

ANALYTICAL PROCEDURES FOR PER/TRI PLANT LABORATORY

1. Test

Moisture Test (Cloud Point Method)

2. Samples

No. S-1 Mixed Organic
No. S-2 Recycle Tank
No. S-3 DH Reflux
No. S-4 DH Bottoms
No. S-5 Dopp Condenser
No. S-6 Heavies Reflux
No. S-7 Per Reflux
No. S-8 Per/Tri Reflux
No. S-9 Tri Reflux Before
No. S-10 Tri Reflux After
No. S-11 Topping Reflux
No. S-12 Dried Per
No. S-13 Dried Tri

3. Apparatus and Reagents

a. Isopropyl Alcohol
b. Dry Ice
c. Thermometer, -36°C to 54°C, 0.2°C Divisions

4. Procedure

a. Place about 20 mL of sample in a clean, dry test tube and immediately stopper with a cork containing a thermometer. The thermometer bulb should reach within 1/4 inch of the bottom of the test tube.

b. The test tube should be cooled down until a cloud first appears in sample. It should then be warmed up and cooled down once or twice more to the cloud point to make the end point more defined.

5. Report

Temperature of initial cloud formation unless it is below -15°C. If the cloud point is below -15°C, report less than -15°C.

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OPERATION OF MITSUBISHI MOISTURE METER MODEL CA-05

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1. Turn power switch on left side of instrument to the ON position.
2. Move Speed Adjust control to 6 on stirrer.
3. If titration speed (on left side of the display) indicates a negative number, inject 1/4 mL of the check solution or 1 mL of a sample and depress the titration button. The titration will begin indicating WAIT. After it has corrected the negative number, an electronic buzzer will sound and the display will display "END". After this, depress the CL button and you are now ready to go to step 4.
4. At this point, depress the Sample button and then the ENT button until a V appears on the display. Then on the keyboard enter the size sample you intend to use (1 mL) and depress ENT again. This will cause SG to come up on the display which stands for specific gravity. Enter the desired specific gravity on the keyboard and depress ENT again. At this point depress the CL button and display will indicate RDY.

Titration of Sample:

1. Rinse the syringe supplied with sample 4-5 times before drawing up the proper amount of sample to be injected into the titration flask.
2. Before injecting the sample into the titration flask, make sure the RDY is displayed on the display. If so, depress the Start button and "Add Sample" will come up on the display. At this point inject the sample into the titration flask. It will automatically start to count out the μg of H_2O . When the buzzer sounds, it will display μg of H_2O and also print out ppm H_2O .

Changing Solutions:

1. Changing C Solution:
 - a. Move speed control (#8) to zero and allow stirring bar (#7) to come to a complete stop.
 - b. Remove drying tube (#1) from cathode solution cell (#3). Using a Dropper Pipette draw out all of the solution in the cathode solution cell (#3).
 - c. Using a funnel inserted into the hole for the drying tube (#1) on the cathode solution cell (#3), pour the contents of one ampule of Aquamicon C.
 - d. Replace drying tube (#1) back into cathode solution cell. (Make sure drying tube has fresh Drierite in it.)
 - e. Move speed control knob on stirrer back to 6 on scale.
 - f. Depress titration button. The titration will begin indicating Wait. After it has dried out the new solution, the buzzer will sound and the display will display End. After this, depress the CL button and you are now ready to go to step 1 of "Titration of Sample."

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2. Changing A Solution:

- a. Move speed control (#8) to zero and allow stirring bar (#7) to come to complete stop.
- b. Open drain port (#6) and allow all of the solution to drain out.
- c. Close drain port (#6).
- d. Remove blind plug (#10) from top of titration cell assembly (#4). Using a funnel, add all of the contents from a Generator Solution A (Anode) bottle to the titration cell assembly (#4) and replace the blind plug (#10).
- e. Be sure to keep fresh Drierite in drying tube #2.
- f. Move speed control knob on stirrer back to 6 on scale.
- g. Depress the titration button. The titration will begin indicating Wait. After it has dried out the new solution, the buzzer will sound and the display will display END. After this depress the CL button and you are now ready to go to step 1 of "Titration of Sample".

Troubleshooting:

1. Open detector indicated.
 - a. Unplug and plug back pin jacks (11,12).
 - b. Turn power off and back on at Power Switch.
 - c. Depress formula key (F), then depress Q on the keyboard and then enter. (This will clear out any error in the calculation formula.)
 - d. Depress formula key (F) again, then depress 5 on the keyboard and then enter. (The correct formula is now in the instrument.)
 - e. Depress print key, then depress 3 on the keyboard and then enter.
 - f. You are now ready to go to step 4 of starting conditions.
2. No end point or stops titrating.
 - a. Change solutions A and C as described in "Changing Solutions".

