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SUBMISSION OF PREPARATIONS

Chemists are invited to submit for publication in *Organic Syntheses* procedures for the preparation of compounds which are of general interest or which illustrate useful synthetic methods. The procedures submitted should represent, as nearly as possible, optimum conditions for the preparations, and should have been checked carefully by the submitter. Full details of all steps in the procedure should be included, and the range of yields should be reported rather than the maximum yield obtainable. The melting point of each solid product should be given, and the boiling-point range and refractive index (at 25°) of each liquid product. The method of preparation or source of the reactants and the criteria for the purity of the products should be stated.

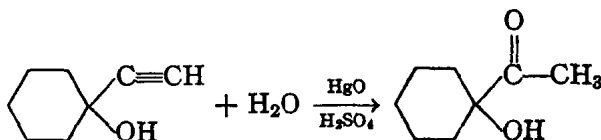
Procedures submitted should be written in the style employed in the latest volume of *Organic Syntheses*. Copies of the current style sheet may be obtained upon request from the Secretary of the Editorial Board. Two copies of procedures which are submitted should be sent to the Secretary. Additions, corrections, and improvements to preparations previously published are welcomed and should be sent to the Secretary.

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1-ACETYLCYCLOHEXANOL

(Ketone, 1-hydroxycyclohexyl methyl)



Submitted by GARDNER W. STACY and RICHARD A. MIKULEC.¹
Checked by JOHN C. SHEERAN, GEORGE A. MORTIMER, and
NORMAN A. NELSON.

1. Procedure

In a 1-l. three-necked round-bottomed flask, equipped with a sealed stirrer, a reflux condenser, a thermometer, and a dropping funnel, is dissolved 5 g. of mercuric oxide (Note 1) in a solution of 8 ml. of concentrated sulfuric acid and 190 ml. of water. The solution is warmed to 60°, and 49.7 g. (0.40 mole) of 1-ethynylcyclohexanol (Note 2) is added dropwise over a period of 1.5 hours. After the addition has been completed, the reaction mixture is stirred at 60° for an additional 10 minutes and allowed to cool. The green organic layer which settles is taken up in 150 ml. of ether, and the aqueous layer is extracted with four 50-ml. portions of ether (Note 3). The combined ethereal extracts are washed with 100 ml. of saturated sodium chloride solution (Note 4) and dried over anhydrous sodium sulfate. The drying agent is removed, the ether is evaporated, and the residue is distilled under reduced pressure through a 15-cm. column packed with glass helices. The 1-acetylcyclohexanol is collected at 92–94°/15 mm. as a colorless liquid, n_D^{25} 1.4670, d_4^{25} 1.0248 (Note 5). The yield is 37–38 g. (65–67%).

2. Notes

1. Mallinckrodt mercuric oxide red (analytical reagent or N.F. 1x grade) was used.

2. 1-Ethynylcyclohexanol is available from Air Reduction Chemical Company, 60 East 42 Street, New York 17, New York, or from Farchan Research Laboratories, Cleveland, Ohio. It can also be prepared as reported by Saunders.²

3. To facilitate subsequent extractions, the solid material remaining after separation of as much of the aqueous phase as possible should be removed by gentle suction filtration and washed with 25 ml. of ether.

4. The sodium chloride solution removes the green color from the ether extract, leaving a yellow solution.

5. The checkers found b.p. 100°/21 mm., n_D^{25} 1.4662–1.4665, d_4^{25} 1.0235–1.0238. Others have reported b.p. 92–94°/12 mm., d_4^{20} 1.0256;³ b.p. 91°/11 mm., n_D^{11} 1.4726, d_4^{11} 1.1033;⁴ b.p. 88.0–88.6°/12 mm., n_D^{28} 1.4712.⁵

Establishing a criterion for the purity of the product is of particular importance because of the known tendency of ethynylcarbinols to undergo rearrangement.^{5,6} The authors have reported that consecutive small fractions of the distillate possess a constant boiling point and refractive index. Further, representative fractions, treated with periodic acid and subsequently with 2,4-dinitrophenylhydrazine, give cyclohexanone 2,4-dinitrophenylhydrazone in 83% over-all yield in a high state of purity.

