identification by mixture melting point method.[3] In about ten other attempts, however, this result could not be accomplished.

Reaction of IV with Morpholine or Diethylamine to Give 1,2-Dibenzoylethane (VIII).—A solution of 2 g. of IV, 10 ml. of morpholine or diethylamine and 10 ml. of ether was allowed to stand at room temperature overnight. The reaction mixture was then diluted with ether, extracted with water and evaporated. Several recrystallizations of the residue from ethanol gave 1.5 g. (85% yield) of material melting at 143–145° which was shown to be 1,2-dibenzoylethane (VIII) by a mixture melting point with an authentic sample.[11]

Reaction of IV with Sodium Hydroxide to Give VIII.—A solution of 2 g. of IV, 70 ml. of ethanol and 75 ml. of 4% sodium hydroxide solution was allowed to stand overnight, after which was added 50 ml. of 0.5% aqueous sodium hydroxide and 10 ml. of ethanol and the mixture was allowed to stand for 6 days. A precipitate formed, was filtered off and triturated with cold ethanol; 0.8 g. (43% yield) of VIII, m.p. 136–140°; recrystallized from ethanol, m.p. 143–145°; identification by the mixture melting point method. From the filtrate, after some evaporation, was obtained 0.7 g. of an intractable oil. The filtrate was diluted with water and acidified with phosphoric acid; some carbon dioxide was evolved and 0.1 g. of an intractable oil precipitated. The filtrate was steam distilled; the distillate was made alkaline and reduced in volume to 10 ml. A test for formic acid, made with S-benzylthiouronium chloride according to the directions of Wild, was negative.[12]

Acknowledgment.—The authors are grateful for a grant from the Research Corporation, New York, N. Y., which helped make this work possible.

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[11] P. S. Bailey and R. E. Lutz, THIS JOURNAL, **70**, 2412 (1948).

[12] F. Wild, "Characterization of Organic Compounds," University Press, Cambridge, England, 1947, p. 148.

AUSTIN, TEXAS

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, WAYNE UNIVERSITY]

Cyclic Dienes. VI. Synthesis of Substituted 1,2-Dimethylenecyclohexanes[1,2]

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By pyrolyzing a diacetate in the final step of the procedure, 4,5-dimethyl-1,2-dimethylenecyclohexane (III) was synthesized in five steps in an over-all yield of 75%. Similarly, 4-methyl-1,2-dimethylenecyclohexane (II) was synthesized in four steps in an over-all yield of 75%. The structures of these two cyclic dienes were proved by analysis, ultraviolet absorption and conversion to known derivatives. The method of synthesis illustrates a procedure for ascending a series by the addition of a cyclohexane ring.

The synthesis of 1,2-dimethylenecyclohexane (I) in high yield by the pyrolysis of a diacetate,[6] was recently reported from this Laboratory. This diene is of interest since it is a cyclic diene that will give multicyclic Diels–Alder adducts and at the same time it is a 2,3-disubstituted butadiene that will undergo free radical polymerization. It was shown that the reaction of two molecules of I with quinone produced a pentacyclic adduct that could be converted to pentacene.[7] It was further noted that this diene should polymerize to yield an all-*cis* diene polymer and should serve as a source of cyclodecane derivatives. In order to show that the pyrolysis of esters is an excellent general method for the synthesis of dienes with rather strained exocyclic double bonds, the synthesis of 4-methyl- and 4,5-dimethyl-1,2-dimethylenecyclohexane (II and III) was undertaken. These dienes would be useful in order to determine the effect of substituents on the properties of an all-*cis* polymer and to produce substituted linear condensed polynuclear hydrocarbons.

