- Thesis -

An Experimental Investigation of the Bi-Sulphite Process of Making Chemical Fibre.

Salmon W. Wilder. Jr.
May, 1891.

— Literature —

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An Experimental Investigation of the Bi-Sulphite Process of Making Chemical Fibre.

For a long time it has been known that all vegetable substances contain more or less of Cellulose, and ever since 1772 (when a German named Schaeffer suggested that this fibrous material be used for the manufacture of Paper) the question of isolating & obtaining this Cellulose in a state suitable for use in Paper Making, experimenters have been trying to solve; until 1853 however when the well known Watt & Burgess patents were taken out, no practical results had been reached.

It is probable however that during the last few years, in no branch of Industry has there been a more rapid development than in the production of this Cellulose commonly known as Chemical Fibre

Of the various vegetable tissues of which Cellulose forms the most important part, wood has been and is now the most extensively employed, and the aim of the various processes which have been and are used at the present time, is to isolate this fibre and obtain it in such a condition that it will be long, fairly white and reasonably pure, and at the same time destroy as few as possible of its "felting" qualities.

Cotton may be taken as a typical example of Cellulose, and the nearer the product approaches Cotton both in its chemical composition and physical properties, the better generally the product is considered.

Now in addition to Cellulose the woods contain varying amounts of foreign matter, consisting of resins, gums, mineral matter, fats and various nitrogenous substances often classed under the head of Lignine.

In the selection of a wood for the manufacture of Chemical Fibre it is necessary to consider the following items, viz:

1° The per centage of Cellulose which it contains.

2° The nature of the foreign matter present in the wood.

3° The ease with which this foreign matter may be separated from the Cellulose.

4° The length and charachter of the fibre.

5° The amount of wood obtainable

and the cost of the same.

Of course in a given locality the wood ~~which~~ will naturally be chosen, which ~~t~~ combines the most desirable features.

The composition of woods varies widely, the amount of Cellulose ranging from twenty to sixty per-cent.

In general the soft woods contain a larger amount than the hard, and for this reason are more employed.

I have not found many analyses of different woods, and probably the results of different analysts are not strictly comparable, owing to the different methods employed and other circumstances. I will give the results of several analysts however.

The following table gives the relative pulping values of different woods, but I am inclined to think that the figures would vary somewhat according to the process which was used. (1)

Hemlock	45 %
Dry Walnut.	42 %
Birch.	40 %
Dry Poplar.	37 %
Green "	30 %
Yellow pine.	36.5 %
White "	33 %
Spruce	32 %
Cherry.	32 %
Chestnut.	30 %
Hickory.	23 %
Ash, Oak & Maple.	21 %

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(1) By Mr. C. M. Cresson, given in advances sheets of report of the Dept. of Agriculture, for 1890.

These results however are somewhat larger than ordinarily reached in Mill practice.

The following is a table of Laboratory analyses of European woods, all of which were air dry and are reported as the mean of the results of several Chemists (1)

Poplar.	62.8 %	Cellulose
Fir.	57.0	"
Willow.	56.7	"
Birch.	55.5	"
Pine.	53.3	"
Bass Wood.	53.0	"
Chestnut	52.6	"
Locust.	48.4	"
Beech.	45.5	"
Oak.	39.5	"

(1) Report by B. E. Fernow.
Dept. of Agriculture 1890.

The following table shows the results of a German Authority (1) and it will be noticed they agree well with those of the Table by Mr. Fernow.

Birch.	55.5 %
Beech.	45.5 "
Lime.	53.0 "
Pine	57.0 "
Poplar.	63.0 "

Neither of the last two tables give an analysis of Spruce, which is largely used in the production of Cellulose.

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(1) Hugo Müller --- Pflanzenfäser."

Numerous processes have been devised which have had for their objects, the dissolving the ligneous matter from the wood and leaving the fibre in such a condition, that it is adapted for the manufacture of paper.

Of the various processes in use. I have chosen the Bi-Sulphite or commonly called "Sulphite Process" on which to make some experiments

My reason for so doing is, that this process is extensively used in this Country, and very favorable results are obtained by it; also it is claimed that the per-cent of fibre obtained is large, and that its physical charachter is not so much altered by the action of the materials used as by some other processes.

Following is an outline of the "Sulphite" Process; The wood (commonly Spruce or Poplar in this country) is first barked and then reduced to small chips; knots are generally bored out; the next operation is the digesting of this wood with some liquor which contains Sulphurous Acid in an available form; the temperature at which the digesting is to be carried on may be varied according to the strength of solution.

This digesting process is commonly carried on in vessels which are heated by steam, and are lined with or constructed of some material which will resist the action of the acid employed, and so arranged that the contents may be kept agitated; Of course the mechanical details of the arrangement differ widely.

The substances composing the ligneous portion of the wood are doubtless of an Aldehyde nature and may perhaps be represented by the formula

X CHO

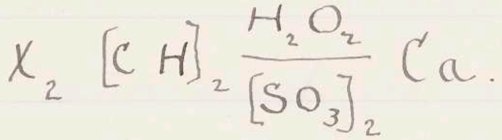
The symbol of X is not known exactly and will vary for different woods.

The action of the Sulphurous Acid ~~re~~ results in the formation of a double salt by union with this Aldehyde, and this may be dissolved out leaving the Cellulose.

The compound which is formed depends on the charachter of the liquor used, and in the case of a solution of

Calcium Bi-Sulphite the reaction is probably this.

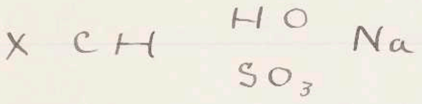
$$CaH_2[SO_3]_2 + 2XCOH =$$



$$X_2[CH]_2\frac{H_2O_2}{[SO_3]_2}Ca.$$

The Sodium Salt would be similar thus.

$$NaH[SO_3] + XCOH =$$



$$XCH\begin{matrix}HO\\SO_3\end{matrix}Na$$

While in the space of time obtainable in the school term I realized that a very extensive set of experiments could not be carried out, yet the following subjects appeared to me as worthy of investigation, and to them I have confined my work.

1° To construct a small apparatus by means of which the solution of Calcium Bi-Sulphite required in this process can be simply made.

2° To examine the liquor obtained from the above apparatus, and to determine the best method for determining the amount of Sulphurous Acid present in this solution.

