The Recovery of Crude Glycerine

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Salt Chamber

The first of the three methods of handling the salt and the oldest, is to have a large chamber in the evaporator below the calandria in which the salt from the entire run is collected. After the finish of a run, the entire charge is dropped into a tank with a false bottom covered with a filter bed of wire screen and filter cloth. The glycerine is then pumped off from below the false bottom. The salt is washed first with lye, then with water, the wash liquors being pumped back to the evaporator feed tank. By this procedure the glycerine content of the salt may be brought down to 0.5 per cent to 2.0 per cent, depending on the amount of washing and the care used.

This method of collecting and handling the salt is used only in single effect evaporation.

Figures 14 and 15 show shop views of such single effect evaporators with a large salt chamber below the calandria section.

Salt Extractors

The second method, which is still extensively used in small or moderate size plants, is to have the bottoms of the evaporators connected to vessels variously known as salt filters, salt boxes or salt extractors.

Figures 2, 3 and 11 show such salt extractors.

These salt extractors are cast iron or steel vessels with a 6-in. or 8-in. inlet at the top connected to the evaporator with a gate valve, and provided with a false bottom with a filter screen, a quick opening discharge door, sight glasses, steam and water connections and a vacuum breaker. For a double effect evaporator there are usually three salt extractors, but sometimes only two are installed. With three salt extractors each evaporator can always be dropping salt while one extractor is being emptied. With only two extractors the salt is allowed to accumulate in the evapora-

