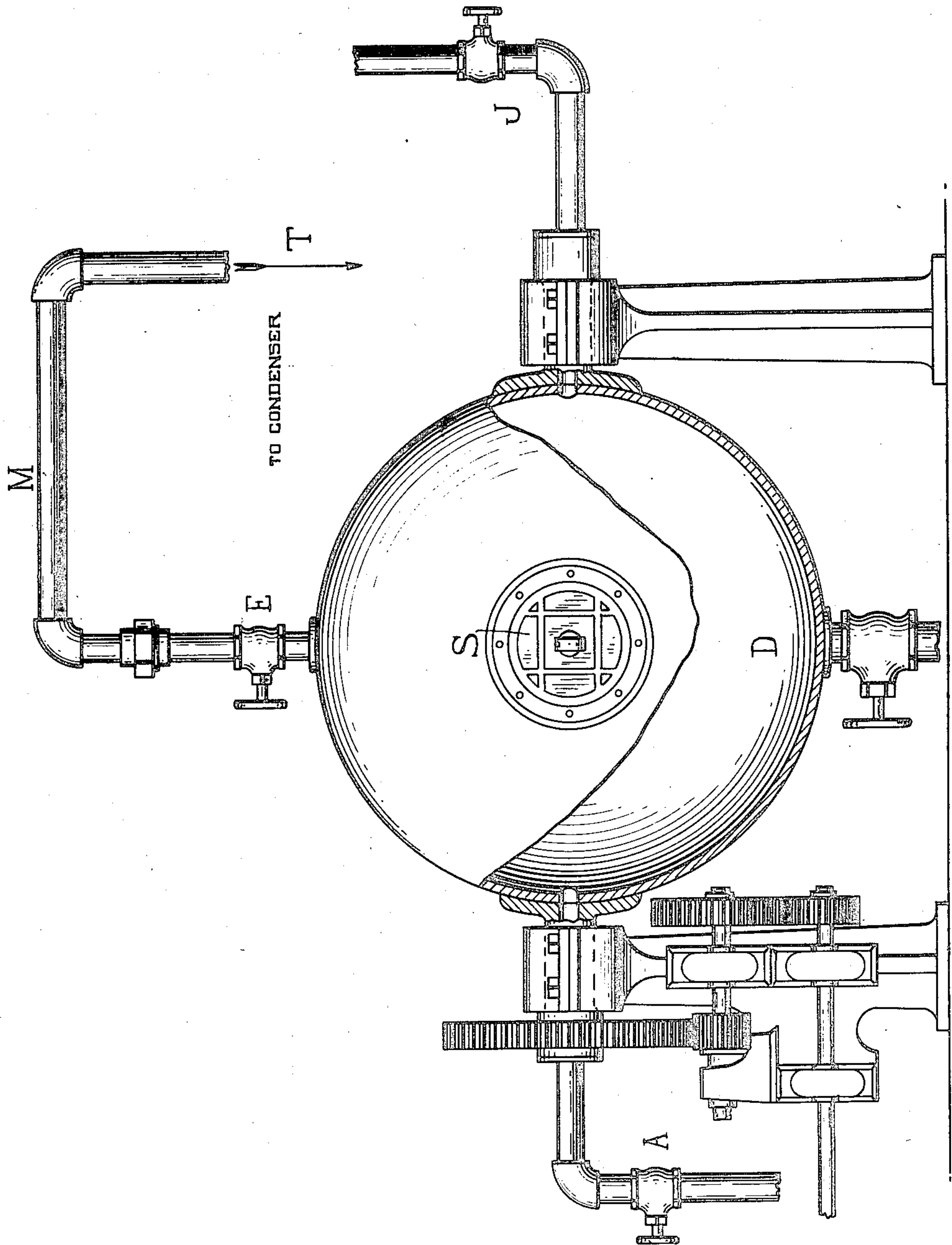


W. J. HOUGH.
 PROCESS OF PRODUCING TERPENES FROM WOOD.
 APPLICATION FILED MAY 14, 1908.

903,471.

Patented Nov. 10, 1908.



Witnesses

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UNITED STATES PATENT OFFICE.

WILLIAM J. HOUGH, OF TOLEDO, OHIO.

PROCESS OF PRODUCING TERPENES FROM WOOD.

No. 903,471.

Specification of Letters Patent.

Patented Nov. 10, 1908.

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To all whom it may concern:

Be it known that I, WILLIAM J. HOUGH, a citizen of the United States, residing at Toledo, in the county of Lucas and State of Ohio, have invented certain new and useful Improvements in Processes of Producing Terpenes from Wood, of which the following is a specification.

My invention relates to a process for the extraction of terpenes from resinous wood, and has as its object the production of said terpenes by a method much more expeditious and economical than any process heretofore.

My invention will now be fully set forth and particularly pointed out in the claims, reference being had to the accompanying drawings forming part hereof, and illustrating a suitable apparatus for carrying out the process.

The resinous wood, such as stumps, roots, slabs, mill waste, fallen and standing timber, is first reduced to small pieces by chipping, shaving, or any of the usual methods of thus preparing wood.

The invention involves a process whereby the wood is treated with a solution of sodium sulfid to neutralize the resinous and other organic acids, and to open the wood cells by dissolving the intercellular adhesive matter. The terpenes are thus enabled to freely escape when the wood is subjected to the action of steam. Reducing the wood to chips not only allows the sodium sulfid to penetrate the wood cells, but enables the terpenes to readily volatilize from the wood, as will appear from the explanations following.

