CONTAINER CORROSION IN HIGH-LEVEL NUCLEAR WASTE REPOSITORIES

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Legacy-70

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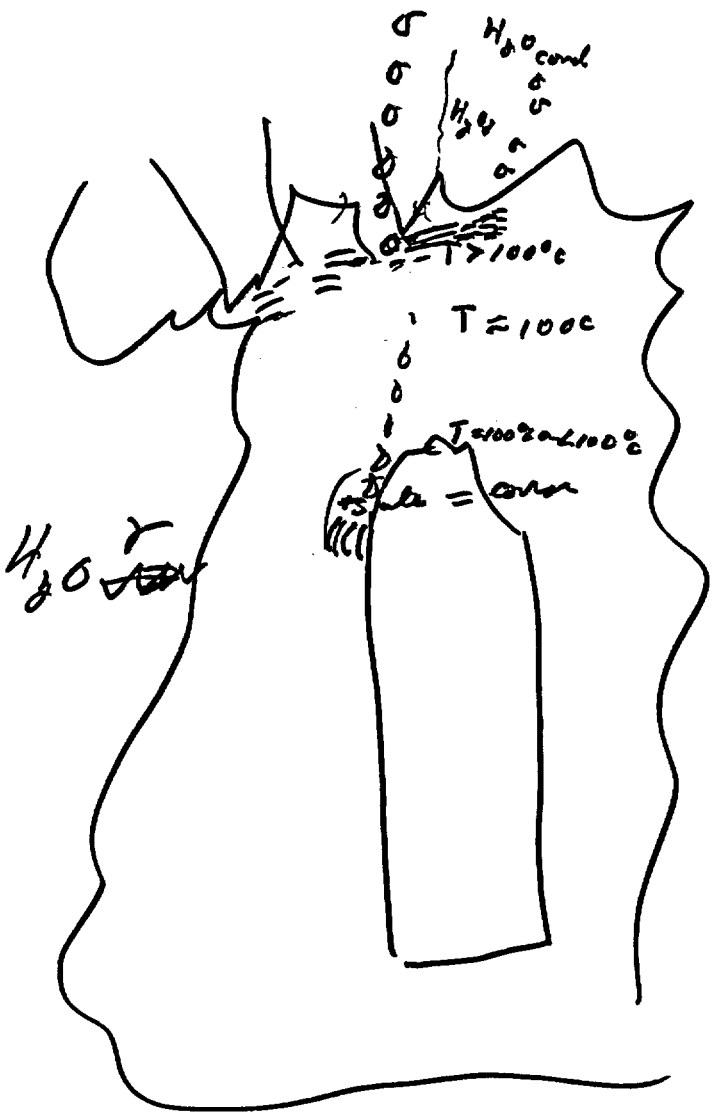
PROGRAM OBJECTIVE

DEVELOP INDEPENDENT EXPERIMENTAL DATA TO ASSIST NRC IN EVALUATING THE UNCERTIANTIES IN DOE's CLAIMS CONCERNING WASTE CONTAINER CORROSION.

ROCK + H_2O → Solut

DISSOLVES



CORROSION

H_2O (corr Sol)

$\boxed{H_2O + \text{salts}}$ → Corr

$Fe \rightarrow Fe^{+2} + E^{0}$

EXPERIMENTAL SCOPE

- o UTILIZE ACCELERATED TEST TECHNIQUES TO EXAMINE POSSIBLE FAILURE MODES,
  - ELECTROCHEMICAL TECHNIQUES,
  - SLOW-STRAIN-RATE, AND
  - MECHANICAL-TEST TECHNIQUES.

- o CONFIRM SHORT-TERM TEST RESULTS WITH LONG-TERM EXPOSURES.

- o EXPAND RANGE OF ENVIRONMENTAL VARIABLES TO CONSIDER PROCESSES THAT AFFECT GROUNDWATER COMPOSITION,
  - ROCK-WATER INTERACTION,
  - LOCAL BOILING, AND
  - RADIATION.

*Environment could not be described in detail

ORIGINAL

PROGRAM TASKS

TASK 1 – REVIEW OF PROBLEMS IN THE TUFF REPOSITORY

TASK 2 – POTENTIODYNAMIC POLARIZATION STUDIES,

TASK 3 – VAPOR-PHASE CORROSION STUDIES,

TASK 4 – PITTING-CORROSION STUDIES,

TASK 5 – STRESS-CORROSION-CRACKING STUDIES,

TASK 6 – STUDIES OF OTHER FAILURE MODES,

TASK 7 – LONG-TERM EXPOSURE STUDIES, AND

TASK 8 – EXAMINATION OF MODELING EFFORTS.

TASK 1 - REVIEW OF PROBLEMS IN THE TUFF REPOSITORY

PURPOSE:

- o COMPILE SITE-SPECIFIC DATA TO BE USED TO DEVELOP AND UPDATE THE WORK PLAN.
- o PROVIDE CONTINUITY BETWEEN INDIVIDUAL TASKS.
- o COORDINATE PROGRAM WITH OTHER NRC PROJECTS.

TASK 1 - REVIEW OF PROBLEMS IN THE TUFF REPOSITORY

STATUS AND RESULTS:

- o COMPLETED SURVEY OF THE LITERATURE,
- o PREPARED TASK REPORT "ENVIRONMENT EFFECTS ON CORROSION IN THE TUFF REPOSITORY".
- o MODIFIED WORK PLAN BASED ON 4-YEAR PROGRAM.

TASK 1 - REVIEW OF PROBLEMS IN THE TUFF REPORTS

MODIFICATIONS OF WORK PLAN

TASK 1 - NO CHANGES

TASK 2 - MINOR CHANGES

TASK 3 - NO CHANGES

TASK 4 - PIT-INITIATION STUDIES EXPANDED. PIT-PROPAGATION STUDIES TERMINATED EARLY.

TASK 5 - OMITTED WELDED SPECIMENS FROM PLAN. SHIFTED FOCUS OF WORK TO ASSESSMENT OF VALIDITY OF SLOW-STRAIN-RATE TECHNIQUE.

No SCC

Concerns about methodology

TASK 6 - OMITTED DEALLOYING AND METALLURGICAL EFFECTS SUBTASKS FROM WORK PLAN.

TASK 7 - PLAN TO TERMINATE LONG-TERM TESTS EARLY TO ACCOMMODATE NEW PROGRAM TERMINATION DATE.

TASK 8 - TERMINATED TASK. NO WORK HAS BEEN PERFORMED.

TASK 2 – POTENTIODYNAMIC POLARIZATION STUDIES

PURPOSE:

- o EXAMINE EFFECTS OF ENVIRONMENT AND METALLURGICAL VARIABLES ON ELECTRO-CHEMICAL BEHAVIOR OF CANDIDATE MATERIALS.

APPROACH:

- o UTILIZED CYCLIC POTENTIODYNAMIC POLARIZATION TECHNIQUE.

- o USED A STATISTICAL EXPERIMENTAL DESIGN APPROACH TO HANDLE A LARGE NUMBER OF ENVIRONMENTAL VARIABLES.

Pasted Sacbrwl
- Bone Bone Minum
- 4 of 6 mat

TASK 2 – POTENTIODYNAMIC POLARIZATION STUDIES

APPROACH (CONTINUED):

- o DEPENDENT VARIABLES OBTAINED FROM CYCLIC POTENTIODYNAMIC POLARIZATION (CPP) CURVES

  E_{cor} – CORROSION POTENTIAL
  i_{cor} – CORROSION RATE AT FREE-POTENTIAL
  i_{pas} – PASSIVE-CURRENT DENSITY
  E_{pit} – PITTING POTENTIAL
  E_{prot} – PROTECTION POTENTIAL

TASK 2 – POTENTIODYNAMIC POLARIZATION STUDIES

APPROACH (CONTINUED):

- STUDIED EFFECT OF TEMPERATURE GRADIENTS ON POLARIZATION BEHAVIOR.

Solution 50°
Specimen 90°

- THREE SOLUTIONS STUDIED:
  - SIMULATED J-13 WELL WATER
  - SOLUTION NO. 7; A PITTING SOLUTION FOR Fe-Cr-Ni ALLOYS
  - SOLUTION NO. 22; A PITTING SOLUTION FOR COPPER-BASE ALLOYS

- SOLUTION MAINTAINED AT 50°C

- SPECIMEN MAINTAINED AT 90°C

Table 3. Concentration Ranges For Environmental Species In Tuff Groundwater.