3. Methods of Preparation

1-Acetylcyclohexanol has been prepared by the hydrolysis of 1-bromo-1-acetylcyclohexane³ and of 1-acetoxy-1-acetylcyclohexane oxime,⁷ by the hydration of 1-ethynylcyclohexanol,^{4,5,8,9} and by the treatment of 1-hydroxycyclohexanecarboxylic acid with methyllithium.¹⁰ The present procedure is based on that of Stacy and Mikulec for 1-acetylcyclopentanol.⁶

¹ State College of Washington, Pullman, Washington.

² *Org. Syntheses Coll. Vol. 3*, 416 (1955).

³ Favorskii, *J. Russ. Phys. Chem. Soc.*, **44**, 1339 (1912) [*C. A.*, **7**, 984 (1913)].

⁴ Locquin and Wouseng, *Compt. rend.*, **176**, 516 (1923).

⁵ Newman, *J. Am. Chem. Soc.*, **75**, 4740 (1953).

⁶ Stacy and Mikulec, *J. Am. Chem. Soc.*, **76**, 524 (1954).

⁷ Wallach, *Ann.*, **389**, 191 (1912).

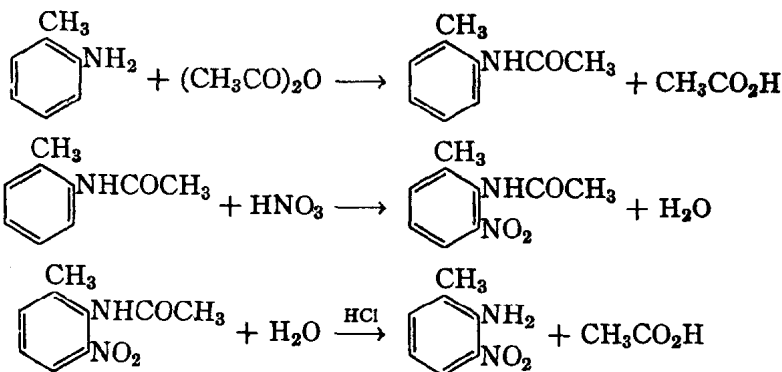
^a Bergmann, Brit. pat. 640,477 [C. A., 45, 1622 (1951)]; U. S. pat. 2,560,921 [C. A., 46, 3072 (1952)].

^b Stacy and Hainley, *J. Am. Chem. Soc.*, 73, 5911 (1951).

¹⁰ Billimoria and MacLagen, *Nature*, 167, 81 (1951); *J. Chem. Soc.*, 1951, 3067.

2-AMINO-3-NITROTOLUENE

(o-Toluidine, 6-nitro-)



Submitted by JOHN C. HOWARD.¹

Checked by CHARLES C. PRICE and JOSEPH D. BERMAN.

1. Procedure

A 1-l. three-necked flask is fitted with a sealed Hershberg stirrer, a reflux condenser, and a dropping funnel. The flask is charged with 650 ml. of acetic anhydride, and 107 g. (107 ml., 1 mole) of o-toluidine (Note 1) is introduced from the dropping funnel. The mixture becomes very warm. After the amine has been completely added, the solution is cooled to 12–13° in an ice-salt bath (Note 2). During the cooling, the dropping funnel and condenser are replaced by another dropping funnel containing 126 ml. (2 moles) of 70% nitric acid and a thermometer which can be read to within 0.5° in the range from 10° to 20° (Note 3).

The nitric acid is added drop by drop to the cold slurry at a rate which maintains the temperature carefully within the limits of

10–12° (Note 4). If the temperature persists in dropping, the addition is stopped after about 5 minutes. The ice bath is removed until the temperature rises 0.5°, the ice-salt bath is replaced, and addition is continued. As the reaction progresses, the acetotoluide which may have precipitated redissolves, and the solution becomes deeply colored. The addition is complete in 1–2 hours, and the nitro compounds may start to separate.

The solution is poured, with stirring, into 3 l. of ice water. The mixture of 4- and 6-nitroacetotoluides precipitates as a cream-colored solid which is collected on a large Büchner funnel. After thorough washing with four 500-ml. portions of ice water, the precipitate is partly dried by suction (Note 5). The moist product is then placed in a steam-distillation apparatus (Note 6), covered with 300 ml. of concentrated hydrochloric acid, and heated until the mixture boils. The acetotoluides are rapidly hydrolyzed, and the solution becomes dark red. Steam is then introduced, and the distillation is thus continued until 36 l. of distillate has been collected (Note 7). The 2-amino-3-nitrotoluene, which separates as bright orange needles when the distillate is cooled, is collected on a large Büchner funnel. The dried product amounts to 75–84 g. (49–55%), m.p. 92–94°. The product may be further purified by a second steam distillation. Ten grams of the amine is distilled from 150 ml. of water, and 3 l. of distillate is collected, yielding 8.7 g. of 2-amino-3-nitrotoluene, m.p. 95–96° (cor.).

