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Technical analysis of corn products

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TECHNICAL ANALYSIS OF CORN PRODUCTS.

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This thesis treats of the technical analysis of the products of corn as used in practical work in the manufacture of starch and glucose. These notes are the results of special investigation carried on along this line.

In the analysis of corn and its products, as applied in industrial works to control the process in the factory, several factors must be considered for the analysis to be of any value. All methods must be rapid and yet accurate enough to form a working basis for comparing and controlling the efficiency of the process. To accomplish this, it is necessary to make the determination on samples containing varying amounts of moisture and correction made for same in final results, as all determinations are reduced to the dry substance basis with the exception of oil and feed.

The rule generally followed is to take a sample large enough to give one-half to two grams of dry substance, depending on the constituent to be determined.

An approximate analysis of storage corn is as follows;

Oil	5%
Carbohydrates	71.5% (of which 55% is starch)
Albuminoids	10.5% (principally Gluten)
Water	11.5%
Ash	1.5%

The process of manufacture consists of steeping the shelled corn from 48 to 74 hours in dilute sulphurous acid,

breaking the steeped corn in burr mills,and separating the germ,starch,gluten and slop(skin of corn) by means of gravity,shakers and reels.

The starch is converted into glucose by hydrolysis with commercial hydrochloric acid,filtered,evaporated in vacuum pans to the desired Beaume' then used in making syrup, jelly,candy,etc. The gluten,slop and steeping water are mixed,dried in rotary kilns,ground in fuss mill and used for cattle feed. The germ is dried,put through expellers and the oil extracted,filtered and is then ready for use. The residue from the germ (oil cake meal) is used for feeding purposes.

Analyses are made of all these products in all steps of the process to determine the efficiency and also to aid in the treatment of same.

Moisture.

Corn. The moisture in corn must be determined accurately as it is used as a check on inspectors and it is also the only means of calculating percentage yields to show the efficiency of the process.

Sampling. A two pound sample is ground in a small disk mill to pass at least an eight mesh sieve. It is then quartered down and a four ounce sample taken for analysis. All samples should be labeled and tightly stoppered from time of sampling in car until analyzed.

Analysis.A five gram sample is weighed into a tarred dish with cover,then dried in a vacuum from four to six hours ,at 115

degrees C. and twenty inches vacuum (time to be determined by experiment). After sufficient heating dish is removed from oven, covered, cooled in dessicator and weighed and loss in weight called moisture.

Notes. Care should be taken in grinding samples as continued grinding with disks close set causes the mill to heat and thus reduces the moisture in sample to a considerable extent before analysis. If many samples are to be ground at one time at least two mills should be used.

Owing to the fact that these compounds oxidize and increase in weight on continued heating, thus giving low results, it is necessary to determine by experiment the correct time of heating in oven.

The moisture varies with the season and also in the different grades of corn used, from twelve percent in second grade to twenty-five percent in no grade. New corn will require from five to six hours, while later in the season, after the corn has been in storage for several months, the time of heating can be reduced to four or four and one-half hours.

The time of heating should be determined every few weeks by means of a series of check determinations made on the same sample and carried on on different days, removing a sample from the oven every fifteen minutes and handling same as in regular determinations. By noting results or plotting curve of same, the correct time to use is easily determined.

Dishes. The dishes should be made of aluminum, nickel or german silver, 45 by 5 c.m. deep and provided with lids made of the same material and only one to two c.m. deep, which fit snugly inside of the dish. The dish and lid should be numbered and much time is saved if all dishes are filed to the same weight. The oxidation of these dishes is very slow and the weights need to be checked and adjusted only about once a month. The dishes should always be covered from time of taking sample out of oven until weighed, as moisture is rapidly taken up even in the dessicator if dish is not covered.

Oven. Experiments were carried on with several ovens. (1) Ordinary air oven with vacuum connection and heated by bunsen burner. (2) Water jacketed vacuum oven heated by (a) bunsen burner, (b) steam. (3) Vacuum oven heated by electricity.

The temperature could not be accurately controlled on any of the ovens of the first or second type and also in the water and steam jacketed ovens leaks were frequent and determinations lost, owing to the high vacuum used, causing very fine sweating leaks.

