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2-HYDROXYCYCLOHEXANEACETIC ACID LACTONES AS INTERMEDIATES IN THE SYNTHESIS OF HYDROGENATED PHENANTHRENES

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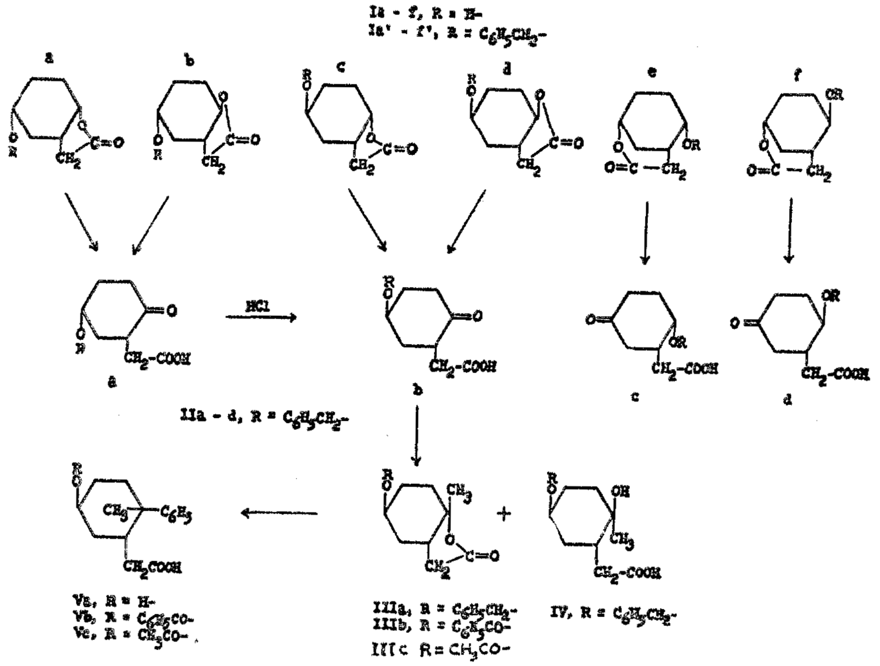
As an intermediate in the synthesis of compounds related to the steroid nucleus, 2,5-dihydroxy-2-methylcyclohexaneacetic acid γ-lactone is attractive in that it appears to offer at least three approaches to the hydrogenated phenathrenes. It should prove to be convertible by conventional methods to a vinylcyclohexene for use in the Diels-Alder condensation; this convenient route to substances related to the steroid nucleus received attention some years ago (1), and is currently of interest again (2). Secondly, the action of an aromatic Grignard reagent on the lactone would be expected to give a substance of a type similar to known compounds which are readily convertible to octahydrophenanthrenes (3). A third possible approach, that utilizing a cyclic lactone in the Friedel-Crafts reaction, was reported by Ghosh (4), but his work became the subject of controversy because of his claim that both 2-hydroxycyclohexaneacetic acid lactone and cyclohexeneacetic acid gave 2-phenylcyclohexaneacetic acid when condensed with benzene. Others showed that the cyclohexeneacetic acid type compound is substituted predominately in the 4-position (5). However, the reported result of the condensation with the lactone was not disputed and Gutsche and Johnson (6) have clarified the confused situation with respect to the other condensation. The present paper is solely concerned with the chemistry of the dihydroxycyclohexaneacetic acid derivatives and the Friedel-Crafts condensation of a derived lactone with benzene.

