

# APPENDIX B

## CHEMICAL ANALYSIS PROCEDURES

### B-1. EXTERNAL TREATMENT PLANTS

**a. Test for Hardness (Colorimetric Titration Method).** This test is based on determination of the total calcium and magnesium content of a sample by titration with a sequestering agent in the presence of an organic dye sensitive to calcium and magnesium ions. The end point is a color change from red to blue. It occurs when all the calcium and magnesium ions are sequestered. NOTE: If the testing procedure of the equipment and/or reagent supplier differs from the following procedure, the supplier's procedure must be used.

(1) **Equipment.** These are supplied by commercial concerns as test kits.  
Equipment includes:

- (a) One 25-ml burette, automatic, complete.
- (b) One 210-ml casserole, porcelain.
- (c) One 50-ml cylinder, graduated.
- (d) One stirring rod, glass,

(2) **Reagents.**

- (a) Hardness indicator.
- (b) Hardness buffer.
- (c) Hardness titrating solution, 1 ml = 1 mg CaCO<sub>3</sub>.

(3) **Procedure.** Measure 50 ml of the sample in the graduated cylinder and transfer it to the casserole. With the calibrated dropper, add 0.5 ml of hardness buffer reagent to sample and stir. Add 4 to 6 drops of hardness indicator. If hardness is present, the sample turns red. Add the hardness titrating solution slowly from the burette, with continued stirring. When approaching the end point, the sample begins to show some blue coloration, but a definite reddish tinge can still be seen. The end point is the final discharge of the reddish tinge. More hardness titrate solution does not produce further color change. In using this procedure, the hardness titrating solution must be added slowly because the end point is sharp and rapid. For routine hardness determination it is suggested that 50 ml of the sample be measured, but only approximately 40 to 45 ml be added to the casserole at the start of the test. Add the hardness buffer reagent and the hardness indicator as directed, and titrate the mixture rapidly to the end point. Then add the remaining portion of the sample. The hardness present in the remainder of the sample turns the contents of the casserole red again. Titrating is continued slowly until the final end point is reached. Keep a record of the total milliliters of hardness titrating solution used.

(4) **Calculation of results.** The ppm hardness is

calculated by the equation:

$$\text{ppm hardness as CaCO}_3 = \frac{\text{ml titrating solution} \times 1,000}{\text{ml sample}}$$

Using a 50-ml sample, the hardness in ppm as CaCO<sub>3</sub> is equal to the ml of titrating solution employed, multiplied by 20.

**b. Test for Alkalinity.** This test is based on determination of the alkaline content of a sample by titration with a standard acid solution. The end points are taken as points of change in color of the organic indicators, phenolphthalein (approximately pH 8.3) and methyl orange (approximately pH 4.3) representing definite points to which the alkalinity of the sample has been reduced by adding standard acid solution.

NOTE: If the testing procedure of the equipment and/or reagent supplier differs from the following procedures, the supplier's procedure must be used.

(1) **Equipment.**

- (a) One 25-ml burette, automatic, complete.
- (b) One 210-ml casserole, porcelain.
- (c) One 50-ml cylinder, graduated.
- (d) One stirring rod, glass.

(2) **Reagents.**

- (a) Acid, sulfuric, N/50.
- (b) Phenolphthalein indicator, 0.5%.
- (c) Methyl orange indicator, 0.05%

(3) **Procedure.**

(a) Measure a clear, 50-ml sample of water in the graduated cylinder and transfer it to the casserole. Add 4 to 5 drops of phenolphthalein indicator. If the sample is alkaline water, which is usual in boiler water, it turns red. If the sample is raw or natural water, it usually remains colorless.

(b) Add the standard N/50 sulfuric acid from the burette drop by drop to the sample in the casserole, stirring constantly until the point is reached at which 1 drop removes the last trace of red and the sample becomes colorless. Stop and record the total milliliters to this point as the P reading.

(c) Add 4 drops of methyl orange indicator. (If no red developed when the phenolphthalein indicator was added to the original sample, the titration may be started with the methyl orange indicator at this point.) Again add the acid, drop by drop, until 1 drop changes the color from yellow to salmon-pink. Record the final burette reading as the M reading. This is a difficult end point to achieve accurately, and some practice may be necessary.

The general tendency is to add too much acid, which changes the sample from salmon-pink to definite red. Record the titration to the P point and the total titration to the M point, as the P and M readings respectively. (Note that M is always greater than P because P is included in M.) If the water sample does not settle clearly, the M reading can best be determined on a filtered sample. However, make the P reading on an unfiltered sample to ensure an accurate result.

(4) **Calculation of Results.** Phenolphthalein and methyl orange alkalinity are calculated by the equations:

$$P \text{ Alk as ppm CaCO}_3 = \frac{\text{ml of H}_2\text{SO}_4 \times 1,000}{\text{ml sample}}$$

$$M \text{ Alk as ppm CaCO}_3 = \frac{\text{ml of H}_2\text{SO}_4 \times 1,000}{\text{ml sample}}$$

Using a 50-ml sample, P Alk in ppm CaCO<sub>3</sub> equals the ml of N/50 sulfuric acid recorded for the P reading multiplied by 20. The M Alk (total alkalinity) equals the ml of N/50 sulfuric acid recorded for the M reading multiplied by 20.