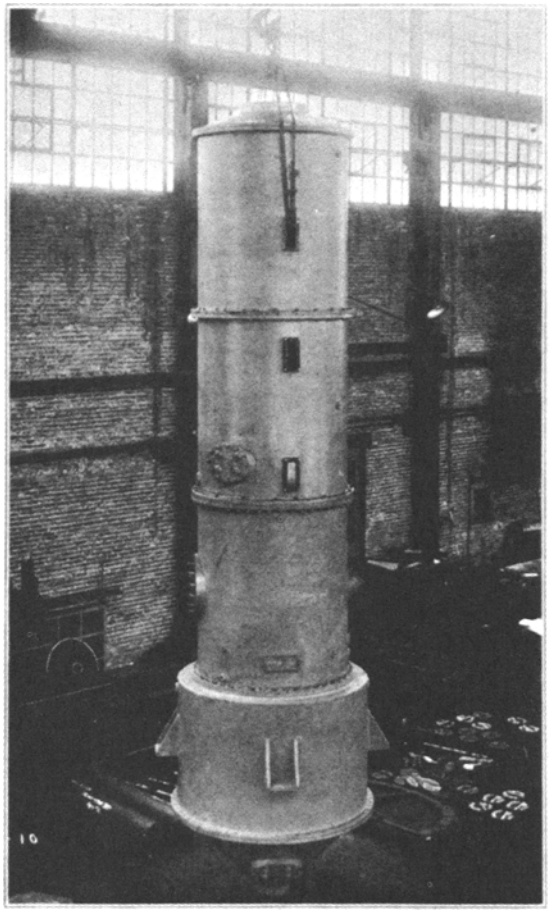

Fig. 14. *Wurster & Sanger 5-ft. Diameter Glycerine Evaporator*

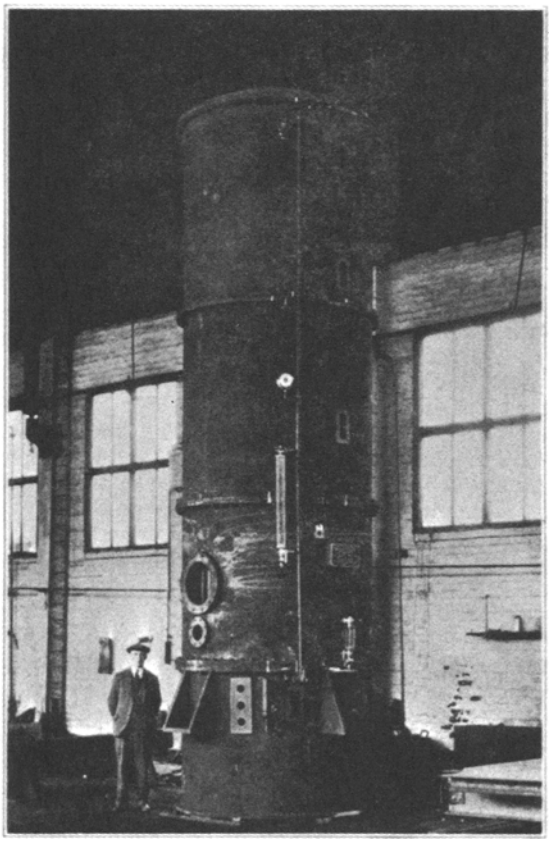

Fig. 15. *Wurster & Sanger 6-ft. Diameter Glycerine Evaporator*

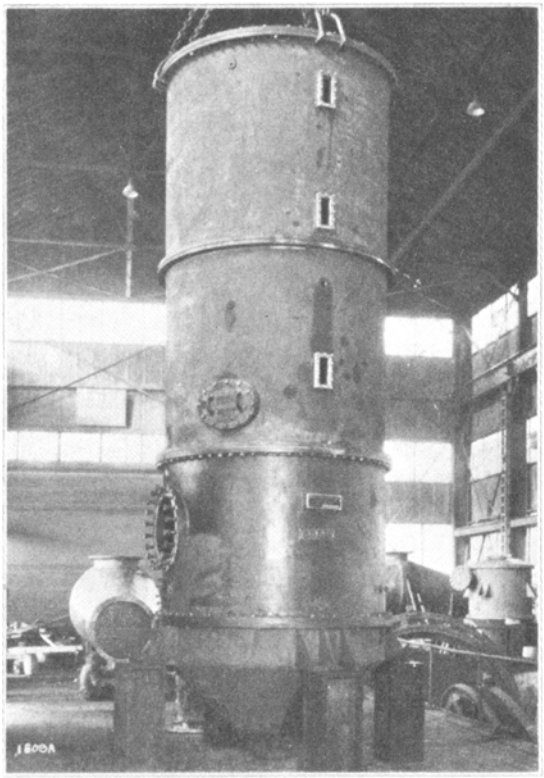

Fig. 16. Wurster & Sanger 7-ft. Diameter Glycerine Evaporator

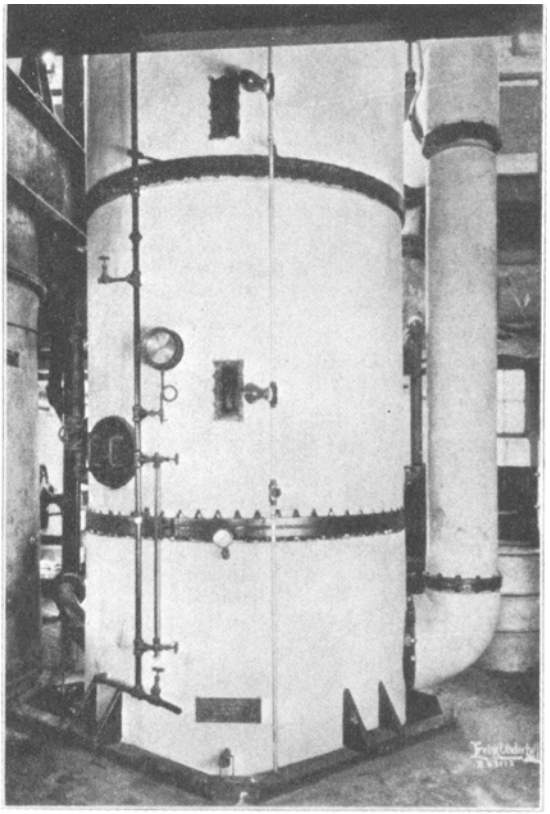

Fig. 17. Wurster & Sanger Single Effect Glycerine Finishing Evaporator

tor during the time required to steam, dry and empty the extractor.

Filling the Salt Extractors

The method of operating the salt extractors is as follows: The valve on the return line from the bottom of the salt extractor to the vapor belt of the evaporator is opened (if it has not been left open since starting up) and vacuum is pulled on the salt extractor. When the vacuum is the same as in the evaporator, this valve is closed. The large salt valve at the top of the salt extractor is opened slowly and the extractor is filled. The salt valve is left open and liquor is fed to the evaporator to restore the normal liquor level.

