

The image shows the front cover of an old book. The cover is decorated with a marbled paper pattern featuring large, irregular brown patches separated by a network of dark blue and reddish-pink veins. A small, rectangular, cream-colored label with a blue border is centered on the cover. The label contains the text 'F.A.S.' in large, bold, red capital letters, followed by 'method' and 'Book.' in smaller, red, cursive script. The left edge of the image shows the dark, worn spine of the book.

F.A.S.
method
Book.

F. A. Simonds 1900



Water etc Analyzers

BRE/1/30

2026

Analysis of Water.

Purity

Preliminary Examination

Ammonia, free & albuminoid.

Oxygen absorption test.

Nitric & Nitrous Acids as nitrates & nitrites.

Chlorine

Poisonous metals. Lead Copper & Iron.

Phosphoric Acid.

~~Total solid matter in solution.~~

Mineral Constituents.

Total solid matter in solution.

Sulphuric Acid.

Silica, Alumina, Iron Lime & Magnesia.

Lime as carbonate

Soda & Potash

Instructions of collecting samples of water.

If sample is to be taken from a tap, allow several gallons to run to waste, & rinse ~~thoroughly~~ with this water, before filling. Tie the stopper tightly down.

Particulars should be given as to where the water comes from & the soils it passes through, & its proximity to cesspools & drains. Corks should be avoided if possible unless quite new.

Analysis.

A water should be analysed as soon as possible after the sample has been taken.

Purity

1. Observe the colour in a long glass cylinder against a white surface.
2. The Taste & Smell of a water should be taken, the latter after warming gently in a closed vessel & opening suddenly.
3. Test the acidity after boiling: alkaline carbonates are often present & the CO_2 will be given off on boiling & excrete the alkalinity.

Microscopic Examination

1. The first step in the microscopic examination of a specimen is to prepare a slide. This involves mounting a small portion of the specimen on a glass slide and covering it with a cover slip. The specimen should be mounted in a liquid medium, such as water or a special mounting medium, to prevent it from drying out and to allow for better visualization under the microscope.

2. The next step is to place the slide on the stage of the microscope and focus on the specimen. This is done by adjusting the coarse and fine focus knobs. Once the specimen is in focus, the observer can begin to examine it at low magnification.

3. After examining the specimen at low magnification, the observer can increase the magnification by rotating the nosepiece to bring a higher power objective into use. This allows for a more detailed view of the specimen's structure.

4. The final step in the microscopic examination is to draw a diagram of the specimen. This involves sketching the observed structures and labeling them. A diagram helps to record the findings of the examination and provides a visual aid for discussion and analysis.

5. The last step is to clean the microscope and store it properly. This involves removing any debris from the stage and objective lenses, and returning the microscope to its original position. Proper maintenance of the microscope is essential for accurate and reliable results in future examinations.

Test for Ammonia

In performing these tests our utensils & reagents must be quite free from ammonia. All the distilled water, KMnO_4 solution etc must be thoroughly tested by distilling small quantities & testing with Nessler, & purified if necessary.

The apparatus must be tested by boiling in it some distilled water free from ammonia & should be purified by boiling in it some alkaline potassium permanganate.

All contact between steam & indiarubber connections should be stopped.

After proving apparatus free from ammonia by distilling with Na_2CO_3 & testing distillate with Nessler, 500 cc of water to be tested is run in with 1 gm of Na_2CO_3 . The whole is distilled & received in Nessler jars. Add to the first jar (containing about 50 cc) 1 cc of Nessler & match it with another jar containing 40 cc ammonia free water ^{a definite volume} & ~~1 cc~~ of standard solution of NH_4Cl . colour the latter with ~~1 cc~~ ^{as required} of Nessler: as required ^{add NH_4Cl} the two jars are of equal tint. ^{Fresh samples of NH_4Cl must be taken every time} The solutions should be allowed to stand a few minutes before judging the tint. A second & third jars of distillate are thus tested.

Test for Albuminoid Ammonia.

When the distillate gives no indication of ammonia, 50 cc. of Potassium Permanganate solution is added to the retort & the boiling continued. The distillate is tested in the same way as before with Nessler.

Calculations of results,

Ammonia free & saline (500 cc)

1st tube requires 3 cc. NH_4Cl to react.

2nd tube — 1 cc. —

3rd tube — Nil —

Total — $\frac{\text{Nil}}{4}$

Oxygen absorption test.