3°. To construct a small digester and produce if possible some wood Cellulose by means of it.

With these objects in view my first undertaking was the construction of a plant to make the acid liquor., and since the form in which the Sulphurous Acid is most commonly applied is as the Acid Sulphite of Calcium commonly known as Bi-Sulphite I arranged to make some of this solution.

The sources of the product were naturally ordinary Quick-Lime and Sulphur.

. From the lime it is easy to form the Hydrate of Calcium corresponding to the symbol

$$Ca\,O_2H_2$$

By causing action to take place

between this and Sulphurous Acid the following equation represents what takes place.

$$CaO_2H_2 + 2[H_2O + SO_2] =$$

$$CaH_2[SO_3]_2 + 2H_2O$$

The simple method of preparing the Calcium Hydrate or Slaked Lime is as follows.

To some Quick lime add a little water, and allow the mass to stand; action soon takes place with the evolution of heat; if necessary add more water or sufficient for the action to be complete, when a thoroughly disintegrated mass of Hydrate of Lime will remain. The action is

$$CaO + H_2O = CaO_2H_2$$

My Sulphurous Acid I obtained by the burning of ordinary Sulphur.

which is described later.

My plan for the production of a solution of the liquor was this. viz:-

I had an iron retort in which I burned the Sulphur, and through it sucked air in sufficient quantity to form Sulphur di-Oxide, but with as little excess of air as possible; from the retort the gas formed was drawn through one or more vessels which contained a solution and mechanical mixture of Calcium Hydrate; at the same time I provided for the agitation of the contents of the vessels, which is necessary because Calcium Hydrate is not very soluble in water and if it is allowed to settle at the bottom of the vessels, the action of the Sulphurous Acid gas will be very slow.

Following is the arrangement I provided, showing the construction of the retort and suction pipe in detail.

Figs 1 and 2 show the retort in front and side elevation, Fig 2 being a longitudinal section along the line SS in Fig 1. Fig 3 is a detailed drawing of the joint W in Figs 1 and 2. The three figures are lettered correspondingly. I will first explain the construction of the retort.

It consists of an iron pipe lettered K and shown in section by Fig 2; it is about 5" inside diam. and made of 5/8" iron.

A is a flange on the end of the pipe and is about 15" outside diameter; the other end of the pipe which is about three feet in length is closed by the plug V, as shown in Fig 2. ; about three inches from this plug the suction pipe N shown in fig. 2. enters; this can be disconnected at I by a brass union shown in both figures.

At the entrance of the pipe is a Plaster of Paris plug lettered B, and of the form shown in both diagrams, and which seals about one half of the opening. ; r r are two iron projections on which rests a sheet iron door lettered D

The method of procedure is as follows.

Sulphur is thrown in at the opening at the end of the pipe and is ignited; it melts, and the pipe K being inclined

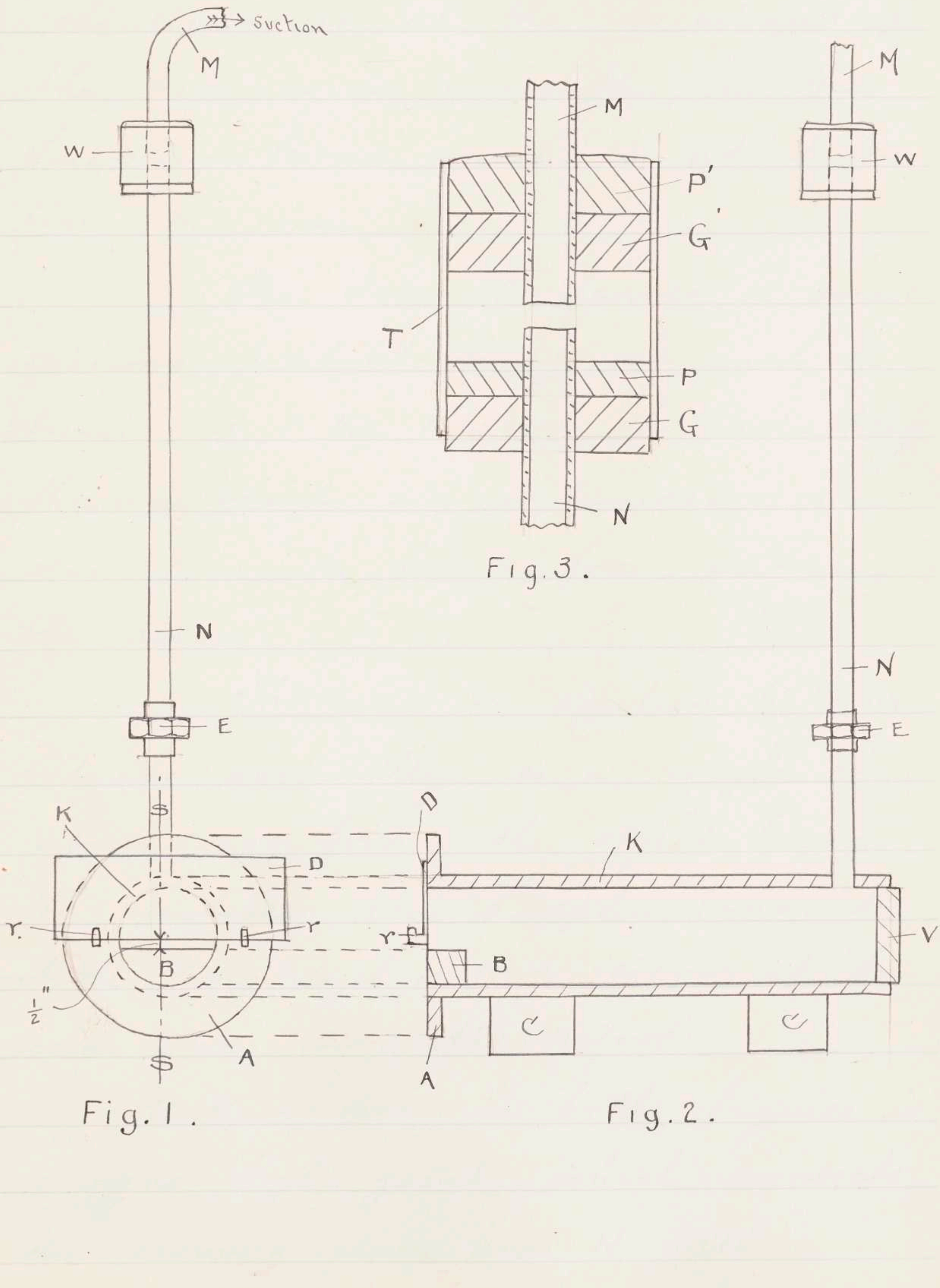


Fig. 1.