Homogentisic acid lactone was converted to its ethyl ester and the benzene nucleus was hydrogenated. Acid hydrolysis followed by distillation converted the stereoisomeric mixture of 2,5-dihydroxycyclohexaneacetic acids into a lactone mixture which could consist of the six forms shown in the chart (two *cis*-γ-, two *trans*-γ-, and two *cis*-δ-lactones; a model indicates the *trans*-δ-lactones could not form). Separation at this point was not attempted because of the apparent physical similarity of the members. Benzylation of the free hydroxyl group resulted in a mixture which could be separated into two crystalline components and an oil, all three of which consisted of isomeric lactones. Hydrolytic opening of the lactone ring followed by oxidation of the alcohol to a ketone eliminated one asymmetric center in each and reduced the number of structural possibilities to the four ketones shown on the chart. One liquid and one crystalline ketone were obtained and the liquid isomer could be converted to the solid by trituration in 9 *N* hydrochloric acid. As there is no asymmetric center in IIc and IId which could be epimerized by this treatment it follows that the ketones may be represented as IIa and IIb, and that the original lactone mixture (Ia–f) is predominately a mixture of γ-lactones. Treatment of the solid ketone with the methyl Grignard reagent followed by relactonization in boiling benzene gave the *cis*-lactone IIIa and the acid IV in nearly equal amounts. Because of the lability of the benzyl group in the Friedel-Crafts reaction it was necessary to replace it with an acyl residue, and it was found that this transformation could be effected in benzene solution by benzoyl chloride and by acetyl chloride in the presence of stannic chloride; subsequent addition of aluminum chloride brought about the addition of benzene to give two isomers, one in very small yield. The predominant isomer is represented in the chart with an indeterminate configuration at the quaternary carbon (Vb). The benzoyl group on Vb was removed by alkaline hydrolysis and an acid melting at 175° obtained. This product could be melted and sublimed without change at 190° and, as a molecular model reveals no obvious reason that a *cis*-δ-lactone could not form under this rigorous condition, the relative configuration of the hydroxyl group and the acetic acid residue will tentatively be called *trans*.[1] Conclusive proof might be had by applying the synthesis to the isomeric ketone IIa.



It is conceivable that the replacement of the benzyl by an acyl group involves a rearrangement and this possibility was investigated. Acetylation of the alcohol derived from IIIa by removing the benzyl group with hydrogen yielded a com-

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[1] A reviewer has kindly pointed out that of the two isomers IIa and IIb the *cis* form IIa is probably the more stable due to the substituents being able to assume the equatorial conformation. However the solubility of the liquid and the insolubility of the crystalline form in the rearrangement medium would insure transformation into the solid regardless of which form is thermodynamically the more stable.

pound identical with that obtained from the benzyl ether by means of acetyl chloride and stannic chloride. On the other hand, benzoylation of the alcohol yielded an unidentified oil. This negative result was shown to have no bearing on the question of inversion. Acetylation of Va, obtained from Vb by hydrolysis, gave the same acetoxy derivative as that from IIIc by condensation with benzene.

Both of the crystalline isomers of the benzyl ether I, after hydrolysis were rapidly reconverted in acid suspension to the lactones. The liquid benzyl ether yielded an acid which resisted this treatment. Hence it is possible to assign *cis*-structures to the two crystalline ethers and a *trans*-structure to the liquid mixture. Furthermore, the crystalline ether melting at 79° yielded the solid ketone while the other ether melting at 102° gave the liquid ketone. Thus it is possible to assign the tentative structure Ic′, *trans*-5-benzyloxy-*cis*-2-hydroxycyclohexaneacetic acid γ-lactone to the ether melting at 79° and Ia′, *cis*-5-benzyloxy-*cis*-2-hydroxycyclohexaneacetic acid γ-lactone to the ether melting at 102°. Because the liquid ether yields both the solid and liquid ketones and appears to resist lactonization it probably is a mixture of Ib and Id.

The formation of δ-lactones in the heat lactonization of 2,5-dihydroxycyclohexaneacetic acid has not been ruled out. Although both the acids and the lactones are water-soluble oils, a considerable amount of oil insoluble in water but soluble in sodium hydroxide solution was left in the distilling flask in which the acid lactonized. This material could be forced to distil on raising the temperature of the bath. The distillate, a water-soluble oil, could be redistilled at the lower temperature range of the main product. This behavior suggests a rearrangement to the γ-lactone and possibly involves a δ-lactone and/or a mixed ester.

Cyclization of either 5-benzoxy- or 5-acetoxy-2-methyl-2-phenylcyclohexaneacetic acid (Vb, Vc) *via* the acid chloride in the presence of stannic chloride probably yielded the corresponding 2-acyloxy-1,2,3,4,4a,9,10,10a-octahydro-9-keto-4a-methylphenanthrene, the skeleton of which was suggested by the isolation of phenanthrene from the products of a palladium-charcoal dehydrogenation of the alcohol resulting from reduction of the ketone.