Two quantities of 200 cc of the sample are poured into clean stoppered bottles capable of holding about 300 cc.

To one is added 10 cc of a standard solution of KMnO_4 containing 0.1 gm of available oxygen per cc. Add a few drops H_2SO_4 & put in forcing tray at 80°F for 4 hrs.

Treat another bottle similarly for $\frac{1}{4}$ hr.

To each bottle add a few drops of potassium iodide solution & 1 cc of clear freely prepared starch. They are then treated with a standard solution of sodium thiosulphate from a burette, & the bottles shaken until the ~~blue~~ blue colour has disappeared. The no of cc employed to decolourise each bottle is then read off & noted.

A blank experiment must be made to standardise the thiosulphate solution with 200 cc of distilled water, & permanganate etc as above.

Nitrous Acid.

Add a few drops of H_2SO_4 , then a few ccs of potassium iodide & a little starch paste to about 100 cc of the water contained in a beaker. A blue colouration will be formed in proportion to the nitrous acid ~~formed~~ present & the amount may be returned as slight trace, trace etc. The mixture should be allowed to stand for 24 hrs before an opinion is formed.

The iodide must be free from iodate & have been kept in coloured bottles in a dark place.

To detect iodates in the presence of iodides acidulate the solution with H_2SO_4 & add a little starch paste: if a blue colour is produced an iodate is present.

Nitrates.

Evaporate ^{to dryness} over a water bath 100 cc of the H_2O to be examined in a porcelain dish & in another evaporate 1 cc of weak standard nitrate solution, & the number of cc of the standard NaCl solution as the Chlorine in the water equals in 100 cc. At once remove from bath & add to each 2 cc standard phenol solution, also a little distilled water & ammonia in excess. A yellow colouration is produced in both: make up to 50 cc & bring to the same tint by taking as many cc of the water as will match the standard when diluted to 50 cc.

Chlorine

100 cc of the sample are poured into a white porcelain basin & one or two drops of potassium chromate solution added.

Standard silver nitrate solution is now run in with constant stirring until a permanent orange tint is just obtained. The number of cc employed is read & noted.

Phosphoric Acid.

It is as a rule only necessary to estimate approximately the quantity of this acid.

Evaporate 500 cc of the water acidified with nitric acid & then add ammonium molybdate solution when about half bulk. The amount of yellow ppt is then judged & returned as 'minute trace', 'trace' or 'heavy trace'.

Total solid matter in solution.

500 cc of the water are evaporated on the water bath in a weighed platinum dish which is kept filled up as the bulk evaporates. After evaporation the dish is placed in a hot air oven at 260°F . which drives off any water of crystallisation but is not hot enough to decompose the organic matter. It is weighed after 2 hrs & every hour after until constant.

The residue is now gently ignited over a spirit flame in such a way that one part after another of the solids is subjected to the heat of the flame. The degree of discoloration etc are noticed. The ignition is continued until the residue is perfectly white or brown in appearance, the latter occurring if iron is present in any appreciable amount, but should not last more than a minute or so or decomposition of salts will result. Cool the dish & moisten with a few drops of ammonium carbonate & dry first on water bath & then in oven. Weigh & dry at intervals until constant.

Quantitative estimation of Iron.

100 cc of the water under examination is evaporated to half bulk having added 5 drops of pure HNO_3 , transfer to a Nessler jar & add a few drops of potassium ferrocyanide. A blue colouration is produced according to the iron present.

It is then nesslerised with a standard solution of iron ($1 \text{ cc} = 0.1 \text{ mgm Fe}$). Fill the other jar with distilled water & the quantity of standard solution considered necessary to equal the blue colour of the water.

Silica, Alumina, Lime & Magnesia.

500 cc of the sample & a few cc HCl are evaporated to complete dryness. After cooling add 40 cc distilled H_2O 2 cc HCl & a few drops of nitric acid, & digest for ~~a~~ half an hour. The Silica will remain insoluble. The whole is filtered & washed with warm H_2O , silica being collected & dried etc as usual. The filtrate is now made strongly ammoniacal & boiled. The iron & alumina are precipitated as hydrates, & collected on a filter paper, & washed with hot H_2O . (see opposite page for quantitative estimation of iron if required) The filtrate is next evaporated to a convenient bulk & then heated with excess of ammonium oxalate, & some ammonium chloride & ammonia. The liquid should be warmed a few minutes & then stood several hours. The calcium oxalate formed is filtered off washed with warm H_2O & dried etc. The dry ppt & paper are incinerated in a tared crucible & allowed to cool, then moistened with ammonium carbonate & gently warmed over a rose burner & weighed as calcium carbonate.