2. Notes

1. Commercially available *o*-toluidine, b.p. 75–77°/10 mm., is suitable. Redistillation of this material gave no significantly better results. The checkers obtained a 42% yield of 2-amino-3-nitrotoluene using practical *o*-toluidine directly, and a 57% yield after redistillation.

2. The flask should be immersed up to the neck in the slurry of ice and salt. During the cooling, the acetotoluide may suddenly precipitate, immobilizing the stirrer; a few turns manually breaks up the mass of crystals and allows the stirring to be continued.

3. A low-temperature thermometer with a range from -15° to $+50^{\circ}$ is suitable.

4. If the temperature is allowed to rise above 18° , violent if not explosive decomposition may ensue.

5. The precipitate can be air-dried to a constant weight of 150–160 g.

6. An efficient steam-distillation apparatus such as that described by Fieser ² is recommended. A 12-l. round-bottomed flask cooled in a tub of ice serves as the receiver, which is equipped with an auxiliary vertical condenser attached to a gas absorption trap ³ to accommodate the hydrogen chloride which distils first.

7. The third 12-l. portion yields about 20 g. of 2-amino-3-nitrotoluene. The residue in the steam-distillation flask, about 20 g. of crude 2-amino-5-nitrotoluene, solidifies when cooled and may be separated by filtration. It can be recrystallized from 2 l. of hot water, yielding 14–15 g. of yellow plates, m.p. $130-131^{\circ}$ (cor.).

3. Methods of Preparation

2-Amino-3-nitrotoluene has been prepared by the nitration of oxalotoluide ⁴ and by the nitration of *o*-acetotoluide in acetic acid with fuming nitric acid ⁵ or with a mixture of nitric and sulfuric acid. ⁶

¹ Cornell University, Ithaca, New York.

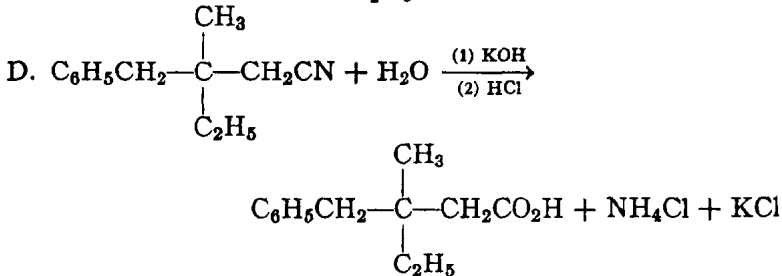
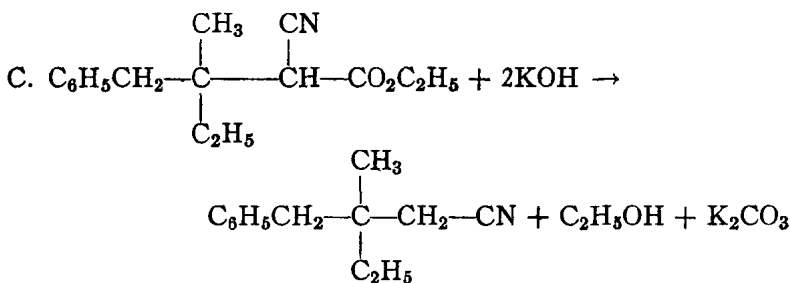
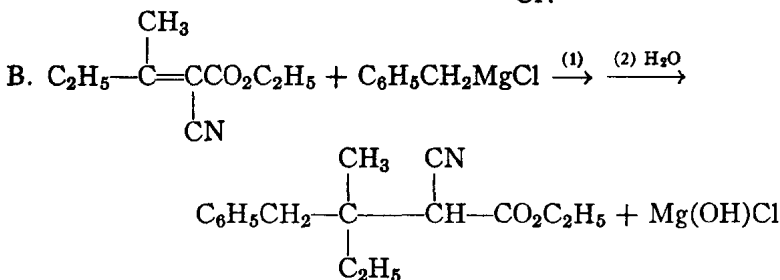
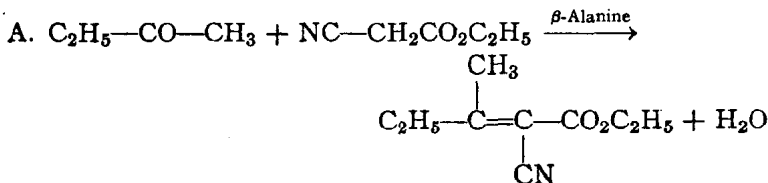
² Fieser, *Experiments in Organic Chemistry*, 2nd ed., p. 159 (Fig. 15), and p. 160 (Fig. 16), D. C. Heath and Company, Boston, Massachusetts, 1941.