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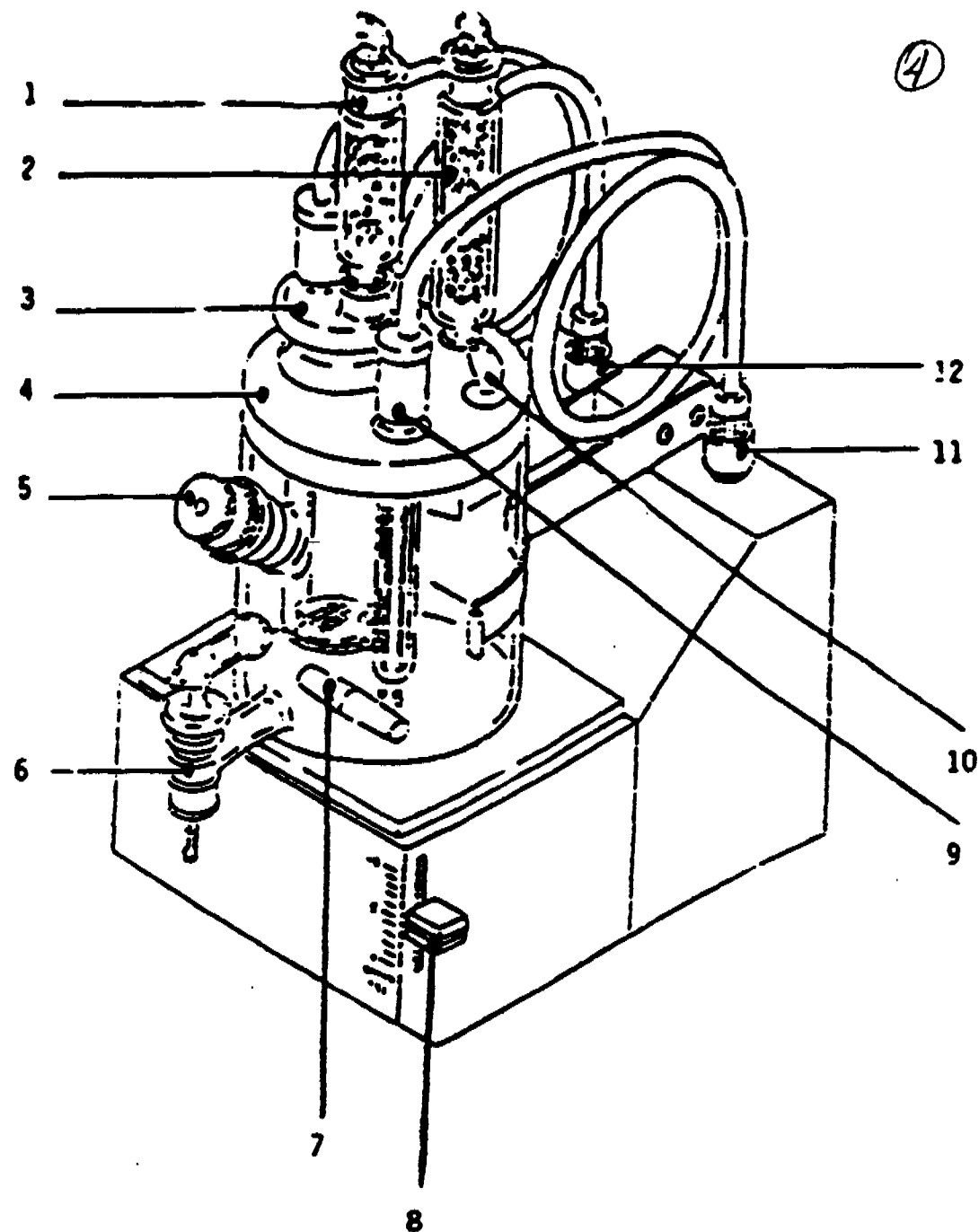


Fig. 3 Cell Unit

- 1-drying tube
- 2-drying tube
- 3-cathode solution cell
- 4-titration cell assembly
- 5-sample injection port
- 6-drain port
- 7-stirring bar
- 8-speed control for stirrer
- 9-electrode
- 10-blind plug

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SPECIFIC GRAVITY
FOR H₂O TITRATIONS

<u>Sample</u>	<u>Specific Gravity</u>
TBL	0.781
NPL	0.806
BLM	0.831
DME	0.861
EC	0.924
VCM	0.969
VDCM	1.212
EDC	1.262
Furnace Feed	1.262
MC	1.326
-314	1.310
-324	1.326
-343	1.308
-348	1.310
-366	1.320
-368	1.625
PER	1.625
-232	1.625
-208	1.625
-229	1.625
-230	1.468
TRI	1.468
-113	1.462
-127	1.465
-138	1.465
-143	1.465
-145	1.460
-147	1.321
MeCl ₂	1.5536
Unsymmetrical	
Mix	
DOL/SBL/DOX	0.930
NRE/MC	1.124
366/208	1.385
366/230	1.460
Dowtherm	1.022
Heavies Still Bottoms	1.330

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY1. Test

pH of Dried Unstabilized Per

2. Sample

No. S-1 Dried Unstabilized Per

3. Apparatus and Reagents

- a. pH meter with electrode assembly
- b. 0.01 N Sodium Hydroxide (approximately)
- c. 0.01 N Hydrochloric Acid (approximately)
- d. Neutral Distilled Water (pH - 7.0)

4. Procedure

- a. Adjust pH of about 50 mL of neutral water to 7.0. (Note D)
- b. Measure 100 mL of sample into a 250-mL separatory funnel. Add 25 mL of neutral water. Shake for two (2) minutes venting as required.
- c. Allow phases to separate and discard organic phase.
- d. Drain aqueous layer into a 150-mL beaker, place on stirrer and allow water phase to stir for 30 seconds with electrode in solution and meter on before taking reading.

5. Report

Report pH of sample as read from meter.

6. Notes

- a. Check standardization of pH meter once each shift. Use pH 7.0 buffer. The buffer should be changed each day, but it is not normally necessary to change it each time the meter is checked. Keep buffer in a tightly stoppered bottle.
- b. Always keep pH electrodes in distilled water when meter is not being used.
- c. Always keep saturated KCl solution in the calomel electrode. Do not cover hole with rubber band.
- d. Use fresh Neutral H₂O each time a set of samples are run, and adjust pH of neutral H₂O just before using. There should be a slow drift through 7.0. This drift should not be less than 10-15 seconds per 0.1 pH reading.