## B-2. BOILER WATER.

**a. Test for Sodium Sulfite.** It is important that sodium sulfite be the first boiler water test performed on any set of samples. Sample exposure to air must be minimized, and test must be performed immediately after collection for accurate results. Cool the sample to 70° F, or below, and expose to the air as little as possible, because oxygen in the air combines with sodium sulfite in the sample and causes low readings. It is desirable to collect a separate sample, using the boiler water sample cooler, with the line reaching to the bottom of the sampling bottle. Allow the boiler water to run until a few bottlefuls overflow to waste. A USAEHSC test kit is used.

### (1) Equipment.

- (a) Two marked test tubes.
- (b) Two plain test tubes.
- (c) One stopper for plain test tubes.
- (d) One stirring rod.
- (e) One 7-in. dropper.
- (f) One ¼ teaspoon.
- (g) One 50-ml beaker.
- (h) One 100-ml beaker.
- (i) One 30-ml acid dropping bottle, with dropper marked at ½ ml for hydrochloric acid 3N.
- (j) One 30-ml starch dropping bottle, with dropper marked at ½ ml for starch indicator.

### (2) Reagents.

- (a) One 2-oz. bottle of potato or arrowroot starch.
- (b) One 8-ml vial of thymol.

(c) One 32-oz. bottle of hydrochloric acid 3N.

(d) One 1-pt. amber bottle of standard potassium iodate-iodide reagent.

(3) **Preparation of Starch Indicator.** This indicator must be prepared locally in the following manner.

(a) Measure out a level ¼ teaspoonful of potato or arrowroot starch and transfer to the 50-ml beaker.

(b) Add a few milliliters of distilled water and stir the starch into a thin paste, using the glass end of the stirring rod.

(c) Put 50 ml of distilled water into the 100-ml beaker. (It is convenient in this step to have the 100-ml beaker marked at the point where it holds 50 ml, or one of the marked test tubes can be used by filling it with distilled water to the fourth mark above the long mark.)

(d) Bring the water in the 100-ml beaker to a boil by any convenient method.

(e) Remove the source of heat and immediately pour the starch paste into the boiling water while stirring the solution.

(f) Put a crystal of thymol into the starch solution and stir. After the solution has cooled, pour off any scum on the surface and transfer 30 ml to the indicator dropping bottle.

(g) The starch solution loses its sensitivity as a indicator after a time. Addition of the thymol preserves it for about two weeks. Date the starch when prepared.

### (4) Procedure.

(a) Transfer 1 ml of hydrochloric acid 3N to a clean, marked test tube by measuring out two ½-ml portions with the dropper of the acid dropping bottle.

(b) From the starch dropping bottle, transfer ½ ml of starch to the marked test tube.

(c) Without disturbing any settled sludge in the sample, pour enough sample into the marked test tube to bring the level up to the first mark (25 ml). Stir the mixture in the tube with the plunger end of the stirring rod.

(d) To add the standard potassium iodate-iodide reagent to the mixture in the marked test tube, it is convenient to have the marked test tube supported and the stirring rod placed in the tube, so that the reagent can be added with one hand while the mixture is stirred with the other. Fill the 7-in. dropper with standard potassium iodate-iodide reagent from the stock bottle by sucking it up with the rubber bulb. (The dropper must be kept clean and reserved for this test only).

(e) Add the reagent to the mixture in the marked test tube, one drop at a time, counting the number of drops and stirring after each is added until a permanent blue color, which is not removed by stirring, is obtained. The standard iodate-iodide reagent reacts with sodium sulfite in the mixture, and formation of the permanent

blue color from the action of excess reagent with the starch shows that all the sodium sulfite in the mixture has been consumed by the iodate-iodide reagent.

(f) Each drop of iodate-iodide reagent used (except the last one) indicates 5 ppm of sodium sulfite in the boiler water sample. To figure the concentration of sodium sulfite in the boiler water, multiply the total number of drops of the standard iodate-iodide reagent used, less one, by 5.

(5) **Recording the Results.** Record the results of the test as ppm in column (4), DA Form 4367, Boiler Water Treatment Log.

**b. Test for Causticity (OH-) Without Tannin.** This test must be run immediately after the sodium sulfite test for any set of boiler water samples. Sample exposure to air must be minimized before this test is performed. The boiler water sample is collected at a temperature of 70° F, or below. The USAEHSC test kit is used.

(1) **Equipment.**

- (a) Two 8-in. droppers with bulbs.
- (b) Four marked test tubes, 22 mm x 185 mm.
- (c) Three plain test tubes, 22 mm x 175 mm.
- (d) Two rubber stoppers, No. 3.
- (e) One 14-in. test-tube brush.
- (f) One test-tube clamp.
- (g) Two 9-in. stirring rods.
- (h) One 1-oz. indicator dropping bottle for phenolphthalein.
- (i) One test-tube rack.

(2) **Reagents.**

- (a) One 32-oz. bottle causticity reagent No. 2
- (b) One 32-oz. bottle causticity reagent No. 1
- (c) One 4-oz. bottle of phenolphthalein indicator.

