[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF EMORY UNIVERSITY]

Hydroxymethylene Ketones. I. The Preparation of β-Ketodimethylacetals and Methoxymethylene Ketones

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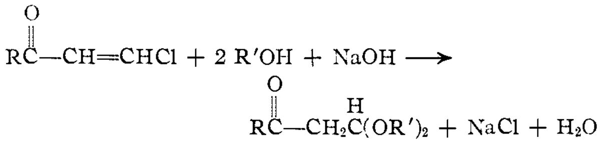
RECEIVED OCTOBER 29, 1952

Sodium hydroxymethylene ketones of the type, $\mathrm{R{-}CO{-}CH{=}CHONa}$, give good yields of β-ketodimethylacetals on treatment with methanolic hydrogen chloride. The sodium salts of hydroxymethyleneacetophenone and hydroxymethylenecyclohexanone give methoxymethylene ketones on similar treatment. Sodium salts of the type $\mathrm{R{-}CO{-}CR'{=}CHONa}$ give mixtures of β-ketodimethylacetals and methoxymethylene ketones. Evidence is presented that the Claisen condensation of methyl formate with methyl ethyl ketone and methyl *n*-propyl ketone occurs at both the methyl and methylene units of the ketones. β-Ketodimethylacetals and methoxymethylene ketones are readily cleaved by aqueous alkali.

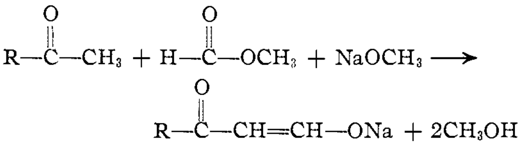
β-Ketoacetals constitute a class of compounds which should be valuable as intermediates in organic synthesis. The most frequently applied method for preparing β-ketoacetals is that of Nelles[1] according to which a chlorovinyl ketone is treated with an alcohol and the stoichiometric amount of alkali



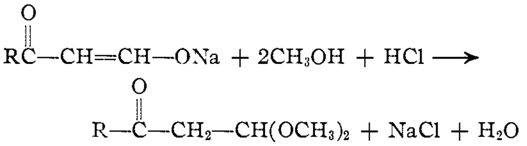
$$\mathrm{R\overset{O}{\overset{\|}{C}}{-}CH{=}CHCl + 2\,R'OH + NaOH \longrightarrow R\overset{O}{\overset{\|}{C}}{-}CH_2\overset{H}{C}(OR')_2 + NaCl + H_2O}$$

Price and Pappalardo[2] and Nesmeyanov[3] have prepared a number of β-ketoacetals by the Nelles procedure. Ruzicka and his co-workers[4] reported the preparation of three β-ketoacetals in the alicyclic series by treatment of the appropriate hydroxymethylene ketone with ethyl orthoformate. Shantz[5] has reported the preparation of the acetal of hydroxymethylene-β-ionone by treatment of the hydroxymethylene ketone with ethanol and calcium chloride. Theimer and Ritter[6] prepared the cyclic 2-methyl-2,4-pentanediol acetal of acetoacetaldehyde (or hydroxymethyleneacetone) by the Oppenauer oxidation of the corresponding acetal of acetaldol. After developing the method of preparation of β-keto acetals described below, it came to our attention that Sugasawa[7] had prepared the diethylacetal derived from hydroxymethyleneacetone in 35% yield by the treatment of the sodium salt of hydroxymethyleneacetone with ethanolic hydrogen chloride, a method essentially the same as ours. In fact, Sugasawa[8] had much earlier reported the preparation of ethyl β,β-diethoxypropionate in 60% yield by an analogous procedure, *i.e.*, by treatment of the sodium salt of ethyl hydroxymethyleneacetate with ethanolic hydrogen chloride. Hata and co-workers[9] have prepared the dimethylacetal of hydroxymethyleneacetone in 45% yield by the addition of sodium hydroxymethyleneacetone to a mixture of methanol and concentrated sulfuric acid. More recently, Richmond[10] has described the preparation of hydroxymethyleneacetone diethylacetal in 47.5% yield by a method analogous to that of Sugasawa.

