COMMITTEE FOR STANDARDIZATION OF CHEMICAL CONTROL.

Report for the Year Ending April, 1940.

Introductory.

Due to certain difficulties met by the Association regarding procedure in experimental work, the nomination of the personnel of the different committees by the Council was not undertaken until well on into last season; consequently it has only been possible to hold one meeting of this Committee.

In view of the fact that since 1932 a very large proportion of the Committee’s time has been spent in discussing strictly “official methods of analysis used in the determination of sucrose in cane, not to mention the lengthy side arguments that have arisen in connection with the little niceties and technicalities of mixed juice analysis, it was decided in the short time at our disposal to deal with a fresh subject.

The field of boiler control work has been rather overlooked in the past by most of the sugar mill laboratories. It is work in which the importance and functions of the chemist and his co-operation with the plant engineer, have come to be clearly understood in most important manufacturing industries.

When one considers the quality and the value of steam generating plant now installed by the Natal Industry, one will realise the desirability and the vital importance of a really strict control. It is doubtful if there is another industry in the world with the same amount of capital tied up in boiler equipment, which spends so little on a correct laboratory supervision.

By setting out these brief preliminary notes on methods the Committee on no account wishes it to be thought that there is any desire to interfere in a purely domestic control at the different mills; nor is it our intention to lay down anything in the way of “official” methods. But by bringing this matter up for discussion, we hope to bring into prominence a neglected subject. These ideas have already been put before the Boiler Committee, and the convener attended by invitation a meeting on October 13th, 1939, at which he was assured of their enthusiastic support in all work of this nature.

Before proceeding with the suggested methods of boiler and feed water analysis, this report will first deal with some other items of factory chemical control which the Committee desires to bring to the notice of all concerned.

Bagasse Sampling.

In connection with the recommended methods published last year (chap. 5, para. 2), the Committee is of the opinion that there should be no bar to the extension of the use of satisfactory automatic bagasse samplers, and expresses the hope that the Recommended Methods may be so amended as to make this possible.

Bagasse Analysis.

Cast iron digesters made to specification have been found to be perfectly satisfactory, and are recommended as an alternative to those made from sheet copper.

Condensers. Particular care must be taken to see that the condenser is working efficiently, and that there is no escape of steam during the boiling. The “Storch” Condenser has been found reliable provided that there is an ample, and continuous, supply of cold water. If this is at all doubtful it would be better to substitute larger condensers of the Liebig or a “worm” type, or even double surface compound condensers such as the Davies, or the Hopkins. The Christianson condenser as described in the Methods has the disadvantage that the cover of the digester is liable to warp. The latest modification of the Christianson condenser with a double top appears to overcome this difficulty. If the cover of the digester shows any sign of warping it would be as well to replace it with a heavier section metal than the prescribed ⅛”, as a good joint is essential.

Saccharimeter Tubes.

Tubes with push-on-ends, although eliminating the danger of cover glass distortion through overtightening, should be maintained in good condition regarding the frictional fit of the ends. If these become slack, causing leaks, they should be repaired or replaced.

Water-jacketed tubes with the side filling tubule are most reliable, the only weak point being in those tubes with an unprotected tubule.

An appeal is again made for vigilance against the overtightening of end-caps. The evidence is conclusive as to the very appreciable optical activity of cover glasses when so stressed. The chemist must be certain that only perfectly plane-parallel glasses are used.
Hydrometer Jars.

Hydrometers varying somewhat in their bulb dimensions, ample clearance should be allowed to take care of any differences in new stocks.

Horne's Dry Lead.

To date this defecant has proved most satisfactory. Difficulties which have arisen have usually been traced to lead subacetate that did not come up to the required standard. As has been previously emphasised at the time of the change-over to the use of the dry salt, it is necessary that the shaking should be vigorous and thorough, so that no large lumps remain in the solution, and several minutes standing should be allowed before filtration.

Clerget Sucrose in the Analysis of Mixed Juice.