Environmental Species	J-13 Well Water mg/l	Solution No. 7 mg/l	Solution No. 22 mg/l
pH	7.6	5.0	10.0
Si	58	100	100
HCO_3	125	10	10
F	2.2	1.0	1.0
Cl	6.9	1000	1000
NO_3	9.6	5.0	1000
SO_4	18.7	*	*
NO_2	---	0.0	0.0
H_2O_2	---	0.0	0.0
Al	0.012	0.01	0.01
Fe	0.006	0.0	0.0
Ca	12.5	20	20
Mg	1.9	20	20
K	5.1	0.0	0.0
Na	44	*	*
P	0.12	20	20
Oxalic	---	200	200

* Na and SO_4 were used to balance composition.

Table 4. Comparison Of Polarization Parameters Of The Candidate Container Alloys From Isothermal And Heat-Transfer Specimens.

Alloy	Test Solution	Solution Temperature °C	E_{cor} V, SCE	I_{cor} $\mu A/cm^2$	E_{pit}/E_b V, SCE	E_{prot}/E_{rp} V, SCE	Comments
CDA 102	J-13	90	-0.030	2.00	+0.140	-0.035	Changes in oxide
CDA 102	J-13	50	-0.020	0.30	+0.110	-0.090	Changes in oxide
CDA 102*	J-13	50	-0.021	0.48	+0.103	-0.078	Pitting
CDA 102†	J-13	50	-0.015	0.43	+0.119	-0.015	Pitting
CDA 102†	J-13	50	-0.028	0.31	+0.113	-0.075	Pitting
CDA 715	J-13	90	-0.265	0.41	+0.180	+0.080	Pitting
CDA 715	J-13	50	+0.025	0.60	+0.190	-0.080	Pitting
CDA 715*	J-13	50	-0.015	0.18	+0.180	-0.050	Pitting
304L	J-13	90	-0.125	0.12	+0.800	+0.800	No pitting
304L	J-13	50	-0.075	0.05	+0.825	+0.825	No pitting
304L*	J-13	50	-0.050	0.90	+0.845	+0.815	Local etching
1825	J-13	90	-0.650	0.08	+0.700	+0.700	No pitting
1825	J-13	50	-0.060	0.05	+0.840	+0.840	No pitting
1825*	J-13	50	-0.050	0.08	+0.820	+0.700	Pitting
1825†	J-13	50	-0.015	0.08	+0.870	+0.870	Pitting
304L	7	90	-0.035	0.21	+0.090	-0.120	Pitting
304L	7	50	-0.080	0.09	+0.170	-0.010	Pitting
304L*	7	50	-0.013	0.43	+0.200	-0.006	Pitting
1825	7	90	-0.090	0.27	+0.600	+0.150	Pitting
1825	7	50	-0.190	0.08	+0.460	+0.070	Pitting
1825*	7	50	-0.080	0.20	+0.435	+0.280	Pitting
CDA 102	22	50	+0.060	5.60	+0.060	—	Pitting, oxide growth
CDA 102*	22	50	+0.100	0.62	+0.013	—	Pitting
CDA 715	22	50	+0.073	1.00	+0.020	—	Pitting, active areas
CDA 715*	22	50	+0.113	1.40	+0.038	—	Pitting, etching

* Heat Transfer Specimen, 90°C

† Heat Transfer Specimen, 90°C, Repeat Test

TASK 2- POTENTIODYNAMIC POLARIZATION STUDIES

APPROACH (CONTINUED):

- o STUDIED THE EFFECT OF WELDING ON POLARIZATION BEHAVIOR.

Just No SCC

① PPC

- THE FOLLOWING SOLUTIONS WERE USED:
  - SIMULATED J-13 WELL WATER
  - SOLUTION NO. 7
  - SOLUTION NO. 25; AN ACTIVE CORROSION SOLUTION FOR Fe-Cr-Ni ALLOYS
  - SOLUTION NO. 25; A PITTING SOLUTION FOR COPPER-BASE ALLOYS

TASK 2 – POTENTIODYNAMIC POLARIZATION STUDIES

STATUS AND RESULTS (CONTINUED):

- o WELDING EFFECTS STUDIES:
  - LITTLE EFFECT OF WELDING ON CPP BEHAVIOR OF Fe–Cr–Ni ALLOYS.
  - WELDING WAS DETRIMENTAL TO PERFORMANCE OF COPPER–BASE ALLOYS IN SIMULATED J–13 WELL WATER AND IN SOLUTION NO. 29.

– Some pitting, Not seen Mark gen

I 825 and 304L

Table 5. Compositions Of J-13 Well Water And Selected Selected From The Task 2 Matrix Used For The Welding-Effect Studies.

Environmental Species	J-13 Well Water mg/l	Solution No. 7 mg/l	Solution No. 25 mg/l	Solution No. 29 mg/l
pH	7.6	5.0	5.0	5.0
Si	58	100	100	100
HCO_3	125	10	2000	2000
F	2.2	1.0	200	200
Cl	6.9	1000	5.0	1000
NO_3	9.6	5.0	1000	5.0
SO_4	18.7	*	*	*
NO_2	---	0.0	0.0	200
H_2O_2	---	0.0	0.0	0.0
Al	0.012	0.01	20	0.1
Fe	0.006	0.0	0.0	0.0
Ca	12.5	20	20	20
Mg	1.9	20	20	0.1
K	5.1	0.0	0.0	0.0
Na	44	*	*	*
P	0.12	20	0.1	0.1
Oxalic	---	200	0	200

* Na and SO_4 were used to balance composition.

Table 6. Comparison Of Polarization Parameters Of The Candidate Container Alloys From Wrought And Welded Specimens.

Alloy	Test Solution	Solution Temperature °C	E_{cor} V, SCE	I_{cor} $\mu A/cm^2$	E_{pit}/E_b V, SCE	E_{prot}/E_{rp} V, SCE	Comments
CDA 102	J-13	90	-0.030	2.00	+0.140	-0.035	Changes in oxide.
CDA 102*	J-13	90	-0.052	0.33	+0.153	-0.109	Incipient pitting.
CDA 715	J-13	90	-.265	0.41	+0.180	+.080	Pitting.
CDA 715*	J-13	90	-.115	0.08	+0.130	-.150	Pitting.
304L	J-13	90	-0.125	0.12	+0.800	+0.800	No pitting.
304L*	J-13	90	-0.226	0.06	+0.821	+0.821	No attack.
1825	J-13	90	-0.650	0.08	+0.700	+0.700	No pitting.
1825*	J-13	90	-0.142	0.03	+0.744	+0.744	Slight etching.
CDA 102	7	90	-0.140	6.60	—	—	No pitting, local oxide growth.
CDA 102*	7	90	-0.127	11.00	—	—	No pitting, possible very slight etching.
CDA 102* Repeat	7	90	-0.138	1.30	—	—	No pitting, possible very slight etching.
CDA 715	7	90	-0.150	23.00	—	—	No pitting, active corrosion.
CDA 715*	7	90	-0.169	12.00	—	—	No pitting, possible very slight etching.
304L	7	90	-0.035	0.21	+0.090	-0.120	Pitting.
304L*	7	90	-0.150	0.06	+0.100	-0.100	Pitting.
1825	7	90	-0.090	0.27	+0.600	+0.150	Pitting
1825*	7	90	-0.123	0.12	+0.691	+0.136	Pitting
CDA 102	29	50	-0.070	2.20	+0.160	0.000	Pitting & oxide growth.
CDA 102*	29	50	-0.126	10.00	—	-0.076	Oxide growth.
CDA 715	29	50	-0.060	0.91	+0.280	+0.020	Pitting & active attack.
CDA 715	29	50	-0.113	7.00	—	-0.188	Oxide & darker spotting.
304L	25	90	-0.075	0.23	+0.820	+0.790	No pitting.
304L	25	90	0.000	0.09	+0.989	+0.989	No attack.
1825	25	90	-0.420	0.11	+0.700	+0.600	Local active attack.
1825	25	90	-0.063	0.05	+0.690	+0.620	Iridescent oxide.

* Welded Specimens.

NOTE: J-13 is simulated J-13 well water.