Fig. 3.

Fig. 2.

slightly forward, it runs down against the plug B; this keeps the Sulphur at the front end of the pipe as is desirable; as the Sulphur burns more is added from time to time.

The door D leaves a small opening of about half an inch between it + the Plaster of Paris plug through which air is sucked; it also prevents fumes from entering the room.

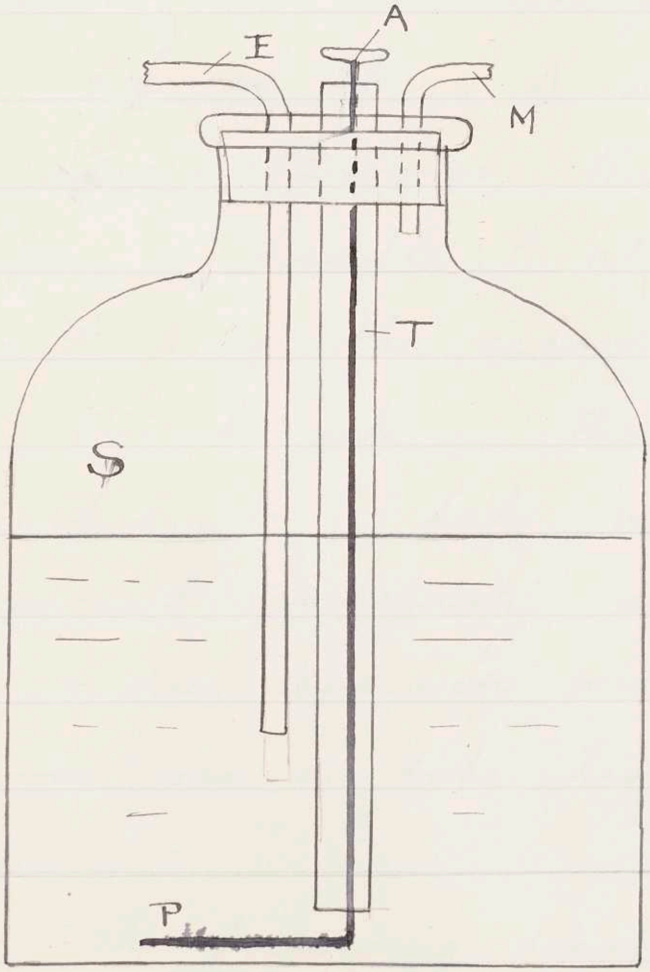
The exit or suction pipe N extends vertically to a height of about six feet, the object being to condense any Sulphur (should it be volatilized) and allow it to run down into the retort.

At W shown in both figures is a gas tight joint which connects the iron suction pipe N with a glass tube lettered M which leads to the vessels holding the Calcium Hydrate.

The arrangement of this joint is shown in detail by Fig 3.

I is a glass tube of about "2½" inside diameter, and 3½" in length; in the lower part is inserted a cork G, through which passes the suction pipe N as shown. Plaster of Paris is now poured on the cork forming a seal as is shown at P. At the other end of the tube, after making this joint a cork G' is inserted so it fits tightly, and the glass tube M passed down through it as shown; another Plaster of Paris seal covers this cork and thus a firm perfectly air tight joint is produced.

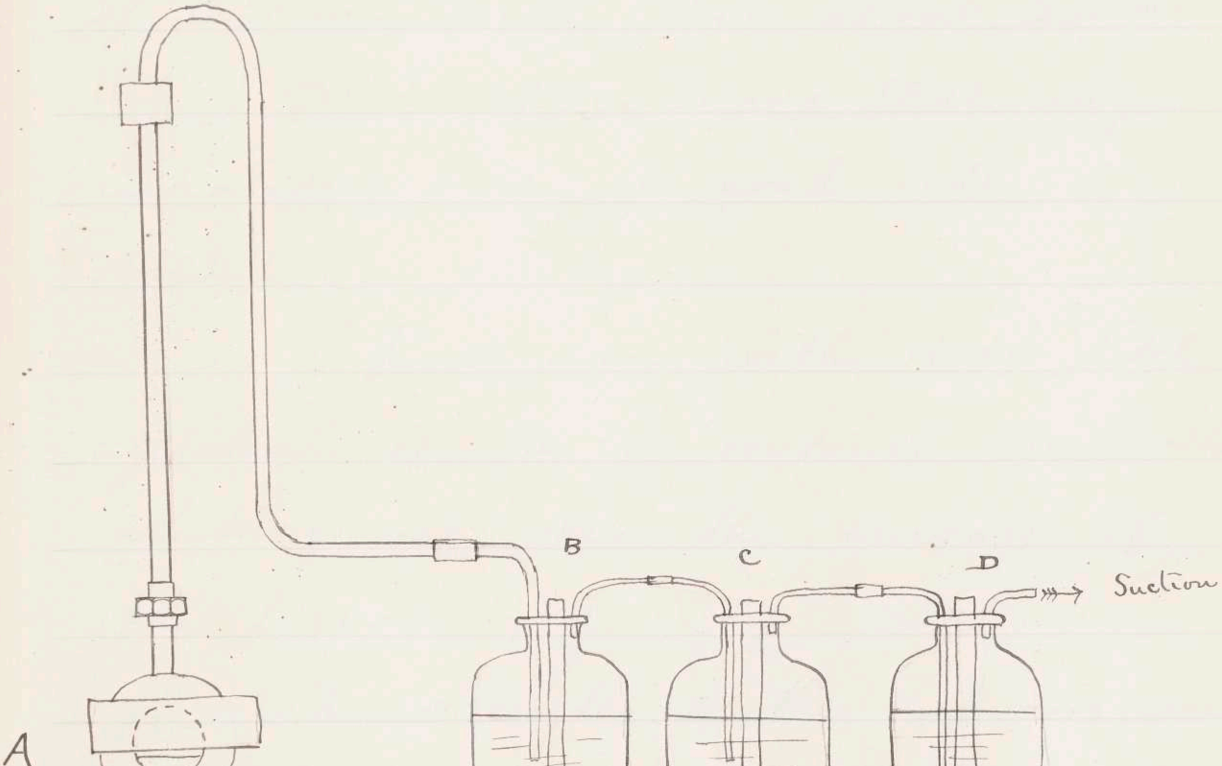
The glass tube M is conducted to a bottle in which is some of the lime liquor ; this bottle is connected to a second , and a third from the latter a pipe leading to the suction tube ; the method of passing the gas through the bottles may be understood by the diagram following