³ *Org. Syntheses Coll. Vol. 2*, 4 (1943).

⁴ Hadfield and Kenner, *Proc. Chem. Soc.*, **80**, 253 (1914).

⁵ Cohen and Dakin, *J. Chem. Soc.*, **79**, 1127 (1901).

⁶ McGookin and Swift, *J. Soc. Chem. Ind.*, **58**, 152 (1939).

3-BENZYL-3-METHYLPENTANOIC ACID**(β -Benzyl- β -methylvaleric acid)**

Submitted by F. S. PROUT, R. J. HARTMAN, E. P.-Y. HUANG,
C. J. KORPICS, and G. R. TICHELAAR.¹

Checked by JAMES CASON, K. C. DEWHIRST, E. J. GAUGLITZ, JR., and
WILLIAM G. DAUBEN.

1. Procedure

A. *Ethyl sec-butylidenecyanoacetate*. In a 1-l. round-bottomed flask fitted with a 24/40 joint are placed 0.45 g. of β -alanine, 113 g. (106 ml., 1.0 mole) of ethyl cyanoacetate (Note 1), 87 g. (108 ml., 1.2 moles) of butanone, 20 ml. of glacial acetic acid, and 100 ml. of benzene. A Barrett-type water separator (Note 2) and a condenser are attached to the flask, and the mixture is heated briskly under reflux until water ceases to be collected in the trap (7–12 hours).

The reaction mixture is decanted into a 500-ml. round-bottomed flask which is attached to a fractionating column (Note 3). The solvent is removed at atmospheric pressure while the oil bath is heated finally at 160°. The residue is distilled at reduced pressure to furnish four fractions: (a) acetic acid and other materials boiling below 95°/16 mm.; (b) ethyl cyanoacetate, b.p. 95–110°/16 mm.; (c) intermediate, b.p. 110–124°/16 mm.; and (d) ethyl *sec*-butylidenecyanoacetate, b.p. 124–126°/16 mm., n_D^{25} 1.4640–1.4648. Fraction *d* amounts to 117–122 g., and re-fractionation of fraction *c* yields an additional 18–24 g.; total yield, 135–146 g. (81–87.5%) (Note 4).

B. *Ethyl 3-benzyl-2-cyano-3-methylpentanoate*. A 2-l. three-necked round-bottomed flask, fitted with a tantalum wire Hershberg stirrer, a condenser, and a separatory funnel, is arranged for use of a nitrogen atmosphere.² Magnesium (19.2 g., 0.79 g.-atom) and 100 ml. of dry ether³ are placed in the flask, and a solution of 100 g. (91 ml., 0.79 mole) of benzyl chloride in 500 ml. of dry ether is added in a period of 1.5–2.0 hours, with stirring, while the mixture boils spontaneously. The mixture is boiled for 15 minutes after completion of the addition, then a solution of 110 g. (0.66 mole) of ethyl *sec*-butylidenecyanoacetate in 130 ml. of benzene is added over a 30-minute period with spontaneous reflux. The reaction mixture is stirred and heated under reflux for an additional hour. A precipitate separates after about 30 minutes.

The reaction mixture is poured onto about 400 g. of cracked ice and is made acidic with 20% sulfuric acid. After two clear phases

have formed the mixture is poured into a separatory funnel, and the lower layer is removed. This aqueous layer is extracted with two 100-ml. portions of benzene and discarded. The three organic extracts are washed separately and successively with 125 ml. of water and 125 ml. of saturated sodium chloride solution, then filtered successively through a layer of anhydrous sodium sulfate.