TASK 2 – POTENTIODYNAMIC POLARIZATION STUDIES

REGULATORY RELEVANCE OF RESULTS:

- OBSERVATION OF LOCALIZED CORROSION WITH ALL OF THE CANDIDATE ALLOYS IN SOME REGIONS OF THE FACTOR SPACE DEMONSTRATES A NEED TO BETTER DEFINE REPOSITORY ENVIRONMENT. (*) Chemical Environment

- OBSERVATION OF DETRIMENTAL EFFECTS OF WELDING AND HEAT TRANSFER ON CORROSION BEHAVIOR, IN SOME CASES, DEMONSTRATES NEED TO CONSIDER THESE FACTORS IN LONG-TERM TESTING

IMMERSION STUDIES — V, L, Alternate Immersion

TASK 3 – ~~VAPOR-PHASE COR~~ROSION STUDIES

"U" Bends

PURPOSE:

- EVALUATE THE CORROSION BEHAVIOR OF CANDIDATE MATERIALS IN THE VAPOR PHASE AND UNDER ALTERNATE-IMMERSION CONDITIONS.

Liquid (Soln) and upon Vapor

APPROACH:

- INTERMEDIATE-TERM EXPOSURES (2,000 HOURS) OF SEVERAL TYPES OF CORROSION TEST SPECIMENS.

- ELECTROCHEMICAL MONITORING OF INSTANTANEOUS CORROSION RATES AS A FUNCTION OF TIME.

TASK 3 – VAPOR PHASE CORROSION STUDIES

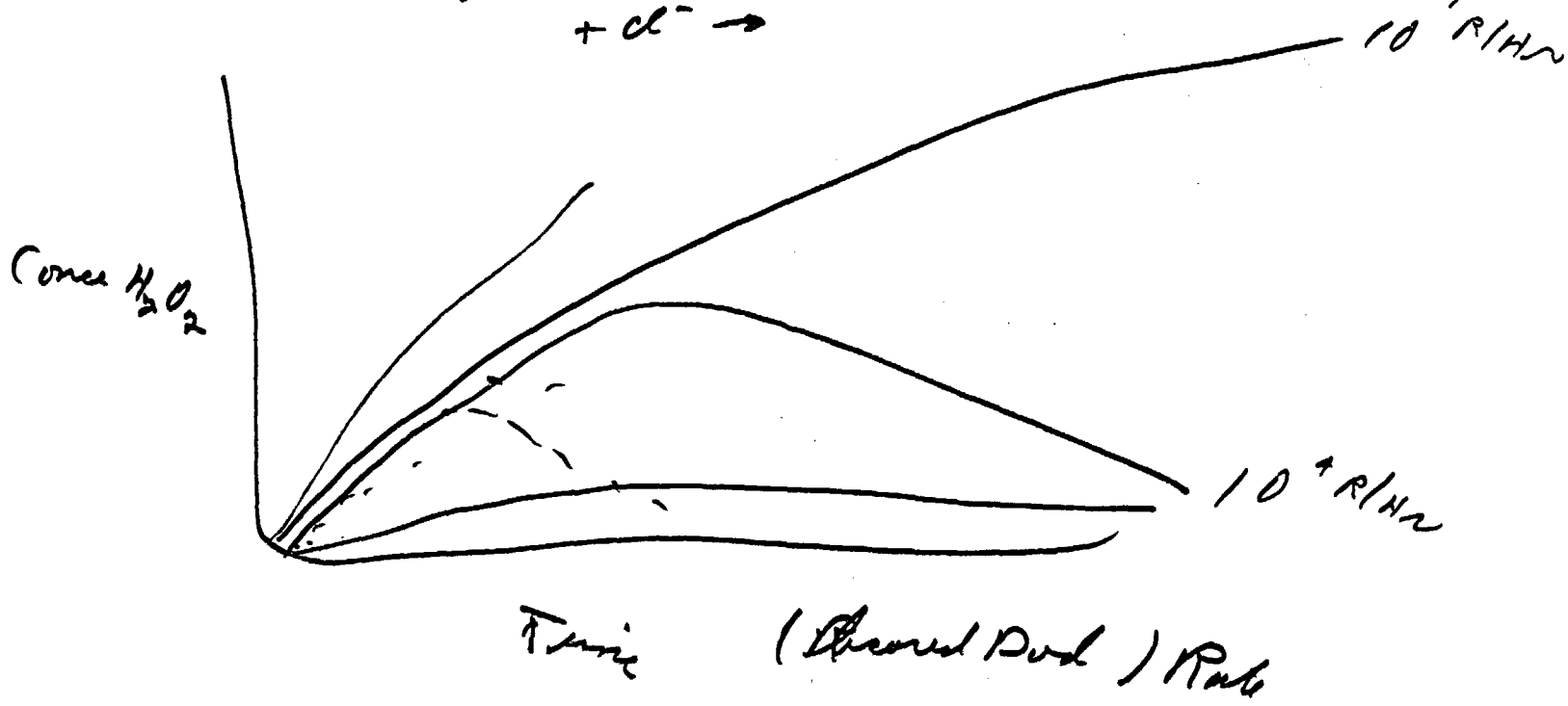
APPROACH (CONTINUED):

- THREE ENVIRONMENTS EVALUATED:
  - SIMULATED J-13 WELL WATER
  - SOLUTION NO. 20; A PITTING SOLUTION FOR Fe-Cr-Ni ALLOYS
  - SOLUTION NO. 7; AN ACTIVE CORROSIVE SOLUTION FOR COPPER-BASE ALLOYS

$H_2O_2 \rightsquigarrow H_2O_2,\ e^-,\ H\cdot,\ I$

$H_2O_2 + H^- \longrightarrow$

$+ Cl^- \rightarrow$



$(H_2O + \text{Chemicals}) \rightsquigarrow$

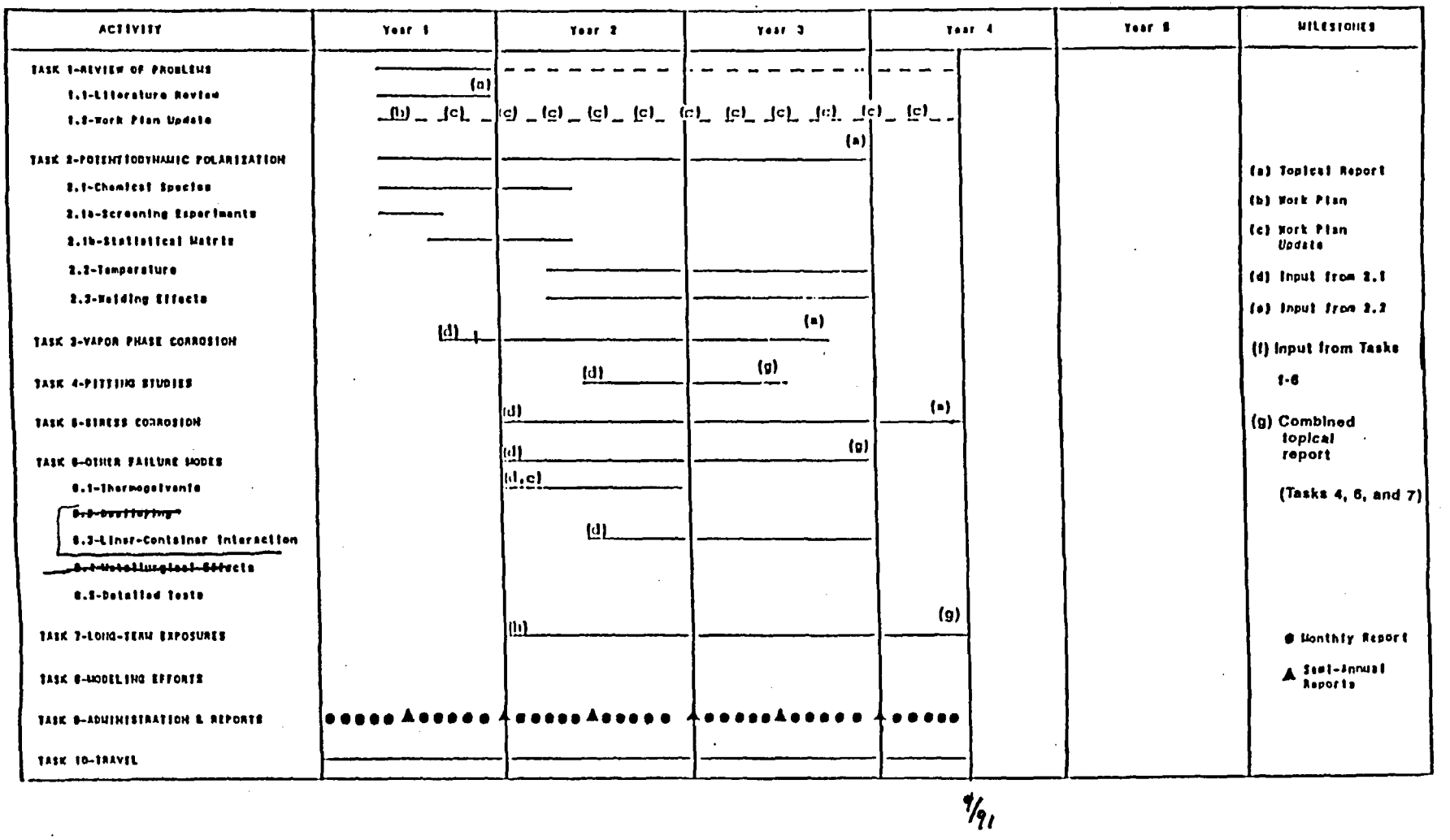


Figure A2. Modified Schedule Of The Work Plan For Each Task For The Remainder Of The Project.

