tion rate as revealed by the decline of the toluene yield. It appears therefore that here the dehydrogenation rate is even more affected by the poisoning action than the cyclization, but the decline still runs parallel to that of the cyclization.

The interpretation given to the results discussed leads to the conclusion that both reactions, dehydrogenation and cyclization, are affected by the poisoning of the catalyst. Where the poisoning action is due to accumulation of polymer, which is probably the reason for the decline of activity in the course of a run, the two reactions are affected to much the same extent. When water and particularly when ethylene is the poison the dehydrogenation rate is repressed even more than that of cyclization, but the decline is still parallel.

These conclusions are contrary to the views of Taylor and Fehrer mentioned previously. It follows that their further deductions concerning the relation of the two reactions and the seat of catalytic action need revision. In their view, because of the very different effect of catalyst poisons on the two reactions, the two reactions take place on different active centers of the catalyst. The present interpretation leads to the opposite conclusion—which also appears the simpler—that, because of the close relationship revealed by the parallel effect of poisons on both reaction rates, it seems likely that they proceed on identical centers of the catalyst surface.

MANCHESTER OIL REFINERY, LTD.
MANCHESTER, ENGLAND
RECEIVED JUNE 30, 1945

## Methylation of Methyl 6-Hydroxydehydroabietate

BY HAROLD H. ZEISS

Methyl 6-hydroxydehydroabietate can be satisfactorily methylated with dimethyl sulfate in an ethereal solution of methylmagnesium chloride.[1] It has been reported that methylation with dimethyl sulfate in alcoholic alkali gives small or vanishing yields. This difficulty was attributed to the steric hindrance exerted by the adjacent isopropyl group, although this explanation appeared surprising in view of the reactivity of the 6-position of the dehydroabietic structure.

It has now been found that the use of dimethyl sulfate in aqueous alcoholic alkali gives a good yield with ease and rapidity.

### Experimental

To a solution of 3 g. of methyl 6-hydroxydehydroabietate (m. p. 158–159°) in 60 ml. of ethanol and 42.5 ml. of 1% aqueous sodium hydroxide 1 g. of dimethyl sulfate is added at room temperature and the turbid mixture refluxed for two and one-half hours. Ten ml. of 12% sodium hydroxide is then added to destroy the excess dimethyl sulfate. After distilling off the alcohol and cooling, the residue is extracted with several portions of ether and the extract washed with water until neutral. Replacement of the ether with ethanol and the addition of water to turbidity gives broad white plates of methyl 6-methoxydehydroabietate in a pure state. The yield in three crops has been 2.1 g. (68%); m. p. 65–66°.

The method was repeated with another 3 g. of methyl 6-hydroxydehydroabietate (m. p. 158–159°). From this run methoxy ester of the same m. p. was obtained again in 68% yield.

RIDBO LABORATORIES, INC.
PATERSON 3, NEW JERSEY
RECEIVED JULY 10, 1945

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[1] (a) Campbell and Todd, THIS JOURNAL, **62**, 1287 (1940); (b) Campbell, U. S. Patent 2,359,826; (c) L. F. Fieser, "Organic Chemistry," D. C. Heath and Co., Boston, Mass., 1944, p. 994.

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# NEW COMPOUNDS

### Some Polysubstituted Benzene Derivatives

**3-Benzoylmesitonitrile.**—A mixture of 10.1 g. of 3-bromomesityl phenyl ketone,[1] 4.5 g. of cuprous cyanide and 6 ml. of pyridine was heated for twelve hours under reflux in a metal bath maintained at 220–225°. The hot reaction mixture was poured into a dilute ammonium hydroxide solution and, after several hours, a mixture of 100 ml. of ether and 70 ml. of benzene was added to the resulting tarry mass. The organic layer, after being washed with dilute ammonium hydroxide and with water, was filtered. Removal of the solvent left the nitrile as an oily residue which crystallized when allowed to stand with methanol. It was recrystallized from methanol; m. p. 107–108°; yield 83%.

*Anal.* Calcd. for $C_{17}H_{15}ON$: C, 81.90; H, 6.07. Found: C, 81.70; H, 6.18.

