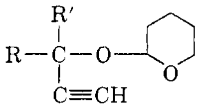
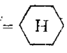
TABLE I

TETRAHYDROPYRANS:[a] 

R	R′	B.P.°	Press., mm.	Formula	Carbon, %		Hydrogen, %	
					Calcd.	Found	Calcd.	Found
CH_3	CH_3	64.5–65.5	8	$C_{10}H_{16}O_2$	71.39	71.16	9.59	9.45
CH_3CH_2	CH_3	62.5–64.5	3.3	$C_{11}H_{18}O_2$	72.49	72.62	9.95	9.67
$(CH_3)_2CHCH_2$	CH_3	47–50	0.6–0.2	$C_{13}H_{22}O_2$	74.24	73.94	10.55	10.24
$CH_3(CH_2)_5$	CH_3	76–77	0.12	$C_{15}H_{26}O_2$	75.58	75.08	10.99	10.45
	CH_3	99–100.5	0.02	$C_{15}H_{18}O_2$	78.22	78.37	7.88	7.85
R, R′= 		101.5–102.5	3.6–3.7	$C_{13}H_{20}O_2$	74.96	74.67	9.68	9.52

[a] Yields ranged from 60.4 to 84.4%.

of the Grignard derivative of the ethynyl alcohols, the magnesium derivatives of the free alcohols are so insoluble, even in tetrahydropyran, as to present contact problems and thus inordinate reaction times. The dihydropyran adducts, however, are quite soluble, even at −70°.

EXPERIMENTAL

General procedure. The appropriate alcohol (1 mole) and dihydropyran (1.2–2 moles) are mixed in a round-bottomed flask fitted with a thermometer or thermocouple well and a reflux condenser with drying tube. A few crystals of *p*-toluenesulfonic acid are added and dissolved by swirling. With the lower molecular weight alcohols, the exothermic addition begins almost at once and the reaction is usually complete within 0.5–1 hr. With higher molecular weight alcohols, the mixture may be heated on the steam bath for 0.5–1 hr. to ensure complete addition.

A gram or two of anhydrous potassium carbonate is added to the cooled mixture and stirred well for 0.5 hr. or allowed to stand overnight. A magnetic stirrer is most convenient for stirring. The salts are removed by filtration, excess dihydropyran recovered by distillation at atmospheric pressure and the product by distillation at reduced pressure (Table I).

The products are colorless, slightly to very viscous and indefinitely stable in the absence of acid.

It should be mentioned that, in a test tube experiment, evidence was obtained that even *tert*-butyl alcohol adds readily to dihydropyran, though no attempt was made to isolate the product.

Since an equimolar mixture of the two reactants would contain the same carbon and hydrogen values as the products, the infrared spectrum of each product was obtained. The spectra were consistent with their formulation as adducts; no hydroxy groups were detected and the acetylenic linkage and its reactive hydrogen were undisturbed.

MIDLAND, MICH.

[CONTRIBUTION FROM THE INDIAN ASSOCIATION FOR THE CULTIVATION OF SCIENCE]

Synthesis of 3-Methyl-5,6,7,8-tetrahydro-1-naphthol

DILIP K. DATTA AND P. BAGCHI

Received August 26, 1959

Cyclization of the dienic acid obtained by dehydration followed by hydrolysis of ethyl γ-(Δ′-cyclohexenyl)-β-methyl-β-hydroxybutyrate with phosphorus pentoxide leads to the formation of 3-methyl-5,6,7,8-tetrahydro-1-naphthol whose structure was proved by dehydrogenation to 3-methyl-1-naphthol and also by an unambiguous synthesis.

A case of cyclization reaction involving an oxocarbonium ion belonging to an unsaturated side chain forming the part of a ring system and an ethylenic double bond was first studied by Bagchi, Bergmann, and Banerjee[1] in connection with the synthesis of 9-hydroxy-*sym*-octahydrophenanthrene. This observation has now been extended to the study of a case involving the cyclization of an ethylenic double bond present in a six-membered ring and a carboxyl group in a linear unsaturated side chain resulting in the formation of 3-methyl-5,6,7,8-tetrahydro-1-naphthol.[2]

As a suitable system, the dienic acid (III, R = H) obtained by the dehydration and hydrolysis of ethyl γ-(Δ′-cyclohexenyl)-β-methyl-β-hydroxybutyrate (I) was prepared for this work. Cyclohexenyl acetone required as the starting material was prepared according to the method of

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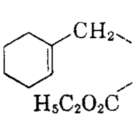
[1] P. Bagchi, F. Bergmann, and D. K. Banerjee, *J. Am. Chem. Soc.*, **71**, 989 (1949).