(Several weighings till constant should be taken.)

The filtrate is next evaporated to convenient bulk, rendered strongly alkaline with ammonia & then sodium phosphate is added. The contents of the beaker are gently warmed & well stirred for a few minutes & the whole allowed to stand 12 hrs before filtering. The ppt should be well washed with dilute ammonia, & then ^{dried} strongly ignited & weighed as usual as pyrophosphate of ammonia.

Sulphuric Acid

500 cc of the sample are evaporated in a porcelain dish with the addition of about 12 drops HCl until only $\frac{1}{3}$ rd is left

Barium Chloride is now added, the whole digested for an hour.

The precipitated barium sulphate is thrown on to a filter washed with warm water, dried ignited & weighed as usual.

Soda & Potash.

500 cc of the sample are evaporated in a porcelain dish with the addition of a few drops HCl to partial dryness. Add Barium Hydrate solid in excess until the reaction is just alkaline & a scum forms. The whole is then digested for one hour & then filtered, the ppt being well washed with hot H_2O & discarded. To the filtrate add excess of ammonium carbonate (solid) & digest for one hour: filter & well wash with H_2O (hot) & discard f-paper. The filtrate is then evaporated in a tared platinum dish to 25 cc: a few cc of ammonium chloride added & the whole evaporated to dryness. When all is evaporated the dish is gently heated over a rose-burner for 3 or 4 hrs to expel ammoniacal fumes, but should never be red hot. Cool & weigh. The residue is next extracted with hot H_2O into a f-paper which is dried & ignited & weighed in the same platinum dish. The difference between this & the former weight is weight of the combined chlorides of sodium & potassium. The washings containing the sodium & potassium chlorides are made up to 100 cc.

Evaporate with about 3 cc platinum chloride to dryness in porcelain dish. Dissolve in alcohol & filter on to a counterbalanced filter paper, wash with alcohol, & ^{dry} filter at $212^\circ F.$ & weigh.

Analysis of Invert Sugar

Invert Sugar.

Dissolve about 1 gm of the sugar in 100 cc of water; boil 50 cc of Fehling's solution diluted with 50 cc water in a bath for 10 minutes: then 10 cc of the sugar solution is added & the whole boiled for exactly 10 minutes. It is then filtered, washed until the last traces of blue disappear & burnt in a weighed crucible.

It is then allowed to cool & moistened with sufficient nitric acid to dissolve the whole of the copper, then well warmed till all the nitric acid fumes are driven off: it is then heated over a full burner for about 15 mins, allowed to cool in a desiccator & weighed.

(The latter part is sometimes omitted & the crucible weighed directly after heating)

Uninverted Cane Sugar.

When cane sugar is digested at 150 F for 20 mins with a little hydrochloric acid, it is completely inverted, & the amount is determined by taking the polarimeter reading before & after inversion.

A preferable method is to invert with about 1 gm of yeast at 125-130 F for 1-2 hrs

First Method (Acid Inversion)

26.048 of the sample are dissolved in 100 cc of water & the reading determined in a 1 decimetre tube at a known temperature.

50 cc of the same solution are digested with 5 cc HCl at 150 F for 20 minutes: Cool & make up to 55 cc & the reading again determined.

2nd Method. Yeast Inversion.

This is carried out in a similar way to the acid method using yeast as the inverting agent; the inversion is carried on for 2-3 hrs at 125°F ; at the end of that time it is cooled, alumina added & made up to the mark. Filter, & take the opticity of the filtrate.

It is as well to add a little thymol dissolved in dilute alcohol to prevent fermentation.

Mineral Matter

About 5 grams of the sample are carefully incinerated in a tared platinum dish & the ash weighed after cooling in desiccator.

Albuminoids (Kjeldahl's)

This is determined in the ^{same} way as malts: 2 grams of sugar should be taken & acid added at once: possibly another 10 cc acid may be required if green coloration is not obtained with permanganate.

Acidity

This may be neglected in sugars.

Moisture & total solid matter in solution

About 20 grams of the sugar are dissolved in ~~100~~ 200 cc warm water & made up to 100 cc at 60°F . (the same solution as for inversion will do \therefore 40 grams in 200 will be required)
The Specific Gravity is then determined.

