	(22)	1.08	(43)	0.17	(6.8)
Type 316 SS	1.52	(60)	0.89	(35)	1.01	(40)
Aluminized 304 SS	1.17	(46)	1.41	(55)	0.88	(34)
Type 304 SS	2.22	(88)	1.37	(54)	1.31	(52)
Incoloy 800	1.81	(71)	2.92	(115)	2.72	(107)
Type 410 SS	2.63	(103)	1.09	(82)	3.45	(136)
Carbon steel	>10.7	(>422)	6.4	(250)	4.99	(196)

[a]Corrosion rates calculated from weight changes, assuming uniform removal of material.

Table 19. Exposure periods and nominal operating conditions for coupons exposed in the reboiler areas of the Fort Lewis light ends and wash solvent columns

Installed	Removed	Exposure (h)	Nominal conditions			
			Temperature		Pressure	
			(°C)	(°F)	(MPa)	(psig)
		Wash solvent column reboiler				
May 1980	October 1980	2422	293–	560–	0.15	22
November 1980	April 1981	3190	357	675		
		Light ends column reboiler				
May 1980	October 1980	2407	227–	530–	0.15	22
November 1980	April 1981	3163	300	570		

Table 20. Corrosion rates of coupons exposed in the reboiler area of the Fort Lewis wash solvent column

Sample	Corrosion rate[a] [mm/year (mils/year)]			
	May 1980–October 1980 2442 h		November 1980–April 1981 3190 h	
Aluminum	2.58	(102)		
Nickel			0.21	(8.3)
Titanium	*b*			
Carbon steel			0.84	(33)
Type 410 SS	1.14	(45)	1.01	(40)
Type 304 SS			0.010	(0.4)
Aluminized 304 SS			*b*	
Type 321 SS			0.005[b]	(0.2)
Type 316 SS	*b*		<0.003	(<0.1)
Type 317LM SS	*b*			
Incoloy 800			0.36	(14)
Incoloy 801			0.26	(10)
Incoloy 825	*b*			
Haynes 20 modified	*b*			
Carpenter 20Cb-3	*b*			
Crutemp 25	*b*			
904L	*b*		0.004	(0.2)
Crucible 6M	*b*			
Udimet 720			0.077	(3.0)
Inconel 600	*b*		0.20	(7.8)
Inconel 601			0.98	(39)
Inconel 625	*b*		*b*	
Inconel 671	*b*			
Inconel X750			0.023	(0.9)
Hastelloy G	*b*			
Hastelloy N			<0.003	(<0.1)
Haynes 263	*b*		0.004	(0.2)
Hastelloy B-2			<0.003	(<0.1)
Hastelloy C-276			*b*	

[a]Corrosion rates calculated from weight changes, assuming uniform removal of material.

[b]Weight gain indicating presence of a tightly adherent scale, which was not removed by wire brushing or ultrasonic cleaning in an organic solvent.

Table 21. Corrosion rates of coupons exposed in the reboiler area of the Fort Lewis light ends column

Sample	Corrosion rate[a] [mm/year (mils/year)]			
	May 1980–October 1980 2442 h		November 1980–April 1981 3190 h	
Aluminum	3.88	(153)		
Nickel			>1.22[b]	(>48)
Titanium	[c]		[c]	
Carbon steel			3.15	(124)
Type 410 SS	0.45	(18)	0.35	(14)
Type 304 SS	0.020	(0.8)	0.029	(1.1)
Aluminized 304 SS			0.019	(0.8)
Type 321 SS			0.007	(0.3)
Type 316 SS	0.009	(0.4)	0.026	(1.0)
Type 317LM SS	0.021	(0.8)		
Incoloy 800	0.189	(7.4)	0.39	(15)
Incoloy 801			0.074	(2.9)
Incoloy 825	0.013	(0.5)		
Haynes 20 modified	0.005	(0.2)		
Carpenter 20Cb-3	0.019	(0.8)		
Crutemp 25	0.029	(1.1)		
904L			0.021	(0.8)
Crucible 6M	<0.003	(<0.1)		
Udimet 720			0.023	(0.9)
Monel 400	0.313	(12)		
Inconel 600	<0.003	(<0.1)	0.076	(3.0)
Inconel 601			0.152	(6.0)
Inconel 625			<0.003	(<0.1)
Inconel X750			0.055	(2.2)
Hastelloy N			<0.003	(<0.1)
Haynes 263	<0.003	(<0.1)	0.009	(0.4)
Hastelloy B-2			<0.003	(<0.1)
Hastelloy C-276	<0.003	(<0.1)	<0.003	(<0.1)

[a]Corrosion rates calculated from weight changes, assuming uniform removal of material.