(3) **Procedure.** In conducting a test for causticity when tannin is not used, follow the procedure outlined below. **NOTE:** Avoid exposure of sample to air as much as possible to minimize absorption of CO<sub>2</sub>. If the sample in the tube is warm, cool it by flowing cold water on the outside of the tube.

(a) Without disturbing any settled sludge, fill a marked test tube exactly to the first mark (25 ml) with some of the original boiler water sample.

(b) Shake causticity reagent No. 1 (barium chloride solution saturated with phenolphthalein) thoroughly and add enough to the graduated tube to bring the level exactly to the second, or long mark (30 ml). Stir the solution with the stirring rod which must be kept clean and reserved for the causticity test only. Do not strike the end of the stirring rod against the bottom of the test tube, as it is easily punctured. If the mixture remains colorless or does not turn pink, the causticity in the boiler water is zero. In this case the test is finished.

(c) If the mixture turns pink, causticity is present.

(If the pink color is not very deep, intensify it by adding two drops of phenolphthalein indicator to the mixture in the tube). Add causticity reagent No. 2 (standard 1/30 normal acid), using the dropper, which must be kept clean and reserved for the causticity test only. Causticity reagent No. 2 is sucked from the reagent bottle into the dropper by its rubber bulb and added, drop by drop, to the test tube. After each addition, stir the mixture with a stirring rod. After sufficient reagent has been added, the pink color disappears, the change point usually being very sharp. As soon as the pink color just fades out, stop adding reagent.

(d) The amount of causticity reagent No. 2 required to make the pink color disappear indicates the concentration of hydroxide (OH-) or causticity in the boiler water. The amount of reagent used is shown by the marks on the test tube above the long mark (30 ml). The distance between any two marks on the test tube equals 5 ml, and readings less than 5 ml can be estimated. For example, if only 3/5 the distance between the long mark and the next mark above was filled, then 3 ml were added. If the distance filled was past one mark plus 3/5 the distance to the next, then 5 + 3 = 8 ml were used. To obtain the actual ppm of hydroxide or causticity shown by the test, multiply the number of ml by 23. Thus, for 8 ml of causticity reagent No. 2, there are 8x23=184 ppm hydroxide or causticity in the water.

(4) **Recording the Results.** Record the results of the test as zero or in ppm, as the case may be, in column (2) of DA Form 4367, Boiler Water Treatment Log.

**c. Test for Causticity (OH-) With Tannin.** This test must be run immediately after the sodium sulfite test for any boiler water samples. Sample exposure to air must be minimized before this test is performed. For this test, it is desirable to start with a warm sample at about 160° F. It may be reheated by placing the sample-collecting container in a stream of hot boiler water drawn through the boiler water cooler connection. **NOTE:** Avoid exposure of sample to air as much as possible to minimize absorption of CO<sub>2</sub>.

(1) **Equipment and Reagents.** These are the same as B-2a(1) and (2) where tannin was not used.

(2) **Procedure.** In conducting a test for causticity when tannin is used, follow the procedure outlined below.

(a) Fill two test tubes to the first mark (25 ml) with some of the original boiler water sample, taking care not to disturb the settled sludge in the container. (It is important that as little sludge as possible be transferred from the sample-collecting container to the test tubes).

(b) Shake causticity reagent No. 1 thoroughly and add enough to each of the two marked tubes to bring the levels up to the second, or long mark (30 ml). Stir both with the stirring rod, which must be kept clean and

reserved for the causticity test only. Stopper both tubes and let them stand until any sludge formed has settled to the bottom. The sludge carries with it much of the tannin or other colored matter in the solution. Settling takes a few minutes if the sample is warm. Without disturbing the sludge at the bottom, pour enough solution from the tubes into the third marked tube to fill it to the second, or long, mark. Discard the mixture left in the first two. If the sample in the third is still warm, cool it by letting cold water run on the outside of the tube. It is sometimes possible to intensify the pink color by adding two drops of phenolphthalein from the indicator dropping bottle to the sample in the tube. Stir the solution. If it is not pink, the causticity in the boiler water is zero and the test is finished.

(c) If the mixture turns pink, proceed in the same manner as B-2a (3) (c), (d), and (e) when no tannin is used.

(3) **Alternate Procedure.** Use a graduated cylinder, marked for 50 or 60 ml., instead of two standard marked test tubes used in steps B-2a(a) and (b). With the graduated cylinder, the warm (160° F) sample is added up to 50-ml mark and causticity reagent No. 1 up to the 60-ml mark. Stir the mixture and lightly stopper the tube or graduated cylinder. After the sludge settles, pour off enough of the solution into one of the standard marked test tubes to fill it to the long mark (30 ml). If the sample is warm, cool it by letting cold water run on the outside of the tube. The pink color may be intensified by adding two drops of phenolphthalein. If the solution is not pink, the causticity in the boiler water is zero. If it turns pink, proceed in the same manner as B-2a (3) (c), (d), and (e), when no tannin is used.

(4) **Titrimetric Method.** Use the titrimetric method of determining causticity when greater accuracy is desired than is given by either of the above methods. This method utilizes a burette which permits measuring very small quantities of causticity reagent No. 2. Procedure is as follows:

(a) Admit 40 ml of hot boiler-water sample to a graduated cylinder. Shake causticity reagent No. 1 thoroughly and add 10 ml of reagent to the graduated cylinder. Stir the mixture and lightly stopper the graduated cylinder. Permit precipitate to settle and then transfer 30 ml of clear solution to a white casserole or other suitable vessel. Add 2 to 3 drops of phenolphthalein indicator.