The vacuum oven, heated by electric current, was found to be greatly superior to all others, owing to the ease of controlling the temperature which was found to be uniform throughout the oven and the chance of loosing determinations by leaks or parching of sample at high temperature is avoided.

The oven should be provided with shelves for the dishes, an accurately fitted door hinged and clamped so as to avoid the use of rubber gaskets, thermometer tube extending into interior of oven, stop cock for regulating the vacuum in oven, which is supplementary to valve and water trap on main vacuum line. This connection should be made in back of oven and near top, above first shelf. There should also be a small pipe running through the back of the oven near the bottom and extending to within one or two inches from the front, with outside cock at back to regulate a slow circulation of dry air taken from two sulphuric and one caustic acid wash bottle provided with safety valve to keep sulphuric acid from being mechanically carried over into oven. If this circulation of dry air is not provided for, the time of heating with require a much longer time.

The oven should have a rheostat connection to regulate temperature and after regulating a constant temperature can be maintained for hours without requiring attention.

Moisture in gluten, slop and press feed. The sample is prepared the same as for corn determination except the gluten which is reduced in porcelain mortar to pass 60 mesh. Weigh three gram sample into weighted porcelain crucible and heat for four hours in an air oven at 110 to 115 degrees C. Cool in dessicator, weigh, loss in weight regarded as moisture.

Starch. Prepare sample as for gluten and heat a three gram sample in air oven four hours at 120 degrees C.

Oil Cake,Germ. Heat a five gram sample in air oven four hours at 105 degrees C.

Sugar,Glucose,Dextrose. A five gram sample is heated in a water jacketed air oven to constant weight at 97 degrees C or until weighing are within two milligrams. Dishes should be of the same material and pattern as is used for corn moisture.

Feed. A five gram sample as taken from fuss mill is heated five hours in water jacketed air oven at 97 to 98 degrees C. The same dish used as for corn determination and dishes should be covered from time of taking from oven until weighed. Feed is easily scorched,owing to the large amount of evaporated steep water that it contains and also when dry the steep water takes up moisture very rapidly,giving results that are too low unles dish is well covered.

Glucose,Syrup and Jelly. Take one to three grams of sample and dissolve in water and mix well in flat aluminum dish, containing short glass rod and about twenty grams of clean, sharp quartz sand which will pass through at least a ten mesh sieve,all of which has been dried to constant weight. Mix sample well with sand and dry to constant weight with frequent stirring in a water oven at 95 to 98 degrees C. Seven to ten hours will be required for determination.

Steep Water and Press Muds. A 50 c.c. sample is evaporated to dryness in a weighed platinum dish on steam bath and then dried to constant weight in water oven at 98 degrees C. One to two hours drying being sufficient.

STARCH.

Samples. The samples for starch determination should be ground as fine as possible or beat up in mortar and put through at least a forty mesh sieve. If the precentage of starch is low as in slop,it is not necessary to have the exceptionally fine frind,owing to the nature of the sample.

Determination. Take one to five grams finely ground sample,representing from one-half to one gram of dry substance,in a nickel or german silver beaker,add 50 c.c. hot water and boil for 45 minutes or until well parted,stir frequently,cool to 50 or 55 degrees C. and add 60 cc. of freshly prepared malt extract. Keep the temperature even for from thirty to forty-five minutes,stirring frequently, or until a drop of solution from beaker gives no starch reaction with dilute iodine. When malted,heat again to boiling,stirring frequently and boil for forty or fifty minutes. The longer this last boiling is carried on,without loosing time or going to dryness,the better it will be as time will be saved in filtering and washing and better results are obtained. Filter on weighed 12 cm. filter papers,using suction bottle,wash a few times(if well malted three is sufficient) with boiling water,dry

in air oven four hours at 120 degrees C. cool in desiccator,using sulphuric acid,weigh and calculate starch by difference,taking into account water soluble and moisture.