The combined extract (about 1 l.) is flash-distilled at atmospheric pressure from a 250-ml. Claisen flask. After the solvent and a small amount of fore-run (ca. 15 g., b.p. $45^{\circ}/3$ mm.) have been removed, the product is distilled to yield 157–162 g. (92–95%), b.p. 150 – $162^{\circ}/3$ mm. (bath temperature, 180 – 190°), n_D^{25} 1.5053–1.5063 (Notes 5, 6, and 7).

C. *3-Benzyl-3-methylpentanenitrile*. Sixty-seven grams of potassium hydroxide is dissolved by heating in 360 ml. of ethylene glycol and is added to a 1-l. round-bottomed flask containing 155 g. (0.6 mole) of ethyl 3-benzyl-2-cyano-3-methylpentanoate (above). A condenser is attached with a rubber stopper, and the mixture is heated under gentle reflux for 3 hours (Note 8). The resulting two-phase mixture is cooled, diluted with 350 ml. of water, and extracted with three portions of ether (250 ml., 100 ml., 100 ml.). The three extracts are washed successively with 100 ml. of water and 100 ml. of saturated sodium chloride solution, then filtered through a layer of anhydrous sodium sulfate (Note 9). The combined extracts are flash-distilled at atmospheric pressure from a 250-ml. Claisen flask to remove the ether. The residue is distilled at reduced pressure to furnish 102–105 g. (91–93%) of nitrile, b.p. 150 – $160^{\circ}/11$ mm. (bath temperature, 190 – 200°), n_D^{25} 1.5111–1.5128 (Notes 10 and 11).

D. *3-Benzyl-3-methylpentanoic acid*. A solution of 112 g. of potassium hydroxide in 400 ml. of ethylene glycol is added to 93.6 g. (0.5 mole) of 3-benzyl-3-methylpentanenitrile in a 1-l. round-bottomed copper or stainless-steel flask. A condenser with a rubber stopper is attached, and the solution is heated under brisk reflux for 6 hours (Note 12). The reaction mixture is cooled, diluted with 400 ml. of water, and extracted with three portions of ether (250 ml., 100 ml., 100 ml.). The ether

extracts are washed successively with two 75-ml. portions of water and then discarded (Note 13).

The combined aqueous phases are acidified to Congo red with 200 ml. of concentrated hydrochloric acid and extracted with three portions of benzene (200 ml., 75 ml., 75 ml.). The benzene extracts are washed successively with 100 ml. of water and 100 ml. of saturated sodium chloride solution, then filtered through anhydrous sodium sulfate. The combined extract is flash-distilled from a 250-ml. Claisen flask at atmospheric pressure (bath temperature, up to 160°). The residue is distilled at reduced pressure to give 94–96 g. (91–93%) of acid; b.p. 173–177°/7 mm. (bath temperature, 207–220°), n_D^{25} 1.5160–1.5163 (Notes 14 and 15).

2. Notes

1. Ethyl cyanoacetate was obtained from Kay-Fries Chemicals, 180 Madison Avenue, New York, New York.

2. The submitters used a Barrett Distilling Receiver, Corning No. 3622, Corning Glass Works, Corning, New York.

3. The submitters used a 60-cm. heated Vigreux column to effect this fractionation. The checkers used a similar column with partial take-off head.

4. Fractions *b–d* consist entirely of ethyl cyanoacetate and the product. Pure ethyl cyanoacetate and ethyl *sec*-butylidenecyanoacetate have n_D^{25} 1.4151 and 1.4650, respectively. The purity of fractions *b–d* can be estimated by their indexes of refraction, which are proportional to the weight per cent.

5. The pure product obtained by fractional distillation has n_D^{25} 1.5052. The product obtained by distillation from a Claisen flask is contaminated mainly with bibenzyl, b.p. 122–125°/3 mm., f.p. 44°. The purity of the product can be estimated by determination of the saponification equivalent in ethanol.

6. The use of dibenzylcadmium gave no improvement in yield.

7. Phenylmagnesium bromide gives a 79% yield of product, b.p. 178–180°/11 mm., n_D^{25} 1.5063; and *n*-propylmagnesium bromide gives 33–42% yields, b.p. 150–153°/22 mm., n_D^{25} 1.4429, of alkylation product when essentially the same procedure is used.