Although 1,2-dimethylenecyclohexane (I) was synthesized from diethyl phthalate, the substituted phthalic acids required for the synthesis of II and III were not readily available. However, the substituted tetrahydrophthalic acid derivatives were readily available through the Diels–Alder reaction of a diene plus maleic anhydride. Thus 2,3-dimethylbutadiene (IV) was treated with maleic anhydride to give a 95% yield of the adduct 4,5-dimethyl-Δ^4-tetrahydrophthalic anhydride (V). Reduction of V with lithium aluminum hydride gave a good yield of 4,5-dimethyl-Δ^4-tetrahydrophthalyl alcohol (VI), provided that care was taken never to let the solution become more acidic than *p*H 2 in working up the product. VI was esterified with acetic anhydride in a 98% yield to form the unsaturated diacetate VII, which on catalytic hydrogenation gave a high yield of 4,5-dimethylhexahydrophthalyl diacetate (VIII). This diacetate VIII was dropped through a helix-packed pyrolysis tube at 520° under such conditions that only 74% of the theoretical acetic acid was cracked out. Under these optimum conditions, a 65% conversion to 4,5-dimethyl-1,2-dimethylenecyclohexane (III), 19% yield of the olefin acetate IX, and a 13% recovery of the diacetate VIII were obtained. The yield of diene III, based on unrecovered VIII and IX, was 96%. If more vigorous conditions are used in order to crack out more acetic acid, a lower yield of a less pure diene is obtained. This again emphasizes that the optimum conditions for the pyrolysis of a diester are those that crack out less than 100% of the acetic acid. The over-all yield of the diene III from IV was 75%.

The structure of III was proved by analysis, ultraviolet absorption and conversion to a known derivative. The ultraviolet absorption spectrum, which shows no maximum above 220 mμ but increases sharply and appears to reach a maximum

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[1] Previous paper in this series, THIS JOURNAL, **76**, 1940 (1954).

[2] Presented at the 119th National Meeting of the American Chemical Society, April, 1951, Cleveland, Ohio.

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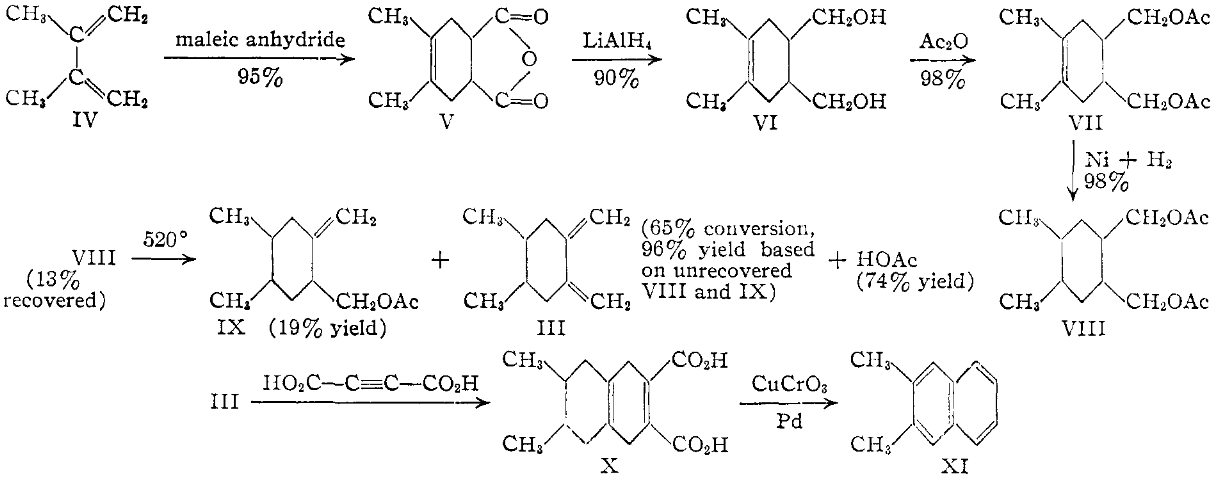
[5] Office of Naval Research Fellow, 1949–1950; Atomic Energy Commission Fellow, 1950–1952.

[6] W. J. Bailey and H. R. Golden, THIS JOURNAL, **75**, 4780 (1953).

[7] W. J. Bailey and M. Madoff, *ibid.*, **75**, 5603 (1953).