Hydroquinone dimethyl ether was used in attempted Friedel-Crafts condensations with the lactone IIIb but the results were inconclusive. The intended compound 2-benzoxy-1,2,3,4,4a,9,10,10a-octahydro-9-keto-5,8-dimethoxy-4a-methylphenanthrene is closely related to 4,4a,9,10-tetrahydro-5,8-dimethoxy-4a-methyl-2(3*H*)-phenanthrone, which was synthesized by Grob, Jundt, and Wicki (7) in part by the Robinson-Mannich method.

Acknowledgment. The interest and encouragement of Dr. Erich Mosettig substantially aided the work reported herein and is gratefully acknowledged.

EXPERIMENTAL[2]

2,5-Dimethoxyphenylacetic acid. The procedure used was essentially that of Abbott and Smith (8). *p*-Dimethoxybenzene (500 g.) was acetylated to give 567 g. (87%) of crude 2,5-dimethoxyacetophenone, distilling at 135–150° at 8 mm. with the major portion distilling at

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[2] All microanalyses are from the Institutes service analytical laboratory under the direction of Dr. William C. Alford.

140–143°. The Willgerodt reaction was carried out substantially as reported with the exception that the crude reaction mixture in ethereal solution was extracted with 6 *N* hydrochloric acid to remove morpholine and then with sodium hydroxide to remove acidic material. The alkaline hydrolysis required 27 hours. After removal of the alcohol an unidentified solid formed and was removed with ether. Acidification precipitated the solid acid which, after washing with alcohol and with benzene, melted at 120–123°. An additional ten percent was recovered from the washings. In this way 497 g. of the ketone yielded 366 g. (67%) of 2,5-dimethoxyphenylacetic acid.

Homogentisic acid ethyl ester. A solution of 382 g. of 2,5-dimethoxyphenylacetic acid in 840 ml. of 48% hydrobromic acid was refluxed under a distillation column so that water could be removed. Methyl bromide was rapidly evolved and 250 ml. of an aqueous acid distillate was collected in 70 minutes. The solution was cooled and the precipitated crystalline lactone was filtered and washed with alcohol. A little sulfur was mechanically removed. The mother liquor was concentrated on the steam-bath under reduced pressure and a second crop obtained. The crude lactone weighed 271.5 g. after drying in an oven at 110°. The lactone was converted to the desired ester by suspending it in 500 ml. of absolute ethanol and passing in dry hydrogen chloride until the solid dissolved in the hot solution. After removing the alcohol-hydrogen chloride under reduced pressure the product was recrystallized from toluene (600 ml.). The ester weighed 340 g. (89%) and melted 115–117°. An additional 2 g. was obtained by esterifying the 16 g. of dark oily material remaining from the HBr hydrolysis and lactonization. This ester has been reported to melt at 119–120° (9).

2,5-Dihydroxycyclohexaneacetic acid γ-lactone (Ia–d). Homogentisic acid ethyl ester (36 g.), m.p. 116–118° in 35 ml. of absolute ethanol along with approximately 22 g. of moist (absolute ethanol) Raney nickel was reduced with hydrogen at 3000 p.s.i. at 150°. The reduction was virtually complete in one hour. After the catalyst was filtered, 30 ml. of 3 *N* hydrochloric acid was added and the solution was refluxed in order to hydrolyze the ester. Alcohol was removed by distillation through a column and water was added toward the end of the 1½ hour heating period to prevent the solution from becoming too concentrated. After removal of the aqueous hydrochloric acid under reduced pressure the product was distilled at 150–190° at 1 mm. On redistillation the range was narrowed to 150–160° at 1 mm. and yielded 25.3 g. (89%) of a colorless viscous water-soluble oil. The distillation temperature is rate-sensitive and can be lowered to *ca.* 135° in a slow distillation. It was observed that the last of the crude material to distil was a very heavy oil, insoluble in water but soluble in sodium hydroxide. It yielded the lighter-bodied water-soluble oil on heating and this phenomenon accounts for the higher vapor temperature at the end of the first distillation. Reductions using slightly impure material (m.p. 115–117°) or old catalyst were incomplete, the best results being obtained with ester recovered from an incomplete reduction and freshly prepared catalyst. The need for pre-treatment with catalyst is thereby indicated.