As the evaporation continues the liquor again becomes a supersaturated salt solution and salt crystals will fall into the bottom of the evaporator and drop into the salt extractor. Evaporation is continued and when the salt extractor is full of salt, as seen in the top sight glass, the salt valve connecting this extractor to the evaporator is closed.

The same operation as above described is now repeated with the other salt extractor by opening the salt valve, so that each effect is at all times connected to one salt extractor and the salt is removed from the evaporators continuously.

Drying and Removing the Salt

The salt in the extractor is now ready for drying. The valve on the return line from the bottom of the salt extractor to the vapor belt of the evaporator is opened. The vacuum breaker, which is a valve connected to the top head of the extractor and opening to the air, is opened. This breaks the vacuum and puts atmospheric pressure on top of the salt and liquor in the salt extractor, whereas the bottom of the extractor is under vacuum through being connected into the evaporator which is under vacuum. The liquor mixed with the salt will draw through the salt and pass back into the evaporator body. It can be determined when the liquor is passing through by feeling the return line, which becomes warm. The level of the liquor as it drops may also be seen in the sight glasses on the salt extractor.

After the liquor starts to pass off from the salt extractor, the vacuum breaker is closed and steam is turned on to the top of the salt. Enough steam is kept on to show a low pressure on the extractor. After the liquor has all been drawn off into the evaporator body, steam is kept turned on for 15 to 30 minutes to dry the salt. When the salt is dry, steam is turned off and the valve on the salt extractor return line is closed. The salt extractor is now completely shut off from the evaporator. The vacuum breaker is opened. The salt door is opened and the dry salt removed with a hoe into a truck or wheelbarrow.

When the salt extractor is empty, the salt door is closed, and the valve on the return line to the evaporator is opened very slowly to pull vacuum on the salt extractor so that it will be ready for dropping the next charge from the evaporator.

Salt recovered by means of salt extractors as described above and carefully steamed will have a glycerol content of from 1% to 2%. If the salt in the extractor is not thoroughly steamed the glycerol content may be considerably above 2% and portions of the salt may even be quite wet. The remedy is longer steaming.

Salt Drums and Centrifuge

The third method of handling the salt is by means of salt drums and centrifugals.

Figures 12 and 13 show the salt drum arrangement.

The salt drums are of steel or cast iron and one is attached to each effect by means of a 6″ or 8″ salt valve. It is not necessary to have two salt drums attached to each effect because the time of emptying is too short to cause any great amount of salt to collect in the evaporator.

The filling of the salt drums is similar to filling the salt extractors as described above. When salt starts crystallizing in the evaporator, the salt valve is opened and the salt drum filled with liquor and salt from the evaporator. The liquid level in the evaporator is restored to normal by feeding. As evaporation continues, the salt formed drops down into the salt drum. When the salt shows at the top sight glass, the salt valve is closed. The salt sludge in the salt drum is now ready to be blown to the salt sludge tank above the centrifuge.

Steam from the blow-out line is turned on to the salt sludge line to clear the line and make sure that it is open to the salt sludge tank. The steam is then turned off and the gate valve at the bottom of the salt drum is opened. Air under pressure is then turned into the top of the salt drum and the charge of salt sludge is blown to the salt sludge tank. Only sufficient pressure should be used to remove the charge. When the salt drum is empty, the air is turned off and steam is turned on to blow out the line. The steam is turned off and the valve at the bottom of the salt drum is closed.

The salt valve between the salt drum and evaporator is opened, the salt drum is filled again and the cycle repeated.

Handling the Salt Sludge

The salt sludge is run from the salt sludge tank to a centrifuge. After the basket has been filled and spun to remove the heavy glycerine liquor, the salt is given a lye wash and a final water wash with a few quarts of water. The wash liquors are returned to the evaporator feed tank. The salt is then removed through the bottom of the basket by means of an automatic unloading device. It requires about 12 minutes to handle one charge in a centrifuge with a 40″ diameter basket, so that 5 charges per hour may be run.

Little washing is required in centrifuging the salt and the glycerine content of the salt is brought down to 0.25% to 0.5%. To avoid using too much wash water, it is well to do this by hand, using a small sprinkler can. The simplicity and convenience of this method of handling the salt and the low glycerine content of the recovered salt has resulted in the extensive use of this method and, all factors considered, it is probably the most satisfactory method in present use.