We have prepared β-ketodimethylacetals from ketones in good yields by the sequence of reactions



$$\mathrm{R{-}\overset{O}{\overset{\|}{C}}{-}CH_3 + H{-}\overset{O}{\overset{\|}{C}}{-}OCH_3 + NaOCH_3 \longrightarrow R{-}\overset{O}{\overset{\|}{C}}{-}CH{=}CH{-}ONa + 2CH_3OH} \quad (1)$$



$$\mathrm{R\overset{O}{\overset{\|}{C}}{-}CH{=}CH{-}ONa + 2CH_3OH + HCl \longrightarrow R{-}\overset{O}{\overset{\|}{C}}{-}CH_2{-}CH(OCH_3)_2 + NaCl + H_2O} \quad (2)$$

The experimental procedure is convenient in that isolation of the intermediate sodium salt is not necessary, and the sequence of reactions may be run in a single reaction vessel.

The products obtained are shown in Table I. Hydroxymethylene ketones of the type $\mathrm{R{-}CO{-}CH{=}CHOH}$, *i.e.*, those which arose from the condensation of a methyl ketone with methyl formate at the methyl group of the ketone, gave good yields of β-ketodimethylacetals on treatment with methanolic hydrogen chloride. Hydroxymethyleneacetophenone and hydroxymethylenecyclohexanone gave methoxymethylene ketones rather than β-ketoacetals.

Hydroxymethylenediethyl ketone, which is derived from a symmetrical ketone through condensation at a methylene group, gave a sharply boiling fraction of the β-ketoacetal, while the methoxymethylene ketone was obtained by rapid distillation of the still residue after the β-ketoacetal had been removed. Our results indicate that the unsymmetrical methyl ethyl and methyl *n*-propyl ketones give products arising from condensation with methyl formate at both methyl and methylene groups under our experimental conditions.[11] Frac-

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(1) Johannes Nelles, U. S. Patent 2,091,373 (1937).

(2) Charles C. Price and Joseph A. Pappalardo, THIS JOURNAL, **72**, 2613 (1950).

(3) A. N. Nesmeyanov, N. K. Kochetkov and M. I. Rybinskaya, *Izvest. Nauk. Akad. S.S.S.R., Otdel. Khim. Nauk*, 395 (1951) [*C. A.*, **46**, 3007 (1952)].

(4) L. Ruzicka, C. F. Seidel, H. Schinz and M. Pfeiffer, *Helv. Chim. Acta*, **31**, 422 (1948).

(5) Edgar M. Shantz, THIS JOURNAL, **68**, 2553 (1946).

(6) Ernst Theodore Theimer and John J. Ritter, Abstracts, Organic Division, 110th Meeting, American Chemical Society, Chicago, Ill., Sept., 1946.

(7) S. Sugasawa, Yoshio Ban and R. Mochizuki, *J. Pharm. Soc. Japan*, **69**, 82 (1949); S. Sugasawa, Japanese Patent 177,821 (1949).

(8) S. Sugasawa, *J. Pharm. Soc. Japan*, No. 545, 551 (1927) [*C. A.*, **21**, 3601 (1927)].

(9) Junji Hata, Shun-ichi Yamada, Jun-ichi Iwao, Nobuyuki Kato, Norio Sugimoto and Rynji Inoye, *ibid.*, **69**, 477 (1949).

(10) Henry J. Richmond, U. S. Patent 2,570,713 (1951).

(11) Erich Benary, *Ber.*, **59**, 2198 (1926), presented evidence that the condensation of methyl ethyl ketone with ethyl formate occurs at both the methyl and methylene positions. At the same time Benary could detect only one isomer from the condensation of methyl *n*-propyl ketone with ethyl formate, *i.e.*, that corresponding to methyl condensation. A. H. Tracy and R. C. Elderfield, *J. Org. Chem.*, **6**, 63 (1941), detected only the methylene condensation product in the