A bottle S of about 10 litres capacity is provided with a cork as shown, and is about half filled with lime liquor; the tube E from the retort enters as shown in the diagram and dips about half way to the bottom of the liquor; the large central tube T dips nearly to the bottom of the vessel so that no gas can escape through it. A is a rod which passes through this tube and has a paddle P at the bottom for keeping the sediment of $CaOH_2$ stirred up.; the pipe M is attached to a tube which leads to a second similarly arranged bottle, + this connects with a third and from here a tube leads to the suction.

The following diagram shows

the whole arrangement.



A shows the retort, with the pipe leading the Sulphurous Acid through the flasks B, C and D, and the last flask D connecting with the suction.

Results from running this apparatus.

My first batch of Lime I made up with seven kilos of water to 170 gr. of Calcium Oxide, (i.e about 2.4% CaO.) and after seeing that my apparatus was in good order I started.

I first ran with with little agitation of the contents of the bottles, white fumes over the surface of the liquor.

The strength of the solution rose very slowly only reaching 2° Twaddel after running 9 hours.; at this point I closed the suction somewhat and kept the sediment in the bottles well stirred up, which was difficult however owing to its density.

The strength rose slowly however, and in about six hours, the first bottle showed 7° Tw. and the second $3\frac{1}{2}$° Tw; while the third had gained little or none; I ran the gas through the bottles for 20 hours longer to observe the effect, testing the liquors from time to time; the strength of the first bottle was stationary all of that time, while that in the second only gained $\frac{1}{2}$° Tw. giving a solution of 4° Tw.

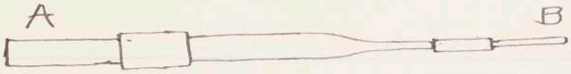
I conclude that this phenomenon was due partly to the oxidation of Sulphite to Sulphate and partly to the fact that the agitation was not violent enough to allow the SO_2 to come into contact intimately with the particles of Calcium Hydrate.

At the close of another 10 hours run, the solution in the first bottle fell off $\frac{1}{2}$° Tw.

From these results I concluded it was necessary for me change my bottles and provide an arrangement which would permit of a more complete agitation of the Lime liquor.

Accordingly I arranged to run the gas into smaller bottles which were arranged so as to permit a complete agitation; In place of the first two bottles and which only gave me a strength of 7½° Tw., I placed two smaller ones, each of about 3 litres capacity, and arranged as shown in the diagram.

In place of the ½" tube which I passed to the bottles from the retort, I used a small one which was connected to the larger by a drawn out tube as as shown in the accompanying diagram. A is the tube leading from the retort



and B the one which leads to the bottles.; this small bottle I connected to a second, and left the third as before; I omitted the paddle in both small bottles.; the only difference between this arrangement and the other was the substitution of the two small bottles for the larger ones.

I now took some of the solution from the first large bottle which after standing several days had a strength of 6° Tw., and filled the small bottles about 2/3 full, and added an excess of Calcium Hydrate, and started running.

In about three hours I hydrometered the solutions and found No. 1 was 10° Tw. and No. 2. was 8° Tw. while No. 3 was not changed.

After running two hours longer I obtained a strength of 11° Tw. which was as strong as I desired. The small entrance tube dipping nearly to the bottom of the bottle kept the contents shaken up all of the time, and once in a while I shook the bottles well, they being connected to each other and to the retort tube by flexible tubes.

With this arrangement I find it only takes a few hours to make a solution of good strength say 10° – 11° Tw.

The second part of my work was the examination of this Bi-Sulphite solution, and to experiment upon a method or methods for determining the amount of Sulphurous Acid [SO_2] contained in solutions of different Specific Gravities; to this end I consulted the literature on the subject.

Sutton --- A Systematic Hand Book
of Volumetric Analysis.
Blakiston & Co. Phila. Pa. 1886

Fresenius --- System of Instruction
in Quant. Chem. Analysis. New System
Wiley & Sons. N. Y. 1888.

Fleischer.
Vol. Analysis.
Mac Millan & Co. London. 1877

Mohr.
Titremethode.
Fr. Vierwig & Sohn.
Braunswick. Ger. 1886

Jour. Soc. Chem. Industry.
Vol. 3. p. 197

Jour. Soc. Chem. Industry.
Vol. 4. p. 303.

The various works describe more or less definitely methods in which the active agents are Iodine and Sulphur Dioxide., and I first tried that one sometimes called Bunsen's method and which is essentially as follows.

Titrate the solution of the sulphite directly with a one hundredth or a one tenth normal solution of Iodine.

For this ~~A~~ analysis it is necessary that the acid solution should not contain more than 0.05 % of SO_2 according to Sutton, and either the per-centage present must be approximately known, or a preliminary titration made to determine it roughly; when the solution is in this state of dilution the following or Dupasquier's reaction takes place.

$$I_2 + SO_2 + 2H_2O = 2HI + H_2SO_4$$

The Iodine is run directly into the solution until a drop will give a blue color with the Starch indicator

It will first be necessary to give the methods of making up solutions &c.

1°. A deci-normal solution of Iodine is required, and is made up as follows, where a strictly accurate solution is required.

Mix carefully some commercial Iodine with about 25% of its weight of pure Potassium Iodide. Heat the mixture gently between two large watch glasses; the Iodine which sublimes should be dried in a dessicator and is then pure.

Weigh out of this carefully 12.65 gr. and dissolve this together with 18 gr. (about) of Potassium Iodide, and make the solution up to one litre.

An approximately deci-normal solution may be made up with ordinary Iodine + standardized against this accurate solution or by the methods which will be given

1° Standardization in terms of Thio-sulphate of Soda $Na_2S_2O_3$
For this purpose a deci-normal solution of $Na_2S_2O_3$ is required, & is made as follows.