A continuous, rotary, vacuum filter may be used in place of the centrifuge. The continuous filter probably requires less attention than the centrifuge. More washing is required to bring down the glycerol content of the salt, thus producing considerably more wash liquor. The power requirements of the continuous vacuum filter are slightly higher than for a centrifuge. For the same capacity the cost of a continuous filter is higher.

Continuous centrifuges, recently improved, are now recommended for this service and may be a future development. At present these machines are costly and have not been tried out on this work.

Concentrating and Finishing

The operation of removing the salt from the evaporators is repeated as often as necessary until the glycerine in the second effect reaches the half-crude stage. The half-crude stage is readily recognized, because at this time, maintaining the same levels in the evaporators and the same amount of steam on the calandria of the 1st effect, the vacuum on the 2nd effect tends to rise, and on the 1st effect the vacuum drops and the steam pressure on the calandria rises. There are several methods of proceeding from this stage to finished crude glycerine.

Finishing in Double Effect

It is possible to continue to operate the evaporator as a double effect and finish to 80% crude in the 2nd effect. In this case feeding lye to the 1st effect and feeding from the 1st to the 2nd effect is continued. The steam pressure on the calandria of the 1st effect rises gradually to about 10 lbs. and the vacuum drops to zero or slight pressure is shown. The finishing is very slow and time consuming on account of the slow rate of evaporation and therefore this procedure is not generally followed.

Finishing in 2nd Effect

The charge may be finished from semi-crude to crude in the 2nd effect. When the stage of half crude is reached, as described above, and the vacuum drops on the 1st effect and the steam pressure rises, the rate of evaporation slows down and time is saved by discontinuing double effect evaporation and finishing in single effect.

Steam is shut off from the 1st effect, the vapor line from the 1st effect to the calandria of the 2nd effect is shut off, and the condensation drain of the 2nd effect is shut off from the condensate pump and connected to the steam trap. Live steam is then turned on to the calandria of the 2nd effect. The liquid in the 1st effect is gradually fed into the 2nd effect and any partly concentrated liquid from a previous evaporation is drawn into the 2nd effect.

In preparation for such finishing in the 2nd effect, the liquid level in each effect may be raised slightly higher than the normal level as the semi-crude stage is reached. This will insure having sufficient liquor to finish a charge of crude without boiling down below the top tube sheet or feeding in fresh lye.

The charge in the 2nd effect is concentrated until the liquor has a temperature of about 160° F. under 27″ of vacuum, 165° F. under 26″ of vacuum, or 170° F. under 25″ of vacuum.

When this concentration is reached, the salt valve is closed and no further salt is removed. With salt drums, the drum is emptied as usual. When using salt extractors, the extractor is steamed and emptied as usual. The charge in the evaporator is then concentrated until it has a temperature of 170° to 200° F. under 27″ of vacuum.

The steam pressure on the calandria need not exceed 15 lbs. per square inch, gauge pressure. If it is desired to use a lower pressure, as with exhaust steam, this may be done, but the time for finishing will be longer.

If the spent lye has been properly treated, this finishing condition will produce a crude glycerine containing in excess of 80% glycerol. Analysis of the crude produced should be made from time to time and the finishing temperature decreased if possible, as there is some loss of glycerine when finishing at 200° F. Good crude glycerine can be obtained with a finishing temperature of 170° F.

Finishing in Single Effects

When the liquor in the 2nd effect has reached the semi-crude stage, the evaporator may be shut down,

the vacuum broken, and the semi-crude liquor dropped into a semi-crude tank. This allows considerable salt to settle out.

The evaporator is then started up again, the liquor from the 1st effect run to the 2nd effect and fresh spent lye fed to the 1st effect.

After sufficient semi-crude has been collected to make several finished charges of crude, the evaporator is emptied and the valves in the vapor lines set so as to operate as two single effects. The unit is then started up and semi-crude glycerine is fed to each effect. The evaporators are run as two single effects and the semi-crude is finished to crude in each effect. The finishing of the crude is carried out in each effect in the same manner as described above for finishing in the 2nd effect only. The same vacuum, pressure and temperature conditions should be maintained as there given.

This procedure of running to semi-crude in double effect and finishing from semi-crude to crude in two single effects, keeps the equipment operating at its maximum capacity at all times and gives the plant its highest possible capacity.