TABLE I

THE PREPARATION OF β-KETODIMETHYLACETALS AND METHOXYMETHYLENE KETONES FROM KETONES

Ketone	Product	Yield, %	B.p., °C.	B.p., Mm.	$n^{25}D$	Equivalent weight Calcd.	Equivalent weight Found	Equivalent weight Found	Carbon, % Calcd.	Carbon, % Found[f]	Hydrogen, % Calcd.	Hydrogen, % Found[f]
$CH_3—CO—CH_3$	$CH_3—CO—CH_2CH(OCH_3)_2$[a]	65.4	39	2.3	1.4143	132.2	132.0	132.4	. . .	. . .	. . .	. .
$(CH_3)_2CH—CO—CH_3$	$(CH_3)_2CH—CO—CH_2CH(OCH_3)_2$	63.3	60	4.8	1.4193	160.2	160.0	160.4	59.97	60.06	10.07	9.84
$(CH_3)_2CH—CH_2—CO—CH_3$	$(CH_3)_2CH—CH_2—CO—CH_2CH(OCH_3)_2$[b]	69.4	73	4.9	1.4212	174.2	174.6	174.5	. . .	. . .	. . .	. .
$C_6H_5—CO—CH_3$	$C_6H_5—CO—CH═CH—OCH_3$	56.3	112	1.8	1.5777	162.2	161.8	161.4	74.05	73.86	6.22	6.32
Cyclohexanone	2-Methoxymethylenecyclohexanone[c]	34.0	85	3.2	1.5025	140.2	142.0	143.1	68.54	67.77	8.63	8.76
$CH_3—CH_2—CO—CH_2—CH_3$	$CH_3—CH_2—CO—CH(CH_3)—CH(OCH_3)_2$	51.7	57–58	4.0	1.4178	160.2	160.7	160.5	59.97	59.73	10.07	9.99
	$CH_3—CH_2—CO—C(CH_3)═CH—OCH_3$	12.6	62–65	3–4	1.4733	128.2	131.7	131.6	65.59	64.72	9.44	9.42
$CH_3—CH_2—CO—CH_3$	Mixture of β-ketoacetals and methoxymethylene ketone[d]	*ca.* 57[e]										
$CH_3—CH_2—CH_2—CO—CH_3$		*ca.* 61[e]										

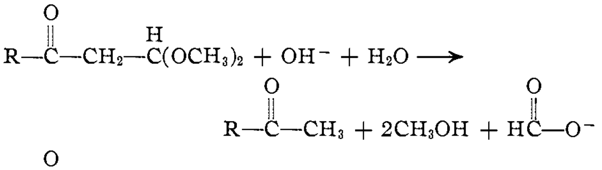
[a] Price[2] reports b.p. 38° at 2.0 mm. and $n^{25}D$ 1.4139. [b] Price[2] reports b.p. 81° at 7.0 mm. and $n^{25}D$ 1.4204. [c] Robert Robinson and James Walker, *J. Chem. Soc.*, 1530 (1935), report this compound from hydroxymethylenecyclohexanone and dimethyl sulfate. They report b.p. 75–80° at 13 mm., $n^{18}D$ 1.4854, unsatisfactory analyses, and that the compound turns green in a few minutes after distillation. It appears that their product was largely hydroxymethylenecyclohexanone; see Experimental section. [d] See Experimental section for details. [e] Calculated as β-ketoacetal; the actual yield is slightly higher since the product is a mixture. [f] Microanalyses by Dr. G. Weiler and Dr. F. B. Strauss, Oxford, England.

tional distillation of the crude products from both of the latter ketones gave in each case a lower boiling fraction which gave good analytical results for, and which we believe to be, the β-ketoacetal derived from the methylene condensation product. Higher boiling fractions were obtained in each case which on the basis of refractive index and analytical data appear to be mixtures of the β-ketoacetal derived from methyl condensation and the methoxymethylene ketone, presumably from the methylene condensation product. In the case of methyl ethyl ketone, distillation of the still residue through a short path still gave the methoxymethylene ketone. Work is now in progress to establish more firmly the structures of these products.

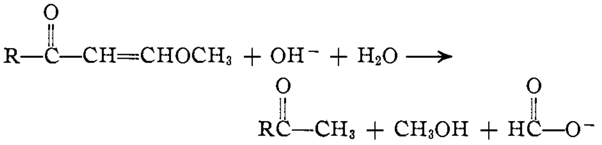
Inasmuch as our procedure failed to give the β-ketoacetal derived from hydroxymethylenecyclohexanone, we attempted to use the method of Ruzicka.[4] Although his experimental procedure was followed as closely as possible, in our hands it gave only the alkoxymethylene ketone when either methyl or ethyl orthoformate was used.