Brewer's Extract

This is determined from the Specific Gravity of 20% solution

Inert Matters

The so-called inert carbohydrates are obtained by adding together the above constituents as determined by direct analysis & then subtracting from the total solids in solution.

Analysis of a Glucose

Make 200 cc of a 20% solution (neglect 3rd place of decimals in weighing out sugar).

On this determine

- (1) Extract. Determine Specific Gravity.
- (2) Optical Activity in a 100 mm tube
- (3) Reducing power after fermentation

Take 100 cc of 20% solution, put in flask (forcing tray), add about 50 cc H₂O & 10 cc yeast water; also about 1 gm fresh yeast. Place on forcing tray from 2-3 days at 80°F. After fermentation boil off alcohol (avoid wide mouthed beaker) & cool: wash out into a 200 cc flask, add a few drops of alumina; make up to mark at 60°F & filter. Determine { Reducing power on 20°C fermentation
also { Optical Activity

~~##~~ Determine also the Reducing Power of 10 cc of a 1% solution.

(on 2 grams)
Albuminoids are determined as with invert sugar.

Ash & Moisture also as above.

Calculation of an Invert Sugar.

To reduce the excess wt over 1000 to lbs per bbl, x by .36.

Now this is a 20% solution & each bbl of such solution contains 72 lbs of sugar so $\frac{112}{72} = 1.555$ which factor gives extract per cwt. $1.555 \times .36 = \underline{.56}$.

.4535 grms dextrose reduce 1 gm Cwt.

Calculation of a Glucose (Garton Hill's)

Extract

Sp. Gravity of 20% \times 10.6330

$$633.0 \times .56 = 35.44 \text{ extract per 1 cwt.}$$

CuO 10% solution.

1% solution of Glucose gave .1786 CuO. = 17.86 %

$$\begin{array}{r} \times 17.86 \text{ multiplied by} \\ 4535 \\ \hline 80.99 \end{array} = \text{Total reducing sugars \%}$$

expressed as dextrose.

Optical Activity.

In a 20% solution = +22.0 in 100 mm tube

$$= 110 \text{ aD \%}$$

Residue Reducing & Optical Activity after fermentation.

100 cc 20% solution taken & fermented, alcohol boiled off,
washed into a 200 cc & filtered: reducing power in 20 cc.

$$20\% = 1010 \text{ CuO}$$

$$\begin{array}{r} 50 \\ \hline 5090 \text{ grams CuO \%} \\ \times 4535 \\ \hline 25450 \\ 15270 \\ \hline 25450 \\ 20360 \\ \hline 23083150 \end{array} \text{ dextrose reducing power.}$$

~~78.6~~

$$\begin{array}{r} 80.99 \\ 2.30 \\ \hline 78.69 \end{array} = \text{Total reducing sugars removed by fermentation.}$$

Analysis of a Caramel.

Required to know:-

Tint of 1% solution in $\frac{1}{4}$ inch cell.

Cane Sugar.

Other fermentable sugars.

Unfermentable sugars.

Containing dextran

Ash. Albo: Moisture & Brewer's Extract

Cane Sugar

If the sugar is directly sweet it should be tested for cane sugar by the yeast method & alpric reduction.

(1) 2% solution. ex 10 cc for CuO.

(2) 2% solution inverted with yeast for 3-4 hrs at 130°F (add thymol) & made up to mark, ex 10 cc for CuO.

Fermentable & unfermentable sugars.

10 grams or (50 cc 20% solution) are fermented for 3 days without yeast food, the whole made up to 200 cc, filter off 100 cc, boil off alcohol & make up to 100 cc again.

Determine the S.G.

50 cc in 100 cc flask is heated to for 1 hr with HCl in the boiling bath; cool & neutralise with Na_2CO_3 , make up to 100 cc & determine CuO on 10 cc.

Extract - Moisture. Take the S.G. of a 20% solution.

Determine ash ^(3 grams) & Albo ^(5 grams) as in other sugars.

S.S.