Some chemists appear to have had difficulty in bringing the solutions for direct and invert polarization to the same temperature. There is no objection to leaving over the direct pol. until the completion of the inversion, then reading both tubes together, thus lessening likelihood of differences, and the Committee recommends the use of separate tubes for the two readings, these tubes being reserved for this item of analysis only. There are many small local difficulties in this matter which may be met with at different laboratories, but it would appear that this is a little problem which can be left to the ingenuity of the chemist concerned, the only stipulation being the reading of both solutions at temperatures within the limit stated.

Brix figure used in referring to Schmitz table. The Committee is agreed that if local conditions are such that there is a bigger variation than 5°C. between the temperatures of the four Brix determinations, then the four values corrected to 20°C. should be averaged, and adjusted back to uncorrected Brix at the temperature of the solution, taken just before pipetting the 50 ml. for direct pol.

Filtration of Crusher Juice Samples.

There have been some instances where considerable difficulty has been experienced in the filtration of crusher juice samples from abnormal or damaged canes. Some little time back the Committee was approached by the Central Board on this matter, and it was agreed that the following means for dealing with a cloudy filtrate in the testing of crusher juice samples should be tried in the order stated, but that these methods should only be adopted when it had been found impossible to obtain a sufficiently clear solution by the official procedure.

(1.) Use the official "Horne's Dry Lead" method but add a little kieselguhr to the solution before adding the reagent, then, after addition of the lead and thorough shaking, allow to stand for a few minutes, agitate once more and pour the solution on to a filter paper containing a few grams of kieselguhr.

(2.) Use Method (1) in sec. 4, p. 20, S.A.S.T.A. Methods, 1938, but for basic lead acetate solution use about 6 ml. of stock solution (S.A.S.T.A. 1), add a few drops of alumina cream, make to 110 ml., shake thoroughly, allow to stand for a few minutes, then filter through a paper containing some kieselguhr.

(3.) Use S.A.S.T.A. Polarization Method No. (3), taking one normal weight per 100 ml., using a fairly large excess of basic lead acetate solution and a few drops of alumina cream before making to the mark. Add a little kieselguhr, shake thoroughly and allow to stand a few minutes. Then filter through a paper containing a few grams of kieselguhr.

It is the opinion of the Committee that one or other of the above methods should yield a filtrate sufficiently clear for polarizing, and that it would be an extraordinarily refractory material that could not be so dealt with. In these cases, where the strictest accuracy must of necessity be abandoned for the sake of obtaining a reading, it may be necessary to deviate from the official procedure and return the filtrate to the filter paper several times. A small quantity of sodium phosphate added to the solution before making to the mark may also be of help.

(4.) However, should it be impossible to obtain a clear filtrate, the sucrose may be approximately estimated by determining the reducing sugars chemically before and after inversion. Use the Eynon and Lane method taking suitable quantities of the juice to be tested to bring the titration within range of the tables. Invert using Walker's method (S.A.S.T.A. Methods, 1938, p. 21, second para., sec. 5). Then the difference between reducing sugars before and after inversion will be the sucrose as invert sugar. Multiply by 0.95 to convert to sucrose.

It must be emphasised that this method should only be adopted in extreme cases.

Errors in Present Editions of Recommended and Official Methods.

In addition to the errata already supplied, the following should be noted:

Page 9, Ninth line from top:—For "millimetre" read "millilitre."

Page 13, eighth line from top:—For "each tank and weighed" read "each tank as weighed."
Page 18, fourth line from top:—Change (0.2 × 0.0067 = 29.85 ml) to (0.2 ÷ 0.0067 = 29.85 ml).

Page 21, seventeenth line from bottom:—Delete words “... the reciprocal of ...”

Page 29, thirteenth line from top:—For “32 gm.” read “32.5 gm.”

Standard Filter Cloth for Use in Revised Elliott Filtration Test.

