In both these experiments, the separation obtained was dependent on sedimentation rates, *i.e.*, centrifugation was not continued long enough for the virus to reach a density equilibrium position.

Density gradient centrifugation is being applied in this Laboratory to the purification of plant viruses. With potato yellow-dwarf virus, whose zones are visible by scattered light, it has served as a rough but very convenient criterion of purity at the same time that it is being used as a separation procedure.

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Isolation of *d*-3-Octanol from American Oil of Spearmint

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In the distillation of American spearmint oil (*Menthis spicati*), it was observed that certain portions of the monoterpene fractions possessed a heavy, fruity odor which could not be accounted for by the reported constituents of these fractions, *i.e.*, *l*-α-pinene, *l*-α-phellandrene, and, mainly, *l*-limonene.[1] The ceric nitrate alcohol test on these fractions was positive. Although the new compound could not be obtained in any degree of purity by fractional distillation, its allophanic ester (m.p. 182°) was readily precipitated when cyanuric acid gas was passed into the terpene fraction. Analytical data on the allophanate indicated that the new compound was an octanol. Saponification of the allophanate gave a dextrorotatory alcohol (b.p. 88–92° (33 mm.)) which was negative in the unsaturation test with bromine in carbon tetrachloride and in the iodoform test. Analysis of the alcohol and its α-naphthylurethan (m.p. 79–80°) supported the view that it was an octanol.

d-3-Octanol has been found in Japanese peppermint oil,[2] in Brazilian peppermint oil[3] and, both free and as the acetate, in European pennyroyal oil.[4] The α-naphthylurethan was reported[2,3] as melting at 81–82°. *l*-3-Octanol occurs in French lavender oil[5]; it forms an allophanate melting at 167° and an α-naphthylurethan melting at 81°. Racemic 3-octanol has been synthesized[2,6,7] (α-naphthylurethan,[7] m.p. 54°) and resolved.[6] The *d*- and *l*-forms were prepared also from *l*- and *d*-amylvinylcarbinol, respectively[8]; the α-naphthylurethan of the synthetic *l*-3-octanol was found to melt at 79–80°. In the work reported here *dl*-3-octanol was prepared by the reaction of *n*-amylmagnesium bromide and propionaldehyde. This compound was practically identical with the unknown alcohol in odor and physical properties.

The semicarbazone of 3-octanone has been reported[6] to melt at 112° (slow heating) and also[4] at 117–117.5°. Oxidation of the alcohol from spearmint oil with chromic acid gave an octanone (b.p. 160–165°) which formed a semicarbazone melting at 112°. The semicarbazone derived in the same manner from synthetic *dl*-3-octanol melted at 114°. A mixed melting point determination showed that the two compounds were identical.

On the basis of the weight of the allophanate obtained, *d*-3-octanol makes up about 1.1% of American oil of spearmint.

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Experimental[9]

Isolation of *d*-3-Octanol.—Oil of spearmint (A. M. Todd Company, Kalamazoo, Michigan) was distilled through a short Vigreux column under reduced pressure until the distillate amounted to about 25% of the original oil. This fraction was cooled in an ice-bath and treated with cyanic acid gas generated by heating cyanuric acid at 325–330° in a slow stream of carbon dioxide. When precipitation was complete, the allophanate was collected on a filter, washed with a small amount of petroleum ether, pressed out on porous paper, and allowed to dry. After recrystallization from ethanol, it melted at 182°. From 1460 g. of spearmint oil there was obtained 27 g. of crude allophanate.

Anal. Calcd. for $C_{10}H_{20}O_3N_2$: C, 55.53; H, 9.32; N, 12.96. Found: C, 55.74; H, 9.18; N, 13.21.

The allophanate was hydrolyzed by boiling under reflux for ten hours with aqueous sodium hydroxide solution (10%). The reaction mixture was extracted with ether and the extract was washed with water and dried over anhydrous sodium sulfate. After removal of the solvent, the alcohol was distilled under reduced pressure; b.p. 88–92° (33 mm.), n^{25}_D 1.4259, $[\alpha]^{25}_D$ +8.23° (abs. alc.).

Anal. Calcd. for $C_8H_{18}O$: C, 73.78; H, 13.93. Found: C, 73.95; H, 13.65.

Treatment of a small portion of the alcohol with α-naphthyl isocyanate in the usual way gave the α-naphthylurethan which, after recrystallization from Skellysolve B, melted at 78°.

Anal. Calcd. for $C_{19}H_{25}O_2N$: C, 76.22; H, 8.42. Found: C, 76.28; H, 8.38.

Oxidation of *d*-3-Octanol.—A portion of the alcohol (2 g.) was dissolved in acetic acid (15 ml.) and treated dropwise in the cold with a solution of chromic anhydride (2 g.) in a small amount of acetic acid. Then the reaction mixture was heated on the steam-bath for one hour, allowed to cool and neutralized with sodium hydroxide solution (10%). The product was removed from the reaction mixture by steam distillation and extracted from the distillate with benzene. After the solvent was removed, the residue was distilled from a small, modified Claisen flask. The material boiled at 160–165° and weighed 1.5 g. Further purification of this ketone was not attempted; instead it was converted to its semicarbazone which, after recrystallization from alcohol-water, melted at 112°.

Anal. Calcd. for $C_9H_{18}ON_3$: C, 58.66; H, 10.39. Found: C, 58.56; H, 10.08.

A sample (7.5 g.) of synthetic *dl*-3-octanol (b.p. 75° (20 mm.), n^{25}_D 1.4256), prepared by the reaction of *n*-amylmagnesium bromide and propionaldehyde, was oxidized with chromic anhydride in acetic acid in a manner identical with the oxidation of the octanol from spearmint oil. Five grams of 3-octanone was obtained; a portion of this ketone was converted to the semicarbazone, m.p. 114°. The melting point of a mixture of this semicarbazone and that derived from the octanol from spearmint oil was 113–114°.

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