Chart I

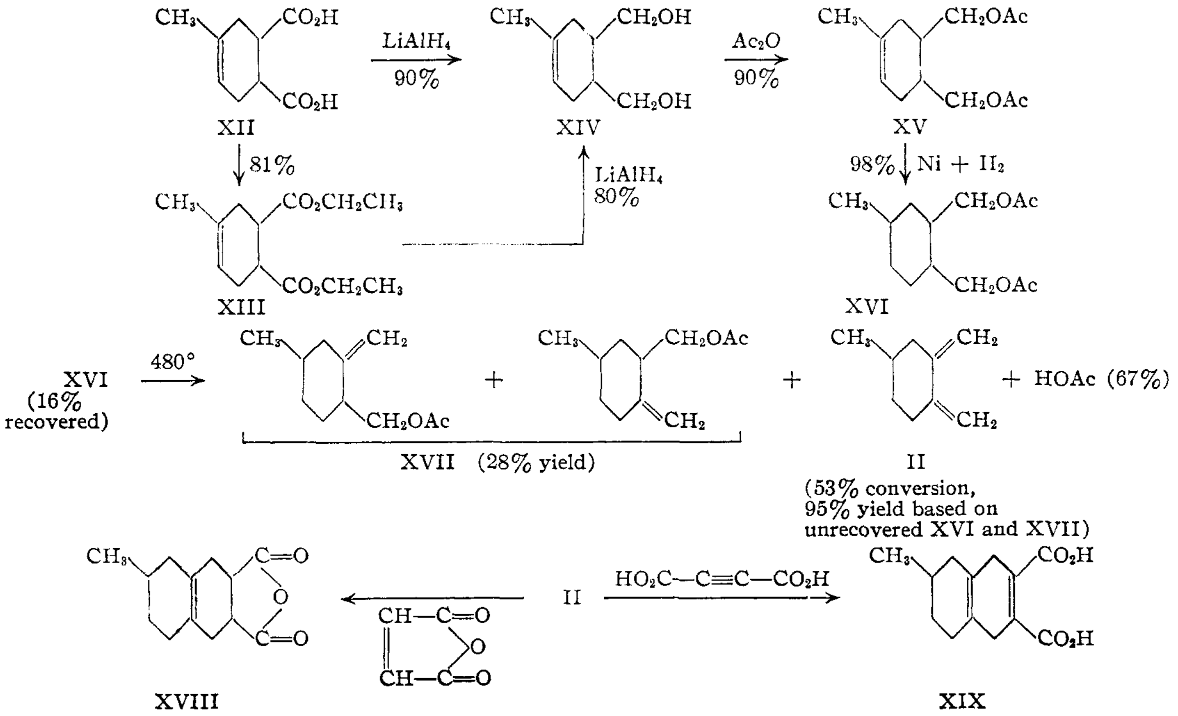


just below 220 mμ, indicates the presence of conjugated double bonds. 1,2-Dimethylenecyclohexane showed a maximum at 218 mμ.[6]

A Diels–Alder reaction between the diene III and acetylenedicarboxylic acid produced the 6,7-dimethyl-$\Delta^{2,4a(8a)}$-hexahydronaphthalene-2,3-dicarboxylic acid (X). Simultaneous dehydrogenation and decarboxylation of X produced the known 2,3-dimethylnaphthalene (XI).

In a similar synthesis, 4-methyl-Δ^4-tetrahydrophthalic acid (XII), which is the Diels–Alder adduct of isoprene and maleic acid, was converted to its diethyl ester XIII. Reduction of XIII with lithium aluminum hydride produced 4-methyl-Δ^4-tetrahydrophthalyl alcohol (XIV) in an 80% yield. Direct reduction of XII to XIV gave satisfactory results only if the reduction time was long. Thus, if the ether solution of XII was heated with lithium aluminum hydride for 3 hours, only a 60% yield of XIV was obtained, but if the reduction was carried out for 2 days using a large volume of ether, the yield of XIV was increased to 90%. Acetic anhydride converted the glycol XIV in good yield to 4-methyl-Δ^4-tetrahydrophthalyl diacetate (XV), which was hydrogenated in a 98% yield to 4-methylhexahydrophthalyl diacetate (XVI). XVI was dropped through a helix-packed pyrolysis tube at 480° under such conditions that only 67% of the theoretical acetic acid was cracked out. Under these optimum conditions, a 53% conversion to 4-methyl-1,2-dimethylenecyclohexane (II), a 28% yield of a mixture of two olefin acetates XVII, and a 16% recovery of the diacetate XVI were obtained. The yield of the diene II, based on unrecovered XVI and XVII, was 95%. The over-all yield of the diene II from the adduct XII was 75%.

Chart II



The structure of II was proved by analysis, ultraviolet absorption and conversion to two solid derivatives. The ultraviolet absorption spectrum, which rapidly increases as the wave length is decreased to 220 mμ and appears to reach a maximum just below 220 mμ, shows the presence of conjugated double bonds. II undergoes a Diels–Alder reaction with maleic anhydride to form 6-methyl-$\Delta^{9(10)}$-octahydronaphthalene-2,3-dicarboxylic anhydride (XVIII). Acetylenedicarboxylic acid, similarly, reacts with II to form 6-methyl-$\Delta^{2,9(10)}$-hexahydronaphthalene-2,3-dicarboxylic acid (XIX).