The terpenes exist in the resinous wood together with resinous acids as an oleo-resin, from which the terpenes are volatilized at temperatures ranging from 155 to 185 degrees C. However, they have the property of distilling with steam, as has also a heavier oil, native to the wood, boiling between 185 and 250 degrees C., and known to commerce as "pine oil." While the terpenes distil with steam of a temperature of about 100 degrees C., the pine oil requires a somewhat greater degree of heat. It is impossible for steam to completely penetrate the pores of the wood when the same is in large pieces. The wood must be reduced to chips in which condition each chip may receive a thorough steaming and the terpenes and pine oil distilled from the same.

After the reducing of the resinous wood to chips, for reasons aforesaid, they are

placed in digester or retort "D," through door "S." Valve "E" is provided at the top of the digester, said valve being connected with pipe "M," which leads to condenser "T." Sodium sulfid liquor is now allowed to enter the digester by opening valve "J." The amount of sodium sulfid is largely determined by the resinous character of the wood, that is, the solution should contain just enough sodium sulfid to neutralize the resinous and other organic acids. For the treatment of ordinary "light wood" and stumps of the *Pinus australis* (long-leaf pine) and *Pinus resinosa ait* (Norway pine) about 150 pounds of sodium sulfid for every ton of wood treated, is sufficient. Less of the sodium sulfid is required for most other species of pines, firs, and other coniferous woods. The sodium sulfid should now be made into about a one per cent. solution. It will be noted that the solution is not of sufficient strength to pulp the wood, as the sulfid salt will be exhausted in the neutralization of the wood acids. This liquor neutralizes the resinous acids contained in the oleo-resin, such as abietic, pinic, pimaric, sylvic, and colopholic acids, the reaction being as follows:



The dissolving of these resinous acids and other gelatinous gums of an acid character, forming the intercellular adhesive matter, opens the wood cells and permits the free escape of the terpenes when steam is now applied by opening valve "A."

The terpenes distil with the steam for reasons aforesaid, and escaping through pipe "M" are condensed together with the steam in condenser "T" and then collected.

It is well to carry a digester pressure of about 40 pounds, as this will insure rapid penetration of the wood cells by the sodium sulfid liquor, and hence hasten the complete extraction of the terpenes from the wood.

A boiler pressure of fifty pounds is carried, as steam of this pressure is of sufficient temperature to extract all the pine oil. A boiler pressure much greater than this should be avoided, as the steam will have a temperature sufficient to cause the terpenes to change into inferior bodies. The terpenes are all isomerids of the formula $C_{10}H_{16}$, and change from one class into another with slight provocation. Turpentine is composed mostly of pinene, boiling at 156° C.

When subjected to a much greater heat these terpenes change into dipentene, also a terpene, but having a boiling point of 178° C. Consequently it is of importance to avoid
5 steam of high pressure and necessarily of high temperature.

The steaming of the chips is continued until the extraction of the terpenes is completed. This requires about two hours
10 time. The liquor is now drained off and recovered as follows, when it may be used again on subsequent charges of wood.

Advantage is taken of the fact that sulfate of soda is reduced to sulfid of soda in
15 the process of igniting sulfate of soda with the evaporated liquor containing the organic matter dissolved from the wood. Consequently sodium sulfate is added to the "spent" liquors to provide for the loss of
20 soda occasioned in extracting the terpenes. The liquor is now evaporated to the desired density, the evaporated lyes mixed with limestone and coal-dust, and after drying, the mass is ignited, whereby the sulfate of
25 soda is reduced to sodium sulfid.

It will be noticed that sodium sulfate supplies all loss of soda, and being an exceedingly cheap commodity, makes a very economic method of extracting terpenes by a
30 chemical method. The oil resulting from this treatment is entirely of different character from that produced by treating resinous wood with a sulfite solution, such as is used in the manufacture of wood pulp by
35 the sulfite process.

In my process the resulting crude oil consists of about eighty-five per cent. terpenes, boiling between 155 C. and 185° C., the remainder being an oil known to commerce as
40 pine oil, the chemistry of which is little known. This oil boils between 185 and 250° C., and can, of course, be readily separated

from the terpenes by fractional distillation. However, the oil resulting from the treating of resinous wood with the ordinary pulping
45 sulfite liquors, that is, the bisulfite of lime and magnesia, does not consist of terpenes, but of cymene, (see *Paper Trade Journal* of February 6, 1908, page 223). This is due
50 to the oxidation of the terpenes by the bisulfite liquors. Cymene, $C_{10}H_{14}$, is a member of the benzene series of hydrocarbons, and is in no sense a terpene. It boils at 175 to 176° C.

Cymene does not absorb oxygen on exposure to air, as do the terpenes, which gradually become viscid and ultimately resinous. It is this property which gives the terpenes their peculiar value—not shared by cymene—to the painter and varnish maker,
60 since the terpenes act as an oxygen carrier to the drying oil, and the resin formed acts as a binding material for the pigments with which it is mixed.

What I claim is:

1. The method described, which consists in treating resinous wood with sodium sulfid to neutralize the resinous acids of the wood and dissolve the non-cellulose constituents thereof, and then volatilizing the ter-
70 penes with steam.

2. The method described, which consists in treating resinous wood with a weak solution of sodium sulfid to neutralize the resinous acids of the wood and dissolve the non-
75 cellulose constituents thereof, and then volatilizing the terpenes with steam.

In testimony whereof I affix my signature, in presence of two witnesses.

WILLIAM J. HOUGH.

Witnesses:

R. PEALE HERRICK,
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