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KIRK. HERE ARE A FEW THINGS THAT MAY CLEAR UP SOME OF OUR PROBLEMS. IF THERE ARE ANY QUESTIONS GIVE ME A CALL

Rickey

The pH instrument should be buffered once a shift.

1. Check and make sure the temperature is set at 25 degrees centigrade.
2. Buffer instrument with buffer 7 first. Then check with buffer 4 and buffer 10. A reading of 4.2 or lower and 9.8 or higher should be observed. If not, check (and fill if necessary) the KCl solution in the reference electrode. If there is still a problem, check for crystals (which may be interfering) in the reference electrode. At this point an instrument person should be contacted to insure proper cleaning and refilling of electrode.

*ANY TIME ELECTRODES ARE REMOVED FROM LIQUID, THE METER SHOULD BE IN THE STANDBY POSITION.

Keep the meter in the standby position when not in use.

Do not leave the electrodes immersed in the sample.

Keep the electrodes in clean distilled water when not in use.

It is very important to follow the job breakdown as written. (Shake the proper length of time, take a reading after 30 seconds, ect.) No exceptions.

Do not neutralize large quantities of "neutral" water and let sit. "Neutral" water should be neutralized just prior to use.

There should be a good response when .01 N NaOH is added to neutralize the "neutral" water. This means there should be noticable needle deflection. No more than a couple of drops (2-4) of .01 N NaOH should be needed to neutralize a volume of 100 mls. of water. If more is needed, the dropper bottle should be emptied, rinsed with distilled water, and filled with fresh .01 N NaOH. If there is still poor response, the "neutral" water is suspect and should be changed out.

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FILENAME: PHPROB

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY

1. Test

pH of Dried Unstabilized Tri

2. Sample

No. S-1 Dried Unstabilized Per

3. Apparatus and Reagents

- a. pH meter with electrode assembly
- b. 0.01 N Sodium Hydroxide (approximately)
- c. 0.01 N Hydrochloric Acid (approximately)
- d. Neutral Distilled Water (pH = 7.0)

4. Procedure

- a. Measure 75 mL of neutral water into a 250-mL beaker and adjust pH to 7.0 (Note D).
- b. Raise electrodes and pipette 25 mL of sample into beaker and mix well with stirrer for one minute.
- c. Slow stirrer and allow phases to separate.
- d. Lower electrodes into aqueous phase but not into organic phase.
- e. Wait a few seconds and switch meter to proper range and read pH.

5. Report

Report pH of sample as read from meter.

6. Notes

- a. Check standardization of pH meter once each shift. Use pH 7.0 buffer. The buffer should be changed each day, but it is not normally necessary to change it each time the meter is checked. Keep buffer in a tightly stoppered bottle.
- b. Always keep pH electrodes in distilled water when meter is not being used.
- c. Always keep saturated KCl solution in the calomel electrode. Do not cover hole with rubber band.
- d. Use fresh Neutral H₂O each time a set of samples are run, and adjust pH of neutral H₂O just before using. There should be a slow drift through 7.0. This drift should not be less than 10-15 seconds per 0.1 pH reading.

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY

1. Test

Bromothymol Blue Test for Acidic Impurities

2. Samples

No. S-1 Dried Per

No. S-2 Tri Before Dryer

3. Apparatus and Reagents

- a. 35-mL glass vials with plastic polyseal caps
- b. Bromothymol blue indicator solution

4. Procedure

- a. Pipette 10 mL of sample into a clean glass vial containing 10 mL of the bromothymol blue solution.
- b. Cap with the plastic cap and shake vigorously.
- c. Allow phases to separate.
- d. Compare the color of the aqueous (top) phase to the color standards.

5. Report

Sample Color

yellow - pH below 6.0; increase ammonia addition

green - pH between 6.0 and 7.5

blue - pH greater than 7.5; decrease ammonia addition

6. Notes

- a. The bromothymol blue indicator solution must remain green when placed in the empty glass vial. A change of color could indicate dirty glassware.
- b. Any sample color in the green range (chartreuse through blue-green) indicates a good sample and no change is needed in the neutralizer.

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MC ANALYTICAL CONTROL TEST1. Test

Color of Product

2. Samples

- a. Dry Per
- b. Dry Tri
- c. 1 Per Bottoms
- d. 2 Per Bottoms

3. Apparatus and Reagents

- a. Hellige Aqua Tester
- b. Nessler Tube with Plunger
- c. Hellige Color Discs

4. Procedure

- a. Fill one Nessler tube to mark with distilled water.
- b. Fill one Nessler tube to mark with sample.
- c. Place sample in the right position of comparator and distilled water in left position of comparator.
- d. Insert plunger in each tube.
- e. Rotate disc until color intensity matches.
- f. Record the APHA color of sample.

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MC ANALYTICAL CONTROL TEST1. Test

Sodium Iodide Test

2. Sample

No. S-1 Dried Unstabilized Per
No. S-2 Dried Unstabilized Tri

3. Apparatus and Reagents

- a. 2.5% Sodium Iodide in Acetone
- b. 0.01 N Sodium Thiosulfate
- c. Acetic Acid

4. Procedure

- a. Measure 50 mL of sample and pour into a 250-mL flask. Add 40 mL of 2.5% sodium iodide-acetone solution and 4 drops of acetic acid.
- b. Loosely stopper and place in dark for 30 minutes.
- c. At the end of 30 minutes observe for any yellow color formation. Add 50 mL of ice water to sample with color and place on magnetic stirrer.
- d. Titrate colored sample with 0.01 N $\text{Na}_2\text{S}_2\text{O}_2$. The disappearance of all color is the end point.