Extract

$$\begin{array}{r} 53412 \times \times \\ 6222 \\ \hline \end{array}$$

$$= 33233 \times 18^*$$

$$= \underline{\underline{59.81\% \text{ extract}}}$$

Cane Sugar Ans before inversion .0959

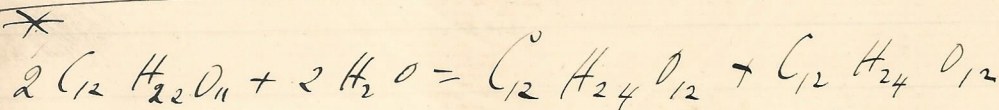
— after — $\frac{.1097}{.0138 \times 4715}^*$

.4715 grams pure invert
reduce (gram Ans.)

$$= 0.0619 \times 50$$

$$= 0.31\% \text{ cane sugar.}$$

$$* \text{ less } \frac{1}{20} = 0.295$$



Cane Dextrose Levulose.

$$604 + 36 = 360 + 360$$

$$\frac{36}{720} = \frac{1}{20}^*$$

Total Analysis

Titr in 1 inch cell 336

Cane Sugar 0.29

Fermentable Sugars 9.0

Unfermentable 57.74 }

containing Dextrose 23.05 }

Water 30.8

Ash 2.11

Albuminoids .35

$$\underline{\underline{100.29.}}$$

Calculation of a Caramel

Fermentable & Unfermentable Matters.

20% solution.

Sp. Grav. 1053412.

$$\frac{53412 \times 5}{3.86} = 69.2 \text{ solid matter} = \underline{30.8\% \text{ water.}}$$

5% solution.

Sp. Grav 1011.60

$$\frac{11.60}{3.86} =$$

3.01 solid matter

$$\frac{3.01 \times 100}{5} =$$

~~60.2~~ 60.2% solid matter

$$\begin{array}{r} 69.2 \\ 60.2 \\ \hline 9.0 \end{array}$$

$$69.2 - 60.2 = \underline{9.0\% \text{ fermentable matter}}$$

50 cc fermented solution boiled with 1 cc HCl made up to 100 cc ex 10 cc.

$$\text{Wt of CuO} = \frac{1447}{1125} \text{ grams}$$

$$\text{Calculated as Dextrose} = \frac{1447}{1225} \times 4535 = \underline{\underline{23.05 \text{ Dextrose}}}$$

$$\underline{\text{ash}} = \frac{.0656 \times 100}{3.0142} \text{ grams} = \underline{\underline{2.11\% \text{ ash}}}$$

$$\underline{\text{albs}} = 6 \text{ cc } \frac{N}{20} \text{ Na}_2\text{CO}_3 \quad 10 - 6 \text{ cc} = 4 \times .0007 \times 6.3 = \underline{\underline{.35\% \text{ Albs}}}$$

in 5 grams.

$$\begin{array}{r} \text{ash} \quad 2.11 \\ \text{albs} \quad 135 \\ \hline 2.46 \end{array}$$

$$\begin{array}{r} 60.2 \\ 2.46\% \\ \hline 57.74 \text{ unfermentable matter} \end{array}$$

4715 grams pure Invert reduce 1 gram Ash.

Typical analysis of an Invert Sugar.

Required to determine the actual Invert Sugar, the ~~un~~inverted cane sugar, the mineral matter, the albuminoids, acidity, inert matters & moisture.

Determination of the Invert Sugar.

$$\begin{aligned}\text{Wt of Beaker} &= 21.9412 \\ \text{--- + sugar} &= 22.9842 \\ \text{Sugar} &= 1.043.\end{aligned}$$

$$\text{Wt of Cmc. \& Cud} = 18.584$$

$$\text{Wt of Cmc} = 18.410$$

$$\begin{array}{r} \text{less paper} \\ \hline .174 \\ .003 \end{array}$$

$$\text{Cud} = .171 \text{ on } 10 \text{ cc} \\ \text{ex } 100\%$$

$$.171 \times .4715^*$$

$$= .0806255 \text{ grms invert sugar}$$

$$\frac{.0806255 \times 100}{1.043} = \underline{\underline{77.3\% \text{ invert sugar.}}}$$

Uninverted Cane Sugar. Yeast Method.

26.048 grms invert sugar dissolved in 100 cc

Opticity at 60 F = -9.0 divisions.

Opticity after ^{Inversion} fermentation = -4.1 $\times 2$ (only 50 cc taken) = 8.2

Difference in reading = .8 or 1.6 for 2 decimetre tube.