The yield obtained with *n*-propylmagnesium bromide depends upon the efficiency of separation from the reduction product, ethyl *sec*-butylcyanoacetate, b.p. 126°/22 mm., n_D^{25} 1.4277.⁴

8. After 30 minutes of reflux the second phase begins to separate. The formation of this nitrile layer is probably complete after 2.5–3.0 hours. A small amount of solid, presumably ammonium carbonate, collects in the condenser during the heating.

9. When the combined aqueous washes from three runs were acidified, extracted, and distilled, there was obtained 1.8 g. of 3-benzyl-3-methylpentanoic acid, n_D^{25} 1.5158.

10. This nitrile is contaminated with some lower-boiling bibenzyl and some higher-boiling amide. The pure nitrile, obtained by fractional distillation, has n_D^{25} 1.5110.

11. Hydrolyses of ethyl 2-cyano-3-methyl-3-phenylpentanoate and ethyl 2-cyano-3-ethyl-3-methylhexanoate (cf. Note 7) by essentially this procedure gave 71% (b.p. 149–151°/16 mm., n_D^{25} 1.5149) and 68% (b.p. 103–104°/31 mm., n_D^{25} 1.4291) yields of nitriles, respectively. In the second case, about 12% additional yield of nitrile could be obtained from the acidic fraction, which contains some undecarboxylated product, 2-cyano-3-ethyl-3-methylhexanoic acid.

12. The two-phase solution becomes homogeneous after 1.5–2.0 hours of boiling. This alkaline solution is very corrosive, and a glass flask can be used only a few times in this reaction. A stainless-steel or copper flask is preferable.

13. If emulsions are encountered, the addition of a few milliliters of saturated aqueous sodium chloride clears them readily. The combined ether extracts contain 4–5 g. of solid, neutral material. This product is mainly bibenzyl, b.p. 138–143°/7 mm., f.p. 40°.

14. The best sample of this acid obtained by fractional distillation had n_D^{25} 1.5160; neut. equiv., 207.3 (calcd., 206.3).

15. Hydrolyses of 3-methyl-3-phenylpentanenitrile and 3-ethyl-3-methylhexanenitrile (cf. Note 11) by the described procedure gave 88% and 95% yields of 3-methyl-3-phenylpentanoic acid (b.p. 190–194°/26 mm., n_D^{25} 1.5182) and 3-ethyl-3-methyl hexanoic acid (b.p. 136–137°/15 mm., n_D^{25} 1.4377), respectively.

3. Method of Preparation

Ethyl *sec*-butylidenecyanoacetate has been prepared by this condensation using various amino acids,⁵ ammonium acetate,⁶ sodium sulfate-piperidine,⁷ and zinc chloride-aniline.⁸

The ethyl 3-benzyl-2-cyano-3-methylpentanoate, 3-benzyl-3-methylpentanenitrile, and 3-benzyl-3-methylpentanoic acid preparations follow the procedure given by Prout, Huang, Hartman, and Korpics.⁹

¹ Department of Chemistry, De Paul University, Chicago 14, Illinois.

² Cason and Rapoport, *Laboratory Text in Organic Chemistry*, p. 312, Prentice-Hall, New York, New York, 1950; cf. *Org. Syntheses Coll. Vol. 3*, 601 (1955).

³ Cason and Rapoport, *Laboratory Text in Organic Chemistry*, p. 319, Prentice-Hall, New York, New York, 1950.

⁴ Prout, *J. Am. Chem. Soc.*, **74**, 5915 (1952).

⁵ Prout, *J. Org. Chem.*, **18**, 928 (1953).

⁶ Cope, Hofmann, Wyckoff, and Hardenbergh. *J. Am. Chem. Soc.*, **63**, 3452 (1941).

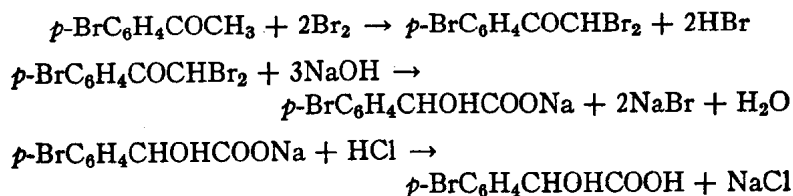
⁷ Cowan and Vogel, *J. Chem. Soc.*, **1940**, 1528.

⁸ Scheiber and Meisel, *Ber.*, **48**, 259 (1915).