The method is based on the fact that fresh extract of malt will convert starch into a soluble diastase which is easily washed from residue. Malt extract is made by digesting 100 grams of finely ground fresh malt (obtained from brewers) with 600 cc. water for three or four hours at room temperature,filtered and is then ready for use. Malt should be freshly made for each set of determinations and not allowed to stand long before using. As there is a residue in the filtered extract on evaporation,60 cc. or the same amount used in the determination is run as a blank test along with each set of starches and correction made for same in the final calculation.

In filtering,use suction flasks,very fine holed cones in funnel all connected to suitable vacuum apparatus.

Filter papers (12cm.) are weighed in marked weighing bottles 30 by 70 cm. in height,which should be kept covered except when in the oven. One to two hours heating in air oven at 120 degrees C. is sufficient to drive all moisture from papers. Bottles are then covered,cooled and weighed and are ready for the filtration of malted sample. After filtering and washing,the paper containing residue is placed in same weighing bottle and heated in oven four hours at 120 degrees C.,covered,cooled and weighed.

The above method gives satisfactory results on all products containing from five to seventy percent starch (such as gluten,slop,feed,etc.)although the results are slightly lower than theoretical. For process starch (90 to 92 %) the method can be used but very careful manipulation is required to obtain accurate results..

WATER SOLUBLE.

All products are soluble in water to some extent,which must be determined and corrected for in final results.Two methods are used. The samples are prepared the same as for starch determinations.

1 A two gram sample is digested at room temperature for two hours with 100 cc. water,filtered same as in case of starch,but washed with cold water,dried,weighed and loss called water soluble after making correction for moisture.

2 Twenty grams of sample is digested with exactly 200 cc. water in nickel beaker for two hours at room temperature, filtered and 50 cc. of the filtrate evaporated to dryness on steam bath. Dried in oven one hour,cooled,weighed and percentage soluble calculated from weight of residue,correcting for moisture in original sample.

The second method gives lower results than the first and is found tobe more accurate,but when the percentage soluble is small,method number one is used as the error is small and determination can be carried along with starches,thus saving time.

Protein (NH_3)

Solution required.

Tenth normal sulphuric acid standardized with barium chloride

" " sodium hydroxide exact strength as N/10 acid

Sulphuric Acid.C.P. 1.84 sp.gr.

Mercury,metallic or mercuric oxide.

Cochineal solution,-3 grams cochineal,50 cc.alcohol,200 cc. water are digested with frequent shaking two days,filtered and ready for use.

Potassium permanganate,powdered.

Zinc dust.

Potassium sulphide,four percent solution.

Sodium hydroxide.41½ Beaume' or stronger,made by boiling powdered caustic in covered vessel to get rid of as much carbonate as possible,filtering through glass wool and keeping in tightly stoppered bottles.

Determination. Weigh one to five grams of finely ground sample into 800 cc. Kjeldahl flask,add one drop of metallic mercury or .7 grams mercuric oxide and 25 cc. sulphuric acid 1.84 sp. gr. (in case of starch use 35 cc. acid) allow to stand 15 minutes then boil in hood over direct flame with flask slightly inclined until all organic matter is oxidized and solution is perfectly clear,requiring from one to two hours. When clear remove from heat,hold upright and add one to two-tenths gram of potassium permanganate for final oxidation,cool,add 300 cc. water,½ gram zinc dust to prevent bumping,and a couple of drops

of parafine oil to reduce frothing. Add 100 cc. of alkali solution, containing 75 cc. caustic, 41½ Beaume' and 25 cc of 4 percent potassium sulphide. This amount of solution will completely neutralize the sulphuric acid used and take care of the mercury so that no mercury-ammonium compounds are formed. Be sure to have solution alkaline before distilling.

Before putting the alkali solution into flask all connections should be tried and a suitable amount of N/10 sulphuric acid in titrating flask connected to the condenser. The ammonia begins to come over as soon as solution is made alkaline therefore care must be taken in adding the caustic to prevent loss, This is generally done by allowing caustic to run down side of flask so as not to disturb the contents of flask, and immediately connecting flask to the condensers. After all connections are tight the contents of flask is mixed by shaking and slow heat applied for a few minutes, then temperature raised to a rapid boil until about 300 cc. of distillate has been collected in the receiving flask. Cochineal is added to the flask, containing the distillate and the excess of acid is titrated with N/10 solution hydroxide. One cc of N/10 acid is equal to .00875 protein.