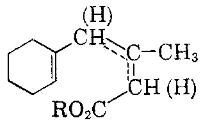
[2] D. K. Datta and P. Bagchi, *Sci. and Culture (Calcutta)*, **17**, 525 (1952).

Jupp, Kon, and Lockton[3] with modifications. The ketone showed the following ultraviolet absorption characteristics: λ max 235–245 mμ (log ϵ 2.8), indicating the presence of cyclohexylidene acetone to the extent of about 6.%. Our product is, therefore, essentially cyclohexenyl acetone. The ketone underwent a smooth reaction with ethyl bromoacetate in the presence of zinc in benzene solution to give ethyl γ-(Δ'-cyclohexenyl-β-methyl-β-hydroxybutyrate) (I) in 53% yield. This on dehydration with phosphorus pentoxide furnished a dienic ester (II, $R = C_2H_5$; yield 87%), which in turn on hydrolysis with ethanolic alkali gave the dienic acid (III, R = H) as a thick liquid which failed to crystallize. It may be noticed that the second double bond in II and III might assume a position of conjugation with respect to either the double bond of the cyclohexene ring or the carbethoxyl or carboxyl. The actual product might also represent a mixture of the bond and/or geometric isomers. Because of these reasons and our inability to obtain the acid III in a crystalline form we have not assigned the position of the second double bond with certainty in our products.

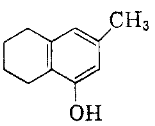
The dienic acid on heating with phosphorus pentoxide in benzene solution underwent cyclization to yield after treatment with ethanolic potassium hydroxide, acidification and washing with bicarbonate and distillation, a solid which on crystallization melted at 98.5° and had as analysis corresponding to methyltetrahydronaphthol. That the above compound possesses structure IV was proved by dehydrogenation over palladium-charcoal at 250° when 3-methyl-1-naphthol(V), melting point 91–92° was obtained.[4] The structures of IV and V were also confirmed by independent unambiguous syntheses described below:



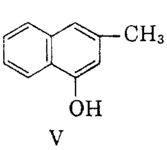
I



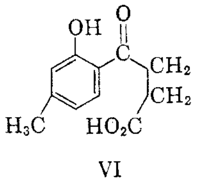
II. $R = C_2H_5$
III. R = H



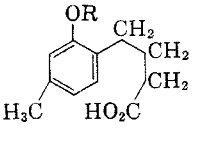
IV



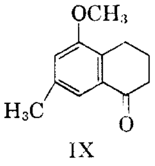
V



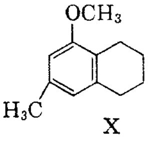
VI



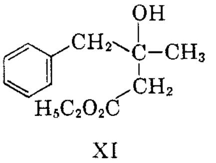
VII. R = H
VIII. $R = CH_3$



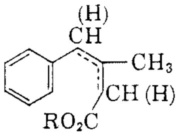
IX



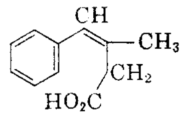
X



XI



XII. $R = C_2H_5$
XIII. R = H



XIV

Synthesis of 3-methyl-5,6,7,8-tetrahydro-1-naphthol.[5] For the preparation of a suitable starting material for the synthesis, the condensation between *m*-cresol and succinic anhydride was investigated; this led to the isolation of β-(2-hydroxy-4-methylbenzoyl)propionic acid (VI), m.p. 154°. The proof of the structure of the condensation product will be published in a subsequent communication. The keto acid (VI) was then reduced by Clemmensen's method using benzene as the immiscible solvent to yield γ-(2-hydroxy-4-methylphenyl)butyric acid (VII, R=H) almost quantitatively. Attempts to cyclize this acid directly with sulfuric acid under various experimental conditions failed, presumably due to sulfonation of the benzene nucleus. The acid was, therefore, methylated with dimethyl sulfate and alkali to γ-(2-methoxy-4-methylphenyl)butyric acid (VIII, $R = CH_3$) in an almost quantitative yield. Methylation of this acid was smooth, unlike the methylation of the keto acid (VI). The difficulty of methylation of VI no doubt arises from the existence of an internal hydrogen bond between the hydrogen of the phenolic hydroxyl and the oxygen of the carbonyl. The acid (VIII, $R = CH_3$) was cyclized with phosphorus oxychloride in tetrachloroethane to 1-methoxy-3-methyl-5-keto-5,6,7,8-tetrahydronaphthalene (IX, yield 64%). Reduction of IX according to the Hueng-Minlon method gave 3-methyl-5,6,7,8-tetrahydro-1-naphthol methyl ether (X, yield 60%) with only slight demethylation. The latter on demethylation with hydrobromic acid–acetic acid furnished 3-methyl-5,6,7,8-tetrahydro-1 naphthol, m.p. 98° (yield 80%) identical in every respect with the cyclized product IV.