The previous standard which was internationally agreed upon (“Facts About Sugar,” 29.1.34, p.p. 7—12), was the cloth manufactured by the California Cotton Mills, Oakland, known under the trade name M-451, Calcot, and specified as “2 yard, 40” drill 70 × 84 weave.” However, when a recent order was sent for the cloth it was learnt that manufacture had been discontinued. After exhaustive inquiries and the testing of many samples of cloth submitted, it was found at the Refinery that an exceedingly close match to the previous standard was obtained in a cloth supplied by The Filter and Press Cloth Co., of Stoke-on-Trent, England, which was in a bolt 29½” in width. This information is given for the benefit of mills that may be requiring to renew their supplies.

ANALYSIS OF FEED AND BOILER WATERS.

At mills where no control or examination of waters and water supplies has as yet been undertaken it must be realised that the first duty of the chemist will be the careful sampling, and complete analysis, of the waters from all the sources available to the factory.

The so-called “rapid methods,” including in this term soap, alkalinity, salt, pH and conductivity tests do not in themselves give sufficient information regarding the suitability or danger of a water as a boiler feed. But they are extremely valuable for routine testing when sufficient data from full analyses has been compiled.

For the purpose of these notes however, the control tests will be dealt with first, and the full water analyses, which come more under the heading of orthodox quantitative work will be briefly outlined subsequently.

Sampling.

The necessity for a strictly representative sample in ordinary sugar house control work is appreciated by all, and is no less important in this particular sphere. Local conditions will alter the form that actual samplers may take, and the exact points at which the various samples are drawn. The following principles should nevertheless always be borne in mind.

Where a softener or treatment plant is employed, time must always be allowed for a change in chemicals to become effective. Experience with the particular plant will soon indicate how long after a change a sample will give a true indication of the new conditions. Where external treatment of the make-up water is in existence, fairly complete analyses of raw and treated waters should be undertaken daily. These samples may well be collected in glass stoppered Winchesters and the sampling vessel should always be plunged well below the surface of the water, thus avoiding surface contamination.

A sample which is obtained from a tap, cock, pump or sampling line more than a few feet in length should only be taken after sufficient water has been run to waste to ensure a thorough flushing.

The best point for the taking of feed samples is not from the feed tank, but from the discharge side of the pressure feed pump.

Where estimation of dissolved gases is to be made, the sample should always be passed through a suitable cooling coil before bottling, and should be collected, at no higher than atmospheric temperature, in glass bottles fitted with rubber stoppers and glass tubing arranged in such a way that the air is displaced and the sample water flows from the bottom to the top. The minimum rate of filling should be 250 ml. per 30 secs., and the sample bottle should be permitted to overflow not less than 5 minutes. After this time the rubber connecting tubes of the sample may be clipped, and the sample removed to the laboratory in this original container, where the analysis should be done with the least possible delay.

Boiler samples drawn from a water guage cock should be passed through a metal gauze filter and efficient cooling coil, and care should be taken to see that the top steam cock is closed, otherwise the sample will be diluted with condensed steam. Metal vessels should not be used for these samples.

Units.

There appears to be some quite unnecessary confusion regarding the meaning of the various units employed in reporting water analyses. Many engineers have become accustomed to hearing results only in terms of grains per gallon, and regard other expressions as somewhat unintelligible. It should be realised that most results are converted to the equivalent in parts of calcium carbonate, even though this substance may not be present in the water at all. It then becomes a question as to the total volume against which these “parts” are to be shown. Grains per gallon, Clark’s degree, the English degree, are synonymous terms indicating
1 part in 70,000. The usual chemical expression is in terms of parts per 100,000, and this unit is now coming into almost universal use in engineering and chemical literature. It is quite useful as a method of routine control.

**Dissolved Oxygen.**

**Rough Sorting Test.**—This method is suitable for dissolved oxygen in quantities greater than 0.5 cc. per litre, and as all the errors of sampling and analysis are invariably on the side of high results, it is quite useful as a method of routine control.