As all caustic and chemicals used may contain some nitrogen products it is necessary to run blank determinations on each lot of caustic made up, using C.P. cane sugar as the reducing agent and treating the same as in the regular determination.

This blank of caustic is well prepared, seldom uses more than three tenths cc N/10 acid which must be corrected for in final calculation.

Most of the ammonia distils over with the first 100 cc. of distillate and practically all will be in the first 200 cc. but for safety it is advisable to collect 300 cc. as this will always keep the apparatus clean for future determinations.

Mercuric oxide is used in place of metallic mercury in some cases but the time of sulphuric acid digestion will be longer. Pumice stone may be used in place of zinc dust. Potassium permanganate is not absolutely necessary but as it does not effect reaction in any way it should be used as it insures complete oxidation of any organic matter that might be left after digesting with sulphuric acid.

Cochineal may be added to the N/10 acid before distilling. This is advantageous when the amount of N/10 acid to be used is not known as it shows at once if the acid has been used up and more of the standard acid can be added without loss of ammonia. There is some loss of ammonia if the distillation is carried on for any length of time after the receiving flask becomes alkaline. It is advisable to have the N/10 acid used up as near as possible, that is within two or three cc. as this reduces time of titrating and also the chance of error if the two standard solutions are not of the same strength.

The distillation should be carried on as rapidly as possible after the first few minutes of heating. It is almost impossible to get high results by this method as all chances of errors tend to give low results.

Protein in steep water and all light liquors. Take 50 c.c. of solution to be tested in 800 c.c. kjeldahl flask and connect to suction pump with rubber stopper, placing flask in hot water, using about 25 inches of vacuum. The contents of flask will evaporate to dryness in 15 minutes. This evaporation reduces the chance of frothing, also shortens time of digestion with sulphuric acid.

Glucose, syrup and Jelly. Weigh 10 grams in watch glass and wash in kjeldahl flask with as small an amount of water (hot) as possible, not over 20 cc. Use 50 c.c. concentrated acid and therefore double the amount of caustic for distillation. Several hours are required for digestion and the heat must be applied very slowly or flask will boil over when carbonization takes place.

Purity. (Dextrose).

In neutralized liqours the purity is determined by means of Brix spindle and Fehling solution. Saple is placed in hydrometer cylinder and temperature reduced to 17½ degrees C if possible, to avoid corrections in calculations. If correction for temperature is necessary, correct according to standard sugar tables.

The Beaume' scale may be used in place of brix spindle, allowing 1.8 degree brix for each degree Beaume' at 17½ degrees C. Take 25 c.c. of this solution and dilute to

500 c.c. in graduated flask and titrate against 25 c.c. Fehling solution in 250 cc. Florence flat bottom flask, adding the sugar solution from 25 cc. burrette and boiling between each addition of solution. The boiling should occupy the same time,about two minutes,for complete determination,as the time of boiling effects results,especially if in standardizing the fehling solution a different time is used than in regular determination. The end point is reached when a straw colored solution appears,the cuprous oxide separating out clear.

The color changes,taking place while titrating,are blue, brown,green to straw color and by noting these colors the end point can be reached in two or three additions of sugar solution. By lightly tapping flask on titrating counter the cuprous oxide will settle very rapidly,leaving solution clear in a few seconds.

Fehling Solution. No.1. 71 grams of copper sulphate is dissolved in water and made up to one liter.

No.2. Dissolve 346 grams of Rochelle salts and 100 grams of pure sodium hydroxide in water and make up to one liter. Mix equal volumns of each solution and standartize.

Standardization. Weigh one gram C.P. Dextrose into 100 cc. graduated flask,fill to mark with water and titrate 25 cc fehling solution with this solution as in regular determination. As dextrose is seldom C.P. purity must be determined then .

