

TEST REPORT

REMOVABILITY OF CORTEC VCI-309 VAPOR PHASE CORROSION
INHIBITOR FROM PIPING SYSTEMS

TENNESSEE VALLEY AUTHORITY

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1.0 Background

The process of replacing the feedwater heaters at Watts Bar Nuclear Plant has required the sectioning of the pipes within the system. Due to the extent and duration of this project, it was decided that a method should be employed to layup as much as possible the carbon steel piping and tanks.

A vapor phase corrosion inhibitor "Cortec VCI-309" was chosen as a method to retard the corrosion of the carbon steel surfaces. The inhibitor was applied as a powder on a weight-per-volume basis (20 grams/ft³) by dusting and piling the inside of the pipe and tanks. Once the surfaces have been exposed and all accesses to the pipes sealed, the inhibitor sublimates and forms a film on the surfaces which impedes the corrosion process.

Following the heater replacement, the system will be flushed to remove all contaminants. Information is needed on how well this inhibitor can be removed by water flushing and if any harmful residue will remain. It was decided that laboratory tests could provide valuable information on the removability and solubility of the inhibitor.

2.0 Introduction

The test was divided into two major tasks. The purpose of the first task was to determine quantitatively the levels of chlorides, sulfates, and phosphates contained in the inhibitor and the amount of those constituents remaining on the metal surfaces exposed to the inhibitor following water flushing.

The purpose of the second task was to determine the removability and solubility of the inhibitor by water under specified flow and temperature conditions.

A relationship between inhibitor concentration and specific conductance was investigated to aid in the removal determination.

3.0 Discussion

3.1 Task I

Carbon steel coupons were exposed to two concentrations of inhibitor solutions, 0.1 percent (w/w) and 1.0 percent. The coupons were allowed to air dry. Then each coupon was rinsed individually with demineralized water flow inside the test apparatus (figure 1, appendix A). The specific flow parameters were:

- a. Flowrate = 2.0 gallons/minute
- b. Water temperature = 70°F
- c. Rinse period = 5 minutes

One coupon exposed to the 1.0 percent solution was swipe tested without prior rinsing. All coupons were swiped and tested according to the procedure in appendix A. The results of the chloride, sulfate, and phosphate analyses are listed in appendix B.

3.2 Task II

Task II involves two subtasks. The first subtask was a determination of the solubility of the powdered inhibitor in water.

A measured amount of the inhibitor was placed in a known volume of water and timed until the inhibitor totally dissolved. The temperature of the test solutions was varied with some samples being agitated. The inhibitor samples used for this test were taken from a section of stored pipe at Watts Bar. The inhibitor had been exposed to moisture which tends to cause recrystallization and hardening. In addition, some grinding slag was mixed with the samples. The results are given in appendix C.

The second subtask was a measurement of the removal rate of the inhibitor from the test apparatus using a flow of water. Approximately 10 grams of the inhibitor were placed in section A of the test apparatus, then a 2-gpm flow was established across the test device. Specific conductance measurements were made at definite time intervals to indicate a removal rate. The test was repeated with the inhibitor being placed in section B of the apparatus.

The portion of the test involving removal of the material from section B exemplified a stagnant flow condition.

Because the possibility of charring some of the inhibitor during the reconstruction phase of the piping system exists, a test was needed to determine the removability characteristics of the charred residue. Several grams of the inhibitor were placed on two metal coupons and ignited. The coupons were placed in demineralized water. Visual observations were made to determine if the charred material could be easily removed. In addition, the conductance was checked on the water exposed to these coupons.

4.0 Conclusions

4.1 Quantitative Analysis

The results of the chloride, sulfate, and phosphate analyses are listed in appendix B. Examination of the results of the blank coupon and the unrinsed 1.0 percent exposed coupon indicate the inhibitor does not contribute contaminants. Appendix B also gives the results of the aforementioned contaminants contained within the inhibitor.