Take some pure $Na_2S_2O_3$, grind it to a powder and dry carefully. Of this powder weigh out carefully 24.8 gr. dissolve in pure water and make up to one litre.

Starch solution is made by mixing one part of Potato Starch with a little water, then adding about two hundred parts of boiling water and boiling for a minute; allow to stand and use the clear liquid of which only a few drops are necessary.

If the solution of Iodine is just deci-normal it will require just 1.c.c to neutralize 1.c.c of the $Na_2S_2O_3$ solution, and strength of an approximate solution may be easily determined in terms of the correct one.

The action is

$$2Na_2S_2O_3 + 2I = 2NaI + Na_2S_4O_6$$

The result is that by the formation of Thio-Sulphuric acid which takes O from the water, tetrathionic Acid and Hydriodic Acid in combination Sodium are produced, and at the moment when this action ceases, and a trace of free I is present, the blue color due to the Iodo-Starch compound is produced

Previous to taking any careful readings I carefully calibrated my burettes.

I first standardized my approximate Iodine solution against the pure $Na_2S_2O_3$ solution both being deci-normal solutions.

– Readings –

$\frac{n}{10}$ I (approx)	$\frac{n}{10}$ $Na_2S_2O_3$.	Actual strength of Iodine
18.04 c.c	18.02	0.999
11.15 "	11.15	1.000
15.81	15.76	0.997
14.34	14.31	0.998
		3.994
		0.998

This shows the strength of the approximate solution is 0.998 deci-normal

I next tried the method of titrating against the pure Iodine solution, i.e finding the ratio of the number of c.c required to neutralize a given solution of $Na_2S_2O_3$, to the number of c.c of the exact solution. The previous table shows the approximate solution and the following the exact.

$\frac{n}{10}$ I	$\frac{n}{10}$ $Na_2S_2O_3$	Ratio
20.41	20.39	0.999
15.08	15.11	1.002
15.12	15.15	1.002
14.23	14.24	1.000
17.92	17.91	0.999
		5.001
		1.0002

$$\frac{0.998}{1.0002} = 0.998$$

which agrees with the first.

The following readings will serve to show the discrepancy from using the Bunsen method.

I used a solution of Bi-Sulphite of Calcium and $Na_2S_2O_3$, but could not get check readings with the former.

The solution was 10½° Tw. and I diluted 100 times to obtain a small per-cent of SO_2; I used 25 c.c of this solution and a twentieth normal solution of Iodine

– Readings –

$CaH_2(SO_3)_2$	$\frac{n}{20}$ Iodine	
25.00 c.c	6.95 c.c	
"	6.44 "	
"	6.22 "	
"	6.01 "	
	25.60	

Mean = 6.40

This shows a difference of 0.94 c.c between the extreme readings or 14.7 % difference.

The reason is plainly due to the fact that the strength of this dilute solution rapidly drops off, due perhaps to escape of SO_2 and to oxidation.

The results I concluded could not be trustworthy and adopted another method.

The difference consisted in adding Iodine in excess and then titrating back the excess by $Na_2S_2O_3$ I also used a twentieth normal solution of Iodine + $Na_2S_2O_3$

Sp. G. 1.046 $CaH_2[SO_3]_2$	Iodine	
25.00 c.c	6.01	
"	5.94	
"	5.84	
"	5.80	
	Mean = 5.90	

This method also shows a dropping off in the strength and might be expected to give a per-cent of SO_2 far too small.

Also owing to the dilution of the solution any error is multiplied

This table would give for the per-cent of SO_2 present according to the readings, since 1.c.c of decinormal Iodine is equivalent to 0.0032 gr. SO_2 - - -

$\frac{5.90}{2} \times 4 \times 100 \times .0032 = 3.78\ \%\ SO_2$

and this from later work is evidently incorrect.

I now used a strong solution of $CaH_2(SO_3)_2$, and decinormal solutions of Iodine and Thio-sulphate of Soda, in order to find if I could obtain readings which would check. Following is the table.

$CaH_2(SO_3)_2$	Iodine	
25.00 c.c	61.45	
" "	61.30	
" "	61.41	
" "	61.38	
	61.385	

The mean of the 4 readings and the variation of the highest and lowest reading from the average is only 0.07 c.c and 0.08 c.c or 0.013 of one per-cent which is sufficiently accurate for ordinary work.

I adopted solutions of the Bi-Sulphite which were diluted 10 times and used deci-normal solutions of Iodine & $Na_2S_2O_3$

For the determination of my Specific gravities I used an accurate direct reading Hydrometer

Being desirous how accurate it was I determined to find the Sp. G. directly and then take the reading of the Hydrometer for that solution. I used an ordinary Sp. G. flask.

