



Designation: D 889 – 99

Standard Test Method for Volatile Oil in Rosin¹

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1. Scope

1.1 This test method covers the determination of the volatile oil content of rosin or similar material. The oil may consist of naturally occurring terpene oil, such as heavy fractions of turpentine, resulting from incomplete distillation in the processing of the rosin, or of foreign nonterpene oil resulting from incomplete removal of mineral or coal-tar solvent used to extract the rosin from wood or still wastes. In certain cases the volatile oil could consist of decarboxylated rosin formed during the processing of the rosin.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 233 Test Methods of Sampling and Testing Turpentine²

E 1 Specification for ASTM Thermometers³

3. Significance and Use

3.1 Rosin and similar materials such as rosin derivatives often contain volatile material derived from the raw material used or formed as a result of the processing of the rosin. This volatile material can have a significant effect on the physical and chemical properties of the rosin and so a standard method for its determination is required.

4. Apparatus

4.1 *Flask*, 500-mL round-bottom having a thermometer well and a 24/40 standard-taper ground joint for connection with the trap (see Fig. 1).

4.2 *Trap* (Fig. 2), designed so as to overcome the tendency for droplets of oil to remain below the surface of the water, due to the greater viscosity of the volatile oils recovered from rosin, and thus be returned to the flask (Note 1). The trap shall be

fitted with 24/40 standard-taper ground joints to provide tight connections with the flask and condenser, in order to avoid vapor loss.

NOTE 1—This trap is a modification of the original Clevenger trap used to recover oils lighter than water. In that trap the oil and water condensate drop directly into the graduated part of the trap. Due to the viscous character, density, and surface tension of the oils recovered from rosin, cylindrical columns of oil were formed below the surface of the water in the graduated section of the trap, which were not penetrated by the water condensed immediately thereafter. This resulted in alternate columns of oil and water in the graduated section. These were returned to the distilling flask in the same order as they occurred, through the side arm of the trap. As this condition continued indefinitely, it was impossible to completely remove all the oil from the rosin. By raising the opening of the side arm of the trap to the position shown, to bring the surface of the liquid into the wide part of the trap above the narrow graduated section, the oil is collected in a thinner film that can be penetrated readily by the droplets of water falling from the end of the condenser, and only the water is thus collected in the narrow graduated section. At the end of the test, the oil is slowly brought down into the graduated section and its volume read. The system or apparatus loss amounts to not more than 0.1 mL of oil.

4.3 *Condenser*, straight-tube, 300-mm, water-jacketed reflux type, with a 24/40 standard-taper ground joint for connection with the trap.

4.4 *Heat Source*—An oil bath containing high-temperature-resistant oil, or an electric heater of the mantle type in which the heating elements are encased in a glass cloth mantle of such shape as to partially or completely surround the flask being heated.

4.5 *Thermometer*, having a range from 30 to 200 °C, ASTM 16C or equivalent (see Specification E 1).

4.6 *Glass Beads*.

5. Procedure

5.1 Place 50 g of the crushed sample in the flask, add 125 mL of a glycerin-water solution (4 + 1), add a few glass beads, insert an ebullition tube, and connect the flask with the trap. Fill the trap (Fig. 2) with water through the top opening until the water level is even with the bottom of the seal of the small return tube to the side arm. Insert the thermometer, and connect the condenser.

5.2 Regulate the applied heat until the liquid in the flask is brought to a boil and distillation continues at a constant temperature, which will be about 125°C at the start. At this stage, open the stopcock slightly and withdraw the water from

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² *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 14.03.