4.2 Solubility

The results of the solubility test (appendix C) indicate that the combination of heat and agitation significantly improve the rate of solubility on the inhibitor in water when compared to the rate achieved by heat or agitation applied separately. The tests were performed at an inhibitor concentration maximum of 0.25 percent. The manufacturer indicates that the inhibitor is soluble in water up to 10 percent (w/w) at 70°F.

4.3 Removability

Approximately 10 grams of inhibitor were placed in a pile in section B of the test apparatus (figure I). A 2-gpm flow was established across the device. Visual observations indicated that approximately one-half the inhibitor was removed during the time required to establish the flow (30 seconds). After 10 minutes of flow, the conductance was checked on the effluent. The conductance value (140 mhos) indicated no inhibitor was being removed. The water background conductance was 140 mhos. Visual observations showed a significant amount of the inhibitor was remaining. The flow was halted; a sample of effluent was taken as the device was being drained. A conductance check indicated that the effluent contained inhibitor (175 mhos). The tee was then filled and allowed to soak for 15 minutes. The flow was reestablished and the effluent conductance checked. The check (150 mhos) indicated a slight amount of inhibitor had been picked up. After an additional 10 minutes of flow, another check was made (140 mhos) which indicated no inhibitor was being removed; however, the inhibitor could be seen in section B.

Ten grams of inhibitor were placed in a pile in section A of the device. A 2-gpm flow was established across the test device. Effluent samples were taken and checked for conductance at 1-minute time intervals (see appendix E). The results indicate that the movement of the water during the initial filling removed a large portion of the inhibitor; however, once the flow was stabilized, decreasing quantities of the inhibitor were being removed. After 6 minutes of flow, the test device was agitated and effluent samples taken and checked. The checks indicated that inhibitor was being removed. At this point, the agitation was halted and effluent samples checked. The checks indicated no inhibitor was being removed. The internals of the test device were examined--showing no inhibitor remained.

4.4 Charred Residue

The majority of the inhibitor placed on the coupons and ignited volatilized--leaving only a minute amount of residue.

The coupons with the charred residual which was loose and easily removed were visually examined then immersed in demineralized water. A portion of the loose material and the two coupons were agitated in the water, and the conductance of the solutions were checked. The results (appendix F) indicate the residue does not significantly contribute to the conductivity of the water. The ease of removability demonstrated by the test indicates that water flushing should be an acceptable method to remove the majority of charred residue from carbon steel surfaces.

Attachments

DSH:WDA

B2351C.WA

PART I

RESIDUAL CHLORIDES, SULFATES, AND PHOSPHATES
ON CARBON STEEL AFTER EXPOSURE TO VCI-309
A VAPOR PHASE CORROSION INHIBITOR

I. Purpose

The purpose of the test is to determine quantitatively the levels of chloride, sulfate, and phosphate remaining on carbon steel test coupons after exposure to Cortec VCI-309 vapor phase corrosion inhibitor. In addition, this test will determine if residual inhibitor remains on the tested surfaces after water rinsing.

II. Procedure

A. Preparation of Test Solutions

Prepare two concentrations of Cortec VCI-309. A 0.1% (w/w) solution is prepared by adding 1 gram of Cortec concentrated powder to a liter of deionized water. Mix thoroughly. Prepare a 1% solution by add 10 grams of Cortec VCI-309 to a liter of deionized water. Mix thoroughly.

B. Preparation of Test Coupons

1. Thoroughly rinse five 2-inch by 2-inch test coupons with DI water.
2. Allow coupons to dry.
3. Using tongs, immerse two test coupons in the 0.1% Cortec solution.
4. Using tongs, immerse two test coupons in the 1.0% Cortec solution.
5. Let coupons drain vertically and dry for 30 minutes.
6. The fifth coupon will be used as a control (blank).

C. Sampling

Clean opaque polyethylene or rubber gloves will be worn when preparing the samples for analysis. (Clear, powder-free polyethylene gloves may be used provided they are used in the laboratory only.)