With lyes from high quality stock and good lye treatment, the glycerol in the finished crude may be run as high as 88% but it is not recommended that the crude be finished above 82% glycerol. With higher concentrations there is some loss of glycerine by distillation with the water vapor.

Separate Finishing Evaporator

A separate single effect finishing evaporator may be used in conjunction with the double effect evaporator. Such an arrangement is shown in Figure 12. In this case the double effect operates continuously and the semi-crude in the 2nd effect may be pumped out, instead of shutting down to drop the charge with consequent loss of time. The semi-crude is finished to crude, as described above, in the single effect finishing evaporator. This results in efficient operation, as all equipment is in constant use under the most favorable operating conditions. Figure 16 shows a single effect finishing evaporator.

Flexibility of Operation with Double Effect

A double effect evaporator unit with the vapor lines and valves so arranged that the unit may be operated as a double effect or that either or both effects may be operated as single effects, gives considerable flexibility to the plant.

The evaporation to half-crude may normally be carried out in double-effect. Finishing from half-crude to crude can be carried out in single-effect using either one or both effects. When it becomes necessary to re-tube one effect or make any repairs, the other effect may be operated as a single effect, thus avoiding the accumulation and fermentation of lyes. Should the production of lyes at times exceed the capacity of the unit when operated as a double effect, the capacity of the evaporators can be increased by operating as two single effects.

If a double effect evaporator is installed to use live steam and, subsequently exhaust steam becomes available, the evaporators can be run as two single effects on exhaust steam.

Correct Operation of Plant

With the equipment in good condition, suitable steam pressure, adequate supply of condensing water and correct vacuum on each effect, the plant will be operating at its rated capacity and normal efficiency.

If the vacuum drops the trouble may be with the pump or ejector, whichever is used, and this equipment should be examined and cleaned or adjusted as required. It is well to have a valve in the line to the vacuum pump or ejector so that the vacuum equipment can be tested against a blanked suction. If the fault is not with the pump, it can at once be looked for elsewhere.

An inadequate supply of cooling water is a frequent source of trouble. The cooling water supply line must be so arranged that air can not be drawn in through the water line in case of water shortage

The steam trap on the 1st effect and the condensate pump on the 2nd effect must be watched to see that they are functioning properly. All condensate must be removed but no steam allowed to escape. If condensate shows in the gauge glasses on the calandrias, some of the heating surface is covered with water and the equipment is not operating at its full capacity.

The steam pressure for the ejector must be uniform and not below the pressure required to operate the ejector, else the vacuum will drop rapidly.

The valve in the vent line connected from the top of the calandria of the 2nd effect to the vapor belt of the 2nd effect must be kept open sufficiently to remove any non-condensable gases, else the top of the tubes will be fouled by these inert gases and the full heating surface will not be in use. At the same time, this valve must not be open so far as to draw any considerable quantity of steam up into the 2nd effect vapor belt.

The vents at the top of the calandria of the 1st effect must also be opened slightly to remove any non-condensables introduced with the steam. This is especially important when the exhaust steam from the ejector is returned to the calandria of the 1st effect, since this exhaust steam contains all of the non-condensable gases removed from the 2nd effect by the ejector.

Priming of the liquor in the evaporators is not a fault of the evaporators but of the liquor and the condition of the lyes should be investigated. If the lyes are too alkaline they may foam. Lyes which have fermented and contain gases in solution also foam. To evaporate such lyes, the liquor level should be held low and the steam supply curtailed to evaporate at a lower rate. The vacuum should be held uniform and the feed regular.

A rapid rise of the vacuum must be carefully avoided, especially following a sudden drop. The sudden expansion of steam in hot liquor due to a sudden rise of vacuum may lift the entire charge out of the evaporator.

Regular examinations should be made for leaking tubes in the calandrias, especially with old tubes. If the rate of evaporation slows down, it may be due to steam leaking into the charge. The condensate can be tested for salt and glycerine. The evaporators can be evacuated and all valves closed. If vacuum shows on the gauges attached to the lines connected to the calandrias a leak is indicated. A water pressure test can be made on the calandrias and leaks will readily show.

If the tubes become salted, the evaporator should be boiled out with water before any tubes are filled solid. In the latter case the salt must be bored out which is tedious and may be harmful to the tubes. Salting out is usually due to improperly treated lyes and the cause should be remedied at once. Carrying the liquor level in the evaporators too low may also cause salting out on the tubes.