The use of methyl orthoformate with the hydroxymethylene ketone derived from diethyl ketone according to Ruzicka's procedure gave a mixed product similar to that obtained by our method, but much richer in the methoxymethylene ketone.

The equivalent weights listed in Table I are based on alkaline hydrolyses of the products. We have found that both β-ketoacetals and the ethers of hydroxymethylene ketones are quantitatively hydrolyzed in aqueous alkali[12] according to the overall equations



$$R—\overset{O}{\overset{\|}{C}}—CH_2—\overset{H}{C}(OCH_3)_2 + OH^- + H_2O \longrightarrow R—\overset{O}{\overset{\|}{C}}—CH_3 + 2CH_3OH + H\overset{O}{\overset{\|}{C}}—O^-$$



$$R—\overset{O}{\overset{\|}{C}}—CH═CHOCH_3 + OH^- + H_2O \longrightarrow R\overset{O}{\overset{\|}{C}}—CH_3 + CH_3OH + H\overset{O}{\overset{\|}{C}}—O^-$$

Thus, the equivalent weights of our products were determined by a simple "saponification" procedure. Johnson[12a] has shown that some alkoxymethylene ketones are not stable toward prolonged distillation, a fact which we have verified, and that satisfactory carbon–hydrogen analyses are not obtained with these compounds. It is apparent that saponification equivalents have more meaning in these cases.

Distillation of a sample of acetoacetaldehyde

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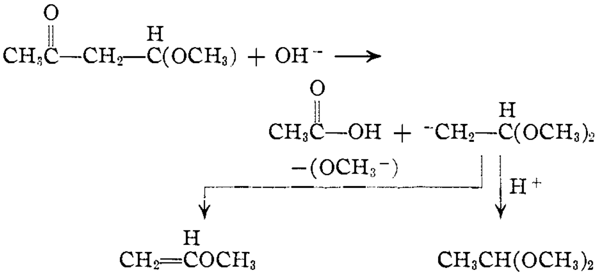
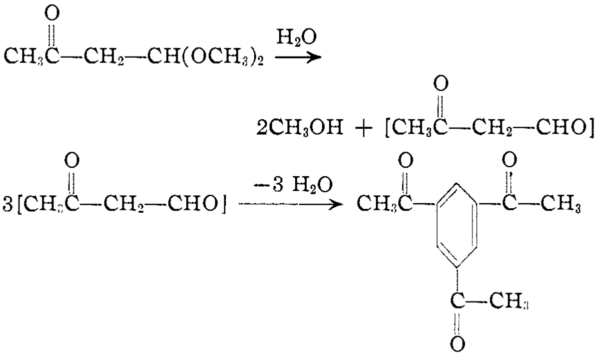
methyl ethyl ketone–ethyl formate reaction; their conclusion was drawn on the basis of a multi-step reaction sequence in which a minor isomer could easily be lost. This latter work is frequently quoted in support of the statement that formates condense at the methylene unit of methyl ethyl ketone; *e.g.*, R. P. Mariella, THIS JOURNAL, **69**, 2670 (1947).

[12] (a) W. S. Johnson and Harvey Posvic, *ibid.*, **69**, 1361 (1947), used the isopropyloxymethylene grouping as a blocking agent, its removal later being accomplished by treatment with acid and then base. (b) R. B. Woodward, F. Sondheimer, D. Taub, K. Heusler and W. M. McLamore, *ibid.*, **74**, 4223 (1952), have reported removal of the methylanilinomethylene group by base alone.

dimethylacetal with 10% aqueous sodium hydroxide gave within the limits of accuracy of the determination the theoretical amounts of acetone and methanol. We were unable to detect any of the products which might arise from alkaline cleavage at the carbonyl group



It should be pointed out that cleavage at the carbonyl group would not interfere with the equivalent weight determination. When acetoacetaldehyde dimethylacetal was refluxed with distilled water for one hour no acetone was detected; 1,3,5-triacetylbenzene crystals appeared in the still-pot within 15 minutes, and on cooling the residue a 75% yield of triacetylbenzene was obtained



Experimental

Materials.—Methyl formate, acetone and methyl ethyl ketone were dried by distillation over phosphorus pentoxide. The higher ketones were dried by distillation through a short Fenske column. Technical grade methanol and Matheson sodium methoxide[13] were used.

