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PINE GUM DISTILLATION

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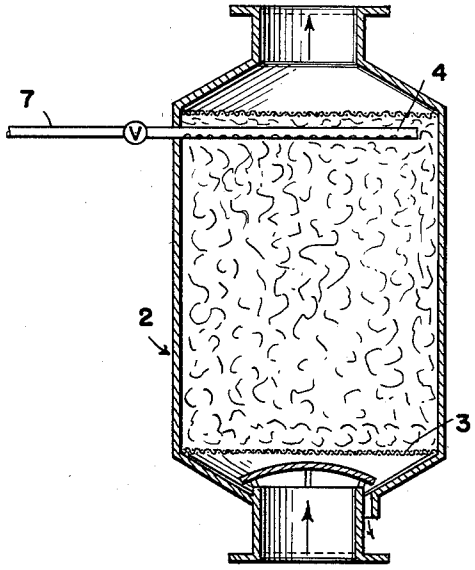


Fig. 1.

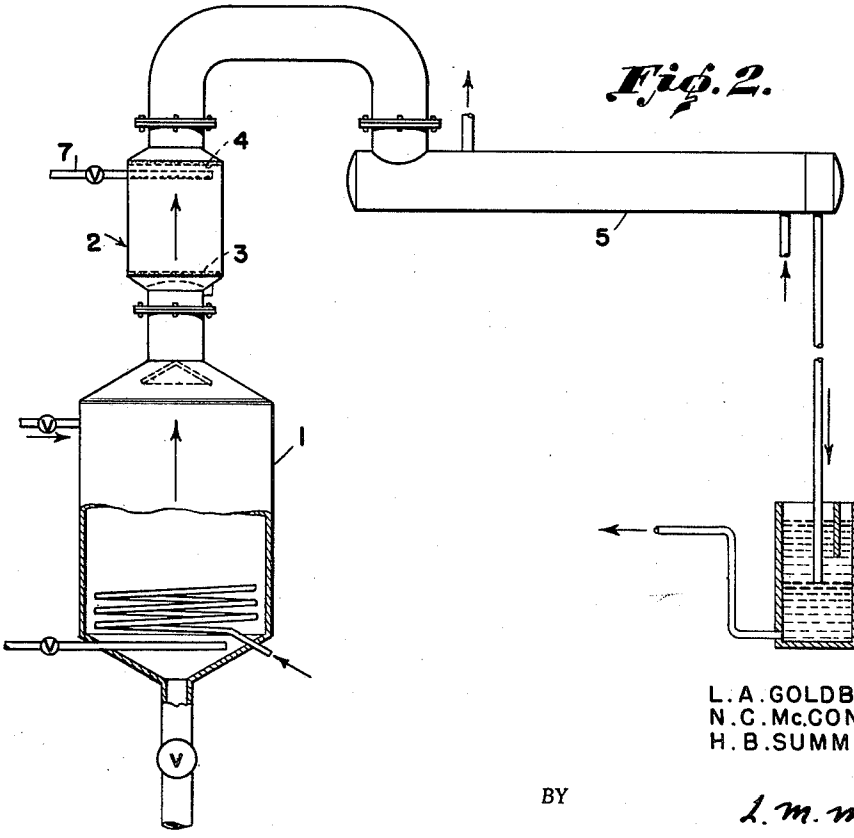


Fig. 2.

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PINE GUM DISTILLATION

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This invention relates to the distillation of pine gum, and more particularly to the production of gum spirits of turpentine having improved quality. It has among its objects a purification treatment of the vapor stream exiting from either batch or continuous steam stills to produce a turpentine of improved color and exceedingly low acidity.

When pine gum is distilled in conventional equipment used in the industry the turpentine obtained (gum spirits of turpentine) contains acidic impurities. The acids present are of two types, water-soluble acids, which are chiefly acetic and formic acids, and water-insoluble acids, which are chiefly resin acids of the formula $C_{20}H_{30}O_2$. This acidity is one of the principal causes of turpentine deterioration and discoloration when placed in metal containers.

The amount of acid present in the turpentine varies considerably depending chiefly upon the care taken during distillation of the pine gum. Nevertheless, even when the greatest care is taken the turpentine will typically have an acid number of about 0.5 or greater with present methods. The acidity of certain samples of commercial gum turpentine has been found to exceed the acid number of 3.1. Typical acid numbers of turpentine obtained with the best commercial practice available prior to our invention are about 0.6 to 0.7, of which the water-soluble acid number is usually about 0.1 and the water-insoluble acid number is about 0.5 to 0.6.