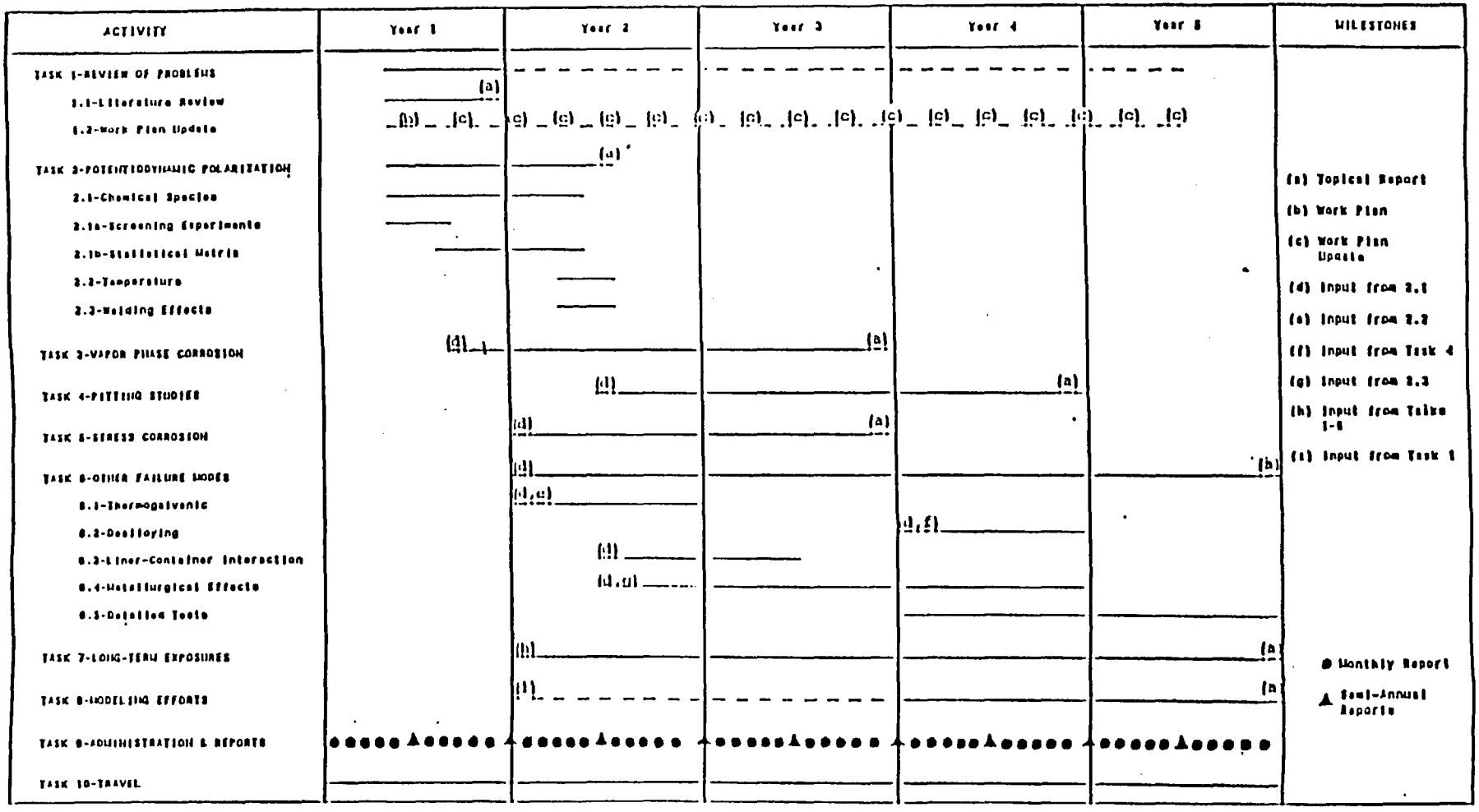


Figure 1. Schedule Of Work Plan For Each Task For The Five Year Projects.

TASK 3 – VAPOR PHASE CORROSION STUDIES

STATUS AND RESULTS

- THE POLARIZATION RESISTANCE (PR) TECHNIQUE WAS FOUND TO OVER-ESTIMATE CORROSION RATES OF COUPONS. compared to weight loss.

- GENERAL CORROSION RATES OF Fe–Cr–Ni ALLOYS WERE BELOW DETECTION LIMIT IN J–13 WELL WATER AND IN SOLUTION NO 20. J-13 wonderful corrosion inhibitor

- IN J–13 WELL WATER, SOME INCIPIENT PITS WERE FOUND ON ALLOY 825 SPECIMENS. - Photo photo

- H_2O_2 PROMOTED PITTING OF ALLOY 304L. — ~~Vapor phase~~ — vapor & liquid

- SCC WAS OBSERVED IN VAPOR–PHASE SPECIMEN OF ALLOY 304L ABOVE SOLUTION NO 20. — No 825 Pitting

TASK 3 - VAPOR PHASE CORROSION STUDIES

STATUS AND RESULTS (CONTINUED)

- o FOR THE COPPER-BASE ALLOYS, GENERAL CORROSION RATES WERE LOW TO MODERATE BUT SIGNIFICANT PITTING WAS OBSERVED IN BOTH J-13 WELL WATER AND IN THE ACTIVE-CORROSION ENVIRONMENT (SOLUTION NO.7).
  - H_2O_2 = Big Effect
  - SCC = None
  - CD 102
    CD 715
    NO 613

TASK 3 – VAPOR PHASE CORROSION STUDIES

REGULATORY RELEVANCE OF RESULTS

- o OBSERVATION OF SCC ON ALLOY 304L IN TEST WITH H_2O_2 IS POSSIBLE CONFIRMATION OF DOE RESULTS IN GAMMA RADIATION IN WHICH SCC OF THIS ALLOY WAS OBSERVED

- o OBSERVATION OF SCC SUGGESTS THAT ALLOY 304L IS UNLIKELY TO SURVIVE IN THE REPOSITORY

- o OBSERVATION OF EXTENSIVE PITTING WITH COPPER-BASE ALLOYS INDICATES THAT PIT PROPAGATION IS THE CRITICAL ISSUE FOR THIS ALLOY SYSTEM. No 613

- o OTHER SIGNIFICANT OBSERVATIONS;
  - (Little) just Study INCIPIENT PITTING OF ALLOY 825
  - OVER-ESTIMATION OF CORROSION RATES BY ELECTROCHEMICAL TECHNIQUE

{ Significance for Field Tests }

TASK 4 - PITTING CORROSION STUDIES

Complete

PURPOSE:

- o STUDY RELATIONSHIPS BETWEEN E_{pit}, E_{prot} AND LONG-TERM PIT-INITIATION BEHAVIOR.
- o EVALUATE PIT-PROPAGATION BEHAVIOR OF THE COPPER-BASE ALLOYS.

APPROACH:

- o INITIATION - POTENTIOSTATIC POLARIZATION TECHNIQUE
- o PROPAGATION - ELECTROCHEMICAL PIT-PROPAGATION TECHNIQUE WITH SIMULATED PIT

- NRC Sponsored Research Product

TASK 4 - PITTING CORROSION STUDIES

STATUS AND RESULTS:

- POTENTIOSTATIC POLARIZATION TESTS COMPLETED ON FOUR CANDIDATE CONTAINER MATERIALS. {# 316SS #CDA615

- RESULTS OF THE CYCLIC POTENTIODYNAMIC POLARIZATION (CPP) TESTS FOR THE COPPER-BASE ALLOYS DID NOT CONFORM WITH CONVENTIONAL INTERPRETATION OF THE CURVES. (*)

- FOR THE COPPER-BASE ALLOYS, THE CPP CURVES DID NOT CORRESPOND TO THE STEADY-STATE BEHAVIOR. (*) so

TASK 4 - PITTING CORROSION STUDIES

STATUS AND RESULTS (CONTINUED):

- o RESULTS OF CPP TESTS FOR Fe-Cr-Ni ALLOYS WERE CONSISTENT WITH CONVENTION INTERPRETATION.