**Solutions required.**—(1) Methylene blue indicator (1 part in 1,000), (2) standard ferrous sulphate solution; 2.2 gms. pure ferrous sulphate (FeSO₄, 7H₂O) dissolved in 500 ml. distilled water and 10 ml. of pure concentrated sulphuric acid are added. The whole is made up to 1 litre. (3) alkaline tartrate solution; 350 gms. of Rochelle Salts and 100 gms. sodium hydroxide are dissolved in about 750 ml. of distilled water, cooled, and made up to 1 litre. Then 1 ml. of the ferrous sulphate solution is equivalent to 1 cc. of oxygen per litre, when operating on 50 ml. of sample.

**Procedure of Test.**—Carefully remove rubber stopper for the sampling bottle, and pipette 50 ml. into a glass cylinder of about 100 ml. capacity. In doing this take great care to cause as little disturbance to the liquid as possible, and there should be no splashing or bubbling whatsoever. Previously wet the outside of the pipette and the inside of the glass cylinder with distilled water to prevent adherence of air bubbles. Now add 5 ml. of the tartrate solution carefully by means of a pipette, and one drop of the methylene blue indicator. In the presence of dissolved oxygen a faint but distinct blue colour will remain. Now slowly run in the ferrous sulphate solution from a 10 ml. burette, stirring the sample meanwhile by means of a small propeller stirrer which just fits into the cylinder, or in the absence of this, with a glass rod having an off-set end. Again, of course, there must be no splashing. Continue the titration until the blue colour just disappears. Repeat the operation and take the mean of the two readings. Then each ml. of ferrous sulphate used is equivalent to 1 cc. of oxygen per litre in the water being treated.

**Winkler's Test.**—This method is to be used where greater accuracy is required or where, after de-aeration, the dissolved oxygen content is below 0.5 cc. per litre.

**Solutions required.**—(1) Manganese chloride; Dissolve 425.5 gms. of crystalline manganous chloride (A.R.) MnCl₂, 4H₂O, in 1 litre of distilled water. (2) alkaline iodide; 360 gms. of sodium hydroxide (A.R.) and 100 gms. potassium iodide are dissolved in distilled water and the solution made up to 1 litre. (3) Sodium thiosulphate; dissolve 6.205 gms. of Sodium thiosulphate (A.R.) in distilled water and make to 1 litre, using only freshly boiled and cooled distilled water. Each ml. of this solution equals 0.2 mg. of oxygen, or 0.1395 cc. of oxygen at N.T.P. This solution should be standardised as indicated for S.A.S.T.A. Reagent 21, or against 0.025 N. potassium bichromate. (4) Sulphuric acid; A dilute acid consisting of equal parts of pure concentrated acid and freshly boiled distilled water. (5) Starch Indicator; S.A.S.T.A. 11.

This method is based upon the oxidation of manganous chloride to the manganic form, and the subsequent reduction of the iodide to liberate free iodine. This iodine is exactly proportional to the amount of oxygen dissolved and is determined by the titration against the standardised sodium thiosulphate.

In the test it is advisable to take the sample in the flask which will be used later when the addition of the reagents is made. A narrow necked flask of 250 to 300 ml. capacity is used, and is fitted with the ordinary two hole rubber stopper when collecting the sample, but when brought to the laboratory this stopper is replaced by one which fits the neck of the flask snugly but may yet be pushed well down into the neck. This latter stopper is bored with a single hole, into which is fitted a short-stemmed dropping funnel of about 10 ml. capacity, having a narrow capillary tube.

**Procedure of Test.**—When the completely filled bottle or flask is brought to the laboratory the stopper and tubes are carefully removed, and 2 mls. each of the manganous chloride and alkaline iodide solution are added from a pipette, the end of which reaches well into the flask. Reagents should be added in the order mentioned. The flask is then closed with the second stopper fitted with the funnel, taking care no air bubbles are entrapped. With the stopper pushed well home the liquid is forced into the capillary tube, and on closing the tap the flask is completely sealed. Now shake gently and permit the precipitate to settle. The solution above the precipitate should be clear and colourless, otherwise further gentle shaking is indicated. Now place 2 mls. of the sulphuric acid in the funnel, open the stopcock and withdraw the stopper very gradually, thus drawing the acid into the flask. Do not allow
air to enter before closing the stopcock. Again mix by shaking, this time thoroughly until the precipitate dissolves. After this, 250 ml. may be transferred to a conical flask and titrated with the 0.025 N sodium thiosulphate. Do not add the starch until the solution becomes a pale yellow colour. If the sample contains oxygen in excess of 0.5 ccs. per litre the water will have a decided yellow colour. If only traces of oxygen are present the water will not appear yellow and the starch solution should then be added immediately.