(b) Add causticity reagent No. 2 from the burette drop by drop until the pink color disappears.

(c) Read the number of ml of causticity reagent No. 2 used directly from the burette. Multiply this figure by 23 to find ppm of causticity of hydroxide.

(5) **Recording of Results.** Record the results on DA Form 4367, Boiler Water Treatment Log, after the test

is complete.

**d. Test for Phosphate.** This is a colorimetric test for phosphate, employing a decolorizing carbon for removal of tannin. Carbon absorbs the tannin, and the carbon and tannin are then filtered out. When tannin is not present, the use of carbon is not required. The USAEHSC Kit is used.

(1) **Equipment.**

(a) One Taylor Comparator Phosphate Slide, 5 to 100 ppm, #9110.

(b) One Taylor Slide Base, #9190.

(c) One Taylor Dalite Lamp, #9195.

(d) Four combination comparator mixing tubes, each marked at 5, 15, and 17.5 ml, with stoppers.

(e) Two short comparator tubes marked at 5 ml.

(f) One filter funnel, 65 mm diameter.

(g) One package filter paper, 11 cm diameter, very fine.

(h) One 15 ml bottle.

(i) One ½ ml dropper.

(j) One ¼ teaspoon measuring spoon.

(k) Two plain test tubes, 22 mm x 175 mm (about 50 mm).

(l) Two rubber stoppers, No. 3.

(2) **Reagents.**

(a) One 32-oz. Comparator Molybdate.

(b) One 2-oz. Concentrated Stannous Chloride.

(c) One Standard Phosphate Test Solution (45 ppm of phosphate, PO<sub>4</sub>).

(d) One lb Decolorizing Carbon. (This special grade of decolorizing carbon has been tested to ensure that it does not affect the phosphate concentration in the sample.)

(3) **Preparation of Dilute Stannous Chloride.** For test purposes, the stannous chloride is supplied in concentrated form. Dilute and prepare the reagent from the concentrated stannous chloride on the day it is to be used, because the diluted solution deteriorates too rapidly for supply by a central laboratory. If not fresh, dilute stannous chloride gives low test results. Concentrated stannous chloride also deteriorates in time and should not be used if more than two months old. Unopened bottles of concentrated stannous chloride are good for six months. Dilute stannous chloride is made by the following method:

(a) Fill the ½-ml dropper up to the mark with the concentrated stannous chloride.

(b) Transfer it to a clean 20-ml bottle.

(c) Add distilled water up to the shoulder of the bottle, then stopper and mix by shaking.

(d) Any dilute stannous chloride not used the day it is made should be discarded.

(4) **Procedure.** The test for phosphate is outlined below.

(a) Without disturbing any settled sludge, transfer sufficient of the sample to the test tube to fill it about

half full.

(b) If tannin is present in the sample, add  $\frac{1}{4}$ -teaspoonful of decolorizing carbon. Stopper the tube and shake vigorously for about 1 minute. The carbon absorbs the tannin so that it can be filtered out.

(c) Fold a filter paper and place it in the filter funnel. Do not wet down the filter paper with water. Filter the shaken sample using a combination mixing tube as a receiver. Avoid jiggling the funnel, as unfiltered boiler water may overflow the edge of the filter paper and go into the tube. The operator may provide a support for the funnel. Filtering is slow because of the action of the carbon.

(d) After 5 ml of sample has filtered through, as indicated by the level in the tube, discard it. Continue filtering to bring the level in the test tube again up to the 5 ml mark. The sample should come through clear and free, or nearly free, of any color resulting from tannin. If it does not, refer to paragraph (i) which describes retesting procedures for cases where more Decolorizing Carbon is needed to remove tannin from the sample.

(e) Add comparator molybdate reagent to bring the level up to the second mark (15 ml). Stopper and mix by inverting the tube several times.

(f) Add fresh dilute stannous chloride up to the third mark (17.5 ml). Stopper and mix by inverting. If phosphate is present, the solution in the mixing tube turns blue.

(g) Place the tube in the center opening in the slide base. Fill the two short comparator tubes with distilled water and insert them into the two holes adjacent to the center hole. Place the base on the Dalite lamp and turn on the lamp.

(h) Find the best match between the sample and the color standards of the phosphate slide by moving the slide along the slide base. Concentrations between those of two color standards can be estimated. Note the reading within one minute after adding the dilute stannous chloride since the color can fade rapidly.

(i) If the color obtained is darker than the 60 ppm standard, the sample should be diluted and tested again. After filtering 5 ml of sample, add 5 ml distilled water and mix. Use 5 ml of this diluted sample to perform the test. Multiply the result by two to obtain the concentration of the original undiluted sample.

(j) Where the use of a  $\frac{1}{4}$ -teaspoon of Decolorizing Carbon does not give a filtered sample which is free or nearly free of color due to tannin, the test can be repeated using more Decolorizing Carbon, say  $\frac{1}{2}$ -Teaspoon. Add it in  $\frac{1}{4}$ -teaspoon portions. Stopper and shake sample for one minute after each addition. With some grades of Decolorizing Carbon, more than this amount should not be used as it can remove phosphate from solution and give low results.