- Results -

Wt. of Sp.G flask full of water	Wt. of Sp. G fsk with Solution
132.875	137.670

Wt. Flask empty

28.276

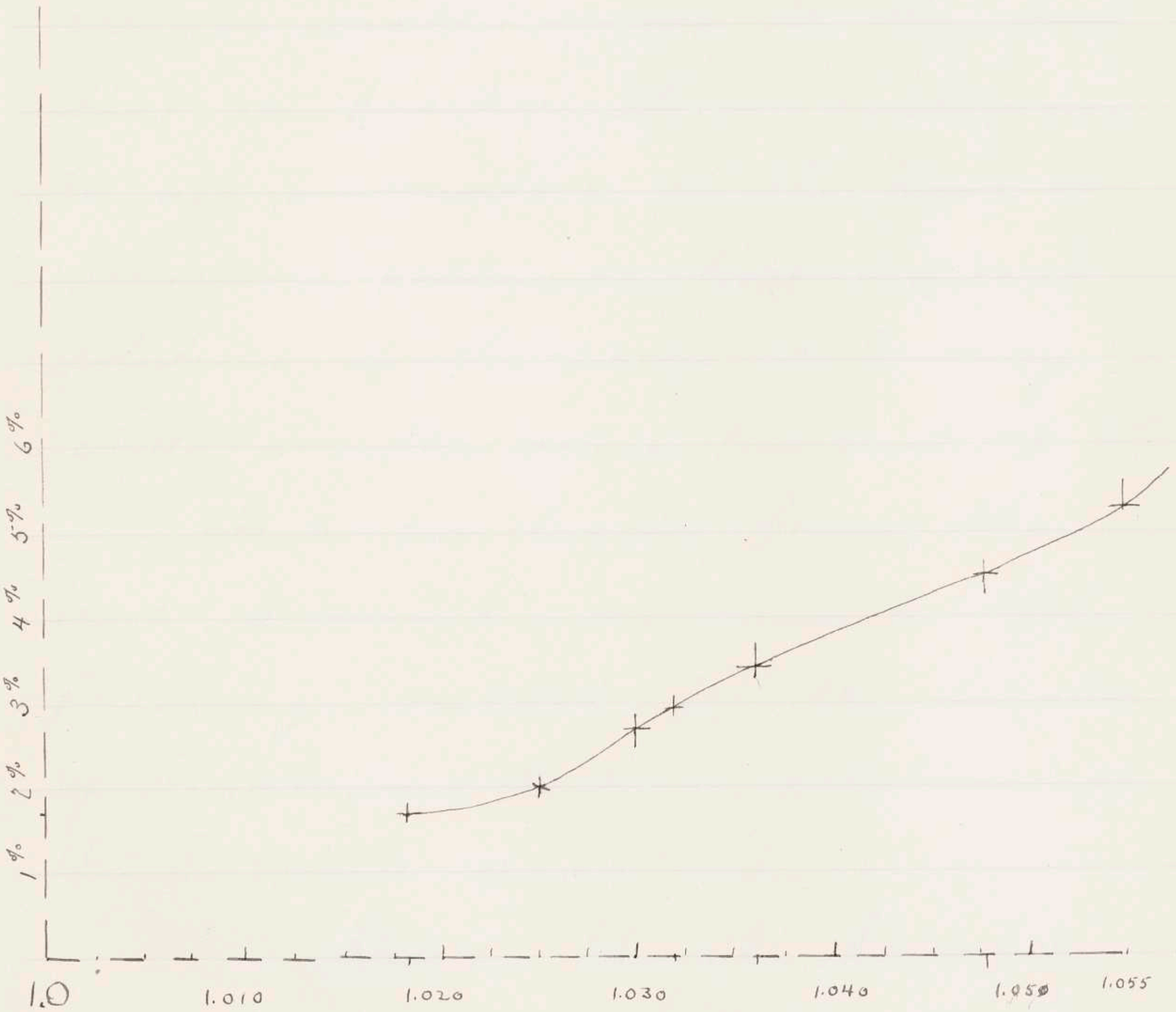
$$Sp.G = \frac{109.394}{104.599} = 1.046$$

The Hydrometer read exactly 1.046 which showed it was accuratily graduated.

The following table shows the number of c.c of deci-normal solution of Iodine required to neutralize 25. c.c of solutions of Bi-Sulphite of Calcium diluted 10 times and of different Sp. G.

Bi-Sul. of Calcium Diluted 10 times	Sp.G	Iodine	Mean	% SO_2
25.00 c.c	1.055	41,42 41,32 41,20 41,28	41.31	5,29
" "	1.048	35.28 35.15	35.22	4.51
" "	1.037	26.79 26.98	26.89	3,44
" "	1,032	23,06 23.20	23.13	2,96
" "	1.028	20.02 19.85	19.94	2.55
" "	1,025	15.79 15.71	15.74	2,01
	1.019	12,80 13,00	12,90	1.65

On the following page will be found a curve plotted with Abscissaes Sp. G. and with per-cents of SO_2 for ordinates.



Abscissae = Specific Gravity
Ordinates = Per-cents of SO_2

While these results may be taken as correct for the solution used, yet no table of great commercial value can be made, owing to the fact that in practice it is customary to use a mixture of the Lime and Magnesium salts, so that different solutions of the same Sp. G may still have the same per-cent of SO_2.

As a conclusion from the work I would recommend the following method for the titration of Sulphurous Acid in solutions of Bi-Sulphite of Calcium.

Dilute the Bi-Sulphite solution to one tenth ($\frac{1}{10}$) of its original strength; make up a deci-normal solution of Iodine and of Thiosulphate of Soda ($Na_2S_2O_3$) having one exactly deci-normal and knowing the strength of one in terms of the other.

Measure out carefully 25 c.c of the Bi-Sulphite, and transfer quickly to a 200 c.c flask, and add a few drops of Starch indicator. Run in Iodine in excess, about the necessary amount being known by previous trial or from the Sp. G. Shake a moment. Now titrate back the excess of Iodine with the Thio-Sulphate solution. The solution will at first be of a greenish hue, but when not much Iodine is left in excess it will become a bright blue; add the Thio-Sulphate carefully until one drop or a fraction of a drop will destroy the blue, leaving a colorless solution.

To find the number of c.c of Iodine, subtract from the Iodine used, the Iodine equivalent of the Thio-Sulphate used.

To find the per-centage of SO_2 in the solution, it is necessary to know the number of grams in 100 c.c. therefore multiply by 4 to correspond to 100 c.c, and then by 10 to allow for dilution, and then by the wt. of SO_2 equivalent to 1 c.c of Iodine and by the no. of c.c of Iodine used, or

40 × no. c.c. Iodine × .0032 = % SO_2

My next plan was to design and have constructed a small digester in which I could make some wood Cellulose.

At first I thought of having one made of Phosphor Bronze or of some other acid resisting material; after examining one of this sort, and taking the cost and manner of working it into account I decided that a preferable one for small work could be made of Iron with a Lead vessel to contain the Sulphite solution and wood.

Accordingly I had one constructed after the designs shown on the following page.