Anal. Calc'd for $C_8H_{12}O_3$: C, 61.52; H, 7.75.
Found: C, 61.59; H, 7.91.

5-Benzyloxy-2-hydroxycyclohexaneacetic acid lactone (Ia′–d′). Dry triethylamine (120 ml.) was slowly added to a solution of 82 g. of 2,5-dihydroxycyclohexaneacetic acid lactone and 126 g. of benzyl bromide in 100 ml. of chloroform. The temperature was not permitted to rise above 50° so as not to lose amine. After the exothermic reaction ceased and all the amine had been stirred into the solution, the temperature was slowly raised to 180° with the loss of the chloroform and excess amine; some red coloration developed at this time. The mixture was held at this temperature (±5°) for three hours, cooled, and then water and ether were added to dissolve amine hydrochloride and the benzyl ether respectively. One of the solid benzyl ethers crystallized from this mixture and was recovered by filtration. It weighed 21.9 g. The oily portion was distilled at 0.6 mm. and the middle fraction boiling in the range 160–200° was collected. It weighed 77.7 g. to give a total crude yield of 99.6 g. (77%). When a solution of the liquid isomer mixture in ether was chilled in a Dry-Ice acetone bath more of the solid isomer crystallized. The total amount of crystalline isomer, m.p. 99–102°, was 23.3 g. (18%). After further purification from alcohol it melted at 100–102°. A second crystalline isomer was obtained in small amount by first hydrolyzing the liquid lactone mixture with a solution of sodium hydroxide, acidifying with hydrochloric acid to precipitate the organic acids as an oil, and then triturating the oil in 6 *N* hydrochloric acid for ten minutes. About half of the material relactonized to give a solid which was separated by chromatographic absorption on alumina followed by fractional crystallization from alcohol into the above-described compound and another isomer melting at 78–79°. The liquid lactone mixture taken for analysis distilled at 167–177° at 0.6 mm.

Anal. Calc'd for $C_{15}H_{18}O_3$: C, 73.15; H, 7.37.

Found for solid, m.p. 100–102°: C, 72.97; H, 7.53.

Found for solid, m.p. 78–79°: C, 73.38; H, 7.19.

Found for liquid: C, 73.37; H, 7.51.

Relactionization of 5-benzyloxy-2-hydroxycyclohexaneacetic acid isomers. Both of the solid isomers were hydrolyzed in sodium hydroxide and the corresponding acids were recovered with ether from the acidified solutions. Both acids relactonized in less than ten minutes on triturating in a 6 *N* hydrochloric acid suspension at room temperature giving the original lactones. The acids derived from the liquid lactone resisted lactonization under these conditions.

5-Benzyloxy-2-ketocyclohexaneacetic acid (IIa–b). (*a*) *From the lactone melting at 100–102°.* 5-Benzyloxy-2-hydroxycyclohexaneacetic acid lactone (25.3 g.), m.p. 99–101°, was heated on the steam-bath with 95 ml. of 2.16 *N* sodium hydroxide (twice theory), and stirred until a solution was obtained. Then 56 g. of monobasic potassium phosphate was dissolved in the hot solution, whereupon two layers separated. The mixture was *p*H 6 by Accutint paper. N-Bromosuccinimide, 20.3 g., was added to the rapidly stirred mixture at 37°. In six minutes the temperature rose to 50° where it was maintained by intermittent cooling. After 18 minutes the yellow suspension suddenly became white, and a starch-iodide test was negative. The mixture was *p*H 5.3 by Accutint paper. The organic oil was dissolved in ether except for a portion which seemed to be a salt. On adding hydrochloric acid the remaining oil dissolved and the ethereal solution became lighter in color. The ketone was recovered as a yellow oil and yielded a *semicarbazone* which was purified from glacial acetic acid, m.p. 179–180.5° (gas).

Anal. Calc'd for $C_{16}H_{21}N_3O_4$: N, 13.16. Found: N, 13.03.