**1,2-Di-(2,4,6-triisopropylbenzoyl)-ethylene.**—A solution of 76.5 g. of fumaryl chloride in 100 ml. of dry carbon disulfide was added gradually over a period of one and one-half hours to a mixture of 245 ml. of 1,3,5-triisopropylbenzene, 300 ml. of carbon disulfide and 140 g. of anhydrous aluminum chloride. The reaction mixture was stirred continuously during the period of addition and for two hours afterward. It was poured on a mixture of 1 kg. of ice and 100 ml. of concentrated hydrochloric acid. Removal of the solvent left the 1,2-di-(2,4,6-triisopropylbenzoyl)-ethylene as a gummy, red solid. It separated from acetone in yellow needles; m. p. 190.5–192°; yield 47%.

*Anal.* Calcd. for $C_{34}H_{48}O_2$: C, 83.55; H, 9.90. Found: C, 83.40; H, 9.76.

**1,2-Di-(2,4,6-triisopropylbenzoyl)-ethane.**—A suspension of 9.7 g. of 1,2-di-(2,4,6-triisopropylbenzoyl)-ethylene in 150 ml. of ethanol was shaken with 0.05 g. of platinum oxide in an Adams hydrogenation machine. One mole of hydrogen was absorbed. Acetone was added to bring the product into solution and the catalyst was removed by filtration. The filtrate evidently contained the expected enediol, 1,4-di-(2,4,6-triisopropylphenyl)-1,3-butadiene-1,4-diol, for it rapidly turned yellow when exposed to the air. It continued to give a positive indophenol test for about twenty minutes. Dilution of the solution with water precipitated the 1,2-di-(2,4,6-triisopropylbenzoyl)-ethane in the form of white crystals. These gave a negative indophenol test. The diketone crystallized from methanol in colorless hexagonal plates; m. p. 140–141°.

*Anal.* Calcd. for $C_{34}H_{50}O_2$: C, 83.21; H, 10.27. Found: C, 83.37; H, 10.42.

**N-Methylmesitamide.**—A solution of 27.5 g. of mesitoyl chloride in 20 ml. of low-boiling petroleum ether was added dropwise, with stirring, to 100 ml. of a 35% methylamine solution. The reaction mixture was kept ice cold during the addition and was stirred at room temperature for one

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[1] Hyde and Adams, THIS JOURNAL, **50**, 2503 (1928).

hour afterward. The amide was recrystallized from benzene-petroleum ether; m. p. 106-107°; yield 75%.

*Anal.* Calcd. for $C_{11}H_{15}ON$: C, 74.54; H, 8.53. Found: C, 74.72; H, 8.37.

**2,2',4,4',6,6'-Hexaethylhydrobenzoin.**—A 2-g. sample of 2,2',4,4',6,6'-hexaethylbenzil[2] was dissolved in 19 ml. of ethanol and hydrogenated at 135° and 2300 pounds pressure in the presence of 2 g. of copper chromite catalyst. The reduction was complete in five and one-half hours. After removal of the catalyst and solvent the hydrobenzoin was recrystallized from aqueous acetone. It separated in colorless crystals; m. p. 225-226°.

*Anal.* Calcd. for $C_{26}H_{38}O_2$: C, 81.62; H, 10.01. Found: C, 81.55; H, 10.02.

**2,2',6,6'-Tetramethylhydroanisoin.**—One gram of 2,2',6,6'-tetramethylanisil[3] in 19 ml. of ethanol was hydrogenated at 130° and a pressure of 1800 pounds per sq. in. in the presence of 2 g. of copper chromite catalyst. The reduction was complete in five hours. After removal of the catalyst by filtration the filtrate was concentrated to 5 ml. The addition of 0.5 ml. of water caused the hydroanisoin to precipitate. It was recrystallized from a mixture of benzene and petroleum ether; m. p. 199-200°; yield 0.65 g.

*Anal.* Calcd. for $C_{20}H_{26}O_4$: C, 72.70; H, 7.93. Found: C, 72.84; H, 8.03.

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[2] Fuson and Corse, THIS JOURNAL, **60**, 2063 (1938).

[3] Fuson, Corse and Welldon, *ibid.*, **63**, 2645 (1941).