The synthesis of the two dienes II and III in high yields illustrates quite emphatically that the pyrolysis of esters is an excellent method for the synthesis of reactive and strained dienes. A method of ascending a series by the addition of a cyclohexane ring was developed in the course of these syntheses. In the first case, 2,3-dimethylbutadiene (IV) was converted to III, and in the second case isoprene was converted to II by the addition of a cyclohexane ring. Also, the tetrahydrophthalic acid derivatives V and XII were converted to the naphthalene-2,3-dicarboxylic acid derivatives X and XIX by the addition of a cyclohexane ring. This process could be repeated and any number of cyclohexane rings added in this series.

Experimental

4,5-Dimethyl-*cis*-Δ^4-tetrahydrophthalic Anhydride (V).—To a mixture of 409 g. (4.17 moles) of maleic anhydride and 300 ml. of dry benzene, contained in a 2-liter, three-necked flask, equipped with a condenser, a stirrer and a dropping funnel, was added, dropwise with stirring, 345 g. (4.21 moles) of 2,3-dimethylbutadiene-1,3 (IV). The mixture was allowed to stand for 24 hours and was then filtered. The filtrate was concentrated to obtain an additional amount of solid. The combined precipitates were recrystallized from a mixture of benzene and petroleum ether to give 716 g. (95%) of colorless needles of 4,5-dimethyl-*cis*-Δ^4-tetrahydrophthalic anhydride (V), m.p. 78–79° (Farmer and Warren reported m.p. 78° and no yield).[8]

4,5-Dimethyl-*cis*-Δ^4-tetrahydrophthalyl Alcohol (VI).—In a 5-liter, three-necked flask, equipped with a stirrer, a condenser and a dropping funnel, were placed 30 g. of lithium aluminum hydride and 300 ml. of dry ether. To this solution 100 g. (0.56 mole) of 4,5-dimethyl-*cis*-Δ^4-tetrahydrophthalic anhydride (V) dissolved in 1000 ml. of dry ether was added dropwise and the reaction mixture was stirred under reflux overnight. To the flask cooled in an ice-bath was added dropwise 100 ml. of water followed by 3 liters of 5% hydrochloric acid, added slowly. Extreme care was taken that the *p*H of the solution was always greater than 2. (If the reaction mixture became strongly acid, a lower boiling, saturated monoalcohol was obtained in which presumably one alcohol group of the glycol had added internally across the double bond.) The water and ether layers were separated and the water layer extracted in an exhaustive ether-extraction apparatus. The ether solutions were combined, dried with anhydrous sodium carbonate, and the ether evaporated to produce the crude crystalline glycol. Recrystallization from a mixture of ether and ligroin produced 85.6 g. (90%) of pure 4,5-dimethyl-*cis*-Δ^4-tetrahydrophthalyl alcohol (VI), m.p. 74°.

Anal. Calcd. for $C_{10}H_{18}O_2$: C, 70.55; H, 10.66. Found: C, 70.48; H, 10.68.

4,5-Dimethyl-*cis*-Δ^4-tetrahydrophthalyl Diacetate (VII).—To 3060.0 g. of refluxing acetic anhydride in a 5-liter, three-necked flask, equipped with a dropping funnel, a stirrer and a condenser, was added dropwise 842 g. (4.95 moles) of 4,5-dimethyl-*cis*-Δ^4-tetrahydrophthalyl alcohol dissolved in 500.0 g. of glacial acetic acid. After the mixture was allowed to reflux for 24 hours, the excess acetic acid and acetic anhydride were removed under partial vacuum and the residue was distilled through a 12-inch, helix-packed column to yield 1229 g. (98%) of 4,5-dimethyl-*cis*-Δ^4-tetrahydrophthalyl diacetate (VII), b.p. 150–152° (3 mm.), $n^{25}\mathrm{D}$ 1.4717, d^{25}_{4} 1.0651.

Anal. Calcd. for $C_{14}H_{22}O_4$: C, 66.11; H, 8.72. Found: C, 66.21; H, 8.70.