D. Apparatus

1. Hotplate
2. Filter papers - Whitman No. 41
3. Funnels - analytical, short stem
4. Beakers - 250 ml and 2 l
5. Water glasses - medium (for 250 ml beakers)
6. Syringe - 50 ml disposable

7. Vials - at least 16-dram capacity with plastic "snap" cap, opaque
8. Rubber sponges - approximately 1 inch by 1 inch (4 per sample swipe)
9. Forceps - stainless steel (at least 8 inches in length)
10. Clean polyethylene or rubber gloves, opaque
11. Flasks - 500 ml Erlenmeyer flasks

E. Sponge Preparation

Prepare the sponges for sampling as follows:

1. Place a number of sponges in a large beaker, add demineralized water, and press the air from the sponges by compression.
2. Refill the beaker to cover the sponges and again compress the sponges, then fill the beaker to within approximately one inch from the top.
3. Bring the water in the beaker to a boil on the hot plate and continue to boil for about 15 minutes.
4. Pour off the excess water.
5. Repeat steps 2, 3, and 4 twice more, except do not pour off the water after step 4 on the third boil.
6. After the third boiling, transfer 20 clean sponges to a 50 ml syringe and compress the water into a clean 500 ml Erlenmeyer flask. Draw additional water from the beaker containing the sponges again using the syringe and add this volume to the flask. Add the additional water from the sponge beaker to the flask to bring the water volume to approximately 200 ml (using the graduations on the flask).
7. Cool and analyze the contents of the flask as described in section III. The acceptable value for the sponge rinse water is 0.05 mg/l Cl. If this value is exceeded, repeat steps 2, 3, and 4 until the acceptable value is obtained. When the rinse water is 0.05 mg/l Cl, the sponges are considered chloride free.
8. Place the clean chloride-free sponges in a pre-numbered sample vial, four per vial.

F. Rinse coupons with DI water.

- a. Rinse each coupon separately in approximately 250 ml of DI water (pH = 7) and save rinses.

- b. Rinse each coupon again separately in approximately 250 ml of DI water (@ pH = 9-10 adjusted with NH_4OH) and save rinses.
 - c. Pour rinses into preweighted evaporating dishes and allow each to evaporate to dryness.
 - d. Weigh residue and save for infrared analyses and/or gas chromatograph.
 - e. Place coupons in the test "tee" shown in Figure II.
 - f. Using a small laboratory pump (2 gpm), circulate DI water of known conductivity through the test "tee" for 4 minutes then test recirculation water for conductivity increase.
 - g. Again recirculate the DI water for 4 minutes and record conductivity.
- G. Obtain the swipes as follows:
1. Swipe both sides of the metal coupon, covering the total 8 in² area.
 2. Swipe the area by moistening one of the four sponges in the bottle with demineralized water and swiping the designated area, then swipe the same area with a dry sponge.
 3. Repeat step 2 with the other two sponges and place all four sponges back in the sample vial.
 4. Repeat steps 1, 2, and 3 as required for each of the remaining sample points using a different set of four sponges for each sample.
- H. Prepare the samples for analyses as follows:
1. Place the four sponges from each sample in a 250 ml beaker and add approximately 100 ml of demineralized water. Rinse the sample vial and add these rinsings to the beaker as part of the 100 ml.
 2. Place the beaker(s) on the hot plate and bring to near boiling (95°-100°C) for approximately 10 minutes. Cover the beakers with a watch glass during this time.
- Note: Perform filtration as described in steps 3 through 7 only if the sample is turbid or cloudy.
3. Cool and filter the solution in each beaker through a rinsed No. 41 Whatman filter paper into a clean 500 ml Erlenmeyer flask. Rinse the watch glasses with demineralized water and filter the rinse water.

4. Using the stainless steel tongs, place the sponges in a 50 ml syringe and compress any water in the sponge through the filter.
5. Aspirate 20 to 30 ml of demineralized water into the syringe containing the sponges and compress the water through the filter to the 500 ml flask containing the sample.
6. Repeat step 5.
7. Rinse the beaker several times with demineralized water and filter the washings.
8. Bring the volume to 200 ml with demineralized water using the graduation markings on the side of the flask.
9. The sample is ready for analysis as described in section III.
10. For each sample or series of samples, perform steps 1 through 8 for a set of four clean sponges as a blank.