- Fig 1 -

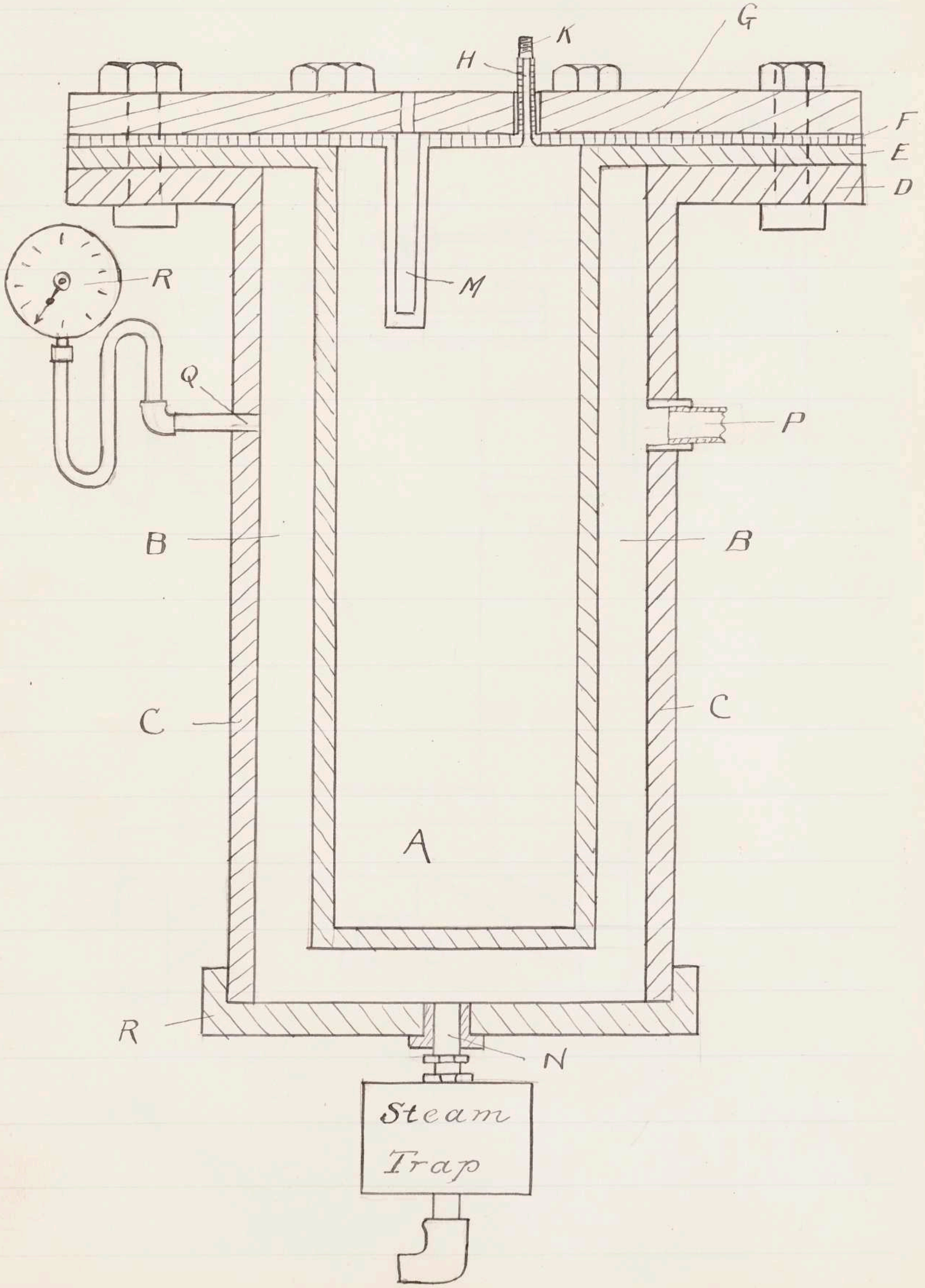


Fig. 2.

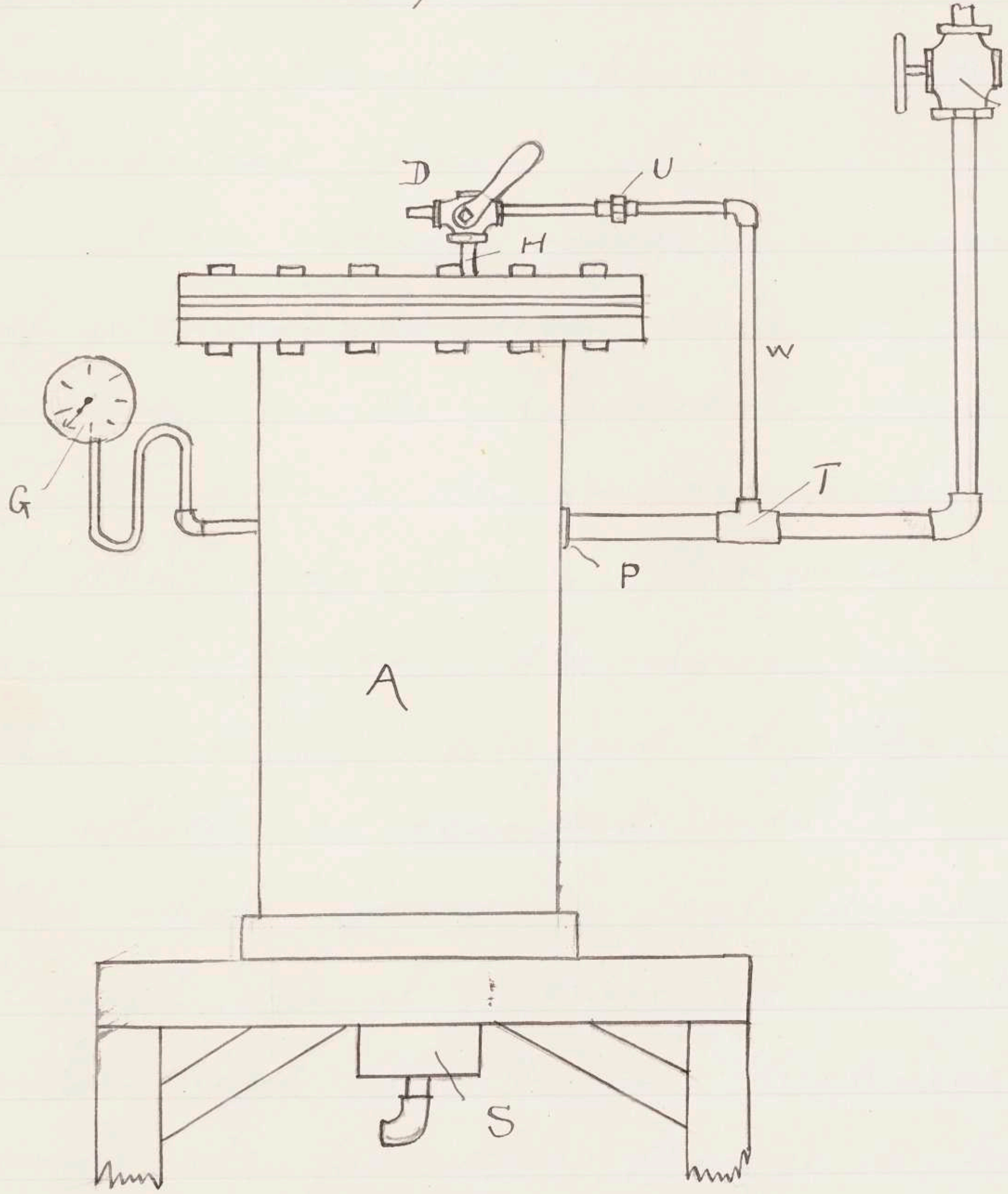


Fig I

This shows a vertical section of the Digester, on a plane passing through the axis of the cylindrical vessel.