4,5-Dimethyl-*cis*-hexahydrophthalyl Diacetate (VIII).—In a high pressure hydrogenation bomb was placed 209 g. (0.823 mole) of 4,5-dimethyl-*cis*-Δ^4-tetrahydrophthalyl diacetate (VII) with 20 g. of W-2 Raney nickel catalyst.[9] The bomb was filled with hydrogen at 1100 p.s.i. pressure and the temperature was raised to 185°. A total drop of 839 p.s.i. of hydrogen (101%) was observed. The catalyst was filtered off and the filtrate distilled through a 6-inch, helix-packed column to yield 206 g. (98%) of 4,5-dimethyl-*cis*-hexahydrophthalyl diacetate (VIII), b.p. 137–140° (3 mm.), $n^{25}\mathrm{D}$ 1.4641, d^{25}_{4} 1.0411.

Anal. Calcd. for $C_{14}H_{24}O_4$: C, 65.60; H, 9.44. Found: C, 65.61; H, 9.45.

4,5-Dimethyl-1,2-dimethylenecyclohexane (III).—At the rate of 1.5 g. per minute, 385 g. (1.504 moles) of 4,5-dimethyl-*cis*-hexahydrophthalyl diacetate (VIII) was added dropwise to a vertical Vycor combustion tube, packed with $^1/_{16}$-inch glass helices externally heated to 528°, as described in a preceding publication.[6] The tube was continuously swept out with a slow stream of nitrogen to prevent oxidation and charring. The pyrolysate was collected in a receiver immersed in a Dry Ice–acetone-bath and was extracted several times with water to remove the acetic acid. Titration of the aqueous extracts indicated the liberation of 74% of the theoretical acetic acid in the pyrolysis which gave the best conversion and material balance. After the organic layer was dried over anhydrous sodium carbonate, it was distilled through a 6-inch, helix-packed column to yield 131 g. (65%) of 4,5-dimethyl-1,2-dimethylenecyclohexane (III), b.p. 98–100° (80 mm.), $n^{22}\mathrm{D}$ 1.4688, d^{25}_{4} 0.8293; 60.2 g. (19%) of 4,5-dimethyl-2-methylenehexahydrobenzyl acetate (IX), b.p. 172–173° (80 mm.); and 50.1 g. (13%) of recovered diacetate VIII. The total recovery of material was 97% and the yield of the diene III, based on unrecovered VIII and IX, was 96%.

Anal. Calcd. for $C_{10}H_{16}$: C, 88.24; H, 11.76. Found: C, 88.20; H, 11.88.

6,7-Dimethyl-$\Delta^{2,4a(8a)}$-hexahydronaphthalene-2,3-dicarboxylic Acid (X).—A solution of 5.7 g. (0.05 mole) of acetylenedicarboxylic acid in 50 ml. of benzene was heated to reflux in a 100-ml., three-necked flask, equipped with a stirrer, a condenser and a dropping funnel, and 6.8 g. (0.05 mole) of 4,5-dimethyl-1,2-dimethylenecyclohexane (III) was added dropwise. After heating under reflux for 1 hour, the mixture was allowed to stand overnight at room temperature. Filtration of the reaction mixture produced the crude adduct. Recrystallization of the crude material from a mixture of acetone and petroleum ether (30–60°) produced 11.9 g. (95%) of 6,7-dimethyl-$\Delta^{2,4a(8a)}$-hexahydronaphthalene-2,3-dicarboxylic acid (X), m.p. 147–147.5°.

Anal. Calcd. for $C_{14}H_{18}O_4$: C, 67.22; H, 7.24. Found: C, 67.01; H, 6.99.

2,3-Dimethylnaphthalene (XI).—Simultaneous dehydrogenation and decarboxylation of 0.2 g. of 6,7-dimethyl-$\Delta^{2,4a(8a)}$-hexahydronaphthalene-2,3-dicarboxylic acid (X) were carried out in the presence of 0.1 g. of palladium-on-carbon and 0.05 g. of copper chromite catalyst. At 270°, 93% of the theoretical amount of hydrogen was liberated. Sublimation of the residue produced 2,3-dimethylnaphthalene, m.p. 104–104.5° (reported[10] m.p. 104–104.5°).