(5) **Testing the Reagents.** Test the comparator molybdate and concentrated stannous chloride reagents at frequent intervals, at least twice a week, to ensure they are giving a correct blue color development in the test. This is done by testing for the phosphate concentration in the standard phosphate test solution. Add sufficient standard phosphate solution (45 ppm  $\text{PO}_4$ ) to a combination mixing tube to bring the level up to the first mark. Add comparator molybdate and stannous chloride reagents as described in the test above. The color obtained should be about midway between 40 and 50 ppm color standards of the comparator. If the color is outside the 40 to 50 ppm range of the standards, the reagents must be replaced.

(6) **Recording the Results.** Record the results on DA Form 4367, Boiler Water Treatment Log, after the test is complete.

**e. Test for Tannin.** When boiler water is treated with tannin, the dosage can be controlled satisfactorily by the depth of brown formed in the boiler water by the tannin. To estimate the depth of the color, which is necessary in adjusting tannin dosages, a sample of the boiler water is compared with the series of brown color standards of successively increased depths of color. The tannin color comparator used for the comparison has five glass color standards: No. 1, very light; No. 2, light; No. 3, medium; No. 4, dark; and No. 5, very dark.

(1) **Equipment.** This test employs a USAEHSC Kit. Required equipment includes:

- (a) One tannin color comparator.
- (b) Two square tubes, 13 mm view depth.
- (c) One plain test tube, 22 mm x 175 mm.
- (d) One filter funnel, 65 mm x 65 mm.
- (e) One package filter paper, 11-cm. diam.

(2) **Procedure.**

(a) Without disturbing any settled sludge in the sample, fill the test tube to within 1 or 2 inches of the top with the boiler water being tested.

(b) Place a square test tube into a slot of the comparator, and set the filter funnel in the tube. Fold a filter paper and place it in the funnel without wetting it down. Filter sufficient sample from the plain test tube into the square tube so that it is nearly full.

(c) Remove the square test tube from the comparator and note the appearance of the filtered boiler water. It should be free of suspended solids and sludge.

(d) If the sample does not come through free of suspended solids or sludge, refilter it, using the same funnel and filter paper. Repeat, using double filter paper if necessary, until it is free.

(e) Set the square tube of filtered sample in the middle slot of the comparator. Compare the color of sample with the five color standards, viewing it against a source of fairly bright daylight.

(f) The color standard most closely matching the color of the filtered sample gives the tannin concentration of the boiler water. For most boiler water conditions, the tannin dosage is usually satisfactory if it maintains a medium (No. 3) tannin color in the boiler water. When boiler water causticity is high, the tannin dosage may be adjusted to maintain a dark (No. 4) tannin color.

(3) **Recording the Results.** Record the results on DA Form 4367, Boiler Water Treatment Log, after the test is complete.

**f. Test for Total Dissolved Solids (TDS).** The electrical conductivity method is a simple and rapid way to determine TDS content.

**(1) Equipment and Reagents.**

(a) Myron L model EP meters can be provided via lateral transfer by USAEHSC which should be contacted for details. This instrument operates from two 9V transistor batteries and has a built-in cell. It has automatic temperature compensation from 32 to 120°F (0-48°C). A variable range selection (0-0.5, 0-5, 0-50, 0-500, 0-5000 micromhos) permits its use for condensate as well as for boiler water. The base temperature of the Myron L is 77°F (25°C). The instrument comes with complete directions for use and these should be followed for general operation.

(b) Alternatively, a Solu-Bridge, Model RD-P4 or SD-P104, for a 105 to 120-V, 50- to 60-cycle AC outlet and one polystyrene dip cell, Model L CEL-52 or CEL-VS2S is required. This Solu-Bridge model has a range of 500 to 7,000 micromhos and a temperature compensation device which adjusts the reading to a base temperature of 64°F. The SD-P104 has a base temperature of 77°F (25°C). This instrument and dip cell may be procured from commercial sources. Note that this conductivity meter can only be used for testing boiler water. To test both condensate and boiler water, an SD-406A range 0-5,000 micromhos meter and CEL-VS2S cell or the 0-5,000 micromhos meter from USAEHSC would be required.

(c) The following equipment and reagents are also needed for TDS testing. They can be obtained from USAEHSC and are referred to as "Equipment used with Conductivity Meter":

- One thermometer, armored.
- Four test tubes, 200 mm x 38 mm.
- One pint conductivity neutralizing solution.
- One 1-oz. indicator dropping bottle with ½-ml dropper.
- One quart conductivity meter test solution, 2,000 micromhos/cm at 64°F
- One quart 3N hydrochloric acid solution (not required with Myron L type meters).
- One quart distilled water.
- Two rubber stoppers, No. 8.
- Four ounces of phenolphthalein solution.