Procedure.—The following general procedure was used in the preparation of the products listed in Table I.

A mixture of 0.5–0.6 mole of the ketone and 0.6–0.75 mole of methyl formate was added to a slurry of 0.5 mole of sodium methoxide in absolute ether at a rate such as to maintain gentle reflux. The minimum amount of ether required to make stirring practicable varied from 200 to 500 ml. with different ketones. Stirring was continued for one hour after which most of the ether was removed without stirring under the vacuum from a water aspirator. Methanol, 3 moles, was then added to the solid sodium hydroxymethylene ketone followed by the addition of a solution of 1 mole of anhydrous hydrogen chloride in 2 moles of methanol.

The temperature, which rose to 35–40° on the addition of the hydrogen chloride, was lowered to 20°. Stirring was continued for 1–2 hours at 20° and a saturated solution of potassium hydroxide in methanol was added until the mixture was just alkaline to litmus paper. The precipitated salts were removed by filtration and the filtrate was distilled from a simple pot still. Methanol was removed at atmospheric pressure until the pot temperature reached 70–75° and finally at *ca.* 150 mm. The pressure was then reduced to *ca.* 30 mm. while a small amount of water was removed.

Finally, at 2–4 mm. the salts which were always present in small quantity were dehydrated with moderate heating, and the reaction product was distilled.

The crude products derived from the following ketones were redistilled through a simple pot still: acetone, methyl isopropyl ketone, methyl isobutyl ketone, cyclohexanone and acetophenone. The products derived from methyl ethyl ketone, methyl *n*-propyl ketone and diethyl ketone were subjected to fractional distillation using a Todd column,[14] 90 cm. (5 mm.), with a monel spiral packing.

The following variations in the general procedure, deviations from normal behavior, and observations are noteworthy.

Acetone.—The use of 0.64 mole of hydrogen chloride (0.14 mole excess) gave a 53.6% yield of the dimethylacetal derived from hydroxymethyleneacetone.

Cyclohexanone.—On addition of hydrogen chloride to the sodium salt of hydroxymethylenecyclohexanone the reaction mixture turned dark green. After the addition of potassium hydroxide the mixture was dark brown. The distillation residue from the reaction mixture was 37 g. of tar.

Diethyl Ketone.—This ketone required 200 ml. of ether for the condensation with methyl formate. When the general procedure was modified by elimination of the ether-removal step, the combined yield of β-ketoacetal and methoxymethylene ketone was 61% as compared to 64% when the ether was removed.

Methyl Ethyl Ketone.—The use of 0.75 mole of hydrogen chloride (0.25 mole excess) gave a 60% yield of crude products, $n^{25}\mathrm{D}$ 1.4330. When 1.25 moles of hydrogen chloride was used, the yield of crude product was again 60% and $n^{25}\mathrm{D}$ 1.4284. Fractional distillation of 40.7 g. of the product obtained using the general procedure gave 32.8 g., b.p. 55–68° at 7–7.5 mm., with the major portion in the ranges 55–58° and 62–64.5°. Successive arbitrarily-cut fractions showed $n^{25}\mathrm{D}$ 1.4142 to 1.4236 and equivalent weights 148.0 to 141.6, indicating increasing contamination of the distillate with methoxymethylene ketone with increasing boiling point.

Anal. For the arbitrary 55–58° fraction. Calcd. for $C_7H_{14}O_3$: C, 57.52; H, 9.65. Found: C, 57.34; H, 9.33.

The distillation residue of 6.1 g. was distilled from a simple pot-still to give 4.0 g. of distillate, b.p. 53–56° at 2 mm., $n^{25}\mathrm{D}$ 1.4693. The equivalent weights found were 119.0 and 118.7. The calculated equivalent weight of the β-ketodimethylacetal is 148.2 and that of the methoxymethylene ketone is 116.1.

Anal. Calcd. for the methoxymethylene ketone $C_6H_{10}O_2$: C, 63.13; H, 8.83. Found: C, 62.38; H, 9.84.