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## 1-(2,4,6-Triisopropylphenyl)-3,3-diphenyl-1-propen-1-ol

Condensation of 2',4',6'-triisopropylchalcone with phenylmagnesium bromide was found to yield 1-(2,4,6-triisopropylphenyl)-3,3-diphenyl-1-propanone, the enol form of which—1-(2,4,6-triisopropylphenyl)-3,3-diphenyl-1-propen-1-ol—was stable in solution.

**2',4',6'-Triisopropylchalcone.**—Fifty grams of benzaldehyde was added, with stirring, to an ice cold mixture of 100 g. of 2,4,6-triisopropylacetophenone, 21 g. of sodium hydroxide and 500 ml. of 95% ethanol. The reaction mixture was stirred during the period of addition and for an additional twenty-seven hours. During the first three hours of this time the temperature of the mixture was kept at 0° and afterward at room temperature. The chalcone crystallized from alcohol in pale yellow needles; m. p. 100-101°; yield 91%.

*Anal.* Calcd. for $C_{24}H_{30}O$: C, 86.17; H, 9.04. Found: C, 86.69; H, 9.32.

**1-(2,4,6-Triisopropylphenyl)-3,3-diphenyl-1-propanone.**—To a solution of phenylmagnesium bromide made from 7.3 g. of magnesium, 47 g. of bromobenzene and 75 ml. of ether was added a solution of 33.4 g. of 2',4',6'-triisopropylchalcone in 150 ml. of ether. The mixture was heated for one hour under reflux and decomposed with cold ammonium chloride solution. The ether solution was divided into two equal parts. Evaporation of the solvent from one of them left the 1-(2,4,6-triisopropylphenyl)-3,3-diphenyl-1-propanone as a residue. It crystallized from ethanol in white needles; m. p. 117-118°; the yield was practically quantitative.

*Anal.* Calcd. for $C_{30}H_{36}O$: C, 87.33; H, 8.80. Found: C, 87.77; H, 8.68.

The ketone gave negative tests with ferric chloride solution, bromine in carbon tetrachloride and potassium permanganate in acetone.

**1-(2,4,6-Triisopropylphenyl)-3,3-diphenyl-1-propen-1-ol Peroxide.**—Half of the ether solution from the foregoing experiment was diluted with about five times its volume of low-boiling petroleum ether and washed twice with ice water. A rapid stream of oxygen was passed through the moist solution. The peroxide separated in white needles which were recrystallized from a mixture of ether and low-boiling petroleum ether. The yield was 11 g. The compound melted, with decomposition, at 156°. In contact with a flame it decomposed with a flash and then burned quietly.

*Anal.* Calcd. for $C_{30}H_{36}O_3$: C, 81.04; H, 8.17; Found: C, 80.73; H, 8.12.

A steady stream of oxygen was bubbled for twelve hours through a solution of 6 g. of the enol peroxide in 200 ml. of acetone. One gram of an acidic product was isolated and purified by sublimation; m. p. 144°. A mixed melting point determination showed the compound to be diphenylacetic acid.[1]

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[1] Dunlap, THIS JOURNAL, **19**, 645 (1897).

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RECEIVED SEPTEMBER 20, 1945

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# COMMUNICATIONS TO THE EDITOR

## OBSERVATIONS ON THE ANGULAR SCATTERING OF LIGHT BY SULFUR SOLS

*Sir:*

Bidhubhusan Ray[1] observed that the light scattered by a sulfur sol produced by mixing dilute solutions of sodium thiosulfate and sulfuric acid exhibited different colors depending on the angle which the scattered light ray made with the incident beam. Ray correctly explained these "axial colors" as due to the asymmetrical distribution of the light scattered by the sulfur particles and to the strong dependence of the shape of the scattering patterns on the wave length of the incident light.

LaMer and Barnes in some unpublished investigations, have shown that purer, more sharply defined colors and hence more uniform particle sizes can be obtained if the reacting solutions are very dilute (about 0.001 *M*). The growth of the liquid sulfur droplets is stopped by the addition of iodine and the sol stabilized.

We have found that although many bands of colors are exhibited, these sols are characterized experimentally most easily by the bands of reddish hue. The angular position and number of these red bands called "orders," are related theoretically to the size of the sulfur particles.

The position of the orders exhibited by such a sol at various times, *i. e.*, stages of growth, are

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[1] B. Ray, *Indian Assoc. Cultiv. Sci.*, **7**, 1 (1921).