Make the ~~change~~ correction necessary for temperature.

on the number representing the change of pure cane sugar.

$$\frac{1.6 \times 134.75}{134.75} - \frac{1.6 \times 100}{134.75} = 1.18\% \text{ cane sugar.}$$

*

The above may be stated shortly thus

$$\frac{100 \times D}{142.5 - \frac{5}{2}} = \text{Percentage of cane sugar}$$

D = Total change of reading in a 2 decimetre tube

Uninverted Cane Sugar Acid Method

26.048 grms sugar dissolved in 100 cc

Opticity at 60°F = -7.2

.. after Inversion = -6.7

To this last reading 10% of the reading must be added in order to correct for the dilution of the solution by the HCl. $6.7 + .67 = 7.37$ divisions.

26.048 grms of pure cane sugar dissolved in 100 cc of water will read in a 2 decimetre tube at 0°C +100; the same after inversion & also at 0° will read -42.5°. The total change of reading owing to the complete inversion is therefore 142.5° at 0°C. But this change of in rotation is one degree less for every 2°C of temperature over 0°.

Difference in reading in 1 decimetre tube = .17 or .34 in 2 decimetre tube. The temperature was 15.5°C. Divide 15.5 by 2 = 7.75 & subtract from 142.5 = 134.75. Since both the standard reading for pure cane sugar & the reading of the sample refer to solutions of the same concentration it will follow that the percentage of cane sugar will be

$$\frac{.34 \times 100}{134.75} = .23\% \text{ Cane sugar.}$$

* Each 1 cc of $\frac{N}{10}$ acid neutralised corresponds to 0.0014
grams Nitrogen

Albuminous matter

Wt of flask & paper = 24.38

Wt of sugar & f + p. = 27.511

Sugar = 3.131

10 cc $\frac{N}{10}$ H_2SO_4 in receiver required after distillation

9 cc $\frac{N}{10}$ Na_2CO_3 for neutralisation.

10.9 cc = 1 cc $\frac{N}{10}$ H_2SO_4 neutralised by the ammonia evolved.

1 \times .0014 = .0014 grms nitrogen in sample.

.0014 \times 6.3 = .00882 grms albuminoids.

$\frac{.000882 \times 100}{3.131} = .24\%$ albuminoids.

Mineral Matter

Wt of pt dish = 30.0434

+ sugar = 33.9204

Sugar = 3.877

Wt of pt dish & ash = 30.0906

Wt of ash = .0472.

= 1.2% ash.

Moisture. Total solids. Sp Gravity of 20% = 1.062.13

$\frac{62.13}{3.86} = 16.09$. $\frac{16.09 \times 100}{20} = 80.45$

80.45% solid matter in solution

= 19.5% of water.

Inert Matters

The so-called inert carbohydrates are obtained by adding together the above constituents, as determined by direct analysis & then subtracting the sum from the solid matter.

Invert Sugar ~~77.3~~ 77.3

Cane Sugar 1.18

Ash 1.2

Albuminoids .24

79.92

$80.45 - 79.92 = \underline{\underline{.53\%}}$ Inert matter.

Brewer's Extract

From 20% solution Sp Gr # 62.13

$$\frac{62.13 \times 360}{1000} = 22.36.$$

$$\frac{22.36 \times 62.22}{20 \times 1} = 69.5 \text{ lbs per 2 cwt}$$

Complete Analysis

Invert Sugar = 77.3%

Uninverted Cane = 1.18.

Ash = 1.2

Albuminoids 0.24

Inert matters 0.53.

Water 19.95

99.95.

Extract 69 lbs per 2 cwt.

Analysis of a finished beer.

Chemical Composition

Required to know the original gravity, the specific gravity of the beer, the percentages of maltodextrins (& their type) of stable dextrin & of low type fermentable maltodextrins (commonly called free maltose) of fermentable sugars which have disappeared during fermentation, ash, albuminoids & acid.

The Acidity is determined by 100 cc & neutralising with $\frac{N}{10}$ or $\frac{N}{20}$ Na_2CO_3 in usual way.

Determination of maltodextrins: dextrin, low type maltodextrins (apparent free maltose) & fermented sugars.

Total reducing power.

Take 5 cc & determine reducing power in usual way.

Opticity

50 cc of the beer are taken, a little alumina added, the whole diluted to 100 cc, filtered & the opticity determined in the usual way in a 1 decimetre tube.

Degradation by malt extract

50 cc of beer are evaporated to about $\frac{1}{2}$ bulk & transferred to a 100 cc measure: 5 cc of cold water malt extract added & the whole digested at 125°F for one hour. It is then cooled made up to 100 cc & the reducing power determined on 10 cc. 10 cc of the malt extract must be treated in the same way for correction.