If 250 ml. of water are titrated then
\[
\frac{1000}{250} \times (\text{ml. of 0.025 N thiosulphate} \times 0.1395) = \text{ccs. of oxygen per litre at N.T.P.}
\]

The above method is satisfactory under normal conditions, but should much organic matter or nitrates be present then the sodium azide modification of the Rideal-Stewart method should be used. (Ruchhoft, Allan Moore and Placak. Ind. & Eng. Anal. 1938, 10, 12, 701).

Note.—A useful apparatus for carrying out the Winkler test is that devised by J. N. Friend, obtainable from laboratory suppliers, also the “Admiralty Testing Set” from Messrs. Griffin & Tatlock.

Free Carbon Dioxide.

This determination may often be useful in the examination of treated waters.

Decant 100 ml. of sample into a stoppered 100 ml. cylinder and use 100 ml. of recently boiled and cooled distilled water as a standard in another cylinder. Add 10 drops phenolphthalein to the blank, then standard sodium carbonate, drop by drop, stopping and gently mixing between additions. When a permanent pink is obtained note the burette reading. Deal with the sample in a similar manner, but in this case the titration may be done more rapidly. Then the mls. of sodium carbonate required by the sample—the quantity required by the blank = sodium carbonate solution used up by the free carbon dioxide. (According to whether results are required in grains per gallon or parts per 100,000 so the sodium carbonate solution will contain either 3.446 gms. Na₂CO₃ per litre or 2.4122 gms. per litre).

Total Hardness.


Solutions required.—(1) N/10 sulphuric acid and Methyl orange indicator.

Procedure for Test.—Take 100 ml. of sample, add two drops of methyl orange and titrate with N/10 sulphuric acid until the colour just changes from yellow to pink. Then the number of mls. used \( \times 5 = \) temporary hardness in parts of calcium carbonate per 100,000.

Permanent Hardness.

This is taken as being equal to the figure obtained by deducting temporary hardness from total hardness.

Magnesia Hardness.

Take 200 mls. of the sample, add methyl orange indicator, neutralise with N/10 nitric acid, and then add 4 drops excess acid. Boil to expel carbon dioxide. Then, while still boiling, add 5 mls. of a saturated solution of sodium oxalate and continue boiling for 1 minute. The calcium present will be precipitated, leaving only the hardness due to magnesium. Now cool, add phenolphthalein, neutralise with sodium hydroxide and acid, and titrate with potassium palmitate exactly as described under “Total Hardness.” Divide the burette reading by 2.
and multiply by 5, thus obtaining the magnesium hardness expressed as parts calcium carbonate per 100,000.

It follows from the above that calcium hardness = total hardness - magnesium hardness.

The methods which have been given above for Dissolved Oxygen, Free Carbon Dioxide, Total, Temporary, Permanent, Magnesia and Calcium Hardness, can well be applied to all feed waters, whether any treatment has been undertaken or not.

With Boiler Concentrates and softened waters it may be desired to obtain further information regarding the nature of the alkalinity, for the purpose of calculating the amount of chemicals required, or deciding on any change in treatment. The following test is then usually undertaken.

Alkalinity.

Deep porcelain dishes are the best vessels to use for the titration. Take 100 ml. of the sample to be tested and add a few drops of phenolphthalein indicator. If no pink colour is produced proceed with the methyl orange titration described below. If pink, then slowly add N/10 sulphuric acid from a burette, stirring with as little agitation as possible, the jet being near the surface of the water. Titrate until the pink colour just disappears. Then mls. acid used \( \times 5 \) gives phenolphthalein alkalinity (Pt) in parts CaCO\(_3\) per 100,000.