5. Calculations

$$\text{a. ppm Perchlor-Oxide} = \frac{T \times N \times 0.0491 \times 10^6}{50 \times 1.62}$$

$$= T \times N \times 1123.4$$

$$= T \times 11.23$$

$$\text{b. ppm Trichlor-Oxide} = \frac{T \times N \times 0.0291 \times 10^6}{50 \times 1.46}$$

$$= T \times N \times 672.3$$

$$= T \times 6.723$$

6. Typical Results

No Color Formation

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY1. Test

Iron Determination

2. Samples

- a. Heavy Still Bottoms
- b. Still Bottoms
- c. Heavy Still Feed
- d. Heavy Still Reflux

3. Apparatus and Reagents

- a. pH meter
- b. PC-600
- c. 60-mL Separatory Funnel
- d. Filter Paper - Whatman No. 541
- e. 520 mm filter
- f. 0.03% ortho-phenanthroline (water matrix)
- g. 5% hydroxylamine hydrochloride (water matrix)
- h. 1:1 Solution of HCl
- i. 6 N Solution of NH_4OH

4. Procedure

- a. In a hood, pipet 25 mL of hydroxylamine hydrochloride into a 60-mL separatory funnel.
- b. Pipet the appropriate sample size into the 60-mL separatory funnel also. (Note 2)
- c. Shake separatory funnel vigorously for 3 minutes to extract the iron from the sample. (3 minutes)
- d. Filter sample mixture through a No. 541 filter paper (that has been moistened with distilled water).
- e. Dilute sample to 50 mL and adjust pH to 3 if necessary.
- f. Add 5 mL of ortho-phenanthroline to solution.
- g. Dilute to 80 mL and adjust pH again to 3.3-3.7 with either 1:1 HCl or 1:1 NH_4OH .
- h. Pour ⁴solution into a 100-mL volumetric and bring to volume with distilled water.
- i. Let stand for 15 minutes.
- j. Blank PC-600 with a reagent blank.¹
- k. Place probe into sample, take reading and record.

CalculationLook up ppm FeCl_3 on chart provided with electrophotometer (Note 3).

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Iron Determination (Continued)NOTES

1. Blank: make blank same as the sample only do not use sample.
2. Sample Size

<u>ppm FeCl₃ Expected in Sample</u>	<u>Sample Volume (mL) to Use</u>
2 ppm	10 mL
2-15 ppm	5 mL
5-50 ppm	2 mL
10-70 ppm	1 mL
20-150 ppm	0.5 mL
100-1000 ppm	0.1 mL
3. If FeCl₃ exceeds limits on chart, rerun sample using a smaller aliquot.

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BRINKMAN PC/600 CHART FOR FECL3

25 ML HYDROXYLAMINE HCL
5 ML O-PHENANTHROLINE

ABSORBANCE READING	MG FE	ABSORBANCE READING	MG FE
0.01	1	0.31	44
0.02	3	0.32	46
0.03	4	0.33	47
0.04	6	0.34	49
0.05	7	0.35	50
0.06	9	0.36	51
0.07	10	0.37	53
0.08	11	0.38	54
0.09	13	0.39	56
0.10	16	0.40	57
0.11	16	0.41	59
0.12	17	0.42	60
0.13	19	0.43	61
0.14	20	0.44	63
0.15	21	0.45	64
0.16	23	0.46	66
0.17	24	0.47	67
0.18	26	0.48	69
0.19	27	0.49	70
0.20	29	0.50	72
0.21	30	0.51	73
0.22	31	0.52	74
0.23	33	0.53	76
0.24	34	0.54	77
0.25	36	0.55	79
0.26	37	0.56	80
0.27	39	0.57	82
0.28	40	0.58	83
0.29	41	0.59	84
0.30	43	0.60	86

FACTOR X MG FE = PPM FECL3

SAMPLE SIZE	FACTOR
0.1	18.125
0.2	9.063
0.3	6.042
0.4	4.531
0.5	3.625
1.0	1.813
2.0	0.9063
5.0	0.3625

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ABSORBANCE READING	MG FE 5.0 ML	0.5 ML
1	0.36	3.63
3	1.08	10.88
4	1.45	14.50
6	2.17	21.75
7	2.53	25.38
9	3.26	32.63
10	3.62	36.25
11	3.99	39.88
13	4.71	47.13
16	5.80	58.00
17	6.16	61.63
19	6.89	68.88
20	7.25	72.50
21	7.61	76.13
23	8.34	83.38
24	8.70	87.00
26	9.43	94.25
27	9.79	97.88
29	10.51	105.13
30	10.88	108.75
31	11.24	112.38
33	11.96	119.63
34	12.33	123.25
36	13.05	130.50
37	13.41	134.13
39	14.14	141.38
40	14.50	145.00
41	14.86	148.63
43	15.59	155.88