Purity of detrose % dry substance X titration = Dextrose factors. Calculation of Purity (Dextrose). By means of standard sugar tables, correct brix reading, if not taken at 17½ degrees C. also from same table get specific gravity corresponding to the corrected brix reading, these

$$\frac{\text{corrected Brix X Sp.Gr. \% titration}}{\text{2 Factor (found by dextrose det.)}} = \text{Purity (\% dextrose)}$$

Purity in sweet water and all light liquors-

Evaporated one to ten liters, according to sugar contents, to 100 cc. and titrate against 5 cc. Fehling solution as above. For anhydrous sugar dissolve 6 grams in 500 cc. water and proceed as in regular determination.

Invert sugar is calculated from dextrose by multiplying by 1.044.

Acidity.

Owing to the phorphoric acid present in corn which is also present in all the liqours it is difficult to get clear end reactions and different end points are obtained with different indicators, that is the end reaction using phenol as indicator requires twice the amount of acid as when acid methyl orange as indicator, resolic acid, litmus and cochineal give end points between the other two, resolic acid being nearest methyl orange. The sulphur dioxide present also interferes with indicator and it is necessary to call the first faint change in end point to avoid over titration. Owing to these conditions and the color of the solutions a large amount of indicator is used.

Acidity in Glucose, Syrup, Jelly and all Heavy Goods.

Weigh 50 grams of sample in tall beaker or glass, dilute with hot water, stir until dissolved and titrate with N/10 sodium hydroxide, using phenol as indicator. End points will be a very slight change in shade of original color.

After dissolving in water it is sometimes necessary to dilute to several hundred cc. to clear the solution so that end point can be determined.

Steep water. Use 10 cc. of sample and dilute to 150cc. use phenol as indicator and titrate with normal sodium hydroxide to first appearance of floculent precipitate which is slightly pink.

Char Acid Water. Determine same as steep water except that a small amount of indicator is used and distinct pink end point is obtained.

All light liquors. Take 50 cc. of sample and dilute until clear (from 100 to 200 cc.) titrate with N/10 sodium hydroxide to first indication of pink, using phenol as indicator.

Feed. Digest 10 grams of sample in 200 cc. of water for two hours, filter, wash and make up to 500 cc in graduated flask. Titrate two portions of 100 cc. each, one with resolic acid and one with phenol as indicator to very faint end points and calculate total acidity.

Sulphur Dioxide.

Solutions. Dissolve 80 grams of Iodine and 160 grams of potassium iodide in water and make up to two liters, keep in dark bottle 1 cc. equals .01 percent sulphur dioxide.

Take 100 cc. of above solution and make up to 1000 cc. 1 cc equals .001 sulphur dioxide.

Starch Water. Dissolve two or three grams of pure corn starch (Oswego or purified table starch) in about 500 cc. of hot water, stir until all lumps are in solution.

Total Sulphur Dioxide. The determination may be made on the same sample in which the acidity was determined or made direct by weighing 50 grams, dissolving in water, adding phenol and making alkaline. In either case make strongly alkaline with potassium hydroxide, allow to stand 15 minutes, then make acid with sulphuric acid, (One acid to two water) add a few cc. of starch water and titrate at once using the stronger iodine solution. Titration should be as rapid as possible.

Free Sulphur Dioxide. Sample is taken in the same manner as for total sulphur dioxide except that after determining the acidity the sample is not made alkaline but starch water is added and titration is made at once, using the dilute iodine solution.

Gluten Water, Separator Solutions and all Wash Waters.

Take 100 cc. of original sample as received unfiltered, add a small amount of starch water when necessary and titrate with strong iodine solution.

Starch. Weigh 10 grams of sample and make thin emulation in cold water and titrate direct with weak iodine solution or if end reaction is not distinct,owing to excess starch present,dilute and titrate an aliquot part and calculate total sulphur dioxide.

Specific Gravity by Beaume' Scale.

Syrup, Glucose and all Mixing Goods are calculated at 100 degrees F. as standard,but owing to the viscosity of the liquids it is necessary to make the determinations at a higher temperature to provide for the easy adjustment of the spindle. Syrup and Glucose are weighed at 142 degrees F. and one degree Beaume' added to actual reading taken so as to correct to 100 degrees F. One tenth degrees Beaume' is allowed for every four degrees F. as above or below 142 degrees F., substracting if below and adding if above that temperature.