The acid concentration in the turpentine is not constant during the distillation of a batch of pine gum, and increases markedly as the distillation progresses. The turpentine obtained at or near the beginning of the distillation is relatively low in acidity. Employing the best previously known methods the initial acid number may be as low as 0.1, whereas the last portion of turpentine obtained has an acid number reaching as high as 5.0 or even more. In a typical case in which special care was taken and all previously known precautions employed to reduce the acidity, it was found that the turpentine obtained at the turning out point (i. e., when the ratio of turpentine to water in the distillate was less than 1 to 10) had an acid number of 4.5.

The presence of appreciable acid in the turpentine is inherently undesirable for most of the purposes for which turpentine is used. The acid attacks metal containers customarily used for storing and shipping, thus causing high corrosion losses, and moreover results in the introduction of impurities in the turpentine. The acidity itself as well as the introduced impurities affects unfavorably the value of turpentine for solvent, paint, medicinal, and other purposes.

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The water-insoluble acidity is believed to be due to the presence of small amounts of resin acids in the turpentine, carried over unavoidably in the vapor stream entering the condenser. In order to reduce the amount of entrained resin acids or rosin in the vapor stream exiting from the stills, it is customary to use centrifugal separators. These devices are effective to remove quantities of rosin carried in the stream as a spray, but as previously mentioned, the best turpentine obtainable with these or other similar devices possesses an acid number of from 0.6 to 1.0 or greater. These devices remove little or none of the volatile water-soluble acids from the vapor stream, yet it is well recognized that they are incapable of reducing the water-insoluble acid number below 0.5 or 0.6 in the final batch or in product processes.

According to our invention we have discovered that the acidity of turpentine can be reduced to the order of 0.1 by employing a section packed with glass fibers in the vapor line of the still. Such a section has a peculiar effect upon the turpentine recovered in the condenser. It acts as a scrubber, yet its total effect is greater than this. For example, the water-soluble acidity as well as the water-insoluble acidity is markedly decreased. This fact is particularly surprising since the water-soluble acids, chiefly acetic acid, and to a lesser extent formic acid, have a lower boiling point than does turpentine itself. One would naturally expect that our packed section would not affect the quantity of water-soluble acids present in the turpentine. We have discovered, however, that our section is capable of decreasing the water-soluble acids by 50 percent or even more.

In following our process we may employ a turpentine still of any of the usual steam distillation types, either batch or continuous, and operating at atmospheric pressure or at pressures above and below atmospheric. We may employ the usual conditions of distillation rates and temperatures, amounts of open steam, and the usual methods of charging and feeding the gum.

Our packed section is preferably placed in the vapor line of the still at a point near the outlet of the distillation chamber or still. The size of the packed section varies with the size and capacity of the still; its specific dimensions depend chiefly on the vapor throughput. In general, the cross-sectional area, measured perpendicular to

the direction of vapor flow, is preferably such that the vapor velocity should be less than 5 feet/second thus, to promote contact with the glass packing. The depth of the packed section should be adequate to remove substantially all entrained material, for we have found that when this result is accomplished, the attending desirable effects of the packing on the vapor stream are also accomplished. It must be kept in mind that our packed section functions not merely as a scrubber since its effect upon the vapor stream is more than merely mechanical removal of spray or other non-vaporous material.

In operation, the vapor passing through the packed section is relieved of spray particles and other non-vaporous material. In addition, a very small amount of condensate usually occurs as a result of thermal losses to the atmosphere at the extreme outside portion of the section. This condensate will then drain back into the still, but the amount is very small and does not affect the successful operation of the distillation.

The temperature maintained within the section may vary within wide limits. For practical purposes no additional temperature control is necessary further than the maintenance of temperature inherently resulting from the heated vapor stream passing through it. The temperature is always below the normal boiling point of the rosin, and as a practical upper limit we mention 350° F. Moreover, the temperature is maintained above the normal melting point of rosin and, with the exception of the extreme outer portions previously mentioned where some insignificant condensation may occur, the temperature is maintained so that the turpentine and water in admixture are in the vapor phase.