⁹ Prout, Huang, Hartman, and Korpics, *J. Am. Chem. Soc.*, **76**, 1911 (1954).

p-BROMOMANDELIC ACID

(Mandelic acid, p-bromo)



Submitted by J. J. KLINGENBERG.¹

Checked by R. T. ARNOLD and C. D. WRIGHT.

1. Procedure

p, α , α -Tribromoacetophenone. In a 1-l. three-necked flask (Note 1) equipped with an efficient mechanical stirrer, a dropping

funnel, and a gas outlet leading to a hood or trap are placed 100 g. (0.5 mole) of *p*-bromoacetophenone (Note 2) and 300 ml. of glacial acetic acid. The resulting solution is stirred and cooled to 20°, and a solution of 26 ml. (0.5 mole) of bromine in 100 ml. of glacial acetic acid is added dropwise (Note 3). Crystals of the mono- α -brominated derivative separate during the addition, which requires about 30 minutes. When the addition is completed, a second solution of 26 ml. (0.5 mole) of bromine in 100 ml. of glacial acetic acid is added dropwise. Slight heating may be necessary to keep the reaction proceeding, as indicated by decolorization of the bromine, but the temperature should be kept as near 20° as possible. During the addition, which requires about 30 minutes, the solid dissolves and crystals of the di- α,α -brominated derivative appear toward the end of the addition. The flask is heated to dissolve the contents, which are transferred, preferably in a hood, to a 1-l. beaker and cooled rapidly by means of an ice-water bath (Note 4). The mixture is filtered with suction (Note 5), and the solid is washed with 50% ethanol until colorless. The air-dried product has a slight pink cast and melts at 89–91°. The yield is 130–135 g. (73–76%) (Note 6). A pure, white solid melting at 92–94° is obtained by recrystallization from ethanol, but the initial product is sufficiently pure for the next step.

p-Bromomandelic acid. In a Waring-type blender are placed 89 g. (0.25 mole) of *p*, α,α -tribromoacetophenone and 100–150 ml. of cold water. The mixture is stirred for 10–15 minutes, and the contents are transferred to a 1-l. wide-mouthed bottle. The mixing vessel is rinsed with 150–200 ml. of ice-cold water. The material from the rinse is combined with the mixture in the bottle, and sufficient crushed ice is added to bring the temperature below 10°. One hundred milliliters of a chilled aqueous solution containing 50 g. of sodium hydroxide is added slowly while the bottle is rotated (Note 7). The contents are stored for approximately 4–5 days in a refrigerator (5°) and are shaken occasionally. During this time most of the solid dissolves, but a slight amount remains as a yellow sludge and the liquid assumes a yellow to amber color. The mixture is filtered, and the insoluble material

is discarded. An excess of concentrated hydrochloric acid is added to the filtrate. The entire resulting mixture containing a white solid is extracted with three 200-ml. portions of ether. The ether extracts are combined, dried over anhydrous sodium sulfate, and filtered into a 1-l. flask. The ether is carefully removed by distillation using a hot-water bath to give a yellow oil which solidifies when cooled. The product is recrystallized from 500 ml. of benzene. The crystals are collected by filtration and washed with benzene until the filtrate is colorless. The air-dried product (Note 8) weighs 40–48 g. (69–83% based on *p*, α , α -tribromoacetophenone), m.p. 117–119° (Note 9). A second recrystallization from 500 ml. of benzene is sometimes necessary.

2. Notes

1. The use of ground-glass equipment is desirable but not necessary.

2. The preparation of *p*-bromoacetophenone is described in *Organic Syntheses*.² The compound is also available from Eastman Kodak Company.

3. Sometimes the initiation of the reaction is slow. The reaction may be started by heating the solution until the bromine is decolorized (approximately 45°), after which the reaction will proceed normally at 20°.

4. The checkers found that maximum yields of product were obtained when cooling was carried to the point where crystallization of the solvent commenced. The trace of crystalline solvent quickly melts during the filtering procedure.

5. These compounds are lachrymatory and should be kept away from the eyes.

6. An additional quantity of less pure material can be isolated from the glacial acetic acid mother liquid and alcohol filtrates by evaporation of the solvent. This, after recrystallization from ethanol, amounts to 15–30 g.

7. It is important that the reaction mixture be kept cold at this point. The amount of sludge and colored material increases as the temperature increases.