Now add 0.5 ml. methyl orange and continue the titration, stirring well meanwhile, till the colour changes from yellow to red.

(Note.-It is helpful to use two dishes in this titration, the one containing a comparison standard consisting of a little over 100 mls. of distilled water, plus methyl orange, and 0.4 mls. of the standard acid. Then if the titration is continued until the colour of the test solution exactly matches this blank, 0.4 ml. must be deducted from the burette reading to obtain the amount required by the water.)

The total number of mls. of acid used in the titration, from the start, \( \times 5 \) = parts methyl orange alkalinity as CaCO\(_3\) per 100,000 (call this Mo).

Then if—

1. \( Pt = Mo \), there are present hydroxides only (caustic alkalinity).

2. \( Pt = \frac{Mo}{2} \), there are present hydroxides and normal carbonates.

3. \( Pt = \frac{Mo}{2} \), there are present normal carbonates only.

4. \( Pt < \frac{Mo}{2} \), there are present normal carbonates and acid, or bi-carbonates.

5. \( Pt = 0 \), the Mo titration represents bi-carbonate only.

In case (2) the alkalinity as carbonate will be 2 (Mo - Pt), and the caustic alkalinity Mo - 2 (Mo - Pt).

In case (4) the alkalinity as carbonate will be 2 Pt, and that due to bi-carbonate Mo - 2 Pt.

Total Chlorides.

**Solutions required.** — (1) N/10 silver nitrate; Dissolve 16.989 gms. silver nitrate (A.R.) and make up to 1 litre. (2) potassium chromate indicator; 10% solution, free from chlorides.

**Procedure for Test.**—For normal feed waters take 300 mls. and evaporate to about 100 mls., and for boiler waters 20 mls. of sample (filtered in latter case). Add 1 drop methyl orange to the water under test, and titrate with N/10 nitric acid until just pink. Then add 2 drops potassium chromate and run in the N/10 silver nitrate from a burette, slowly and with constant stirring. Continue until a permanent brick-red colouration is obtained. It is as well to do a blank titration using distilled water instead of the sample. Deduct the amount of N/10 silver nitrate required for the blank from that used in titrating the sample.

Then
\[
\frac{\text{mls. AgNO}_3 \text{ used}}{3} \times 3.55 = \text{parts Cl per 100,000.}
\]

mls. multiplied by 5.85 gives parts sodium chloride 3

**Sulphate Radical.**

This should be estimated by the usual gravimetric procedure. With feed waters take 500 mls. and evaporate to 100 mls.; with boiler waters take 100 mls. Acidify with hydrochloric acid, add 5 mls. 10% ammonium chloride, bring to the boil and add 10% barium chloride solution to slight excess. Boil for ten minutes, cool, filter, ignite and weigh.

Then
\[
\text{gms. BaSO}_4 \times 411.5 = \text{parts SO}_4 \text{ per 100,000.}
\]

**Carbonate Radical.**

This may be calculated from the alkalinity determination as carried out above with phenolphthalein and methyl orange titrations. The CO\(_3\) radical, in parts per 100,000 = 1.26 (Mo - Pt).
Phosphate Radical.

A colourimetric method is used employing the same apparatus and technique as in the Atkins Method for phosphate in juice.

Solutions required.—(1) Sodium phosphate Na₃P₀₄.12H₂O; 0.32 gms. per litre: (2) Sulphuric acid; 600 ml. water plus 300 ml. A.R. 95% sulphuric acid. (3) Ammonium molybdate, (NH₄)₂MoO₇.4H₂O; 92.3 gms. per litre plus 38 ml. conc. sulphuric acid. (4) Hydroquinone; 23 gms. per litre plus 5 ml. conc. sulphuric acid. (5) Alkaline sodium sulphite: 183 gms. NaOH plus 16 gms. sodium sulphite, Na₂S₀₄, per litre of water.