Diethyl 4-Methyl-Δ^4-tetrahydrophthalate (XIII).—In a 1-liter, round-bottom flask, fitted with a Dean–Stark distilling receiver and a reflux condenser, were placed 184 g. (1 mole) of 4-methyl-Δ^4-tetrahydrophthalic acid (XII), 350 g. of ethanol, 18 ml. of sulfuric acid and 600 ml. of benzene. After the reaction mixture had been heated under reflux for 24 hours, it was poured into 1 liter of ice-water. The organic layer was extracted with sodium carbonate solution, then with water and was dried over magnesium sulfate. The

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[8] W. F. Farmer and H. A. Warren, *J. Chem. Soc.*, 902 (1929).

[9] A. A. Pavlic and H. Adkins, THIS JOURNAL, **68**, 1471 (1946).

[10] H. S. Barnett and L. L. Sanders, *J. Chem. Soc.*, 434 (1933).

solvent was removed on a steam-bath under reduced pressure and the residue was distilled through a 12-inch, helix-packed column to yield 193 g. (81%) of diethyl 4-methyl-Δ^4-tetrahydrophthalate (XIII), b.p. 125–130° (4 mm.), n^{25}_{D} 1.4601 (reported[11] b.p. 135–140° (5 mm.), n^{20}_{D} 1.468).

4-Methyl-Δ^4-tetrahydrophthalyl Alcohol (XIV). A. From Diethyl 4-Methyl-Δ^4-tetrahydrophthalate (XIII).—In a 3-liter, three-necked flask, equipped with a stirrer, a reflux condenser and a dropping funnel, were placed 25 g. (0.66 mole) of lithium aluminum hydride and 500 ml. of dry ether. After solution of the hydride was effected, 122 g. (0.51 mole) of diethyl 4-methyl-Δ^4-tetrahydrophthalate (XIII), dissolved in 500 ml. of dry ether, was added dropwise. The unreacted hydride was decomposed with water and the aluminum complex was hydrolyzed with just enough 10% hydrochloric acid to dissolve the precipitate. The aqueous layer was extracted in an exhaustive extractor with ether and this ether extract was combined with the original ether layer. After the combined ether extracts had been dried over anhydrous sodium carbonate, the ether was removed by distillation. The residue was distilled through a 12-inch Vigreux column to yield 64 g. (80%) of 4-methyl-Δ^4-tetrahydrophthalyl alcohol (XIV), b.p. 155–156° (6 mm.), n^{25}_{D} 1.4988, d^{25}_{4} 1.0567.

Anal. Calcd. for $C_9H_{16}O_2$: C, 69.19; H, 10.32. Found: C, 69.13; H, 10.03.

B. From 4-Methyl-Δ^4-tetrahydrophthalic Acid (XII).—In a 5-liter, three-necked flask, equipped with a stirrer, a condenser and a dropping funnel, were placed 35 g. (0.92 mole) of lithium aluminum hydride and 500 ml. of dry ether. After solution of the hydride was effected, a solution of 100 g. (0.35 mole) of 4-methyl-Δ^4-tetrahydrophthalic acid in 1600 ml. of dry ether was added dropwise. The mixture was allowed to stir for 48 hours and then was worked up as described above. Distillation of the residue produced 49 g. (90%) of 4-methyl-Δ^4-tetrahydrophthalyl alcohol (XIV).

4-Methyl-Δ^4-tetrahydrophthalyl Diacetate (XV).—In a 1-liter, three-necked flask, equipped with a condenser and a dropping funnel, was placed 280 g. (2.75 moles) of acetic anhydride. To the refluxing anhydride, 140 g. (0.90 mole) of 4-methyl-Δ^4-tetrahydrophthalyl alcohol (XIV), dissolved in an equal volume of acetic acid, was added dropwise at a rate sufficient to maintain the reflux temperature. After the mixture was heated under reflux for 24 hours, the excess acetic anhydride and acetic acid were removed under reduced pressure. The residue was distilled under reduced pressure through a 6-inch, helix-packed column to yield 195 g. (90%) of 4-methyl-Δ^4-tetrahydrophthalyl diacetate (XV), b.p. 143–144° (5 mm.), n^{25}_{D} 1.4684, d^{25}_{4} 1.0639.

Anal. Calcd. for $C_{13}H_{20}O_4$: C, 64.96; H, 8.39. Found: C, 64.99; H, 8.22.