Particular advantages characterizing the present invention are a turpentine of vastly improved quality. Furthermore, the packed section may be employed without special attention over long periods of time. In addition, our packed section eliminates special devices such as centrifugal separators designed to operate with particles in the vapor stream.

The apparatus employed in carrying out our invention is described by the accompanying drawing.

Referring to the figure, 1 refers to a conventional pine gum still containing steam heating coils and provided with an inlet for introducing steam or water. The packed section is illustrated by 2. It contains glass wool or fibers loosely packed and held in place by retainer members 3 and 4, which may be screens or perforated plates. Vapors exiting from the still passes through the packed section into condenser 5 of conventional design. The vapors are condensed by the action of cooling water flowing through the condenser. The turpentine is collected at 6 where the turpentine and condensed steam separate into two immiscible layers. An opening 7 is provided to permit washing the glass packing with steam, turpentine, or any suitable solvent for removal of entrained materials.

The following specific examples illustrate the invention:

Example 1.—A packed section containing 55 pounds of Fiberglas insulating wool Type TW–F, packed to a height of 3 feet in a tube measuring 22 inches outside diameter was inserted in the vapor line of a small commercial still. A charge of 2500 pounds of pine gum was distilled over a period of 50 minutes. The turpentine was found to have a total acid number of 0.09, of which the water-soluble acids contributed 0.02 and the water-insoluble acids 0.07.

The same weight charge of pine gum from the same source was distilled in the same still at the same rate, but without the use of the packed section. The acid number of the turpentine from a number of distillations varied from 0.6 to 0.75. The water-soluble acid number of the turpentine averaged about 0.06.

Example 2.—A packed section such as illustrated in the accompanying drawing was installed in a commercial still. This section measured 3 feet in diameter and 4 feet in height. It was packed with 179 pounds of Fiberglas insulating wool, Type TW–F. The turpentine obtained from the still under normal batch operation had an acid number of only 0.09, of which the water-soluble acidity was 0.01 to 0.02, the remaining being water-insoluble acidity. These values are typical and are representative of a large number of distillations carried out on this particular still.

In contrast to the above results, this same still before installation of our packed section produced turpentine consistently possessing an acid number of 0.75, of which the water-soluble acidity was 0.05 and the water-insoluble acidity was 0.70. Before installation of our packed section this still was provided with a centrifugal separator in the vapor line.

The invention herein described may be manufactured and used by or for the Government of the United States of America for governmental purposes throughout the world, without the payment to us of any royalty thereon.

Having thus described our invention we claim:

1. The method of distilling pine gum comprising vaporizing the turpentine in the pine gum by means of heat and steam thereby producing a stream comprising turpentine and water vapors, passing this vapor stream through a zone packed with glass fibers, condensing the vapor stream, and recovering turpentine from the condensate.

2. In the process of steam distilling pine gum in which a condensate comprising water and turpine is obtained and a residue comprising rosin is obtained, the steps which comprise passing the vapor stream exiting from the still and comprising water and turpentine vapors through a zone packed with glass fibers sufficient to remove substantially all non-vaporous particles from said stream thereby purifying the turpentine by reducing its water-insoluble and water-soluble acidity.

3. The method comprising vaporizing the turpentine in pine gum by maens of heat and steam, passing the stream comprising turpentine vapor, water vapor and rosin particles through a zone presenting multiple passages and scrubbing surfaces of glass fibers, the depth of the zone being at least sufficient to remove substantially all entrained material, the temperature in the zone being below the normal boiling point of the rosin and above the melting point of the rosin, the turpentine and water in said zone being in the vapor phase, condensing the vapor stream, and separating turpentine from the water condensate.

4. The method of distilling pine gum comprising vaporizing the turpentine in the pine gum by means of heat and steam, thereby producing a stream comprising turpentine and water vapors, passing this vapor stream through a zone presenting multiple passages and scrubbing surfaces of glass, fibers, the vapor velocity being leess than five feet per second therein, to promote extended contact with the glass surfaces, the temperature

within the glass packed zone being below 350° F. and above the normal melting point of rosin, the water and turpentine being in vapor phase in said zone, and thereafter condensing the water vapor and recovering turpentine, whereby the turpentine is purified by reducing its water-insoluble and water-soluble acidity.

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