Methyl *n*-Propyl Ketone.—Fractional distillation of a 37 g. aliquot of the crude product gave 34.1 g., b.p. 56.5–66° at 7–7.5 mm., with the major portion in the ranges 58.5–60° and 64–66°. Successive arbitrarily-cut fractions showed $n^{25}\mathrm{D}$ 1.4179 to 1.4285 and equivalent weights 160.0 to 155.1.

Anal. For the arbitrary 58.5–60° fraction. Calcd. for $C_8H_{16}O_3$: C, 59.97; H, 10.07. Found: C, 59.77; H, 9.88.

The distillation residue was 2.0 g. The theoretical equivalent weight for the β-ketodimethyl acetal is 160.2 and that of the methoxymethylene ketone is 128.2.

Hydroxymethylenecyclohexanone and Orthoformic Esters.—Freshly distilled hydroxymethylenecyclohexanone, 24.0 g. (0.19 mole), was added to 24.0 g. of absolute methanol and 24.0 g. (0.23 mole) of methyl orthoformate. Two drops of concd. hydrochloric acid was added to the mixture, the temperature rose to 35°, and the mixture turned dark green immediately. The mixture stood 48 hours at room temperature, was taken up in ether, and washed once with 50 ml. of cold 10% potassium hydroxide and twice with 50-ml. portions of distilled water. Distillation gave 14.0 g. (52%) of methoxymethylenecyclohexanone, b.p. 86–88° at 3.2 mm. and $n^{25}\mathrm{D}$ 1.5020. Equivalent weights found were 147.9 and 147.7; calcd. for methoxymethylenecyclo-

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(13) It was observed that when sodium methoxide was used from bottles which had been opened several times, yields were markedly lower even though the sodium methoxide remained perfectly free-flowing. This situation was obviated by strict limitation of the number of exposures to air to a maximum of three or four. Woodward (ref. 12) has reported variable yields using commercial sodium methoxide.

(14) Floyd Todd, *Ind. Eng. Chem., Anal. Ed.*, **8**, 313 (1936).

hexanone, 140.2. The procedure was repeated with hydroxymethylenecyclohexanone, ethanol and ethyl orthoformate. A 70% yield of ethoxymethylenecyclohexanone, b.p. 95–96° at 2.0–2.5 mm., $n^{25}D$ 1.4940, was obtained. Equivalent weights found were 161.3 and 160.8; calcd. for ethoxymethylenecyclohexanone, 154.2.

Attempted fractional distillation of ethoxymethylenecyclohexanone gave much resinification and no well-defined fractions.[12a] It was observed that both methoxymethylenecyclohexanone and ethoxymethylenecyclohexanone slowly became very viscous on standing, the latter much faster than the former.

Procedure for Equivalent Weight Determinations.—A weighed sample of the β-ketodimethylacetal or the alkoxymethylene ketone was refluxed for 0.5–2 hours with excess standard 0.1 *N* sodium hydroxide and back-titrated to phenolphthalein with 0.1 *N* hydrochloric acid. The equivalent weight is then equal to the sample weight in g. × 1000 meq. of base used.

EMORY UNIVERSITY, GEORGIA

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF ROCHESTER]

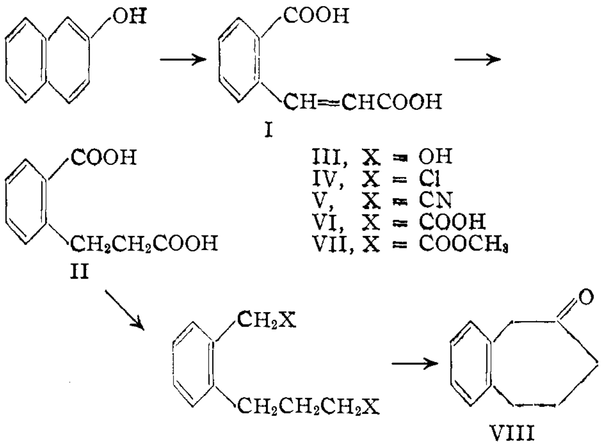
The Synthesis of Benzcyclohepten-6-one[1]

BY G. A. PAGE AND D. S. TARBELL

RECEIVED DECEMBER 1, 1952

The preparation of benzcyclohepten-6-one, a compound of use for syntheses in the general field of colchicine chemistry, is described.