ABSORBANCE READING	MG FE 5.0 ML	0.5 ML
44	15.95	159.50
46	16.68	166.75
47	17.04	170.38
49	17.76	177.63
50	18.13	181.25
51	18.49	184.88
53	19.21	192.13
54	19.58	195.75
56	20.30	203.00
57	20.66	206.63
59	21.39	213.88
60	21.75	217.50
61	22.11	221.13
63	22.84	228.38
64	23.20	232.00
66	23.93	239.25
67	24.29	242.88
69	25.01	250.13
70	25.38	253.75
72	26.10	261.00
73	26.46	264.63
74	26.83	268.25
76	27.55	275.50
77	27.91	279.13
79	28.64	286.38
80	29.00	290.00
82	29.73	297.25
83	30.09	300.88
84	30.45	304.50
86	31.18	311.75

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY
METHOD A-LC-11

1. Test

Dew Point Using Alnor Dew Pointer

2. Sample: Plant Air

3. Apparatus: Alnor Dew Pointer with dew point calculator

4. Procedure

- a. Purge out sample line by opening sample valve to allow an air flow.
- b. Open instrument case. Pull out operating valve handle; open purging valve; turn switch to battery position.
- c. Connect sample line to inlet connection and purge instrument with plant air. Let purge under pressure for one minutes. Operate pump at last 5 times.
- d. Close purging valve. Operate pump until pressure ratio gauge read about 0.3. Press operating valve handle and then pull out. Open purging valve. Repeat this step at least 4 times.
- e. Disconnect sample line or complete close sample valve.
- f. With the index finger tip, depress the gauge valve. While the gauge is depressed, turn the unit adjuster to bring the bottom of the meniscus even with the 1 mark on the gauge. Release the gauge valve.
- g. Repeat Steps B, C and D.
- h. Pump up sample to a pressure ratio of about 0.45; make test as in Step D. Look into the observation window as the operating valve handle is pushed in. Note whether a cloud is formed. If not, repeat this procedure by going to successively lower pressure ratios (0.45, 0.44, 0.43 ... 0.38, 0.37, 0.36 ... 0.33, 0.32, 0.31) each time a fog or cloud is noticed. Repeat at the same pressure ratio. If fog repeats, this is the end point. If not, continue until the end point is reached.
- i. Read thermometer.
- j. Set arrow number 2 on dew point calculator opposite the end point pressure ratio on Scale D. Find the thermometer temperature on Scale F. Read dew point on Scale E opposite thermometer temperature on Scale F.
- k. Leave a small purge on sample line.

5. Report: Dew point temperature from dew point calculator

6. Plant air dew point of 40°F.

NOTE: Step j is to be used only when running dew points on plant air. Check with the Laboratory when other samples are to be run.

7. Change glass wool in Dew-Pointer filter weekly.

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY
METHOD A-LC-11

1. Test

Determination of Specific Gravity by Hydrometer

2. Sample

Refrigerated Brine

3. Interferences

Insoluble materials and suspended air bubbles

4. Apparatus

- a. Hydrometer, 1.200-1.400 Specific Gravity
- b. Thermometer, -40° to 120°F

5. Procedure

- a. Fill graduate with sample, being careful not to entrain air and form bubbles.
- b. Place hydrometer in sample slowly. Do not allow hydrometer to sink much below the final reading. Read at the highest point the liquid comes on the hydrometer.
- c. Measure temperature of sample. Leave thermometer in sample at least one minute.

6. Calculations

- a. Determine the temperature correction for the sample from the correction table.
- b. Determine the freezing point from the table using the corrected specific gravity.
- c. Report corrected specific gravity and freezing point of sample.

7. Typical Results

Specific Gravity - 1.280-1.300

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY
Temperature Corrections for Specific Gravity
of Refrigerated Brine (Calcium Chloride)

Hydro- meter Reading	Corrections to be applied to hydrometer readings of calcium chloride solutions at various temperatures, using a hydrometer calibrated at 60°F/60°F										
	0°F	10°F	20°F	30°F	40°F	50°F	60°F	70°F	80°F	90°F	100°F
1.200	.011	.010	.009	.007	.005	.003	N	.002	.004	.007	.009
1.210	.012	.011	.009	.007	.005	.003	O	.002	.005	.007	.010
1.220	.012	.011	.009	.007	.005	.003		.002	.005	.007	.010
1.230	.013	.011	.009	.007	.005	.003		.002	.005	.007	.010
1.240	.013	.011	.009	.007	.005	.003	C	.002	.005	.008	.010
1.250	.013	.012	.010	.008	.005	.003	O	.003	.005	.008	.010
1.260	.014	.012	.010	.008	.005	.003	R	.003	.005	.008	.011
1.270	.014	.012	.010	.008	.005	.003	R	.003	.005	.008	.011
1.280	.015	.013	.010	.008	.006	.003	E	.003	.005	.008	.011
1.290	.015	.013	.011	.008	.006	.003	C	.003	.006	.008	.011
1.300	.015	.013	.011	.008	.006	.003	T	.003	.006	.009	.011
1.310	.016	.014	.011	.009	.006	.003	I	.003	.006	.009	.012
1.320	.016	.014	.012	.009	.006	.003	O	.003	.006	.009	.012
1.330	.017	.014	.012	.009	.006	.003	N	.003	.006	.009	.012
1.340	.017	.015	.012	.009	.006	.003	S	.003	.006	.009	.012
1.350	.018	.015	.012	.009	.006	.003		.003	.006	.009	.012
1.360			.013	.009	.006	.004		.003	.006	.010	.013
1.370				.010	.007	.004		.003	.006	.010	.013
1.380					.007	.004		.003	.007	.010	.013
1.390						.004		.004	.007	.010	.013
1.400						.004		.004	.007	.010	.013
1.410								.004	.007		