- o THE OCCURRENCE OF LOCALIZED CORROSION IN SOME OF THE MATRIX ENVIRONMENTS FOR ALLOY 304L AS WELL AS FOR INCOLOY ALLOY 825 WAS CONFIRMED IN LONG-TERM POTENTIO-STATIC TESTS.

TASK 4 - PITTING CORROSION STUDIES

REGULATORY RELEVANCE OF RESULTS:

- o RESULTS DEMONSTRATE NEED TO USE CAUTION IN INTERPRETING POTENTIODYNAMIC POLARIZATION TESTS FOR COPPER-BASE ALLOYS

- o PIT-PROPAGATION TEST RESULTS DEMONSTRATE THE DELETERIOUS EFFECT OF H_2O_2 ON PIT PROPAGATION OF ALLOY CDA 102.

- Status

~~Should~~ Writing it up

TASK 5 - STRESS-CORROSION-CRACKING STUDIES

PURPOSES:

- o IDENTIFY ENVIRONMENTAL CONDITIONS THAT PROMOTE SCC OF CANDIDATE ALLOYS
- o EVALUATE THE SLOW-STRAIN-RATE TECHNIQUE FOR SCC ASSESSMENT

APPROACH:

- o SLOW-STRAIN-RATE MECHANICAL TEST TECHNIQUE
- o INTERMEDIATE-TERM U-BEND EXPOSURES

TASK 5 - STRESS-CORROSION-CRACKING STUDIES

STATUS AND RESULTS

NU 613

- o COMPLETED 23 SSR TESTS ON COPPER-BASE ALLOYS IN VARIOUS $NaNO_2$ SOLUTIONS AT 23° AND 90°C.
  - 0.005 M AND 1 M $NaNO_2$ (0.005 M = 200 ppm NO_2).
  - SIMULATED J-13 + $NaNO_2$
  - $NaNO_2$ SOLUTIONS, ANODICALLY POLARIZED
- o SCC OF ALLOY CDA 102 OCCURRED IN DILUTE (0.005 M) $NaNO_2$ SOLUTIONS.

NO FINE SCAN 715

- o ANODIC POLARIZATION STIMULATED CRACKING OF CDA 102.

Table 7. Summary Of Results Of Slow-Strain-Rate Tests Performed On Alloy CDA 102 In The Liquid Phase At A Strain Rate of 1×10^{-6} sec^{-1}.

Sodium Nitrite Concentration	Polarized Potential mV (SCE)	Time To Failure Hours	Reduction In Area Percent	Elongation Percent	Ultimate Tensile Strength MPa	Temp. °C	SCC	Crack Velocity $X10^{-6}$ mm/sec
0M*	—	18.7	55.2	4.9	320	90	None	—
0M*	—	20.1	45.8	4.9	343	90	None	—
1M	(-13)	23.4	53.8	5.0	393	23	TG	†
1M	(+ 8)	21.2	64.0	4.9	337	23	TG	1.97
0.005M	(+12)	26.0	79.2	6.1	348	23	TG	†
0.005M	(+35)	24.4	78.5	5.6	348	23	TG	1.02
0.005M	(+17)	20.5	53.8	4.8	320	90	None	—
0.005M	(+13)	22.3	57.0	5.1	298	90	None	—
1M	(- 2)	20.0	49.3	4.8	309	90	None	—
1M	(-17)	21.2	43.4	3.9	292	90	None	—
0.005M in J-13	(+ 8)	22.0	57.0	4.7	309	90	None	—
0.005M in J-13	(- 7)	20.4	52.7	4.8	309	90	None	—
1M in J-13	(+ 6)	21.0	58.0	4.9	303	90	None	—
1M in J-13	(+15)	21.1	49.3	4.2	309	90	None	—
0.005M	+200	21.4	63.0	5.8	337	23	TG	5.84
0.005M	+174	20.2	58.0	4.8	346	23	TG	5.09
1M	+ 61	18.0	53.8	4.0	337	23	TG	8.02
1M	+ 68	18.5	50.4	4.8	337	23	TG	5.26
0.005M	+234	21.3	58.0	5.0	309	90	TG	0.52
0.005M	+246	21.0	60.1	5.4	315	90	TG	1.19
1M	+122	11.6	10.8	2.7	315	90	TG	20.4
1M	+121	11.0	9.4	2.9	309	90	TG	12.6
0.005M in J-13	(+40)	25.0	76.9	6.5	340	23	None	—
0.005M in J-13	(+29)	26.0	79.2	6.3	351	23	None	—

* Control Test

† Not Sectioned

TG Transgranular

( ) Potential under freely-corroding conditions

Table 8. Summary Of Results Of Slow-Strain-Rate Tests Performed On Alloy CDA 715 In The Liquid Phase At A Strain Rate of 1×10^{-6} sec^{-1}.

Electrolyte	Polarized Potential mV (SCE)	Time To Failure Hours	Reduction In Area Percent	Elongation Percent	Ultimate Tensile Strength MPa	Temp. °C	SCC	Crack Velocity $X10^{-6}$ mm/sec
Oil*	—	69.1	79.2	15.9	410	90	None	—
Oil*	—	67.9	80.6	15.8	393	90	None	—
1M $NaNO_2$	+127	74.6	85.3	18.3	433	23	None†	—
1M $NaNO_2$	+ 64	74.0	81.3	16.9	410	23	None	—
1M $NaNO_2$	+ 33	72.3	76.9	17.3	421	23	None‡	—

* Control Test

† Slip planes, possibly incipient cracking.

‡ Pitting at 30X magnification.

TASK 5 - STRESS-CORROSION-CRACKING STUDIES

STATUS AND RESULTS (CONTINUED):

- COMPONENTS OF J-13 WELL WATER APPEARED TO INHIBIT SCC OF ALLOY CDA 102 IN $NaNO_2$ SOLUTIONS.

- ALLOY CDA 715 WAS MORE RESISTANT TO SCC THAN ALLOY CDA 102.

TASK 5 - STRESS-CORROSION-CRACKING STUDIES

STATUS AND RESULTS (CONTINUED):

- RESULTS OF A SURVEY OF THE LITERATURE ON THE SLOW-STRAIN-RATE TECHNIQUE SUGGEST THAT THE STRAIN RATES USED IN MOST PREVIOUS STUDIES OF Fe-Cr-Ni ALLOYS WERE TOO HIGH FOR THE DILUTE SIMULATED REPOSITORY ENVIRONMENTS EXAMINED.

- THIS THEORY WAS CONSISTENT WITH RESULTS OF SSR TESTS ON ALLOY 304L IN SOLUTION 20; SCC WAS ONLY OBSERVED AT SLOWER STRAIN RATE.

Table 9. Summary Of Results Of Slow-Strain-Rate Tests Performed On Alloy 304L At 90°C.

Electrolyte	Strain Rate sec^{-1}	Potential mV (SCE)	Time To Failure Hours	Reduction In Area Percent	Elongation Percent	Ultimate Tensile Strength MPa	SCC	Crack Velocity $X10^{-6}$ mm/sec
Oil*	$1x10^{-6}$	—	106	77.2	40.0	572	None	—
Oil*	$1x10^{-6}$	—	106	77.0	38.9	606	None	—
Soln. #20*	$1x10^{-6}$	(- 87)	80.3	80.0	17.9	669	None†	—
Soln. #20*	$1x10^{-6}$	(-115)	80.5	76.2	18.7	618	None†	—
Soln. #20*	$1x10^{-7}$	(-74)	935.2	78.5	15.5	618	TG**	.37
Soln. #20*	$1x10^{-7}$	(-115)	550.7	75.4	11.7	646	?	?
J-13 + 100 000 ppm Cl as NaCl	$1x10^{-6}$	(-310)	64.4	73.0	14.3	674	None	—
J-13 + 100 000 ppm Cl as NaCl	$1x10^{-6}$	-174	69.3	68.6	15.5	618	None	—

* Liquid phase, control test.