The outside vessel C consists of a wrought iron tube of nine inches inside diameter, and of 3/8" iron; the upper portion of the tube has a flange D, 5/8" in thickness; the lower end of this tube is plugged by the cap R which is screwed onto it, so the whole forms a cylindrical flanged vessel; there is a cylinder head G, 3/4" in thickness made to fit this flange D, both the flange and head being provided with corresponding holes to allow bolts to pass through and the whole to be screwed together.

Within this iron vessel there is another one lettered A which is made of lead, 3/8" in thickness and 6" inside diameter; on the upper part of this lead tube a flange marked E is burned on, and which is of a size to just fit onto the flange of the iron vessel.

Over this vessel is placed a circular plate (marked F) 1/8" in thickness, and of the same diameter as the flanges of the lead and iron vessels; to this plate is burned a lead tube M, (shown in the diagram,) which dips down into the lead vessel; H is a small tube which projects upwards about two inches, and in the end of which a brass tube K is fused, and is provided with a thread as shown.

Holes are drilled through the lead flange and plate corresponding to those of the cylinder head and iron flange, so the whole may be screwed up together.

Thus it will be seen that the lead vessel is surrounded by the iron one which forms a sort of jacket.

On one side of the vessel shown at P is an opening through which a steam pipe enters; on the opposite side is another opening Q, into which the steam gage pipe is screwed; at the bottom of the iron vessel is an opening N, to which the steam trap is attached as shown by the diagram.

Now the object of this arrangement is to have wood and Sulphite liquor in this lead vessel, and cook the mass at the temperature corresponding to the steam pressure employed. Steam is admitted at P at the given pressure and surrounds the vessel, and from the Tee union shown in Fig 2, the pressure passes inside the vessel thus balancing the pressure on the outside.

Fig 2. shows the arrangement when set up, and the mode of procedure is as follows.

After the lead vessel is placed in the iron cylinder place the wood and liquor in it, place the lead plate on, and finally the cylinder head, a hole in which allows it to pass down over the tube H Fig 1. and there is also a hole directly over the thermometre tube M Fig 1. Now insert the bolts and screw the whole up tightly.

Fig 4. shows the arrangement for furnishing steam; at the opening P there is a bushing, into which screws the main steam pipe; at B is a Tee union with a pipe W leading up and over to the top of the digester as shown in the diagram; this pipe connects with a three way cock D, which is screwed onto the pipe H, leading to the interior of the vessel; at U is a brass union for disconnecting quickly.

The operation is as follows. First arrange the three way cock so it will blow into the air, then turn on the steam gently until all the water in the pipes is blown out; then turn the cock so it opens into the lead vessel, and let the pressure be light until the whole vessel is well heated up; then turn on to the desired pressure.

The steam trap S at the bottom is to take care of the condensation in the iron vessel, and is to be so regulated that there will be a little steam escaping, to be certain that no water is in the cylinder.

The arrangement is now

complete, and the contents may be allowed to digest for the desired time and at the required pressure.

If the temperature inside the vessel is desired by a thermometre, a glycerine bath may be placed in the tube M fig. 1. and the thermometre inserted.

– Results. –

At my first run I relied upon the lead surfaces to form a steam and gas tight joint, but was unable to obtain one; accordingly I unscrewed the, and between each joint I placed packing made of oiled brown paper, and with this arrangement there was no leakage; this paper requires to be renewed at each run, but it requires but a short time however.

I cooked the wood on my first run for six hours at a pressure of seventy eight (78) pounds.

The result was a pulpy mass of light brown color which darkened when exposed for a time to the air; some of the larger pieces of wood were not quite disintegrated.

I used acid of 10½° Tw. and in the proportion roughly of 2 litres of acid to 2½ litres of wood.

I washed this pulp carefully on a 40 sieve, and picked out the large hard pieces before mentioned; the result was a fibrous cotton like mass of a light brown color ; I placed some of this in a beaker and allowed to stand over night in a Bleaching Powder solution (very dilute) and then washed in very dilute Sulphuric Acid bath. ; the result was a pure white cellulose, but the fibres did not appear to be of great length.

The second run was under the same conditions as the first but I cooked seven (7) hours. ; a very complete disintegration took place ; I washed and strained this lot and obtained a fibre similar to the first.

Third run -- I cooked this lot five (5) hours at 80 lbs. pressure ; the wood was only partially disintegrated, and the pulp when washed was poor and mixed with hard pieces of wood.

Runs of three and two hours respectively with a pressure of 80 lbs. produced a very poor pulp, containing pieces of wood which apparently were very little acted upon by the liquor.

The liquor from the six and seven hour run I filtered, and obtained a solution of brown color, with a peculiar odor; I titrated these samples for Sulphurous Acid but found only 0.34 % and 0.39 % percent present.

It would be interesting to know the composition of these liquors.

I will give a few samples of the product obtained, on the following page.

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No. 1 — Sample of Cellulose after cooking six hours at a pressure of seventy eight pounds.

N.º 2 Sample after cooking for seven hours at a pressure of seventy eight pounds.

N.º 3. Sample of an unbleached sample of a seven hours cook at a pressure of seventy eight pounds.

N.º 4. Sample of an unbleached product after cooking five hours at a pressure of eighty pounds.

N.º 5. Sample (bleached) after cooking seven hours at seventy eight pounds; pressed for a day in a copying press.

No. 6. Bleached, sample after cooking seven hours at a pressure of seventy eight pounds.

No. 7. Unbleached sample of a six hours cook at a pressure of seventy eight pounds.

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May, 1891.