**(2) Procedure for Myron L Type Instrument.**

(a) Without disturbing settled sludge, first rinse a clean 200 mm x 38 mm test tube with the sample, then pour in about 60 ml of the sample. The tube can be marked with a file or crayon at the point where it holds 60 ml. This point is about 5 inches from the top of the tube. The test tube rack provided with the causticity test kit can be used to support the tube. Add 1 to 2 drops of phenolphthalein solution, and ½ ml of conductivity neutralizing solution to the sample. Stopper and invert several times to mix. If pink color persists, an additional amount of neutralizing solution should be added until the pink color disappears. In the presence of a dark tannin color, a pink color may not be clearly distinguished but a deepening of the original color will be observed. Upon complete neutralization, the original color will be restored.

(b) Rinse the built in cell cup three times with the sample to be tested, then fill with the sample to at least ¼ inch above the top electrode. NEVER FILL THE CELL BY DIPPING THE INSTRUMENT INTO WATER! DO NOT USE WITH SAMPLES HOTTER THAN 120°F (48°C).

(c) Select the desired range.

(d) Push the button; the pointer immediately indicates the specific conductivity of the sample in micromhos.

(e) When finished with the instrument, rinse the cell cup with clean water, preferably deionized. Be certain that all tannin is removed from the cup and electrodes. A wash bottle containing deionized water is helpful for cleaning the cell. When there are visible films of oil, dirt, or scale in the cell cup or on the top electrode, scrub lightly with a small brush and household cleaner. Rinse out the cleaner and the instrument can be used.

(f) The specific conductance reading is converted to TDS concentration in parts per million by multiplying by one of the following conversion factors, depending on the tannin number of the sample.

Tannin Number	Factor
0	0.7
1	0.8
2, 3	0.9
4.5	1.0

Note the conversion factor for condensate is 0.6. The change in condensate conductivity is important. The TDS number is relative.

(g) Check calibration of the cell at least weekly. This is done by measuring the temperature and specific conductance of the calibration solution, using the same procedure as described above for boiler water and rinsing the cell cup with the calibration solution. Use of the neutralizing solution is omitted. If the reading does not correspond to the specific conductance of the calibration solution at the temperature given on the solution bottle,

adjust the calibration control (accessible through the fingerhole in the bottom cover) until the reading agrees with the micromho value of the standard solution. Measure a fresh sample of the standard solution. The meter reading must again indicate the micromhos value of the standard solution; the instrument is now calibrated on all ranges.

**(3) Procedure for Solu-Bridge Type Instrument.**

**(a)** Without disturbing settled sludge, first rinse a clean 200 mm x 38 mm test tube with the sample and then pour in about 60 ml of the sample. The tube can be marked with a file or crayon at the point where it holds 60 ml. This point is about 5 inches from the top of the tube. If desired a rack or other support for the tube can be made at the boiler plant. Add to the sample two dropperfulls (filled to the mark) of conductivity neutralizing solution. Stopper and invert several times to mix.

**(b)** Connect the terminals of the cell to the conductivity meter. Fill a clean 200 mm x 38 mm test tube about half full with distilled water. Wash the cell by immersing in distilled water. Do not immerse the cable. Move the cell up and down several times in the distilled water. Remove the cell and shake to remove any distilled water. Discard the distilled water in the tube. Immerse the cell in the solution being tested up to a point where the level of the solution is at least 1/2 inch above the vent holes. Move up and down several times to release any bubbles which may adhere to the cell. Set the thermometer in the solution in the test tube.

**(c)** Plug the instrument cord into a 105 to 120V, 50-60 cycle AC outlet and turn on the instrument switch.

**(d)** After the thermometer reading has come to rest, set the temperature adjustment knob of the instrument to the same reading as the thermometer. Remove the thermometer from the solution. Set the cell so that it is centered in the test tube.

**(e)** After the instrument has warmed up, as indicated by the brightness of the electron tube (RD-P4 only), rotate the dial of the instrument until the dark segment of the electron tube reaches its widest opening and a sharp shadow is obtained. The RD-P4 Solu-Bridge reads specific conductance in micromhos/cm at 64° F. With the SD-P104, rotate the dial until both the red and green lamps are lit. The SD-P104 reads specific conductance at 77° F (25° C).

**(f)** Clean the cell by rinsing in fresh distilled water. When not in use, immerse the cell in distilled water in a 200 mm x 38 mm test tube.

**(g)** Note that each small division on the scale between 500 and 4,000 represents 100 micromhos/cm and each small division between 4,000 and 7,000 is 200 micromhos/cm on the RD-P4 meter.

**(h)** The specific conductance reading is converted

to TDS concentration in parts per million by multiplying by one of the following conversion factors depending on the tannin number of the sample:

Tannin Number	Factor with 64° F Base Temperature	Factor with 77° F Base Temperature
0	0.8	0.7
1	0.9	0.8
2,3	1.0	0.9
4,5	1.1	1.0

Example: If the sample has a tannin number of 1, and a specific conductance of 3100 micromhos/cm is obtained, the concentration in the sample is 3100 x 0.9 = 2800 ppm with a 64° F base temperature meter. Record the results of the test as ppm in column (5), DA Form 4367, Boiler Water Treatment Log.

Note the conversion factor for condensate is 0.6 for either base temperature. The change in condensate conductivity is important. The TDS number is relative.