† Pitting at 30x magnification.

** Probably transgranular, occurs in the radius of the reduced area at the vapor-liquid interface.

( ) Potential under freely-corroding conditions.

TASK 5 - STRESS-CORROSION-CRACKING STUDIES

REGULATORY RELEVANCE OF RESULTS

- o DEFINING LIMITS OF APPLICABILITY OF SSR TEST TECHNIQUE.
- o OBSERVATION OF SCC WITH ALLOY CDA 102 AND ALLOY 304L IN SEVERAL ENVIRONMENTS DEMONSTRATES NEED TO BETTER DEFINE FIELD ENVIRONMENT.

TASKS 6 - OTHER FAILURE MODES

PURPOSE:

- o EXPLORE OTHER FAILURE MODES THAT MAY LEAD TO PREMATURE FAILURE OF WASTE CONTAINERS
  - THERMOGALVANIC COUPLES
  - ~~DEALLOYING OF COPPER-BASE ALLOYS~~
  - ~~METALLURGICAL EFFECTS IN Fe-Cr-Ni-ALLOYS~~
  - BOREHOLE LINER-CONTAINER INTERACTIONS

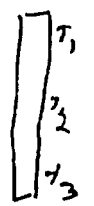
APPROACH:

- o FAILURE MODE SPECIFIC BUT INCLUDES ELECTROCHEMICAL AND WEIGHT-LOSS TECHNIQUES

TASK 6 – OTHER FAILURE MODES

APPROACH (CONTINUED):

- o THERMOGALVANIC COUPLES:
  - – GALVANIC CURRENT MEASUREMENTS BETWEEN HEATED AND ISOTHERMAL SPECIMEN



- o BOREHOLE LINER–CONTAINER INTERACTION:
  - – GALVANIC CURRENT MEASUREMENTS BETWEEN ALLOY 304L AND C1010 CARBON STEEL SANDWICH

– IN 825 x 304 SS

Container

Liner

TASK 6 - OTHER FAILURE MODES

STATUS AND RESULTS:

ALLOY CDA 102 THERMOGALVANIC CORROSION TESTS

- o OBSERVED A SMALL BUT SYSTEMATIC EFFECT OF TEMPERATURE DIFFERENCE (BETWEEN HEATED AND ISOTHERMAL SPECIMEN) ON THERMOGALVANIC CORROSION OF LOW--TEMPERATURE SPECIMEN.

- o H_2O_2 EXACERBATED THERMOGALVANIC EFFECT.

- o INCREASE IN GENERAL CORROSION RATE WITH INCREASING TEMPERATURE DOMINATED THERMOGALVANIC CORROSION EFFECT.

Table 12. Summary Of Polarization Resistance And Weight-Loss Data From Thermogalvanic Corrosion Experiment With CDA 102 In J-13 Well Water.

Condition	Specimen	Polarization Resistance $K\Omega - cm^2$	Corrosion Rate From PR $\mu m/y$	Corrosion Rate From Weight Change $\mu m/y$
Before H_2O_2	Isothermal	1.43×10^2	7.68	-
Before H_2O_2	Heat Transfer	9.35×10^1	11.7	-
After H_2O_2	Isothermal	1.87	26.9	0.97
After H_2O_2	Heat Transfer	0.37	143	1.59

Table 10. Summary Of Results Of Alloy 304L Thermogalvanic Coupled Specimens.

Environment	Specimen	Polarization Resistance* K ohms - cm^2	Corrosion Rate, um/y From PR	Corrosion Rate, um/y Wt.-Loss	Description
J-13	Heated	-	-	3.95	No visible attack.
	Isothermal	-	-	0.17	No visible attack. Rings of scale encircled the top of the specimen due to minor fluctuations in fluid level.
No. 7	Heated	2.34E2	0.87	0.41	Numerous pits on the sides and top associated with halos of rust-colored oxide. Some localized etching.
	Isothermal	4.15E2	0.49	<0.07	No visible attack.

* Polarization resistance (PR) measured after 200 ppm H_2O_2 addition.

TASK 6 - OTHER FAILURE MODES

STATUS AND RESULTS (CONTINUED):

ALLOY 304L THERMOGALVANIC CORROSION TESTS

- GENERAL CORROSION RATES WERE LOW, AS WERE THERMOGALVANIC CORROSION EFFECTS.

- HEATED SPECIMEN EXPERIENCED PITTING ATTACK IN SOLUTION NO.7; NO VISIBLE ATTACK WAS OBSERVED ON ISOTHERMAL SPECIMENS IN EITHER TEST SOLUTION OR ON HEAT-TRANSFER SPECIMEN IN SIMULATED J-13 WELL WATER.

TASK 6 - OTHER FAILURE MODES

RUGULATORY RELEVANCE OF RESULTS

- THERMOGALVANIC CORROSION DOES NOT APPEAR TO BE A SERIOUS PROBLEM, based on limited data.

- HEAT-TRANSFER EFFECTS ON THE HEATED SPECIMENS MAY ACCELERATE GENERAL CORROSION OF COPPER-BASE ALLOYS AND PITTING CORROSION OF Fe-Cr-Ni ALLOYS (FURTHER STUDIES IN THIS AREA ARE WARRANTED).

Need to Run 825

TASK 6 - OTHER FAILURE MODES

STATUS AND RESULTS (CONTINUED):

BOREHOLE LINER-CONTAINER INTERACTION

- o FIRST EXPERIMENT COMPLETED AND TWO ARE IN PROGRESS.
  - EXPERIMENT WITH ALLOYS 304L AND C1010 IN 90°C SIMULATED J-13 WELL WATER COMPLETED AND EXPERIMENTAL SET-UP WAS REVISED.
  - EXPERIMENT WITH ALLOY 304L AND C1010 IN 90°C SIMULATED J-13 + 1000 ppm Cl (NaCl) WAS STARTED.
- o DATA ARE BEING ANALYZED.