Temperatures below 60°F

Observed Hydrometer Reading MINUS Temperature Correction = Specific Gravity
at 60°/60°F

Temperatures above 60°F

Observed Hydrometer Reading PLUS Temperature Correction = Specific Gravity
at 60°/60°F

FREEZING POINTS OF REFRIGERATED BRINE

Sp. Gr.	0	2	4	6	8
1.20	- 6	- 7	- 8	- 9	- 9
1.21	-10	-11	-12	-12	-13
1.22	-14	-15	-15	-16	-17
1.23	-17	-18	-19	-20	-21
1.24	-22	-23	-24	-24	-25
1.25	-26	-27	-28	-29	-30
1.26	-31	-32	-33	-34	-35
1.27	-37	-38	-40	-42	-43
1.28	-45	-47	-49	-52	-55
1.29	-60	-56	-52	-48	-45
1.30	-42	-40	-38	-35	-31
1.31	-28				

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SL 020743

ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY1. Test

Determination of HCl in Vent Gas

2. Sample

Reactor Condenser Vent

3. Apparatus and Reagents

- a. Gas burette, 100 mL
- b. Confining solution

4. Procedure

- a. Put about 200 mL of confining solution in leveling bottle. Change confining solution once per week.
- b. Fill connecting tube by running a small amount of confining solution into burette.
- c. Connect burette to sample line. Purge at least 5 minutes.
- d. Close burette stopcocks and sample valve so as to leave a slight pressure in burette. Disconnect from sample line, and allow burette to come to room temperature.
- e. Equalize sample in burette to atmospheric pressure by rotating stopcock.
- f. Allow confining solution to enter burette and absorb the HCl. After absorption has taken place, read the amount of HCl absorbed directly from gas burette.

5. Report

% HCl absorbed by confining solution

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SL 020744

ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY

1. Test

Determination of % HCl

2. Sample

HCl Absorber Bottoms

3. Reagents and Apparatus

- a. 2.74 N Sodium Carbonate
- b. Xylene cyanole indicator
- c. 20-mL pipette
- d. 250-mL beaker

4. Procedure

- a. Pipette 20 mL of sample into a 250 mL beaker.
- b. Add 50 mL of distilled H₂O and 4 drops of xylene cyanole.
- c. Titrate with 2.74 N sodium carbonate to a steel blue end point and record titer.

5. Calculations

$$20 \text{ mL} - \frac{T \times 2.74 \times 0.0365 \times 100}{20} - \% \text{ HCl}$$

or

$$\text{Titer} \times 0.50 - \% \text{ HCl}$$

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY

1. Test

Determination of Alkalinity of Water

2. Sample

Steam Drum Water

3. Apparatus and Reagents

- a. 0.02 H_2SO_4
- b. Phenolphthalein Indicator
- c. Methyl Orange Indicator
- d. 10% Barium Chloride Solution (BaCl_2)

4. Procedure

a. "P" and "MO" Readings

- (1) Measure 100 mL of sample and pour into casserole. Add 4 drops phenolphthalein indicator.
- (2) Titrate with 0.02 N H_2SO_4 to clear end point. Record this titration as "P".
- (3) Add 4 drops of methyl orange indicator. Without refilling burette, titrate with 0.02 N H_2SO_4 to a reddish orange end point. Record this titration as "MO".

b. Hydrate "PHT" Reading

- (1) Measure 100 mL of sample and pour into casserole. Add 10 mL of 10% BaCl_2 solution.
- (2) Let stand 20 minutes.
- (3) Add 4 drops of phenolphthalein indicator and titrate with 0.02 N H_2SO_4 to a clear end point. Record this titration as "PHT".

5. Calculations

$$\text{a. } \frac{\text{"P" Reading}}{\text{ppm CaO}_3} = \frac{T \text{ ("P")} \times 0.02 \times 0.05 \times 10^6}{100} - T \text{ ("P")} \times 10$$

$$\text{b. } \frac{\text{"MO" Reading}}{\text{ppm CaCO}_3} = \frac{T \text{ ("MO")} \times 0.02 \times 0.05 \times 10^6}{100} - T \text{ ("MO")} \times 10$$

$$\text{c. } \frac{\text{"PHT" Reading}}{\text{ppm CaCO}_3} = \frac{T \text{ ("PHT")} \times 0.02 \times 0.05 \times 10^6}{100} - T \text{ ("PHT")} \times 10$$

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY1. Test

Dissolved Solids in Water

2. Sample

Steam Drum Water

3. Apparatus and Reagents

- a. Nalcometer
- b. Phenolphthalein Indicator
- c. 0.02 N Sulfuric Acid
- d. 600 Micromho Standard (Nalco Solution 298)

4. Standardization

Pour 600 Micromho Standard into cell on nalcometer. Turn range, set knob to 100 and press button. Meter should read 6. If it does not, remove button on bottom of nalcometer and adjust to 6.

5. Procedure

- a. Measure 50 mL of sample into a beaker.
- b. Add two (2) drops of phenolphthalein indicator to sample.
 - (1) If no pink color appears, add sufficient sample to fill cell.
 - (2) If pink color appears, add 0.02 N H_2SO_4 , one drop at a time until the pink color disappears) then add sufficient sample until pink color just reappears. Add to cell and proceed with step "C".
- c. Set range (10, 100, or 1,000) and push button.

6. Calculation

Reading x Range = Micromho

Look up ppm dissolved solids on chart provided using your known Micromho from meter.