**(i)** Inspect the cell at intervals for the following: is wear or cracking noted; is there foreign material on the electrodes; is shield in position, intact, and are electrodes in position; are vent holes free of obstruction; is the black platinum coating present over all the electrode surfaces? If a mechanical defect is noted in the cell or if loss of the black platinum coating on the electrodes is noted, the cell can be returned to the manufacturer for repair or replatinizing of the electrodes. When not in use, the cell can be stored submerged in distilled water.

**(j)** Clean a cell used frequently every few weeks. This may be done by dipping in 3N hydrochloric acid for about two minutes, followed by washing in running tap water, and inverting the cell to wash the electrodes. If any deposits remain in the cell, clean further by swishing in soapy water followed by washing in running tap water and inverting the cell to wash the electrodes. If the dark segment of the electron tube becomes hazy, this condition can sometimes be corrected by cleaning the cell. Where cleaning does not improve the hazy condition, this may mean the black platinum coating on the electrodes has become worn. In this case, return the cell to the manufacturer for replatinizing and calibration.

**(k)** Check calibration of the cell once a week. This is done by measuring the specific conductance of the calibration solution, using the same procedure as described above for boiler waters, including setting the temperature adjustment to the temperature of the solution. Use of the neutralizing solution is omitted. Glassware, cell, and thermometer must be thoroughly cleaned and rinsed first with calibrating solution. The reading obtained should correspond to that of the calibrating solution at the temperature given on the solution bottle. If the reading is low, try cleaning the cell with 3N acid. If the reading

remains appreciably outside the range, return the cell to the manufacturer for repair.

### B-3. MISCELLANEOUS TESTS.

a. **Test for pH.** The value of pH indicates the degree of acidity or alkalinity of a sample. A pH of 7.0 represents the neutral point, lesser values denote acidity, and greater values denote alkalinity. The test is made as soon as possible after taking the sample. Avoid exposure to air as much as possible to minimize absorption of CO<sub>2</sub>. Several methods are available for pH determination.

(1) **Colorimetric Method.** This is represented by the Taylor comparator, which is sold by chemical supply sources in kit form. The method is simple, but to assure accuracy the indicator fluid must be standardized periodically.

(2) **USAEHSC Test Kit Method for pH of Boiler Water.** The test kit includes:

- (a) Two vials indicator paper, hydrions pH 10 to 12.
- (b) Two vials indicator paper, hydrions pH 11 to 12.
- (c) One 50-ml beaker.
- (d) One 2-oz. bottle

(3) **Procedure.**

(a) Remove a strip of pH 10 to 12 indicator paper from the vial and dip it into the sample in the beaker. Keep the paper immersed for 30 seconds and then remove it. If the sample does not change the color of the paper, or colors it yellow or very light orange, the pH of the sample is too low and the test is finished. If the paper turns orange or red, the pH is either satisfactory or too high.

(b) In that case, remove a strip of paper of pH 11 to 12 from the vial and dip it into the sample in the beaker. Keep the paper immersed for 30 seconds and then remove it. If the sample does not change the color of the paper, or colors it a light blue, the pH is satisfactory. If the paper turns deep blue, the pH is higher than necessary.

(c) **Adjusting the Chemical Dosage.** For most cases, a satisfactory pH determined by these directions indicates a pH in the boiler water in the range of about 10.5 to 11.5. If caustic soda treatment is being used to build up pH, a low pH indicates too little chemical is being used. The pH can be built up by increased treatment with the chemical. Too low a pH can also be brought about by excessive blowdown, excessive loss of condensate or improper operation of a zeolite softener. If the pH of the boiler water samples is high and caustic soda is being used, the dosage can be reduced.

b. **USAEHSC Test Kit Method for Condensate pH.**

(1) **Applicability.** This kit is designed for controlling the dosage of volatile neutralizing amines such as

morpholine or cyclohexylamine which are currently used to raise pH of the condensate, thereby reducing corrosion due to carbon dioxide.

(2) **Equipment.** The test kit includes the following:

- (a) One Dalite Lamp, Taylor #9195\*
- (b) One base, Taylor #9190
- (c) One pH Slide Comparator (pH range 6.0 to 7.6), Taylor #9066
- (d) Two four-ounce bottles of Bromthymol Blue, indicator for #9066 slide, Taylor #R-1003 K-D
- (e) One pH slide comparator (pH range 7.2 to 8.8), Taylor #9068
- (f) Two four-ounce bottles of Cresol Red, indicator for #9068 slide, Taylor #R-1003H-D
- (g) One pH slide comparator (pH range 8.0 to 9.6), Taylor #9070
- (h) Two four-ounce bottles of Thymol Blue, indicator for #9070 slide, Taylor #R-1003M-D
- (i) Five combination mixing tubes marked at 5, 15, 17.5 ml and #2 rubber stoppers.
- (j) One Test Tube Brush
- (k) One Stirring Rod
- (l) Three Plastic Droppers marked at 0.5 ml
- (m) Two 100 ml Beakers marked at 50 ml

(3) **Taking the Sample of Condensate.** Condensate return samples should be taken at the following points:

(a) Daily at the boiler plant main return to test for pH. Preferably, conductivity and hardness should also be checked on this sample.

(b) Samples should be taken at least once a week from the endpoints and midpoints of a large distributions system and tested for pH, hardness, and conductivity.