4-Methylhexahydrophthalyl Diacetate (XVI).—A mixture of 232 g. (0.97 mole) of 4-methyl-Δ^4-tetrahydrophthalyl diacetate (XV) and 12 g. of W-2 Raney nickel catalyst[9] was placed in a high pressure hydrogenation bomb and hydrogenated at 150° and 1600 p.s.i. of hydrogen. The cooled hydrogenation mixture was filtered to remove the catalyst and the filtrate was distilled under reduced pressure through a 6-inch, helix-packed column to yield 230 g. (98%) of 4-methylhexahydrophthalyl diacetate (XVI), b.p. 115° (1.5 mm.), n^{25}_{D} 1.4582, d^{25}_{4} 1.0398.

Anal. Calcd. for $C_{13}H_{22}O_4$: C, 64.43; H, 9.15. Found: C, 64.31; H, 9.16.

4-Methyl-1,2-dimethylenecyclohexane (II).—Using the pyrolysis apparatus previously described for the preparation of III, 185 g. (0.76 mole) of 4-methylhexahydrophthalyl diacetate (XVI) was dropped through the vertical Vycor tube packed with $^1/_8$-inch Pyrex helices at a temperature of 480° at the rate of 1 g. per minute. The pyrolysate was dissolved in ether and this ether solution was extracted several times with water. (Titration of the aqueous extracts with standard sodium hydroxide showed that 67% of the theoretical amount of acetic acid had been cracked out.) The ether layer was dried over sodium sulfate and the ether removed under reduced pressure. The residue was then fractionated through a 6-inch, helix-packed column under reduced pressure to yield 49.6 g. (53%) of 4-methyl-1,2-dimethylenecyclohexane (II), b.p. 67° (71 mm.), n^{25}_{D} 1.4585; 38.4 g. (28%) of a mixture of 4- and 5-methyl-2-methylenehexahydrobenzyl acetate (XVII), b.p. 144° (71 mm.), n^{25}_{D} 1.4666; and 30 g. (16% recovery) of 4-methylhexahydrophthalyl diacetate (XVI). The yield of II, based on unrecovered XVI and XVII, was 95%.

Anal. Calcd. for C_9H_{14}: C, 88.45; H, 11.55. Found: C, 88.55; H, 11.41. Calcd. for $C_{11}H_{18}O_2$: C, 72.49; H, 9.96. Found: C, 72.55; H, 10.01.

6-Methyl-$\Delta^{9(10)}$-octahydronaphthalene-2,3-dicarboxylic Anhydride (XVIII).—A mixture of 3.05 g. (0.035 mole) of 4-methyl-1,2-dimethylenecyclohexane (II), 2.54 g. (0.025 mole) of maleic anhydride and 50 ml. of ether in a 100-ml. flask was heated under reflux on a steam-bath for 4 hours. Evaporation of solvent and recrystallization of the solid residue from a mixture of ether and petroleum ether (30–60°) yielded 4.5 g. (81%) of 6-methyl-$\Delta^{9(10)}$-octahydronaphthalene-2,3-dicarboxylic anhydride (XVIII), m.p. 119–120°.

Anal. Calcd. for $C_{13}H_{16}O$: C, 70.90; H, 7.28; sapon. equiv., 110. Found: C, 70.82; H, 7.20; sapon. equiv., 111.

Acidification of the saponification reaction mixture produced 6-methyl-$\Delta^{9(10)}$-naphthalene-2,3-dicarboxylic acid (XIX), m.p. 196–197°.

6-Methyl-$\Delta^{2,9(10)}$-hexahydronaphthalene-2,3-dicarboxylic Acid (XX).—To a solution of 2.85 g. (0.025 mole) of acetylenedicarboxylic acid in 50 ml. of ether was added 3.05 g. (0.025 mole) of 4-methyl-1,2-dimethylenecyclohexane (II). The mixture was heated in a 100-ml. flask for 2 hours on a steam-bath. The solvent was removed by evaporation and the residue was recrystallized from ethyl acetate to give 5.0 g. (85%) of white crystalline 6-methyl-$\Delta^{2,9(10)}$-naphthalene-2,3-dicarboxylic acid (XX), m.p. 111–112°.

Anal. Calcd. for $C_{13}H_{16}O_4$: C, 66.09; H, 6.85. Found: C, 66.03; H, 6.58.

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[11] F. J. Soday, U. S. Patent 2,375,384 (1942).