[b]Coupon completely consumed.

[c]Weight gain indicating presence of a tightly adherent scale, which was not removed by wire brushing or ultrasonic cleaning in an organic solvent.

These tables show that aluminum, nickel, and carbon steel had higher corrosion rates in the light ends column even though it operated at a lower temperature. In both vessels, Inconels 600 and 601 and Incoloys 800 and 801 had higher corrosion rates than did the austenitic stainless steels. In the upper portion of the WSC, these Inconels were consistently more resistant to corrosion than were the austenitics, supporting the idea that two different corrosion processes are at work in the WSC.

From the data collected during this study, the austenitic stainless steels appear to provide ample corrosion resistance in the reboiler areas of the two columns. The only contradictory data came from service in late 1979. That bundle contained tubes of Incoloy 800, type 316 stainless steel, and Hastelloy G and had support members of type 304 stainless steel. The 304 and 800 alloys were severely corroded, and the type 316 stainless steel had a significant amount of pitting, but the Hastelloy G suffered only minor attack. These data suggest that further studies are needed to determine the most suitable alloys for this application.

CATLETTSBURG, KENTUCKY, H-COAL PILOT PLANT

The ORNL involvement with the Catlettsburg pilot plant initially consisted of supplying 13 racks of U-bend specimens and 24 individual corrosion coupons to be mounted on racks provided by IMMR. Subsequent examination of the coupons was conducted by IMMR. In the fall of 1980, Catlettsburg engineers asked ORNL to provide five racks of corrosion coupons to be exposed in the N-202 fractionator, in which they had found severe corrosion like that previously noted at the SRC pilot plants. The exposure schedules for the individually supplied coupons and for the five racks used in the fractionator are given in Table 22. Results for the coupons exposed on IMMR racks were reported.[4,5]

Pitting of a few of the type 410 stainless steel trays in the N-202 fractionator was noted during the November 1980 shutdown. To monitor this corrosion, ORNL supplied five racks of coupons, which were located directly on trays 9, 10, 11, 12, and 13 in the 14-tray column). One rack of corrosion coupons was removed from the fractionator during the March 1981 shutdown. At that time, four other racks, located on trays 9, 10, 12, and

Table 22. Exposure schedule for ORNL-supplied corrosion coupons in the Catlettsburg H-Coal Pilot Plant

Exposure site	Installed	Removed	Exposure (h)
Coupons supplied to IMMR[a]			
N-202 fractionator N-204 atmospheric stripper N-205 vacuum tower	June 1980	November 1980	
	February 1981	December 1981	3397
	March 1982	April 1982	456
	July 1982	*b*	
Coupons exposed on ORNL racks			
N-202 fractionator	November 1980	April 1981	1 rack, 1080; 4 racks, 1176
N-202 fractionator	July 1981	December 1981	2160
N-202 fractionator	March 1982	*b*	

[a]IMMR, Institute for Mining and Minerals Research.

[b]Not yet removed.

13, were left in the tower. Because of operating difficulties, the plant was shut down again a short time later, and the other four racks were removed and examined. Results of the examination of all five racks are given in Table 23. These results show that carbon steel suffered extensive corrosion on all five racks but that corrosion was most severe on trays 10 and 11. Except for Monel, all nickel-base alloys performed very well. Only two samples of type 321 stainless steel were exposed, but they did not perform nearly as well as had the type 321 stainless steel in the fractionation towers at the SRC pilot plants. The iron-base high-molybdenum alloys (904L, Haynes 20 modified, and Crucible 6M) demonstrated very good corrosion resistance.