(c) In a system with several branches, samples should be taken weekly from alternate branches so that condensate chemistry (pH, hardness, conductivity) can be monitored and properly maintained. These samples are in addition to those specified in paragraphs (a) and (b) above.

(d) A sample should not be taken from a collecting tank if other water such as makeup is received in the tank.

(e) A sample is collected in the beaker up to the 50 ml mark. Cooling the condensate is not necessary. However, it should be collected slowly to reduce flashing. In the distribution system, the sample should be taken from a running stream from a heat exchanger.

(f) If the pH is not to be measured immediately, the sample should be collected in a glass bottle which should be completely filled and tightly sealed.

(4) **Operating Limits.** A minimum pH range of 7.5-8.0 is considered adequate for controlling corrosion. If morpholine is used, the minimum pH is to be maintained near the end of the distribution system. If cyclohexylamine is used, the minimum pH point will be observed closer

to the boiler. For the most satisfactory performance, especially in large steam distribution systems, it is recommended that both morpholine and cyclohexylamine be used in a 75/25 or 50/50 ratio of morpholine/cyclohexylamine. When using a mixture of amines, the minimum pH point must be determined locally through on-site chemical analysis of the condensate throughout the system.

**(5) Procedure.**

**(a)** Rinse three of the combination mixing tubes with the sample to be tested. Fill them to the first mark (5 ml) with the sample, wipe dry, and place in the holes back of the three slots in the Taylor base. The two outside tubes act as blanks to eliminate the effect of color and turbidity on the sample.

**(b)** To the middle tube add 0.5 ml of Cresol Red. (Taylor #R-1003K-D).

**(c)** Remove the tube from the base and mix thoroughly, using a clean stirring rod. Replace the tube in the middle hole.

**(d)** Place the Cresol Red pH slide comparator (pH 7.2 to 8.8) on the base so that one of the white lines on the slide is directly above the line on the base.

**(e)** Make comparison with the standards by holding the comparator toward a window or, preferably, placing it on the shelf in front of the Taylor Dalite lamp. Do not use fluorescent light.

**(f)** If the color of the middle tube does not match the color of either of the standards, or lies between the colors of the two standards, move the slide to the right or left, as required, until the next line on the slide is directly above the line on the base.

**(g)** If the color of the middle tube exactly matches one of the standards, read the pH value directly from the slide. If, however, the color of the sample lies between the colors of two consecutive standards, the pH is taken as an average of the two.

**(h)** Never consider a match with the lowest or highest standard as an accurate determination. The actual pH may be beyond the range of the indicator.

**(i)** Should a match appear to be likely with the lowest or highest pH color standard on the slide, repeat the test using a fresh sample and using Bromthymol Blue (Taylor #R-1003H-D) indicator and Bromthymol Blue pH slide (pH range 6.0 to 7.6) or Thymol Blue (Taylor #R-1003M-D) indicator and Thymol Blue pH slide (pH range 8.0 to 9.6).

**(6) NOTE:** Do not filter any samples for pH test. If the sample is too cloudy or turbid, allow it to stand in a closed bottle until the liquid is clear. Decant required amount of sample carefully.

## APPENDIX C HEAT BALANCE CALCULATIONS

### C-1. 100PSIG STEAM HEAT BALANCE

Reference figure C-1. (Note that this figure is a duplicate of figure 1-1.) The following assumptions have been made:

—100 lb of steam at 100 psig saturated conditions will heat water from 50° F to 140° F

—Percent energy is based on the energy in the steam input to the heat exchanger.

—Negligible heat is lost from the insulated piping, heat exchanger, vent condenser, or condensate tank.

—The vent condenser condenses 91% of the flash steam and cools the condensate to 100° F

—The amount of flash steam released is not effected by the water returned from the vent condenser. With this assumption our example serves to establish flash steam losses for systems without vent condensers.

**a.** The following steam table enthalpies in Btu/lb have been utilized:

For 100 psig steam  $hg_{100} = 1189.7$  and  $hf_{100} = 309.0$   
For 0 psig steam  $hg_0 = 1150.4$  and  $hf_0 = 180.0$

For water at 50° F  $hf_{50} = 18.0$

For water at 100° F  $hf_{100} = 68.0$

For water at 140° F  $hf_{140} = 107.9$

For water at 198° F  $hf_{198} = 166.0$

**b.** Percent energy in saturated water at the heat exchanger outlet is calculated as follows:

Base Energy =  $M \times hg_{100} = 100 \text{ lb} \times 1189.7 \text{ Btu}$

Energy at heater exchanger outlet =  $M \times hf_{100} = 100 \text{ lb} \times 309.0 \text{ Btu/lb} = 309,000 \text{ Btu}$

Percent energy at heat exchanger out = (energy at heat exchanger outlet divided by base energy)  $\times 100 = (309,000 \text{ divided by } 118,970) \times 100 = 26\%$

**c.** Energy from the 100 psig saturated water at 309 Btu/lb and 338° F reaches a new equilibrium after it exits the steam trap at 0 psig, 180 Btu/lb, and 212° F by flashing a portion of its mass to steam at 0 psig. The pounds of flash steam  $F_s$  released is calculated as follows:

$M \times hf_{100} = F_s \times hg_0 + (M - F_s) \times h_{f0}$