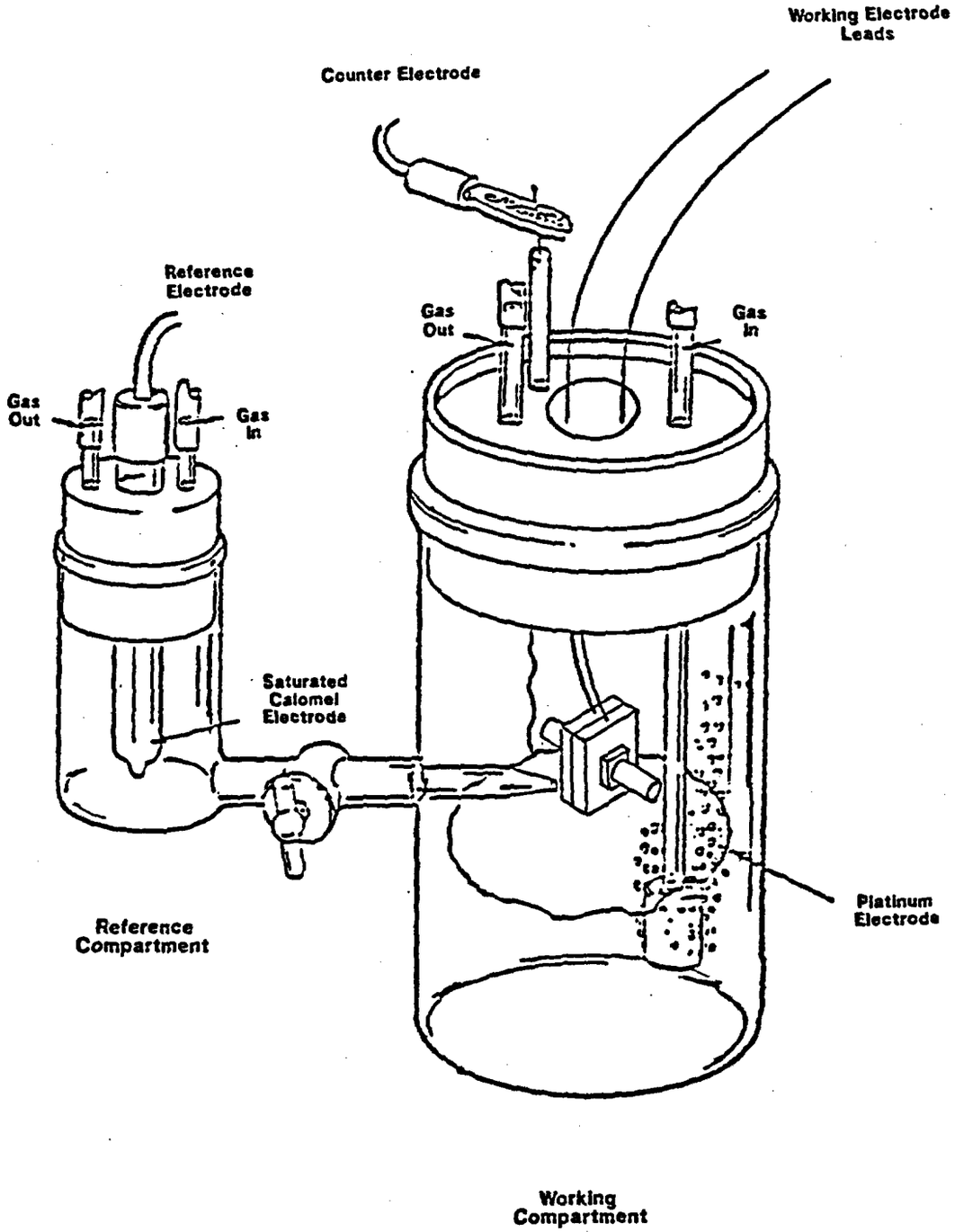
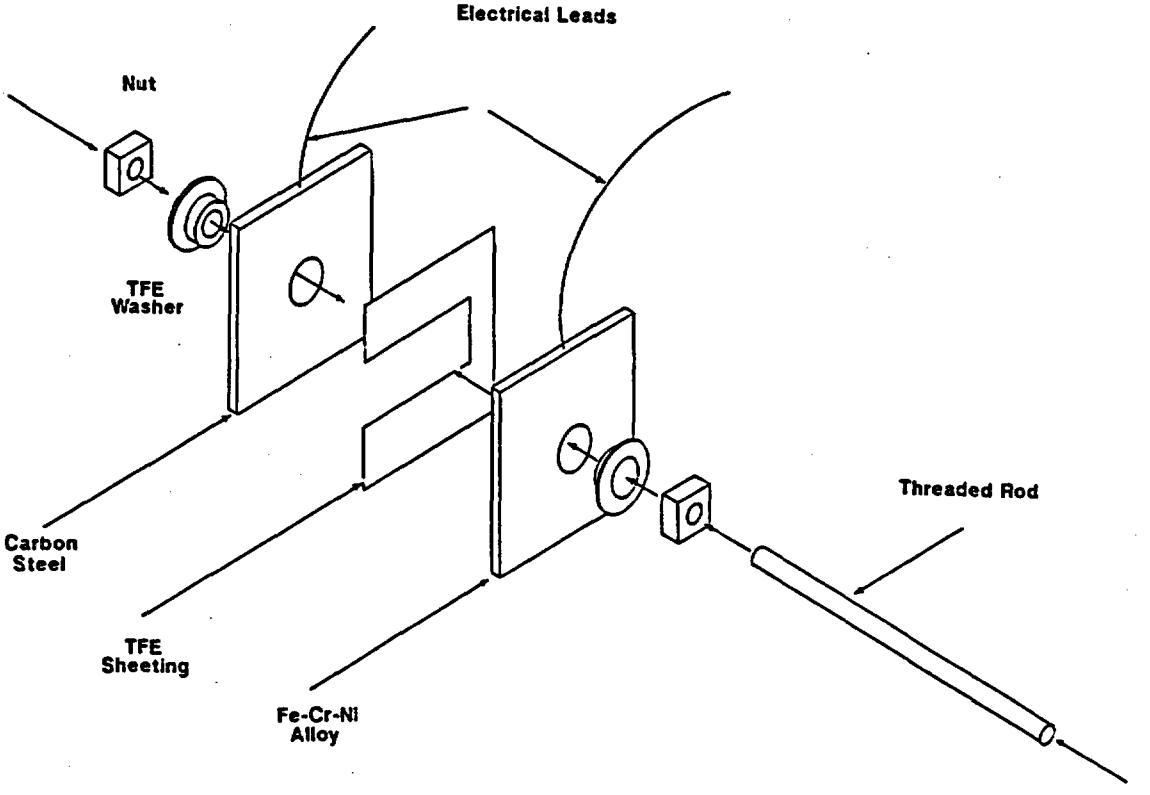


Figure 2. Electrochemical Cell Used For Borehole Liner-Container Interaction Testing


Electrical Leads
Nut
TFE
Washer
Carbon
Steel
TFE
Sheeting
Fe-Cr-Ni
Alloy
Threaded Rod

SANDWICH

TASK 7 - LONG-TERM EXPOSURES

- o PURPOSE - PROVIDE LONG-TERM DATA FOR EVALUATING CORROSION MODES IDENTIFIED IN TASKS 2-6

- o APPROACH - LONG-TERM EXPOSURES IN GLASS VESSELS:
  - GRAVIMETRIC AND OPTICAL EXAMINATIONS OF SPECIMENS
  - ELECTROCHEMICAL MONITORING = Polarization Resistance

TASK 7 - LONG-TERM EXPOSURES

STATUS AND RESULTS:

- o LONG-TERM BOIL-DOWN TESTS FOR EACH OF THE ALLOYS HAVE BEEN CONTINUED FOR 12,000 HOURS (TO DATE) IN SIMULATED J-13 AT 90°C. SPECIMENS WERE REMOVED AFTER 4000 AND 8000 HOURS.
  - THE Fe-Cr-Ni ALLOYS EXHIBITED VERY LOW CORROSION RATES AND NO ATTACK AFTER 8000 HOURS.
  - ALLOY CDA 715 EXHIBITED LOW CORROSION RATES AND LOCALIZED ETCHING AT THE CREVICES.
  - ALLOY CDA 102 EXHIBITED HIGHER CORROSION RATES AND HEAVY ETCHING.

825

TASK 7 - LONG-TERM EXPOSURES

STATUS AND RESULTS (CONTINUED):

Purpose is to correlate

- o LONG-TERM EXPOSURE OF U-BEND SPECIMENS OF ALLOY 304L IN SIMULATED J-13 CONTAINING VARIOUS CONCENTRATIONS OF SALT HAVE BEEN CONTINUED FOR 2000 HOURS; 200 ppm H_2O_2 ADDED DAILY.

  Test Technique confirmed of

  - ONLY VAPOR-PHASE SPECIMENS IN 1000 AND 10,000 ppm Cl (NaCl) AND 10,000 ppm Cl ($CaCl_2$) EXHIBITIED CRACKING. NO SCC OBSERVED IN TESTS EXPOSED TO 100,000 ppm Cl NACl.
  - IN PREVIOUS EXPERIMENT, WITHOUT H_2O_2, SCC OCCURED AT CREVICES UNDER DEPOSIT IN THE VAPOR PHASE OF J-13 + 100,000 ppm Cl (NaCl).

Test Technique

Long Term

Table 13. Summary Of Results Of Exposure Of Creviced Fe-Cr-Ni Specimens In Aerated Simulated J-13 Well Water At 90°C. concentrated J-13

Alloy	Test Time Hours	Specimen Type	Corrosion Rate µm/yr	Maximum Pit Depth µm	Comments	Specimen
I825	4056	Coupon	<0.02	---	No attack; very slight scaling.	I1,I2,I3
I825	8062	Coupon	<0.009	---	No attack; slight scaling.	I4,I5,I6
304L	4034	Coupon	<0.02	---	No attack; slight scaling.	L1,L2,L3
304L	8065	Coupon	<0.009	---	No attack; slight scaling.	L4,L5,L6

Kelter;