Note: Clean all glassware with 50% HNO₃.
 Also, a blank test coupon should be run to ensure that no contamination is in the system.

III. Analysis (Calculations)

- A. Determine the sulfate, chloride, and phosphate levels in the extracts by ion chromatography then calculate the levels of each species on a weight per unit area basis as follows:

$$\text{Chloride} \quad \text{mg} = \frac{(C_s - C_b) \times VT}{A} \text{ dm}^2$$

- where C_s = chloride concentration of sample (mg/l)
 C_b = chloride concentration of sponge blank (mg/l)
 VT = total volume of sample (0.2 l)
 A = area of wiped surface (0.5 dm²) = 8 in²

Determine other species same as above.

- B. Analyze the residue from the evaporating dishes and an aliquot of the swipe solution by infrared spectroscopy at expected IR bands (3413, 1632, 1590, 1516, 1443, 1370, 1066, 1042, 820, 720, and 690 cm⁻¹) or by gas chromatography.

(cont.)

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File _____

ANALYSIS REPORT
Central Laboratories
Chattanooga Power Service Center

Lab Sample No. 82-5247

Submitted by Mark Koss

SAMPLE DESCRIPTION

(Sample C-131) - Cortec VCI 309 - Lot 112480

TEST RESULTS

Chloride - <10 ppm

Fluoride - <5 ppm

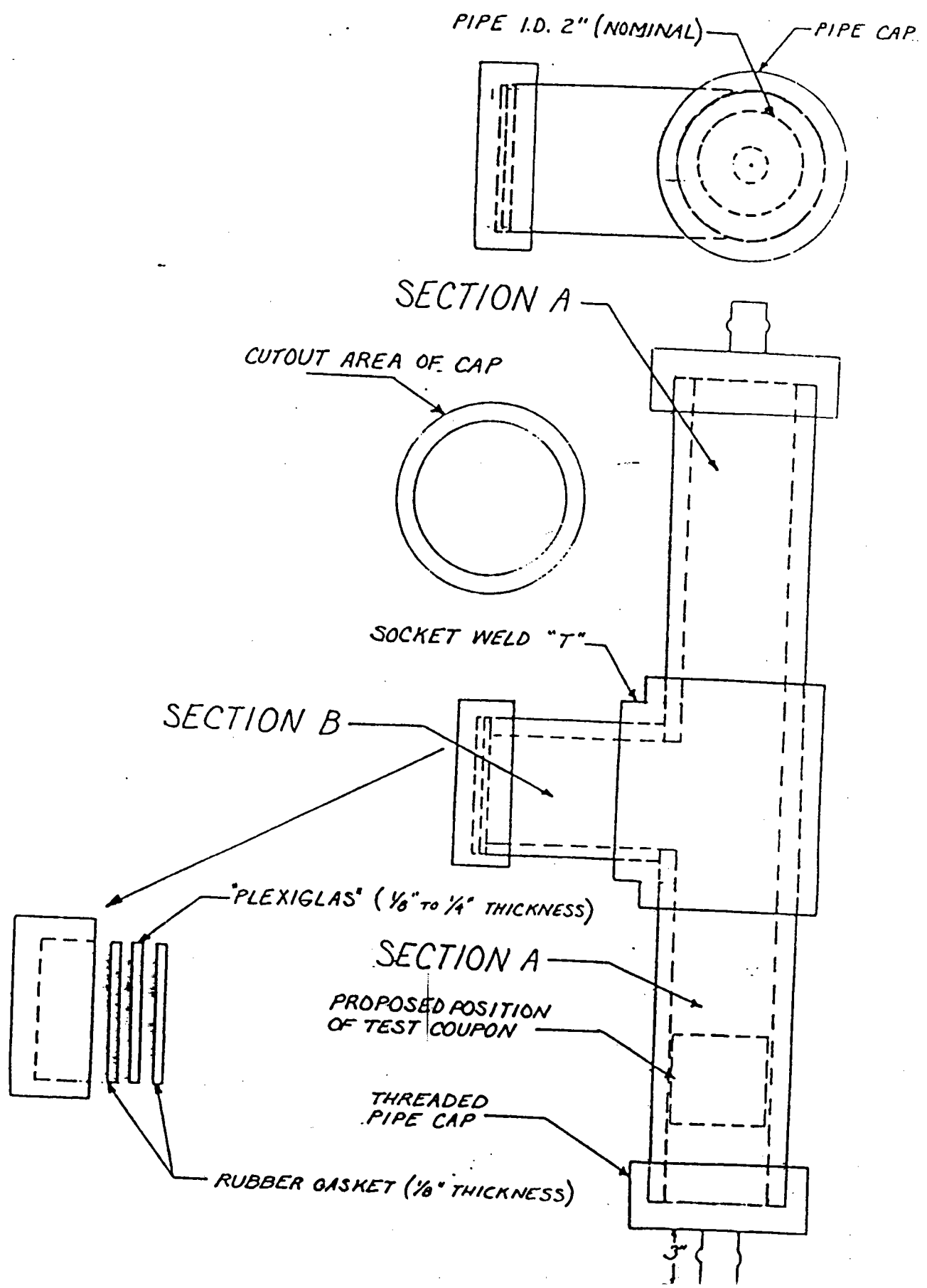
Sulfur - <25 ppm

Reported by Elizabeth B. Smith

Date July 1, 1982

Distribution: David Goetcheus, 1410 CST2-C
Mark Koss, 1410 CST2-C

APPENDIX A, PART II
FIG. I



SAMPLE	Cl (ppb)	PO ₄ (ppb)	SO ₄ (ppb)
RINSE (0.1% Coupon)	49	< 5	13
RINSE (1.0% Coupon)	56	< 5	12
BLANK COUPON	192	< 5	46
0.1% COUPON	207	< 5	25
1.0% COUPON	190	< 5	38
* 1.0% COUPON	200	< 5	23

* Swiped without rinsing

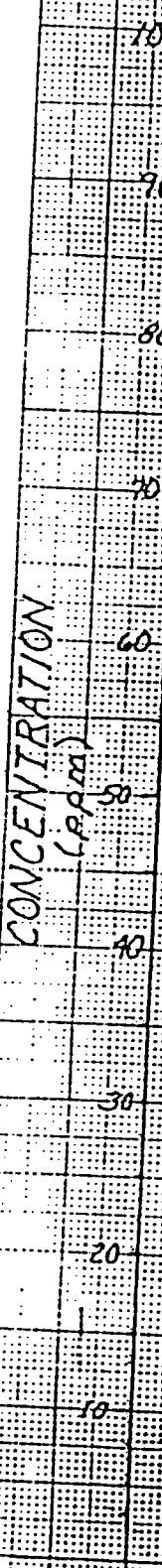
APPENDIX C

SOLUBILITY

Amount (g) of Cortec K1-309	Volume (ml) of H ₂ O	Time required for dissolution	Temperature of H ₂ O (°F)	Agitation (yes or no)
.5	500	2 Hrs.	70	NO
2.0	800	< 25 sec	148	YES
0.8	800	< 10 min.	150	NO
1.9	800	8.5 min.	70	YES

APPENDIX D
PART I

CONCENTRATION
(ppm)



LEGEND

- - - - - O - - - - - Least Squares Line
- - - - - O - - - - - Actual Line

DATA

Sp. Condu.	Conc.
1.68	1.5
3.28	10
14.2	50
25.0	100

SPECIFIC CONDUCTANCE
(micmhos)

12-14-82
CFM

APPENDIX D PART 2

CONCENTRATION
(ppm)