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 MICROMHO/CM TO PPM TOTAL DISSOLVED SOLIDS CONVERSION CHART

μ MHO/CM	PPM TDS	μ MHO/CM	PPM TDS	μ MHO/CM	PPM TDS
2	1	120	68	900	560
4	2.1	140	80	950	600
6	3.2	160	91	1000	630
8	4.2	180	100	1500	970
10	5.2	200	115	2000	1300
				2500	1700
12	6.4	220	127	3000	2000
14	7.4	240	139	3500	2400
16	8.5	260	150	4000	2750
18	9.6	280	164	4500	3150
20	11.0	300	176	5000	3500
25	13.5	350	210	5500	3900
30	16.0	400	240	6000	4300
35	19.0	450	270	6500	4700
40	22.0	500	300	7000	5000
45	24.5	550	335	7500	5400
50	27.5	600	370	8000	5800
				8500	6200
60	33.0	650	400	9000	6600
70	39.0	700	435	9500	7000
80	45.0	750	470	10000	7400
90	51.0	800	500		
100	56.0	850	530		

* Every 50 μ MHO/CM is approximately 30 ppm TDS

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SL 020748

ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY

1. Test

Determination of Calcium and Magnesium in Water as Calcium Carbonate

2. Sample

Steam Drum Feed

3. Apparatus and Reagents

- a. Versenate Solution (EDTA) - 1 mL = 0.001 g CaCO_3
- b. Monover Indicator
- c. Hydrochloric Acid, 1 N (HCl)
- d. Methyl Red Indicator
- e. Ammonium Hydroxide, 6 N (NH_4OH)

4. Procedure

- a. Measure 100 mL of sample into the casserole and add 200 mL of distilled water.
- b. Add 1-2 drops of methyl red.
- c. Titrate with 1 N HCl to the red end point and add exactly 2 mL in excess.
- d. Stir gently for 30 seconds.
- e. Add 2 mL of 6 N NH_4OH and stir to mix.
- f. Add Monover indicator until wine-red color develops.
- g. Titrate with standard versenate until the solution changes from wine-red to pure blue.

5. Calculations

$$\text{ppm } \text{Ca}^{+2} \text{ and } \text{Mg}^{+2} \text{ as } \text{CaCO}_3 = \frac{\text{Titer} \times 0.001 \times 10^6}{100} = \text{Titer} \times 10$$

6. Typical Results

0 ppm

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY1. Test

Calcium

2. Sample

Cooling Tower Water

3. Apparatus and Reagents

- a. Burette, 25 mL
- b. Casserole, porcelain
- c. Cylinder, graduated, 50 mL
- d. Measuring dipper, brass
- e. Pipette, safety bulb, 2 mL
- f. Stirring rod, glass
- g. Calcium indicator
- h. Versenate 1 mL - 1 mg CaCO_3
- i. Sodium hydroxide, 1.0 N

4. Procedure

- a. Measure 50 mL of sample and transfer to a casserole.
- b. Add 2.0 mL of 1.0 N sodium hydroxide to sample and stir.
- c. With the brass measuring cup provided, add one (1) level measure of calcium indicator and stir.
- d. Sample will turn salmon-pink if Ca is present.
- e. Titrate with versenate until the color turns orchid-purple.
- f. Record titer and calculate ppm CaCO_3 .

5. CalculationTiter x 10 = ppm CaCO_3

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY1. Test

Determination of Sodium Sulfite in Water

2. Sample

Steam Drum Water

3. Apparatus and Reagents

- a. Starch indicator
- b. 1.0 N HCl
- c. 0.01587 N KI-KIO₃

4. Procedure

- a. Cool sample to at least 70°F.
- b. Measure 100 mL of sample into casserole.
- c. Add 1 mL of starch indicator and 5 mL of 1.0 N HCl.
- d. Immediately titrate with 0.01587 N KI-KIO₃ until first permanent blue color is obtained.

5. Calculation

$$\text{Sodium Sulfite (Na}_2\text{SO}_3\text{) ppm} = \frac{\text{NT} \times 0.01587 \times 0.0630 \times 10^6}{100} = \text{NT} \times 100$$

Where NT = (Sample Titer - Blank Titer)

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY

1. Test

Determination of Residual Chlorine (Cl_2)

2. Sample

- a. Well Water
- b. Cooling Tower Water

3. Apparatus and Reagents

- a. Wallace and Tiernan Comparator
- b. Residual Chlorine Disc
- c. Ortho-tolidine

4. Procedure

- a. Select two clean comparator cells. Fill one to the mark with a sample and place in right cell space of comparator.
- b. Add .5 mls of O - toolidine from the calibrated dropper into the other cell. Fill to the mark with sample. Place in left cell space.
- c. Immediately hold up to the light and match colors by revolving the disc. Read the value from upper left corner comparator.

5. Report

Value read from comparator.

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY

1. Test

Determination of Ortho-Phosphate

2. Sample

Cooling Tower Water
Make-up Water

3. Apparatus and Reagents

- a. Amino Acid Reagent Powder Pillows (Code-2012)
- b. Molybdate Reagent for Phosphate (code-2044)
- c. Sulfuric Acid Solution, 10N (Code-2033)

4. Procedure

UNFILTERED:

- a. Add 25 ml of sample to a 100-ml graduated cylinder with a ground glass stopper.
- b. Add 1 ml of sulfuric acid solution (Code-2033) and mix.
- c. Add 1 ml of molybdate reagent (Code-2044) and mix.
- d. Add the contents of one amino acid reagent powder pillow (Code-2012) and mix until dissolved.
- e. Allow 10 minutes for full color development which will be blue if phosphate is present. Do not wait more than 15 minutes before taking the readings.
- f. Use an unfiltered cooling water blank to zero Sequoia - Turner spectrophotometer with a 600 nm wavelength setting.
- g. Place sample in instrument, take reading and look up ppm orthophosphate from chart provided.