The racks were removed for a second examination in December 1981. Results from examination of the coupons are given in Table 24. These results show that the previously noted relationship between alloy composition and corrosion resistance still holds: nickel-base alloys and titanium are the most corrosion resistant, followed by Fe-Ni-Cr-Mo alloys, austenitic stainless steels, and ferritic and martensitic steels. The level of worst corrosion was lower in the column, most likely the result

Table 23. Corrosion rates of coupons exposed in the N-202 fractionator at the Catlettsburg H-Coal Pilot Plant

Alloy	Corrosion rate[a] [mm/year (mils/year)]				
	Tray 9[b]	Tray 10[b]	Tray 11[b]	Tray 12[b]	Tray 13[b]
Carbon steel	14.79 (582)	20.43 (804)	19.32 (760)	17.80 (701)	7.50 (295)
Type 409 SS	1.15 (45)	6.65 (262)	7.63 (300)	5.35 (211)	0.005 (0.2)
Type 347 SS	0.82 (32)	0.76 (30)	0.55 (22)	0.49 (19)	<0.003 (<0.1)
Type 321 SS		0.40 (16)		0.53 (21)	
Monel 400	0.33 (13)	0.29 (11)	0.114 (4.5)	0.149 (5.9)	0.112 (4.4)
Type 317 SS	0.060 (2.4)	0.179 (7.0)	0.189 (7.4)	0.029 (1.1)	<0.003 (<0.1)
Carpenter 20Cb-3	0.033 (1.3)	0.036 (1.4)	0.114 (4.5)	0.035 (1.4)	<0.003 (<0.1)
SAF 2205	0.004 (0.2)	0.033 (1.3)	0.038 (1.5)	0.046 (1.8)	<0.003 (<0.1)
Sandvik 2RE69	<0.003 (<0.1)	0.014 (0.5)	0.034 (1.3)	0.014 (0.6)	<0.003 (<0.1)
Incoloy 825	<0.003 (<0.1)	0.011 (0.4)	0.018 (0.7)	0.004 (0.1)	0.003 (0.1)
Inconel 600	0.012 (0.5)	0.014 (0.5)	<0.003 (<0.1)	0.006 (0.2)	<0.003 (<0.1)
Hastelloy B-2	0.007 (0.3)	0.007 (0.3)	<0.003 (<0.1)	0.009 (0.3)	0.007 (0.3)
Crucible 6M	0.006 (0.2)	0.004 (0.1)	0.003 (0.1)	0.003 (0.1)	0.003 (0.1)
RA 333	0.005 (0.2)	0.003 (0.1)	<0.003 (<0.1)	<0.003 (<0.1)	0.004 (0.2)
Haynes alloy 263	0.003 (0.1)	<0.003 (<0.1)	0.004 (0.2)	0.003 (0.1)	<0.003 (<0.1)
Titanium	0.003 (0.1)	<0.003 (0.1)	<0.003 (<0.1)	<0.003 (<0.1)	0.003 (0.1)
Alloy 904L	<0.003 (<0.1)		<0.003 (<0.1)		<0.003 (<0.1)
Haynes 20 modified	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Hastelloy C-4	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Inconel 625	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Hastelloy G	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)

[a]Corrosion rates calculated from weight changes, assuming uniform removal of material.

[b]Exposure times: tray 11, 1080 h; trays 9, 10, 12, and 13, 1176 h.

Table 24. Corrosion rates for coupons exposed 2160 h in the Catlettsburg atmospheric fractionator from July through December 1981