Subsequent to our preliminary publication[5] of the above results, preparations of the compounds VII, VIII, and IX have been described by Cooke and Dowd[6] and Davies and Roberts[7] using almost identical procedures.

Synthesis of 3-methyl-1-naphthol. This compound has been prepared by various workers[4] by more or less roundabout methods. Our method[8] for the synthesis of this compound consists of four steps

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[3] L. G. Jupp, G. A. R. Kon, and E. H. Lockton, *J. Chem. Soc.*, 1638 (1928).

[4] M. Tishler, L. F. Fieser, and N. L. Wendler, *J. Am. Chem. Soc.*, 62, 2866 (1940); J. Cason, *J. Am. Chem. Soc.*, **63**, 828 (1941).

[5] D. K. Datta and P. Bagchi, *Sci. and Culture (Calcutta)*, **18**, 95 (1952).

[6] R. G. Cooke and H. Dowd, *Australian J. Chem.*, **6**, 53 (1953).

[7] J. E. Davies and J. C. Roberts, *J. Chem. Soc.*, 2173 (1956).

[8] D. K. Datta and P. Bagchi, *Sci. and Culture (Calcutta)*, **18**, 243 (1952).

only which are easily carried out. Benzyl methyl ketone prepared according to the method of Julian and Oliver[9] was condensed with ethyl bromoacetate under Reformatsky conditions to give ethyl γ-phenyl-β-methyl-β-hydroxybutyrate (XI, yield 97%). Dehydration of this compound with phosphorus oxychloride in pyridine yielded an unsaturated ester (XII, R = C_2H_5; yield almost quantitative), which furnished on hydrolysis with ethanolic alkali an acid (XIII, R = H; yield 77%) which partially crystallized. The crystalline acid, m.p. 113°, showed the following ultraviolet absorption characteristics: $\lambda_{max.}$ 249 mμ (log ε 4.1), proving it to be an open-chain styrene derivative,[10] and hence to possess the structure XIV. The absorption properties of the gummy portion indicate the presence of both bond isomers in it, since a considerable amount of absorption is observed between 220 and 250 mμ. When the mixture of acids (XIII, R = H) was cyclized with phosphorus oxychloride in tetrachloroethane, 3-methyl-1-naphthol, m.p. 92°, was obtained in 28% yield. This product did not show any melting point depression on admixture with the product V described above.

EXPERIMENTAL

All melting and boiling points are uncorrected.

Cyclohexenyl acetone. To a cooled solution of sodium (23 g., 1 atom) in dry ethanol (300 ml.), a cold mixture of cyclohexanone (98 g., 1 mole) and ethyl acetoacetate (130 g., 1 mole) was added with shaking. After keeping overnight, the solution was refluxed for 24 hr. Water (300 ml.) was added and the resulting mixture was refluxed again for 3 hr. The reaction mixture was then acidified with cold hydrochloric acid and worked up as usual, and the resulting oil was finally distilled. The fraction boiling at 60–110°/15 mm. was collected. The product (66 g.) was carefully fractionated and collected at 58–65°/3 mm.; yield 46 g. *Semicarbazone,* m.p. 144–145° (lit.,[3] m.p. 145°). Under atmospheric pressure, the ketone could be distilled at 195–205° without much decomposition; λ_{max}^{alc} 235–245 mμ (log ε 2.8).

Ethyl γ-(Δ^1-cyclohexenyl)-β-methyl-β-hydroxybutyrate (I). A mixture of cyclohexenylacetone (27.6 g., 0.2 mole), ethyl bromoacetate (50 g., 0.3 mole), and activated zinc wool (26 g., 0.4 atom) in dry benzene (160 ml.) was heated on a water bath, when a vigorous reaction set in. After the initial vigor had subsided, the reaction mixture was refluxed for 2 hr. The greenish-yellow liquid was decanted from the unchanged zinc, acidified strongly with cold hydrochloric acid and shaken vigorously. The benzene solution was then separated and washed consecutively with water, dilute ammonia, and water. The solvent was removed, and the residue distilled to yield 24 g. (53%) of I, b.p. 139–142°/3 mm., n_D^{32} 1.4846.