The oily ketone would not crystallize on seeding with its solid isomer (described below). It was dissolved in 50 ml. of 9 *N* hydrochloric acid at room temperature. Heavy crystallization took place in a few minutes. After two hours the acid was diluted and the product was filtered and washed with water. After freeing from oil by washing with ether chilled in an acetone-Dry Ice bath it was purified from dilute alcohol, m.p. 107–111°, weight 17.0 g. (63%). The analytical sample melted at 109–111°.

Anal. Calc'd for $C_{15}H_{18}O_4$: C, 68.68; H, 6.92.

Found: C, 68.61; H, 7.06.

A *semicarbazone* was prepared and purified from alcohol. It melted at 180–182° (gas); a mixture with the isomeric semicarbazone melted at 178–180° with a slight sinter beginning at 176°. [The three samples were melted side-by-side].

Anal. Calc'd for $C_{16}H_{21}N_3O_4$: N, 13.16. Found: N, 13.11.

(*b*) *From the lactone melting at 78–79°.* A sample of the isomer, m.p. 77–79°, weighing 0.17 g. was hydrolyzed and oxidized in the manner described above. The purified product weighed 0.12 g. (66%) and melted at 109–111°. The melting point was not depressed on admixture with the above-described analytical sample.

(*c*) *From the liquid lactone.* A mixture of the solid and liquid ketones was obtained in a similar oxidation of the hydrolysis product of the liquid lactone. Trituration of the crude mixture with 9 *N* hydrochloric acid yielded 60% of the ketone, m.p. 103–109°. After further purification it proved identical with the above ketone.

5-Benzyloxy-2-hydroxy-2-methylcyclohexaneacetic acid lactone (IIIa). Solid 5-benzyloxy-2-ketocyclohexaneacetic acid (40 g.) was added portionwise to 285 ml. of 1.8 *N* CH_3MgI solution held between −15° and 0°. Gas was liberated and the solid dissolved. Toward the end an oil separated and the solid did not react as easily. After standing an hour at 7° the mixture was poured onto ice and the magnesium hydroxide was dissolved in hydrochloric acid. The ethereal solution was evaporated on the steam-bath until the temperature of the oily product was 88°; 100 ml. of benzene was added and the solution was refluxed 30 minutes during which time 3.5 ml. of water was collected in a trap. The benzene solution was extracted with sodium carbonate solution, the organic acid was recovered by acidification, and the mixture was held on the steam-bath an additional 1.5 hours. The very heavy bodied yellow-orange oil (IV) weighed 19.1 g. (45%). Its sodium bicarbonate solution was slightly cloudy. The acid was held at 180° under reduced pressure until the pressure dropped to a constant 0.6 mm. This required 20 minutes. It was then distilled at 182–184°/0.6 mm. The distillate was no longer soluble in sodium carbonate solution but could be dissolved in hot sodium hydroxide solution. The analysis indicates incomplete dehydration to the lactone.

Anal. Calc'd for $C_{16}H_{20}O_3$: C, 73.82; H, 7.74.
Found: C, 73.22; H, 7.68.

The sodium carbonate-insoluble portion of the product was recovered from the benzene solution and was distilled at 160–170° at 0.4 mm. A small forerun distilled from 150°, total weight 19.9 g. (50%). This compound is designated as the *cis*-lactone in the following experiments.

Anal. Calc'd for $C_{16}H_{20}O_3$: C, 73.82; H, 7.74.
Found: C, 73.96; H, 7.87.

5-Benzoxy-2-hydroxy-2-methylcyclohexaneacetic acid cis-lactone (IIIb). To a solution of 1.0 g. of 5-benzyloxy-2-hydroxy-2-methylcyclohexaneacetic acid *cis*-lactone (IIIa) in 2 ml. of dry benzene was added 0.3 ml. of stannic chloride. The temperature rose from 24° to 40° and an additional 0.5 ml. caused no further temperature rise. The solution was chilled to 10° and 0.5 ml. of benzoyl chloride was added with a temperature rise to 32°. In five minutes ice was added to the solution and the separated oil was stirred until it dissolved in the benzene. The benzene solution was separated and washed with water and with sodium carbonate solution. The solvent was removed on the steam-bath under reduced pressure and 30–60° petroleum ether was added to the oil which thereupon crystallized. It was subsequently purified by triturating in ether chilled in Dry Ice-acetone; weight, 0.6 g. (57%), m.p. 80–83°. After further purification from dilute alcohol it melted at 82–84°.

