

On the Conversion of *l*-Abietic Acid into a *d*-Abietic Acid

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The addition of hydrogen chloride to abietic acid produces dichlorodihydroabietic acid which is crystalline and easily purified.¹ We have found that dichlorodihydroabietic acid may be obtained directly from rosin and in apparently equal purity as when Steele's abietic acid² is used as a starting material. On splitting off of hydrogen chloride from dichlorodihydroabietic acid with sodium ethoxide, we have been able to obtain an abietic acid with the positive sign of rotation. This seemingly new abietic acid was characterized through its di-*n*-amylamine salt. According to Levy,¹ the dichlorodihydroabietic acid produces an abietic acid with the negative sign of rotation when heated at its melting point, and when treated with quinoline.

Experimental

Dichlorodihydroabietic Acid.—This compound was prepared substantially according to the directions given by Levy.¹ Ninety-five grams of "X" grade gum rosin was dissolved in 70 cc. of glacial acetic acid and boiled for four hours. The solution was then cooled to about 0°. The semi-solid mixture was saturated with dry hydrogen chloride, the bottle sealed and the mixture left standing at room temperature for two weeks. It was then filtered and the solid collected; yield 26 g. It was recrystallized from acetone and then from ethyl acetate, m. p. 190.5° dec. (corr.); (α)_D -10° (in ethanol).

Anal. Calcd. for C₂₀H₃₂O₂Cl₂: C, 63.97; H, 8.59. Found: C, 63.76; H, 8.60.

The dichlorodihydroabietic acid prepared from Steele's abietic acid,² m. p. 163.5–165° (corr.); (α)_D -76° (in ethanol) showed the following constants: m. p. 190.5° dec. (corr.); (α)_D -8.1 (in ethanol). Found: C, 63.96; H, 9.16.

***d*-Abietic Acid.**—Ten grams of dichlorodihydroabietic acid (m. p. 190.5° dec. (corr.); (α)_D -10°), was added in portions over a period of five to ten minutes to a boiling solution of sodium ethoxide (17.5 g. of sodium and 175 cc. of absolute ethanol). The mixture was refluxed for one hour. Water was then added and the excess of alcohol was distilled off. As much of the liquid as possible was poured off the remaining solid. The soapy residue was then dissolved in 3 liters of water. The solution was filtered and the filtrate acidified with dilute hydrochloric acid. The white precipitate was filtered off and crystallized from methanol, yield 3.5 g. (40%), m. p. 137–139°; (α)_D +27° (in ethanol), which on successive crystallization yielded an abietic acid melting at 142–143° (corr.); (α)_D +20° (in ethanol). Calcd. for C₂₀H₃₀O₂: C, 79.40; H, 10.05. Found: C, 79.33; H, 10.08; C, 79.57; H, 10.32.

(1) Røy and Simonsen, *Indian Forest Records*, **11**, 211 (1924); Levy, *Ber.*, **64**, 2441 (1931).

(2) Steele, *This Journal*, **44**, 1333 (1922).

The mother liquor from this separation yielded small quantities of an acid melting at 207–212° (corr.), probably identical with the dihydroabietic acid of melting point 217.5–218.5° (corr.), previously described by us.³

A solution of 6 g. of *d*-abietic acid of m. p. 139–143° (corr.) was treated with 4 g. of di-*n*-amylamine (Eastman Kodak Company) in acetone. The solution was boiled for about ten minutes and filtered. On standing 10.6 g. of crude di-*n*-amylamine salt was obtained, m. p. 117.5–119° (corr.). The compound was recrystallized four times, the melting point remaining constant after the third crystallization, being 119–119.5° (corr.), (α)_D +3.3° (in ethanol). The amounts of recovered di-*n*-amylamine salt were too small to be converted into the original acid.

Anal. Calcd. for C₃₀H₅₀O₂N: C, 78.35; H, 11.62. Found: C, 78.61; H, 11.76.

The di-*n*-amylamine salt of *l*-abietic acid shows the following constants: m. p. 141–142°; (α)_D -74.5°.⁴

This investigation is being continued.

(3) Hasselstrom and McPherson, *ibid.*, **61**, 1228 (1939).

(4) Palkin and Harris, *ibid.*, **56**, 1935 (1934).

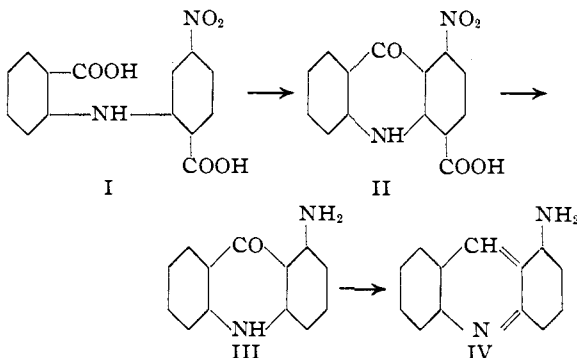
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The Identification of 1-Nitroacridone-4-carboxylic Acid as 1-Aminoacridine

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In a previous paper,¹ it was stated that the nitroacridone-carboxylic acid (m. p. 333° dec.) which was obtained from 5-nitrodiphenylamine-2,2'-dicarboxylic acid (I) by ring closure should have the constitution of 1-nitroacridone-4-carboxylic acid (II) in consideration of the observations that the fluorescence and solubility of the aminoacridone (III), which was converted from the nitroacridonecarboxylic acid in question, exactly coincided with those of 1-aminoacridone but were widely different from those of the 3-isomer.



The present paper indicates the previous interpretation to be altogether correct, but not in harmony with the Lehmstedt conclusion,² on

(1) Matsumura, *This Journal*, **60**, 591 (1938).

(2) Lehmstedt and Schlader, *Ber.*, **70**, 1526 (1937); Lehmstedt, *ibid.*, **71**, 1609 (1938).