Anal. Calcd. for $C_{13}H_{22}O_3$: C, 69.0; H, 9.7. Found: C, 69.3; H, 9.6.

Dehydration to the dienic ester (II, R = C_2H_5). To a boiling solution of the hydroxy ester (I, 18 g., 0.08 mole) in dry benzene (125 ml.), phosphorus pentoxide (17 g., 0.12 mole) was added in three portions during 1 hr. Thereafter, the mixture was refluxed for 2.5 hr. The benzene layer was decanted and the dark-brown residue was extracted three times with boiling benzene. The pentoxide was then decomposed carefully with cold water and extracted with benzene. The combined benzene layer was washed with water and fractionated to yield 14.5 g. (87%) of the dienic ester, b.p. 125–132°/3 mm.; n_D^{32} 1.4903.

Anal. Calcd. for $C_{13}H_{20}O_2$: C, 75.0; H, 9.6. Found: C, 74.8; H, 9.5.

Hydrolysis to the dienic acid (III. R = H). The dienic ester (II, 10.4 g., 0.05 mole) was added to a solution of potassium hydroxide (5.6 g., 0.1 mole) in methanol (60 ml.) and the resulting solution was refluxed for 4 hr. and then evaporated on the water bath. The dark viscous residue was dissolved in water from which any unsaponified matter was extracted with ether. On acidification of the alkaline solution with cold hydrochloric acid, a dark brown oil separated which was extracted with benzene. After washing the benzene solution with water, the solvent was evaporated *in vacuo,* and the dienic acid (*ca.* 8 g.) was obtained as a thick liquid which failed to crystallize.

3-Methyl-5,6,7,8-tetrahydro-1-naphthol (IV). To a boiling solution of the dienic acid (III, 8 g., 0.045 mole) in dry benzene (120 ml.), phosphorus pentoxide (12.7 g., 0.09 mole) was added in three small portions during 1.5 hr. After refluxing for 2.5 hr., the reaction mixture was worked up as usual. The benzene solution was then evaporated and the residue was refluxed with a solution of potassium hydroxide (5 g.) in methanol (45 ml.) for 1 hr. The residue obtained on evaporating the solution was dissolved in water and acidified with dilute hydrochloric acid at 0°, when an oil separated which was extracted with ether. The ethereal layer was washed successively with water, 5% sodium bicarbonate solution and water and dried over anhydrous sodium sulfate. Distillation gave 1.4 g. of a light-yellow thick liquid, b.p. 127–132°/2 mm., which solidified on cooling. After removing the oily material on a porous tile, the solid melted at 97–98°. The solid was crystallized from petroleum ether (40–60°) to yield colorless long needles of the tetrahydronaphthol, m.p. 98.5°; the melting point was not raised on further crystallization.

Anal. Calcd. for $C_{11}H_{14}O$: C, 81.4; H, 8.6. Found: C, 81.2; H, 8.5.

The naphthol produced a deep violet coloration with a drop of ferric chloride in ethanolic solution.

3-Methyl-1-naphthol (V). The mixture of tetrahydronaphthol (IV, 0.5 g.) and palladium-charcoal (0.3 g., 30%) was taken in a small dehydrogenation apparatus, and heated on a metal bath initially kept at a temperature of 220°, and finally at 300°. Carbon dioxide gas was swept through the system and the evolved hydrogen was collected and measured by the displacement of a 40% solution of potassium hydroxide. After heating for 4 hr., the calculated volume of hydrogen was liberated. The residue was extracted with chloroform. The solvent was removed and the residue was evaporatively distilled at 160–165° (bath temperature)/40 mm. The solid distillate was crystallized from petroleum-ether (40–60°) to yield the naphthol as pale-yellow flakes, m.p. 91–92° [lit.,[4] m.p. 91–92°].

Anal. Calcd. for $C_{11}H_{12}O$: C, 82.5; H, 7.5. Found: C, 82.4; H, 7.3.