Fermentation

Two portions of the beer (50 cc) are evaporated to $\frac{1}{2}$ bulk, diluted to about 50 cc with water & about 0.25 gram yeast added. Ferment for 70 hrs at 80°F having added .25 cc of cold water extract to one portion. Transfer to a 100 cc flask, add a little alumina make up to 100 cc, filter & determine the CuO on 10 cc.

ash On 25-30 cc of the beer in the usual way

Albuminoids On 10 cc of the beer evaporated to dryness by Kjeldahl's process as in malts etc
10 cc $\frac{N}{10}$ H_2SO_4 in receiver.

Analysis of a Bisulphite of Lime.

Required to determine :-

- | | |
|----------------------------|--------------|
| (1) S.G | (6) Lime |
| (2) Sulphurous Acid total. | (7) Magnesia |
| (3) Sulphurous acid free. | (8) Alkalies |
| (4) Sulphuric acid | (9) Iron |
| (5) Chlorine | |

Specific Gravity

This is taken in the ordinary manner in an SSG bottle, the only precaution necessary being to weigh as rapidly as possible to obviate alteration in weight from evaporation & oxidation of the free sulphurous acid.

Total Sulphurous Acid.

This may be determined as follows gravimetrically. 20 cc Bisulphite is measured into a 200 cc flask & diluted to mark. Place in a beaker 100 to 150 cc of distilled water & run in about 20 cc Bromine water.

Add 10 cc of the solution of bisulphite (1 cc bisulphite)

After the addition the solution should remain quite brown; a little HCl is added, boiled until it loses its colour & all free bromine is expelled. The sulphurous acid has now been oxidised into sulphuric, the bromine decomposing the elements of water & combining with the hydrogen to form hydrobromic acid, whilst the sulphurous acid seizes the oxygen thus liberated.

The H_2SO_4 is now determined in the solution & the sulphurous acid calculated after deducting the $BaSO_4$ due to the sulphuric acid naturally present.

Sulphuric Acid.

100 cc distilled H_2O + 10 cc concentrated HCl are placed in a small flask & boiled vigorously for 5 minutes to expel the air in the flask & the dissolved oxygen in the water: 50 cc of the bisulphite solution is added & the ebullition continued until the evolved steam no longer smells of S sulphurous acid.

The contents of the flask are now transferred to a beaker, diluted with H_2O , & barium chloride added in excess, the precipitate $BaSO_4$ is filtered dried cooled & weighed.

Lime.

20 cc of the diluted solution (20 cc in 200 cc) is placed in a beaker with about 100 cc H_2O , add ammonium oxalate, ammonium chloride & ammonia each in slight excess.

The liquid is allowed to stand for 3 hrs after gently warming for $\frac{1}{2}$ hr, then filtered & washed with hot H_2O , dried ignited, & recarbonated & weighed as $CaCO_3$.

Magnesia

To the filtrate from the lime estimation is added ammonium phosphate in slight excess & ammonia until the liquid smells strongly ammoniacal.

It is then well stirred & allowed to stand 6 hrs.

Filter the precipitate & wash with dilute ammonia, dry ignite & weigh as Magnesium Pyrophosphate.

Chlorine

20 cc of Bisulphite^(1cc) is evaporated to dryness on a water bath: when dry the residue is moistened with a little distilled H_2O & redried, this operation being repeated 2 or 3 times.

The experiment is carried out as with water on the clear filtrate.

Hyposulphites may be easily detected by adding to the bisulphite strong mineral acid, which causes a precipitation of sulphur on boiling.

Calculating a Bisulphite of Lime.

$$S.G. = 1053.754.$$

H₂SO₃ . 1cc Bisulphite

$$\begin{array}{r} .2644 \\ .003 \\ \hline .2614 \end{array} \begin{array}{l} \text{due to sulphate naturally present} \\ \text{BaSO}_4 \text{ due to Sulphurous acid.} \end{array}$$

$$\begin{array}{l} \text{BaSO}_4 = 233 \quad 64 = .2746 \text{ pts SO}_2 \text{ in 1 pt BaSO}_4 \\ \text{SO}_2 = 64 \quad 233 \quad .2746 \times .2614 = .0717 \text{ SO}_2 \text{ per cc} \\ \quad \quad \quad \underline{7.17\% \text{ SO}_2} \end{array}$$