10,000
9000
8000
7000
6000
5000
4000
3000
2000
1000

200 400 600 800 1000 1200 1400 1600 1800 2000

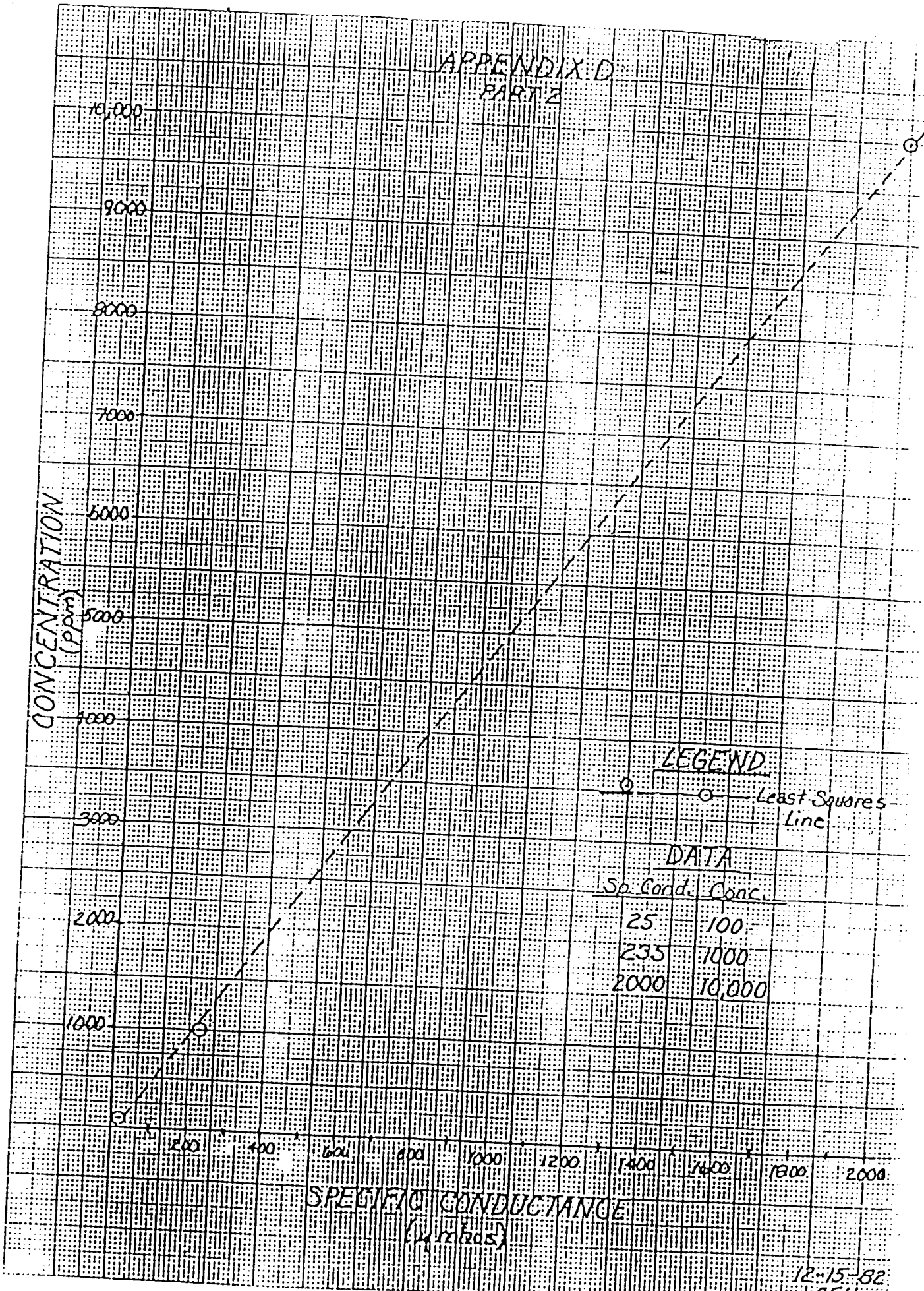
SPECIFIC CONDUCTANCE
(μ mhos)