FILTERED:

- a. Filter 25.0 ml of sample through a 0.20 micron filter paper.
- b. Use steps a-e above.
- c. Use distilled water to zero the spectrophotometer.
- d. Place sample in instrument, take reading and look up ppm orthophosphate from chart provided.

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SL 020753

ORTHOPHOSPHATE
0 - 25 ppm as PO₄

APPARATUS REQUIRED

Comparator, Taylor test tube model, complete with 0-25 ppm phosphate, as PO ₄ , slide	code 401
Measuring Dipper, brass	113
Mixing tube, graduated 10 and 14 ml	170
Millipore filter paper	
50 cc syringe and filter paper holder	

CHEMICALS REQUIRED

Molybdate Reagent	code 236
Stannous Reagent, dry	239

PROCEDURE FOR TEST

Filt red Phosphate

The temperature of the sample should be in the range of 70-100° F. Filter the sample through the millipore filter paper, using the 50 cc syringe.

Using the mixing tube graduated at 10 and 14 ml, fill the tube to the first mark (10 ml) with the filtered sample. Add molybdate to the second mark (14 ml). Stopper and mix well. Add one (1) level measure of stannous reagent. Again stopper and mix well. A blue color develops, the intensity of which is proportional to the phosphate present.

Place the mixing tube in the middle compartment in the base. Fill the two test tubes on either side with filtered sample. With the color slide on the base, hold the instrument toward a source of daylight and move the slide in front of the test sample until a color match is obtained. Read the phosphate value within one (1) minute.

CALCULATION OF RESULTS

When a match is obtained, the phosphate in parts per million as PO₄ is read directly from the color standard slide.

Unfilt red Phosphate

Same as above, deleting the filtering step.

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Cooling Water Test for - Phosphate

Unfiltered

1. Pour 25.0 ml of sample into a 50 ml mixing graduate.
2. Add 1 ml 10N H_2SO_4 and swirl to mix (1 dropper full).
3. Add 1 ml of molybdate reagent for phosphate using the calibrated dropper (1 dropper full).
4. Add 1 amino acid reagent powder pillow, mix well.
5. Read after 10 minutes but wait no longer than 15 minutes at 660 nm.
6. Using unfiltered cooling water zero the spectrophotometer.
7. Read the absorbence of the developed sample and convert obsorbance to ppm phosphate using the chart provided by the PPG Laboratory personnel.

Filtered

1. Filter 25.0 ml of sample through a .22 micron filter paper.
2. Use steps 1 - 5 above.
3. Use distilled water to zero the spectrophotometer.
4. Read the absorbence of the developed sample. Convert absorbence to ppm phosphate using the chart provided by PPG Laboratory personnel.

Control Filtered Phosphate	15 - 20 ppm
Unfiltered Phosphate - Filtered Phosphate	less than 3

Cooling Water Test for - Betz 2020

1. Pour 50 ml of sample into a 50 ml mixing graduate.
2. Add 5 ml of Polymer Buffer and swirl to mix.
3. Set wavelength drive to 420 and zero the spectrophotometer with this solution.
4. Add 1 ml of Polymer Reagent and stir for 5 seconds EXACTLY.
5. Absorbence x 120 = ppm 2020.

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY

1. Test

BETZ 2020

2. Sample

Cooling Tower Water
Make-up Water

3. Apparatus and Reagents

- a. Spectrophotometer
- b. Beaker, 150 ml
- c. Cylinder, graduated, 50 ml
- d. Pipette, 1 ml
- e. Pipette, 5 ml
- f. Stirring Rod
- g. Polymer Buffer, Code 1254 (pints)
- h. Polymer Reagent, Code 1253 (pints)

4. Procedure

- a. Using to 50-ml graduate, measure 50 ml of sample into a 150-ml beaker.
- b. Add 5 ml of Polymer Buffer by pipette and stir for 10 seconds.
- c. Use a wavelength setting of 420 nm for the Sequoia-Turner Spectrophotometer.
- d. Fill sample cell with water solution and use to set photometer to zero absorbance.
- e. Pour solution back into beaker.
- f. Add 1 ml of polymer reagent by pipette and start timing simultaneously. Stir solution for 30 seconds.
- g. Then set timer for 4 1/2 minutes. After 4 1/2 minutes, transfer solution to sample cell and read absorbance. Note that total lapsed time of solution since polymer reagent addition should be exactly 5 minutes before taking reading.

5. Calculation of Results

Determine Betz 2020 treatment concentration in ppm 2020 from calibration curve.

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ANALYTICAL PROCEDURE FOR PER/TRI PLANT LABORATORY1. Test

pH

2. Sample

Cooling Tower Water

3. Apparatus and Reagents

- a. pH meter with Calomel and glass electrodes
- b. Buffer solution (pH 7.0)

4. Procedure

- a. Standardize pH meter with buffer solution.
- b. Rinse electrodes thoroughly with the sample.
- c. Read pH of the sample on a portion sufficient to cover the tips of the electrodes.

5. Report

Direct reading for pH meter

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