A benzcyclohepten-6-one was required as an intermediate compound for use in studies aimed at the synthesis of the three carbon ring system of colchicine. Although the preparation of 2,3,4-trimethoxybenzcyclohepten-6-one has been described,[2] the synthetic route to the parent ketone (VIII), outlined below, appeared worthy of investigation.



o-Carboxycinnamic acid (I), prepared by the oxidation of β-naphthol using peracetic acid,[3] was reduced by means of the Schwenk procedure[4] to give β-(*o*-carboxyphenyl)-propionic acid (II) in excellent yield. Reduction of II using ethereal lithium aluminum hydride gave the expected diol (III),[5] which was obtained pure in relatively low yield. Conversion of the diol to the corresponding dichloride (IV) went smoothly and a satisfactory yield of this compound was obtained from II when the crude diol was treated directly with thionyl chloride in dimethylaniline.[6] Cyanation of IV, using procedures in the literature,[2,7] and modifications thereof, failed to give a pure compound upon fractional distillation. No attempt was made to cyclize this crude dinitrile (V), although our original intention was to effect ring closure at this stage using an established procedure.[8]

Hydrolysis of crude V gave a fair yield of γ-(*o*-carboxymethylphenyl)-butyric acid (VI). Using the Arndt–Eistert procedure as adapted to bis-homologation,[9] an attempt to convert the acid chloride of II to the required acid (VI) resulted in a very low yield of product. Although this method was not considered suitable here for the large-scale preparation of VI, it is felt that considerable improvement of the Arndt–Eistert procedure is possible and would provide a short-path route to the ketone (VIII).

The dimethyl ester (VII) of γ-(*o*-carboxymethylphenyl)-butyric acid was cyclized by means of a Dieckmann-type ring closure[10] and the resultant crude β-ketoester(s) saponified and decarboxylated to give benzcyclohepten-6-one (VIII) in moderately good yield.

Experimental[11]

Oxidation of β-Naphthol; Formation of I.—The procedure used was essentially that reported in the literature.[3] However, the use of a 10% excess of the peracetic acid[12] resulted in slightly higher and more consistent yields of *o*-carboxycinnamic acid, over a period of 4 to 5 days, than when the theoretical 3.0 molecular proportion of oxidant was employed. Starting from 10 g. or 20 g. of β-naphthol, the yield of crude I was 73–75%. After purification by solution in 5% bicarbonate solution and subsequent acidification with mineral acid, the yield of *o*-carboxycinnamic acid, m.p. 205°,[3,13] was 67–70%.

A small portion of the acid was crystallized from aqueous alcohol to give small, white, felted prisms, m.p. 205°, having the correct neutralization equivalent.

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[1] This work was supported by a grant from the National Institutes of Health.

[2] H. Rapoport and J. E. Campion, THIS JOURNAL, **73**, 2239 (1951).

[3] J. Boeseken and Lochmann von Konigsfeldt, *Rec. trav. chim.*, **54**, 318 (1935); F. P. Greenspan, *Ind. Eng. Chem.*, **39**, 847 (1947).

[4] E. Schwenk, *et al.*, *J. Biol. Chem.*, **118**, 792 (1937); *J. Org. Chem.*, **7**, 587 (1942); **9**, 1, 175 (1944).

[5] J. v. Braun and F. Zobel, *Ber.*, **56**, 2139 (1923).

[6] A. Cohen, *J. Chem. Soc.*, 429 (1935).

[7] A. E. Bide and P. A. Wilkinson, *J. Soc. Chem. Ind.* (*London*), **64**, 84 (1945).

[8] E. M. Fry and L. F. Fieser, THIS JOURNAL, **62**, 3489 (1940).

[9] W. E. Bachmann and W. S. Struve, "Organic Reactions," Vol. I, John Wiley and Sons, Inc., New York, N. Y., 1942, pp. 45, 51.

[10] R. E. Beyler and L. H. Sarett, THIS JOURNAL, **74**, 1397 (1952).

[11] All melting points are corrected; microanalyses by Miss Claire King and Miss Viola Williams.

[12] Commercial 40% peracetic acid is available from the Becco Sales Corporation, Buffalo 7, N. Y.

[13] After melting, the acid solidifies and remelts at 150–151° due to formation of phthalideacetic acid, m.p. 153°.