γ-(2-Hydroxy-4-methylphenyl)butyric acid (VII, R = H). To amalgamated zinc (from granulated zinc, 21.4 g., mercuric chloride, 2.14 g., water, 36 ml. and concd. hydrochloric acid, 1 ml.) was added successively water (14 ml.), concd. hydrochloric acid (31 ml.), benzene (36 ml.), and β-(2-hydroxy-4-methylbenzoyl)propionic acid (10.4 g., 0.05 mole). The mixture was gently refluxed over a free flame for 30 hr., adding concd. hydrochloric acid (12-ml. portions) every 6 hr. After decanting the liquid, the benzene layer was separated and the aqueous portion extracted with ether. From the combined organic extract, the solvent was removed and the residue was triturated with a few drops of glacial acetic acid to yield VII as a white powder, weighing 9.6 g. (99%), m.p. 77°. Two crystallizations from benzene-petroleum ether gave white flakes, m.p. 80.5°.

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(9) P. L. Julian and J. J. Oliver, *Org. Syn.,* **Coll. Vol. II,** p. 391.

(10) M. A. Ramart-Lucas and P. Amagat, *Bull. Soc. Chim.,* [4] **51,** 119 (1932).

Anal. Calcd. for $C_{11}H_{14}O_3$: C, 68.0; H, 7.2. Found: C, 68.1; H, 7.1.

γ-(2-Methoxy-4-methylphenyl)butyric acid (VIII, R = CH_3). The hydroxy acid (VII, 8 g., 0.04 mole) was methylated with dimethyl sulfate (15 g., 0.12 mole) in a 10% solution of sodium hydroxide (10 g., 0.24 mole) in the usual manner. After extracting the alkaline solution with ether, it was acidified with cold hydrochloric acid and the precipitate was filtered. The solid weighed 8.3 g. (almost quantitative). The methoxy acid crystallized from light petroleum ether (40–60°) as white flakes, m.p. 53°; the melting point was not raised on further recrystallization.

Anal. Calcd. for $C_{12}H_{16}O_3$: C, 69.2; H, 7.6. Found: C, 68.9; H, 7.3.

1-Methoxy-3-methyl-5-keto-5,6,7,8-tetrahydronaphthalene (IX). To a solution of the methoxy acid (VIII, 6 g.) in dry tetrachloroethane (120 ml.) was added phosphorus oxychloride (3 ml.) dropwise with shaking and the mixture was boiled for 2.5 hr. Water was added and the tetrachloroethane was distilled in steam. The semisolid residue was dissolved in ether, the ethereal layer was washed successively with water, 5% sodium carbonate solution and water, and dried over sodium sulfate. Distillation gave 3.5 g. (64%) of a pale-yellow liquid, b.p. 132–136°/2 mm., $n_D^{32.5}$ 1.5596.

Anal. Calcd. for $C_{12}H_{14}O_2$: C, 75.8; H, 7.3. Found: C, 75.9; H, 7.5.

The *2,4-dinitrophenylhydrazone* crystallized from glacial acetic acid as bright-red needles, m.p. 229–230°, unchanged on further crystallization.

Anal. Calcd. for $C_{18}H_{18}N_4O_4$: N, 15.1. Found: N, 15.1.

1-Methoxy-3-methyl-5,6,7,8-tetrahydronaphthalene (X). A solution of the tetralone (IX, 3.6 g.) in diethylene glycol (10 ml.) was reduced according to the Huang-Minlon method[11] using potassium hydroxide (3.8 g. , diethylene glycol (20 ml.), and 50% solution of hydrazine hydrate (5.6 ml.). After distilling the reaction mixture in steam, the distillate was extracted with ether, the ethereal layer was dried over anhydrous sodium sulfate, and the solvent was reremoved. The residue was distilled to yield 2.1 g. (60%) of a colorless mobile oil, b.p. 106–108°/3 mm., $n_D^{32.5}$ 1.5388.

Anal. Calcd. for $C_{12}H_{16}O$: C, 81.8; H, 9.1. Found: C, 82.2; H, 9.1.

3-Methyl-5,6,7,8-tetrahydro-1-naphthol (IV). A solution of the methyl ether (X, 1.6 g.) in glacial acetic acid (30 ml.) and 48% hydrobromic acid (12 ml.) was refluxed on an oil bath kept at 130° for 6 hr. The cooled solution was then poured into ice water and the pink solid was filtered. The naphthol weighed 1.2 g. (80%), m.p. 94°. The melting point rose to 98° after crystallization from petroleum ether (40–60°) and remained undepressed on admixture with the sample obtained previously through cyclization.