H₂SO₄ 50 cc Bisulphite taken dilute = 5 cc Bisulphite

$$.1556 \text{ BaSO}_4 \text{ due to H}_2\text{SO}_4$$

$$\begin{array}{l} \text{BaSO}_4 = 233 \quad 80 \\ \text{SO}_3 = 80 \quad \frac{80}{233} = .343 \end{array} \cdot 1556 \text{ BaSO}_4 \times .343$$

$$\begin{array}{l} = 10534 \text{ per 50 cc} \\ = \underline{1067\% \text{ SO}_3} \end{array}$$

Lime 2 cc taken .091 CaCO₃

$$\text{CaCO}_3 = 100$$

$$\text{CaO} = 56 \quad .091 \times .56 = .0509 \times 50 = \underline{2.54\% \text{ CaO}}$$

Magnesia 2 cc taken .0044 Mg₂P₂O₇

$$\text{Mg}_2\text{P}_2\text{O}_7 = 222$$

$$\text{MgO} = 40 \quad \frac{80}{222} = .36 \text{ parts MgO in 1 pt Mg}_2\text{P}_2\text{O}_7$$

$$.0044 \times .36 = .00158 \text{ MgO per 2 cc} = \underline{.0792\% \text{ MgO}}$$

Chlorine 20 cc taken .9 cc AgNO₃.

$$.9 \times .001 = .0009 \text{ Cl}_2 \text{ in 20 cc}$$

$$\text{NaCl} = 50.5$$

$$\text{Cl} = 35.5$$

$$\begin{array}{r} .0045 \\ \times 1.65 \\ \hline .00742 \end{array} \text{ NaCl per 100 cc}$$

$$\frac{35.5}{50.5} = 1.65$$

Combinations.

H_2SO_4 combined to calcium

$$SO_3 = 1.077$$

$$SO_3 = 80$$

$$CaO = 56$$

$$\text{As } 80 : 1.077 :: 56 : x$$

$$\begin{array}{r} .753 \text{ CaO} \\ 1.077 \text{ SO}_3 \\ \hline 1.830 \text{ CaSO}_4 \text{ per } 100 \text{ cc} \end{array}$$

H_2SO_3 to Calcium

$$\text{Total CaO} = 2.548$$

$$\text{CaO as CaSO}_4 = \frac{0.753}{1.795}$$

$$CaO = 56$$

$$SO_2 = 64$$

$$\frac{120}{120}$$

$$\frac{1.795 \times 64}{56} = \underline{3.846} \text{ CaSO}_3 \text{ per } 100 \text{ cc}$$

Sulphurous Acid & Magnesia

$$MgO = .079$$

$$MgO = 40$$

$$SO_2 = 64$$

$$40 : .079 :: 64 : x$$

$$40 \overline{) 5.056}$$

$$.126 \text{ SO}_2$$

$$.079 \text{ MgO}$$

$$\underline{.205} \text{ MgSO}_3 \text{ per } 100 \text{ cc}.$$

$$\text{Total SO}_2 = 7.17$$

$$\text{Combined SO}_2 = 2.176$$

$$\text{Free SO}_2 = 4.994$$

$$SO_2 \text{ as CaSO}_3 = 2.051$$

$$SO_2 \text{ as MgSO}_3 = 0.126$$

$$\underline{2.177}$$

$$\text{Total Sulphurous Acid} = 7.17 \text{ per } 100 \text{ cc} \quad 6.80 \%$$

$$\text{Free} \quad \text{---} \quad \text{---} = 4.99 \quad \text{---} \quad \text{---} \quad 4.73$$

$$\text{Combined} \quad \text{---} \quad \text{---} = 2.17 \quad \text{---} \quad \text{---} \quad 2.05$$

$$\text{Calcium Sulphate} \quad 3.84 \quad \text{---} \quad \text{---} \quad 3.64$$

$$\text{Magnesium "} \quad 0.20 \quad \text{---} \quad \text{---} \quad 0.18$$

$$\text{Calcium Sulphate} \quad 1.83 \quad \text{---} \quad \text{---} \quad 1.7$$

$$\text{Sodium Chloride} \quad 0.007 \quad \text{---} \quad \text{---} \quad .006$$

The Specific Gravity of the Bisulphite being
1053.75, the actual weight of liquid is 105.375
... for each figure of the analysis multiplied by
 $\frac{100}{105.375}$ = results in percentages