Procedure for Test.—To 40 mls. of the sample add 5 mls. sulphuric acid and mix thoroughly. Then add 1 ml. each of the molybdate and the hydroquinone. Again shake vigorously and then allow to stand for 5 minutes. Now add 15 mls. of alkaline sulphite. Any phosphate present will develop a blue colour which may be compared with standards in the usual way. Compare the colours immediately, as they fade rapidly and the colour due to silicate appears.

Each 1 ml. of the standard phosphate solution under the conditions of the test is equivalent to ½ part P₂O₅ per 100,000.

The tests for phosphate must always be carried out on perfectly clear, filtered samples.

Hydrogen Ion Concentration.

The usual colourimetric methods already employed in the factory control may be used for this purpose. For boiler water, or blow-down testing, however, two additional indicators will be necessary.

Nitro-yellow ... ... ... 10.0 to 11.6 pH.
Sulpho-orange ... ... ... 11.0 to 12.6 pH.

Cresol red (7.2 to 8.8 pH) is recommended in place of phenol red.

Where the apparatus exists, or can be obtained, it is strongly advised that electrometric methods should be used for the determination of the pH of all waters. The glass electrode is by far the most suitable for use, except in very high concentrations of alkalinity.

Conductivity.

Conductivity control of both feed and boiler waters is exceedingly useful. At this stage, however, no attempt will be made to set out any method, until the Committee receives some information as to the different types of apparatus for this test which may be installed in mill laboratories. When a sufficient number of analyses for total solids and chlorides, in conjunction with the conductivity test, have been done, it becomes possible to plot a curve showing the relationship between conductivity and these two items, thus saving a considerable amount of time in routine analysis work.

It will of course be realised that the curve obtained will only be applicable to that particular type of water, and any considerable change in composition necessitates fresh chemical analyses.

The “Dionic” tester will prove of great practical value in these tests.

Applications and Interpretations of the Above Tests.

It is not the object of the Committee, nor is the space available, to set out now all the accepted safe standards and conditions of water for good working. To do so, and to enter into a discussion of all the pros and cons regarding different theories of corrosion, caustic embrittlement, priming, scale formation, et al, would occupy a good sized volume.

If the discussion on these notes at the Conference displays sufficient interest on the part of factory technologists to proceed further with the pooling of ideas on this work, information could be obtained from the mills as to the different waters, treatments, and types of plant with which they have to deal.

Then it will be the function, not of this Committee alone, but of the Boiler Committee also, to decide on what recommendations to make as to what will constitute good practice.

With co-operation it should be possible to make suggestions of great practical value to the industry, as has already been done in their own sphere by the Boiler Committee.

However, a few notes are made here with the idea of opening discussion.

Medium Pressure Boilers.

Feed Water Conditions.—Dissolved oxygen by “Winkler Test” less than 0.1 ccs. per litre.

Chlorine should be less than 0.43 parts per 100,000. Alkalinity should be definitely caustic soda alkalinity, less than 0.71 parts per 100,000. The pH should be near 8.0. The total solids should be as low as possible. Conductivity will vary with the ratio of make-up of condensate used, and the test is a good basis for calculating the quantities of these materials.

Boiler Water Conditions.—Caustic soda alkalinity should be less than 70 parts per 100,000, chlorine less than 45 parts per 100,000. Hardness should be near zero, and there should be no trace of oil. The ratio of sodium sulphate to alkalinity (total alkalinity as parts CaCO₃) should in no case be less than 2 to 1.
The concentration of total solids permissible can be determined by a comparison of such tests with a record of troubles encountered from excessive moisture in steam, such as sudden drops in superheat temperatures, trouble with engine lubrication or deposits in superheaters and steam pipe-lines, or by noting the quality of steam corresponding to various concentrations, by a throttling calorimeter, or chemical tests on samples of condensed steam.