The sample is put in copper cylinders 10 inches by 2 inches in diameter and placed in a water bath with the cylinders completely surrounded to within one inch of top. After reaching the required temperature the bath is held at 142 degrees for from one to two hours to allow all air bubbles to come to top and to insure uniform temperature throughout sample.

The hydrometer should be kept in the same bath as sample so as to be of even temperature and should not be put into the syrup cold as the surface of sample congeals and keeps spinde from sinking to proper level.

The sample is s'immed just before taking the Be' reading. Hydrometers are allowed to drain but are not dried before placing in the sample. Readings are taken at an angle of 45 degrees, reading through the meniscus and thus having the correct reading at surface level of sample.

After reading is taken the hydrometer is pressed down about three-tenths Be' and allowed to rise and reading again taken to insure against the sticking of spindle.

All hydrometers should be standardized against a set of standards which has been checked by pyknometer determinations or should be checked direct by pyknometer every few weeks and necessary corrections marked on stem. This checking is required owing to the fact that spindles in constant use change in weight and als the enclosed scale may become loosened and slip down a few tenths which isn not noticible by observation.

Cane syrup and Fancy Mixing Goods. Be' readings are calculated to 60 degrees F. The determination is made the same as above except in case of sorghum where seven to ten hours is required to get all air out of sample. Two degrees Beaume' are added tothe reading taken at 142 degrees.

Steep Water and Starch Liquors. Beaume' readings are taken at 60 degrees F. and sample should be well shaken in bath to insure perfect mixing, for every eight degrees above or below 60 degrees F one tenth degrees Beaume is allowed. If foam caused by fermentation interferes with reading of spindle a drop of fusel oil is added.

Weight per Gallon from Specific Gravity.

Syrup,Glucose and all mixing goods. A four inch funnel with long thin stem is stopped with glass rod and piece of rubber tubing. The funnel is then filled and allowed to stand several hours until all air,sticks and impurities come to the top. The plug is removed and sample allowed to flow into weighed 100 cc. flask until filled almost to neck,care being taken not to get any syrup on side of neck. Flask and syrup are weighed,then filled to mark with water and placed in bath at 60 degrees F. for one to two hours to insure uniform temperature. Note that flask is filled exactly to mark then dry and weigh.

Calculation. The weight of flask should be known by several check determinations also the weight of flask plus water to 100 cc. mark at 60 degrees F. These weights need only be checked once in two or three months.

The weight of the syrup plus water,less the weight of syrup equals the amount to be deducted from weight of water when flask is filled to 100 cc. mark. The remainder is divided by the weight of syrup alone,which gives the specific gravity. The specific gravity multiplied by .833 equals pounds per gallon.

Light Liqcurs. The specific gravity of all light liquors, Emulations,etc. is determined by phknometer or by Beaume' reading and calculated from suitable tables (chemical handbook)

In Pyknometer determinations use 25 or 50 cc. pyknometer with capillary side arm graduated every 1/8 or 1/16 inch,

the water value of each mark being determined at 15½ degrees C. several times. The thermometer should be ground into stopper. As none of these liquids are volatile the cap on the capillary arm may have fine hole ground in top to allow for the adjustment of level of liquid in arm. The bottle is filled with sample, capped, cooled to 15½° and line to which liquid extends in arm noted, then dried and weighed. The weight of sample at any mark divided by weight of water at same mark gives specific gravity.

The pounds per gallon may be determined by Beaume' or specific gravity determinations by following formulae.

$$\frac{145 - Be'}{145 \div (145-Be')} \times .833 = \text{Pounds per gallon.}$$

Corn Oil.