Ethyl γ-phenyl-β-methyl-β-hydroxybutyrate (XI). The Reformatsky reaction was carried out as before using benzyl methyl ketone (20 g., 0.15 mole), ethyl bromoacetate (31.4 g., 0.188 mole), activated zinc wool (19.5 g., 0.3 atom), and dry benzene (180 ml.). Distillation gave 32 g. (97%) of a colorless oil, b.p. 140–146°/3 mm., n_D^{22} 1.4994.

Anal. Calcd. for $C_{13}H_{18}O_3$: C, 70.2; H, 8.1. Found: C, 70.2; H, 8.2.

Dehydration to the unsaturated ester (XII, R = C_2H_5). To a solution of the β-hydroxy ester (XI, 8 g., 0.036 mole) in dry pyridine (18 ml.) cooled in ice, was added phosphorus oxychloride (2.8 g., 0.018 mole) dropwise with shaking. After allowing to stand overnight the mixture was warmed on the steam bath for 1 hr. and acidified with cold dilute hydrochloric acid. The organic matter was extracted with ether, the ethereal layer was separated, washed with a solution of sodium bicarbonate and water, and dried over sodium sulfate. On distillation, 7.5 g. (99%) of XII, b.p. 124–126°/1.5 mm. was obtained as a colorless oil, $n_D^{27.5}$ 1.5090.

Anal. Calcd. for $C_{13}H_{16}O_2$: C, 76.4; H, 7.8. Found: C, 76.6; H, 7.7.

Saponification to the unsaturated acids (XIII, R = H, and XIV). To a solution of potassium hydroxide (4.2 g., 0.074 mole) in ethanol (50 ml.), the unsaturated ester (XII 7.5 g., 0.037 mole) was added, and the resulting solution was refluxed for 5 hr., and then worked up as usual. Distillation gave 5 g. (77%) of the acidic material, b.p. 156–162°/3 mm. The distillate solidified to a white mass admixed with an oily material. A portion of the solid was dissolved in petroleum ether (40–60°) from which crystals were deposited, m.p. 110°. Recrystallization from the same solvent yielded the acid (XIV) as white flakes, m.p. 113°; λ_{max}^{alc} 249 mμ (log ε 4.1).

Anal. Calcd. for $C_{11}H_{12}O_2$: C, 75.0; H, 6.8. Found: C, 74.8; H, 6.6.

3-Methyl-1-naphthol (V). Phosphorus oxychloride (2 ml.) was added to a solution of the acid (XIII, 4 g.) in dry tetrachloroethane (80 ml.) and the solution was refluxed for 2.5 hr., and then distilled in steam. The residue was dissolved in ether, the ethereal layer washed with water, and evaporated. The residue was hydrolyzed with a solution of potassium hydroxide (3 g.) in ethanol (40 ml.) and worked up as usual. On distillation, the product (1 g.), b.p. 140–144°/3 mm., solidified to a yellow mass, m.p. 89–90°. Crystallization from petroleum ether (60–80°) raised the melting point to 92°, which was not depressed on admixture with the sample obtained previously.

JADAVPUR, CALCUTTA 32, INDIA

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(11) Huang-Minlon, *J. Am. Chem. Soc.*, **68**, 2487 (1946).

[CONTRIBUTION FROM THE CHEMICAL THERAPEUTICS RESEARCH LABORATORY, MILES LABORATORIES, INC.]

Spiro[cyclohexane-1,9′-fluoren]-4-one and Some 4-Amino Derivatives

DALE A. STAUFFER AND OTIS E. FANCHER

Received December 10, 1959

Spiro[cyclohexane-1,9′-fluoren]-4-one has been synthesized by two methods and a number of 4-amino derivatives have been prepared for pharmacological examination. The compounds showed no outstanding activity.

Spiro[cyclohexane-1,9′-fluoren]-4-one (V) was obtained by two different series of reactions, one starting with 9,9-fluorenedipropionitrile (I).[1] This was cyclized in the presence of metallic sodium and a small quantity of potassium metal to give 4-iminospiro[cyclohexane-1,9′-fluorene]-3-carbonitrile in good yield. The imino compound (II) was hydrolyzed readily to the cyanoketone (III) which, on refluxing with a mixture of acetic and hydrochloric acids, was hydrolyzed with the sub-

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(1) H. A. Bruson, *J. Am. Chem. Soc.*, **64**, 2457 (1942).