**Outline of Tests Required.**

Briefly, these should be:—

**Feed Water.**—Solids, pH, caustic alkalinity, hardness, dissolved O, sulphate-carbonate ratio, conductivity, chlorides (Conductivity curves will eliminate some of these items).

**Boiler Water.**—Solids, alkalinity, pH, sulphate-carbonate ratio, phosphate, conductivity.

**General Analyses.**

As mentioned previously, a full mineral analysis of all waters and supplies should precede any treatment or control work. The Committee feels, that unless a call is made for this information, the details of analysis are so well set out in all the better known text-books on quantitative work that no further publication is needed here.

The items required are:—

1. Suspended matter.
2. Total solids.
3. Organic matter.
4. Silica.
5. Iron and alumina.
6. Calcium.
7. Magnesium.
8. Nitrates.
10. Chlorides.
11. Sulphates.
12. Oil.

(Not.—A useful test for item (12) is as follows: Take 1 litre or more of water, in small quantities at a time, and shake with ether in a separating funnel, keeping the ether layer at least 1" deep. Distil off the ether in a distilling flask and dry the oil to constant weight, preferably in vacuum oven).

**References.**

In the compilation of the above notes the following texts have been referred to, and in some places, drawn upon:—

1 "Fuel, Gas Water and Lubricants" S. W. Parr 1932 (McGraw-Hill).
2 "Boiler Feed Water Treatment" F. J. Matthews 1935 (Hutchinson's).
3 "Boiler Feed Water," P. G. Jackson, 1922 (Charles Griffin).

**COMMITTEE FOR STANDARDISATION OF CHEMICAL CONTROL, 1939-40.**

W. BUCHANAN.
G. GALKRAITH.
G. S. MOBERLY.
J. RAULT.
J. L. du TOIT.
M. VIGER.
F. W. HAYES, Convener.
Mr. HAYES, commenting on the report, stated that the two items referred to during Mr. Dodds' paper, viz. the determination of brix of molasses and the use of the Jackson & Gillis method for sucrose in molasses, could now be enlarged upon. The first item was dealt with by Dr. Hedley and himself in a paper some years ago (Proc. S.A. Sugar Tech. Assoc. 1932, 9) and need not be discussed further.

Regarding item two, he did not hesitate to accept the Jackson & Gillis method No. 4 as the most accurate one they had to date. Mr. Hayes then suggested that discussion on the second part of the paper should be withheld until he read his paper on boiling water treatment.

The PRESIDENT, in opening the discussion, said he was pleased to note the work given to testing boiler feed water.

Dr. HEDLEY stated that the study of boiler feed water was long overdue. The mills went to great expense erecting boilers, and it was time that they recognised the need for controlling the boiler feed water.

Mr. BECHARD, referring to bagasse analysis, said the cast iron digesters were standard and not made to specification. Again, with respect to water-jacketed saccharimeter tubes, they are not made with push-on ends, but are screwed on. Considerable care had to be taken when screwing the ends on. Regarding the brix figure used in referring to Schmitz table, he personally preferred averaging the uncorrected brix and temperature, getting the temperature of polarization and working back from that. It was difficult to get in the specified time a temperature approximately the temperature at which polarization was going to be carried out eventually.

Mr. HAYES referred again to the determination of sucrose in molasses. After many years investigation the Jackson & Gillis method No. 4 had been adopted. With this method one did get comparative results under routine conditions, though they might not be scientifically accurate. Sufficient attention to detail was not always given by mill laboratories, and this was an important source of errors. With regard to the bagasse digester introduced by Mr. Bechard, they were made to a specification which the Committee deemed satisfactory. Regarding the temperature figure for the average brix of the mixed juice sample, he said that this was required for referring a volume measurement back to a weight measurement by means of the specific gravity, so that the temperature taken in the polarisation tube would not be strictly accurate. Mr. Hayes went on to invite Mr. Bechard to submit his silver sulphate method to the Committee. The Volhard method, he said, had not been tried out. Mr. Duchenne's question would be answered during his next paper. Mr. Hayes concluded by thanking all for their criticisms and suggestions.