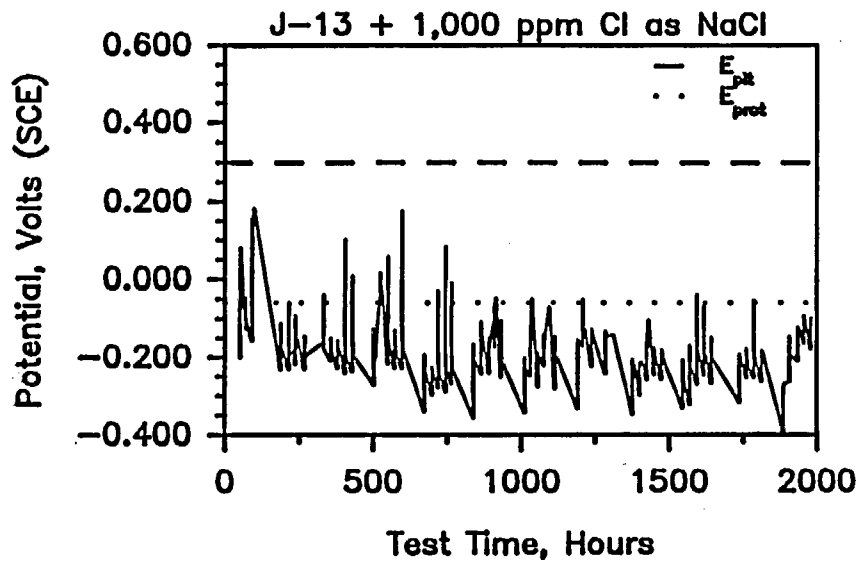
Barlow

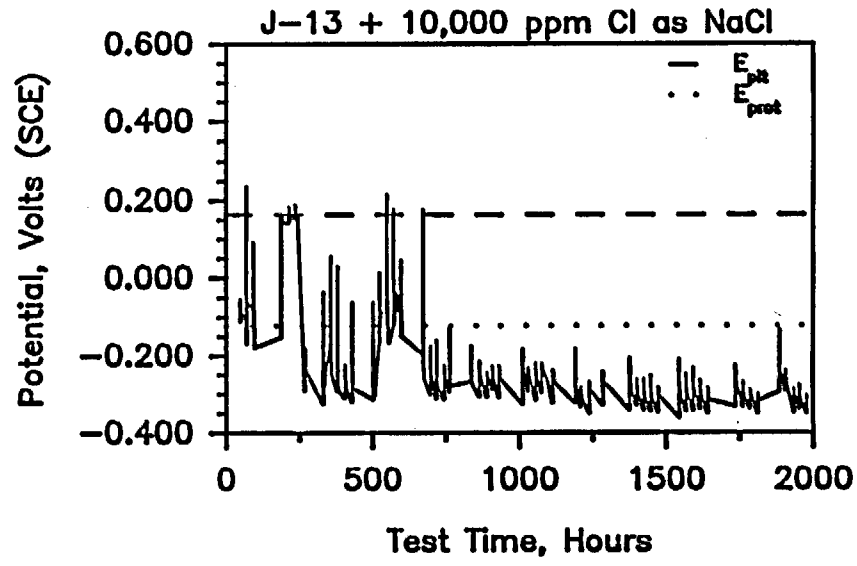
Long-Term

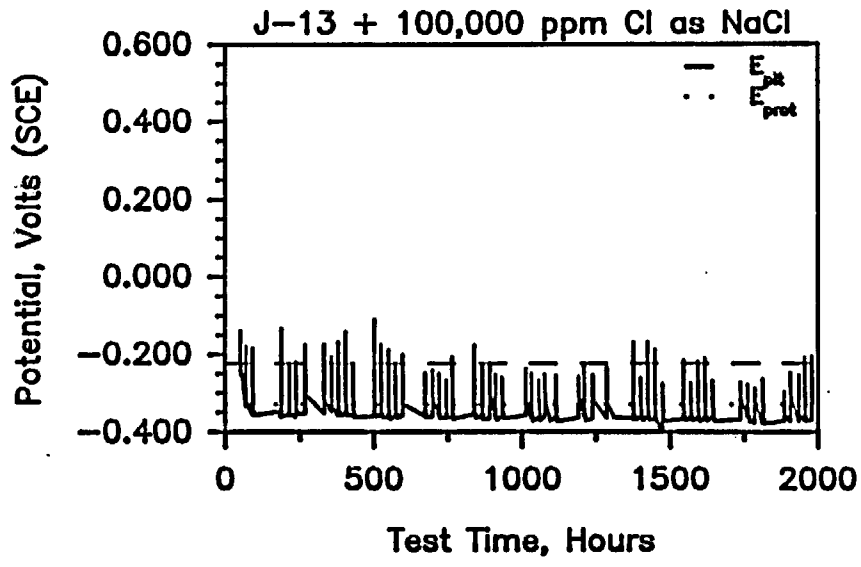
Table 14. Summary Of Results Of Exposure Of Creviced Copper-Base Specimens In Aerated Simulated J-13 Well Water At 90°C.

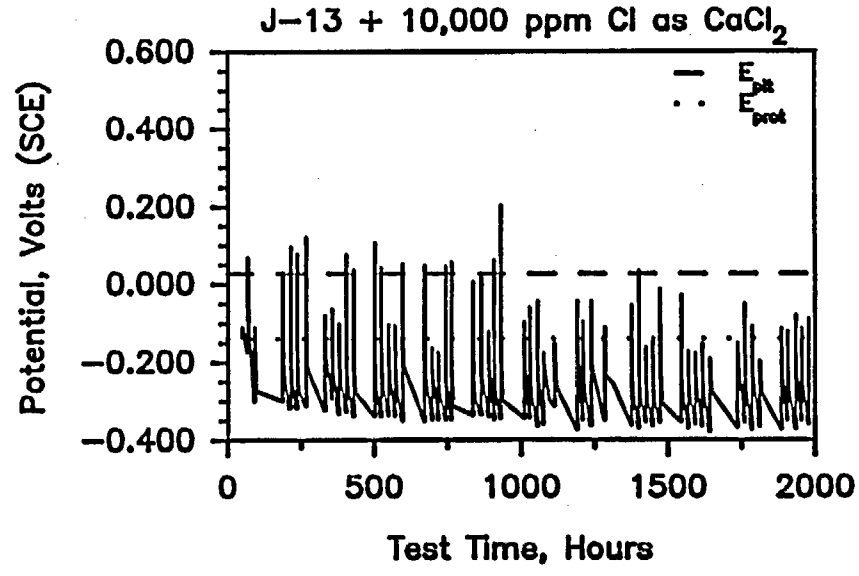
Alloy	Test Time Hours	Specimen Type	Corrosion Rate µm/yr	Maximum Pit Depth µm	Comments	Specimen
715	4034	Coupon	<0.02	---	Localized etching @ crevice. Very slight scaling.	32A,33A, 34A
715	8064	Coupon	0.02	---	Localized etching @ crevice. Very slight scaling.	35A,36A, 37A
102	4033	Coupon	1.16	---	Heavy etching; slight pitting on one specimen.	801,811, 821
102	8064	Coupon	0.65	---	Heavy etching.	831,841, 851

304L


J–13 + 1,000 ppm Cl as NaCl
Potential, Volts (SCE)
0.600
0.400
0.200
0.000
–0.200
–0.400
0
500
1000
1500
2000
Test Time, Hours
E_{pit}
E_{prot}


J–13 + 10,000 ppm Cl as NaCl
Potential, Volts (SCE)
0.600
0.400
0.200
0.000
–0.200
–0.400
0
500
1000
1500
2000
Test Time, Hours
E_{pit}
E_{prot}


J–13 + 100,000 ppm Cl as NaCl
Potential, Volts (SCE)
0.600
0.400
0.200
0.000
–0.200
–0.400
0
500
1000
1500
2000
Test Time, Hours
E_{pit}
E_{prot}


J–13 + 10,000 ppm Cl as $CaCl_2$
Potential, Volts (SCE)
0.600
0.400
0.200
0.000
–0.200
–0.400
0
500
1000
1500
2000
Test Time, Hours
E_{pit}
E_{prot}

FUTURE WORK

TASK 1 - COMPLETED

TASK 2 - EXPERIMENTAL WORK COMPLETED

- COMPLETE PREPARATION OF TOPICAL REPORT.

TASK 3 - COMPLETED

TASK 4 - EXPERIMENTAL WORK COMPLETED

- COMPLETE PREPARATION OF TOPICAL REPORT.

TASK 5 - CONTINUING SCC TESTS ON Fe-Cr-Ni ALLOYS. Add 825

- FOCUS ON ENVIRONMENTAL AND STRAIN-RATE DEPENDANCE OF SCC.

TASK 6 - COMPLETE BOREHOLE LINER-CONTAINER INTERACTION TESTS ON CARBON STEEL-ALLOY 304L.

- START-UP INTERACTION TEST ON ALLOY 304L AND ALLOY 825.

- EXAMINE EFFECTS OF HEAT TRANSFER ON CORROSION OF ALLOY 304L AND ALLOY CDA 102. Include 825

TASK 7 - CONTINUE LONG-TERM BOIL-DOWN TESTS.

- CONTINUE U-BEND SCC TESTS AS SUPPORT FOR TASK 5.

- Start up 825

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