Oil is determined by means of Soxhlet extraction apparatus using carbon tetrachloride as solvent. The sample is weighed and put in fat free thimbles with small piece of cotton on top to hold sample. Put small piece of cotton in extraction funnel, insert thimble and connect to 150 cc. wide necked, flat bottom flask of known weight. The extractor is filled with enough carbon tetrachloride so that 25 to 50 cc. will remain in flask when extractor is full to syphon arm. The apparatus is then connected to condenser and heated on hot plate or in steam bath from four to seven hours, depending on amount of oil present. The process is continuous as the carbon tetrachloride is volatilized in flask then passes through large side arm, extending above thimble is condensed and falling into

thimble percolates through sample extracting the oil which is automatically syphoned through small arm back into flask. The carbon tetrachloride is again volatilized, leaving the extracted oil in flask. At the end of extraction thimbles are removed from extractor and the carbon tetrachloride is distilled as above, collecting in extractor. This distillation is carried on until three to five cc. of chloride is left in flask. The flask is then disconnected and dried in air oven at ninety degrees C. for one hour or until all tetrachloride is evaporated, cooled, weighed and calculated the increased weight of flask as oil. For gluten, oil cake and feed use five gram sample, starch and slop ten grams and germ meal two grams. The moisture should be under two percent to avoid water extraction or extraction of resinous matter.

Fibre.

Fiber in Feed, Oil Cake and Slop. Take two grams of the residue left from oil determination, place in 500 cc. erlemeyer flask and add 200 cc. of boiling sulphuric acid 1.25 percent, loosely cover flask with small watch glass, heat to boiling and boil for 30 minutes, filter on a weighed hardened filter paper, wash with hot water and then wash back into same flask, by removing paper from funnel and opening on large watch glass, with 200 cc. 1.25 percent sodium hydroxide, free from carbonate, or wash into flask with 100 cc. hot water and add 100 cc. boiling 2.5 percent sodium hydroxide, heat at once to boiling and boil thirty

minutes,adding a drop of parafine oil will partially reduce the excessive frothing at this stage. This step must be closely watched and agitated to prevent loss from frothing. After boiling,filter at once on same paper used above. Thoroughly wash with hot water and finally with a little alcohol and ether. Dry to constant weight at 100 degrees C and weigh. Determine the amount of ash by incineration, also the loss in weight of blank paper treated with hydroxide,alcohol and ether and deduct from total weight of residue above. Remainder is calculated as fiber. Care must be taken not to vary time of boiling or strength of solutions used. Also determination should be carried on as rapidly as possible to insure check results.

Ash.

In Feed,Gluten,Oil Cake,etc. A two gram sample is heated in platinum crucible at low heat until most of volatile matter is driven off. The temperature is then raised to red heat until ash appears grey or greyish brown. Cool, moisten with alcohol and heat again. Repeat until constant weight is obtained.

Germ and all products high in oil or phosphoric acid are not treated with alcohol. Also use old crucibles as ash is in nature of resinous fusion and crystallizes platinum very rapidly.

Glucose,Syrup and all heavy liquors and steep waters. A fifty cc. sample is evaporated to dryness in weighed platinum dish on a steam bath,charred at a low temperature over direct flame,then heated to constant weight at high

temperature. Dish should be partly covered by piece of platinum foil throughout determination to avoid mechanical loss.

Carbon Dioxide.

In Syrup, Glucose and Mixing Goods. Fill a 500 cc. Erlemeyer flask to neck with sample, allowing only one or two cc. of air space between sample and bottom of close fitting, one holed rubber stopper. Connect small bore glass tube with flask and pass into test tube of calcium hydroxide solution, also stoppered. Allow to stand from one to five days at room temperature. A white precipitate in tube or white film in glass tubing where air meets surface of calcium hydroxide solution, indicates the presence of fermentation (carbon dioxide).

If fermentation is well advanced connect sample as prepared above to weighed potassium hydroxide bulbs and allow to stand twenty-four hours and weigh. This gives comparative results Only.

Sulphuric Acid.

Determination is made gravimetrically by evaporating 25 to 200 cc. of solution nearly to dryness, adding one to two drops of hydrochloric acid, if necessary, and 50 cc. of water. Heat to boiling and add 1 to 25 cc hot barium chloride solution (1 cc equal to .01 barium sulphate). Boil 10 minutes, cool and allow to stand several hours. Filter and wash with water, containing a few drops of alcohol. Dry, blast in platinum crucible, cool, weigh as barium sulphate and calculate sulphuric acid.