Alloy	Corrosion rate [mm/year (mils/year)][a]				
	Tray 13	Tray 12	Tray 11	Tray 10	Tray 9
Carbon steel	3.54 (139)	6.32 (249)	11.0 (435)	10.5 (414)	>15.8 (>620)
Type 410 SS	0.019 (0.8)	0.068 (2.7)	0.44 (17)	0.53 (21)	1.86 (73)
Monel 400	0.137 (5.4)	0.182 (7.2)	0.196 (7.7)	0.166 (6.5)	0.30 (12)
Type 304 SS	<0.003 (<0.1)	0.010 (0.4)	0.28 (11)	0.31 (12)	0.26 (10)
Type 347 SS	<0.003 (<0.1)	0.022 (0.9)	0.155 (6.1)	0.135 (5.3)	0.30 (12)
Type 321 SS	<0.003 (<0.1)	0.050 (2.0)	0.063 (2.5)	0.141 (5.5)	0.145 (5.7)
Type 317 SS	<0.003 (<0.1)	0.006 (0.2)	0.056 (2.2)	0.065 (2.6)	0.048 (1.9)
Type 316L SS	<0.003 (<0.1)	0.005 (0.2)	0.056 (2.2)	0.084 (3.3)	0.017 (0.7)
Sandvik 2RE69	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	0.068 (2.7)	0.008 (0.3)
904L	<0.003 (<0.1)	0.006 (0.2)	0.009 (0.3)	0.016 (0.6)	<0.003 (<0.1)
SAF 2205	<0.003 (<0.1)	0.003 (0.1)	0.013 (0.5)	<0.003 (<0.1)	0.013 (0.5)
Inconel 600	<0.003 (<0.1)	0.005 (0.2)	0.005 (0.2)	0.004 (0.2)	0.007 (0.3)
RA 333	<0.003 (<0.1)	0.006 (0.2)	0.005 (0.2)	0.007 (0.3)	<0.003 (<0.1)
Carpenter 20Cb-3	<0.003 (<0.1)	<0.003 (<0.1)	0.005 (0.2)	0.005 (0.2)	0.007 (0.3)
Incoloy 825	<0.003 (<0.1)	0.004 (0.1)	<0.003 (<0.1)	0.005 (0.2)	0.004 (0.1)
Crucible 6M	<0.003 (<0.1)	0.004 (0.1)	0.005 (0.2)	<0.003 (<0.1)	<0.003 (<0.1)
Titanium	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Hastelloy C-4	<0.003 (<0.1)	<0.003 (<0.1)	0.004 (0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Inconel 625	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Haynes 20 modified	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)

[a]Corrosion rates calculated, assuming uniform removal of material.

of a lowering of the operating temperature of the column. To allow for this change in location of the most corrosive area, the coupons were reinstalled two trays lower in the column. Subsequently, the column was lengthened and five trays added, so the racks were repositioned in the column. The coupons are scheduled for final examination following the termination of plant operation in November 1982.

BAYTOWN, TEXAS, EXXON COAL LIQUEFACTION PILOT PLANT

At the time of startup of the Baytown pilot plant, ORNL supplied only unmounted U-bend specimens. However, after corrosion problems were encountered near the top of the T-201 fractionator, we were asked to provide five racks of coupons for exposure in the tower. Five racks were supplied in June 1981 for exposure on trays 20, 21, 23, 25, and 26. These racks were removed for examination in November 1981 and again in August 1982, when plant operation was terminated. Corrosion rates calculated after the November 1981 examination are given in Table 25.

During the operating period that ended in November 1981, the pilot plant operated for about one week on Illinois No. 6 (bituminous) coal and for about ten weeks on (subbituminous) coal from the Wyodak mine. On the basis of corrosion probe measurements and knowledge of the chlorine content of the coals, we expected that corrosion would be more severe during operation on Illinois coal than on Wyodak coal. Significant corrosion was noted on the carbon steel and type 410 stainless steel coupons exposed on trays 21 and 23. We speculate that much of this corrosion occurred during the initial operation on Illinois coal.

The second set of coupons was examined after 4920 h at operating conditions. During this operating period, Illinois No. 6 coal was used for 84 d, Wyodak coal was used 45 d, and Texas lignite was used the final 76 d. The corrosion rates calculated from this exposure are given in Table 26. Corrosion of carbon steel was quite severe on trays 20, 21, 23, and 25, but the austenitic stainless steels showed less severe corrosion than they did on comparable trays at the SRC pilot plants.

Table 25. Calculated corrosion rates for first set of coupons exposed 1896 h in the Exxon Coal Liquefaction Pilot Plant T-201 atmospheric fractionator

Alloy	Corrosion rate [mm/year (mils/year)]				
	Tray 26	Tray 25	Tray 23	Tray 21	Tray 20
Carbon steel	0.58 (23)	1.47 (58)	4.86 (191)	4.54 (179)	2.69 (108)
Type 410 SS	0.010 (0.4)	0.045 (1.8)	2.10 (83)	2.62 (103)	0.46 (18)
Type 321 SS	<0.003 (<0.1)	<0.003 (<0.1)	0.166 (6.5)	0.009 (0.4)	<0.003 (<0.1)
Aluminized 304 SS	0.016 (0.6)	0.017 (0.7)	0.011 (0.4)	0.008 (0.3)	0.010 (0.4)
Inconel 600	*a*	*a*	<0.003 (<0.1)	0.003 (0.1)	*a*
Inconel 601	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Type 304 SS	0	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Type 316 SS[b]	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Incoloy 825	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Type 316 SS[b]	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Type 316L SS	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Ferralium	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Haynes 20 modified	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Zirconium	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Type 347 SS	*a*	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Titanium	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)	<0.003 (<0.1)
Inconel 625	*a*	<0.003 (<0.1)	*a*	<0.003 (<0.1)	<0.003 (<0.1)
Hastelloy C-276	<0.003 (<0.1)	<0.003 (<0.1)	0	<0.003 (<0.1)	<0.003 (<0.1)