LEGEND

○ Least Squares Line

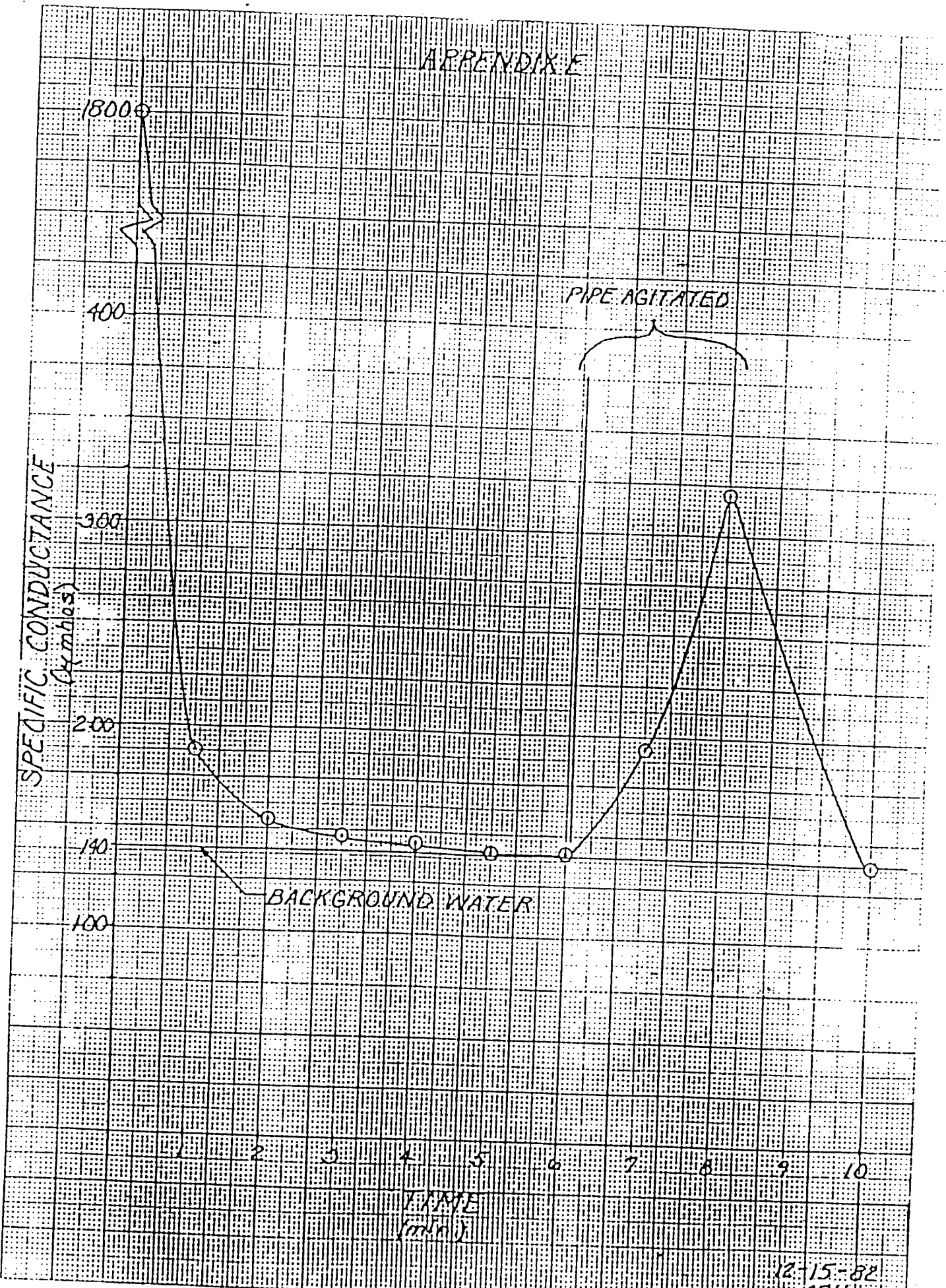
DATA

Sp. Cond.	Conc.
25	100
235	1000
2000	10,000



12-15-82
CFH

APPENDIX E



12-15-82
VFH

APPENDIX F

<u>SAMPLE</u>	<u>CONDUCTANCE (μmhos)</u>
DEMIN. WATER	.74
LOOSE RESIDUE	1.58
COUPON # 1	1.25
COUPON # 2	1.25