[a]Weight gain indicates presence of a tightly adherent scale not removed by ultrasonic cleaning.

[b]Two type 316 stainless steel coupons were exposed.

Table 26. Corrosion rates of coupons exposed at the Exxon Coal Liquefaction Pilot Plant for 4920 h (second exposure)

Alloy	Corrosion rate[a] [mm/year (mils/year)]									
	Tray 20		Tray 21		Tray 23		Tray 25		Tray 26	
Carbon steel	>4.20	(>165)	>4.23	(>166)	>4.22	(>166)	>4.20	(>165)	0.86	(34)
Type 410 SS	0.60	(24)	4.04	(159)	1.96	(77)	0.025	(1.0)	0.004	(0.2)
Type 304 SS	0.040	(1.6)	0.277	(10.9)	0.146	(5.7)	<0.003	(<0.1)	<0.003	(<0.1)
Type 321 SS	0.041	(1.6)	0.196	(7.7)	0.131	(5.2)	<0.003	(<0.1)	<0.003	(<0.1)
Type 316 SS	0.031	(1.2)	0.153	(6.0)	0.140	(5.5)	<0.003	(<0.1)	0.003	(0.1)
Type 316L SS	0.020	(0.8)	0.131	(5.2)	0.072	(2.8)	<0.003	(<0.1)	<0.003	(<0.1)
Type 317LM SS	0.022	(0.9)	0.119	(4.7)	0.069	(2.7)	<0.003	(<0.1)	0.003	(0.1)
Ferralium	0.022	(0.9)	0.094	(3.7)	0.048	(1.9)	<0.003	(<0.1)	0.003	(0.1)
Haynes 20 modified	0.003	(0.1)	0.037	(1.4)	0.012	(0.5)	<0.003	(<0.1)	<0.003	(<0.1)
Aluminized 304 SS	0.003	(0.1)	0.010	(0.4)	0.011	(0.4)	0.007	(0.3)	0.005	(0.2)
Incoloy 825	0.003	(0.1)	0.019	(0.7)	0.006	(0.2)	<0.003	(<0.1)	<0.003	(<0.1)
Inconel X750	<0.003	(<0.1)	0.008	(0.3)	0.005	(0.2)	<0.003	(<0.1)	<0.003	(<0.1)
Inconel 600	<0.003	(<0.1)	0.008	(0.3)	0.006	(0.2)	<0.003	(<0.1)	<0.003	(<0.1)
Titanium	0.004	(0.2)	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)
Hastelloy C-276	<0.003	(<0.1)	<0.003	(<0.1)	0.003	(0.1)	<0.003	(<0.1)	<0.003	(<0.1)
Zirconium	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)
Inconel 625	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)	<0.003	(<0.1)

[a]Corrosion rates calculated by assuming uniform removal of material. In fact, corrosion of type 410 stainless steel was not uniform.

SUMMARY

We have presented the results of exposures of ORNL-supplied corrosion coupons in the direct coal liquefaction pilot plants. Testing of this type provides valuable information on materials behavior, but this testing provides only part of the information required to define the needed materials of construction for a given component in a coal liquefaction plant. Although coupon tests may provide guidance to the appropriate types of material, other forms of performance degradation must be considered, and these are, indeed, often the controlling factors. These factors include erosion, erosion-corrosion, stress corrosion cracking, hydrogen attack and embrittlement, and aging effects on materials properties.

Although the data must be considered qualitative, they nevertheless provide a consistent pattern of materials performance in several major coal liquefaction components and process fluids. When combined with the operating experience of the various pilot plants and of the petrochemical industry, they should provide the materials engineer with most of the information needed to make appropriate choices of materials of construction in bringing coal liquefaction technology up to commercial-scale plants.

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