Anal. Calc'd for $C_{16}H_{18}O_4$: C, 70.05; H, 6.62.
Found: C, 70.26; H, 6.65.

An unsuccessful attempt was made to get this compound by benzoylating the alcohol obtained by removing the benzyl group with hydrogen.

5-Acetoxy-2-hydroxy-2-methylcyclohexaneacetic acid cis-lactone was prepared from the benzyloxy compound in the manner described above but the yield was only 29%. Purified from dilute ethanol it had m.p. 61.5–63°.

Anal. Calc'd for $C_{11}H_{16}O_4$: C, 62.25; H, 7.60.
Found: C, 62.49; H, 7.33.

A 40% over-all yield was obtained on acetylating the alcohol derived from the benzyloxy compound by replacing the benzyl group with hydrogen. This was accomplished in alcohol solution at room temperature using a 5% palladium-on-charcoal catalyst which was fortified by the addition of a little 5% palladium chloride solution.

5-Benzoxy-2-methyl-2-phenylcyclohexaneacetic acid (Vb). A solution of 1.77 g. of 5-benzoxy-2-hydroxy-2-methylcyclohexaneacetic acid *cis*-lactone (IIIb) in 3 ml. of dry benzene was added intermittently to an ice-cold suspension of 3.5 g. of aluminum chloride in 5 ml. of dry benzene. The oily complex which first formed dissolved on stirring. The temperature was kept below 15° during the time it took the oil to dissolve, then was allowed to come to room temperature. After ten minutes at room temperature the solution (some excess solid aluminum chloride present) was poured onto ice and the benzene was separated and extracted with sodium carbonate solution. The crude crystalline product, weight 1.95 g. was recovered after acidification of the salt solution. Fractional crystallization from dilute ethanol yielded two fractions, the one, 1.54 g., m.p. 152–154°; and the other 0.05 g., m.p. 147–148°. A mixture of these compounds melted at 125–135°.

Anal. Calc'd for $C_{22}H_{24}O_4$: C, 74.98; H, 6.87.
Found for the compound melting 152–154°: C, 75.13; H, 6.92.
Found for the compound melting 147–148°: C, 75.07; H, 6.71.

5-Hydroxy-2-methyl-2-phenylcyclohexaneacetic acid (Va) was obtained in 90–93% yield by the action of hot sodium hydroxide on a solution of the sodium salt of the above-described benzoyl derivative (Vb). In one experiment in which the benzoyl derivative was not isolated the yield of the corresponding alcohol of m.p. 161–170°, was 68%, calculated from the lactone (IIIb). Purified from alcohol it had m.p. 174–176°.

Anal. Calc'd for $C_{15}H_{20}O_3$: C, 72.55; H, 8.12.
Found: C, 72.81; H, 8.14

The alcohol (50 mg.), m.p. 172–175°, was melted and sublimed at 190°/0.3 mm.; 43 mg. m.p. 173–176°, was recovered and shown to be unchanged by a mixture melting point determination, and by its solubility in sodium bicarbonate solution.

5-Acetoxy-2-methyl-2-phenylcyclohexaneacetic acid (Vc) was prepared in 96% yield by the action of acetic anhydride-pyridine on the alcohol Va for four hours at room temperature. It was purified by dissolving in ether, concentrating the solution, and then adding petroleum ether. The acid appeared to be dimorphous with the melting range 112–119° or 111–113°, depending upon the particular crystallization.

Anal. Calc'd for $C_{17}H_{22}O_4$: C, 70.32; H, 7.64.
Found: C, 70.00; H, 7.67.

5-Acetoxy-2-hydroxy-2-methylcyclohexaneacetic acid lactone (610 mg.) dissolved in one ml. of dry benzene was added slowly to a chilled suspension of 1.5 g. of aluminum chloride in 2 ml. of dry benzene. The complex slowly precipitated as a heavy oil which became friable in 15 minutes. After 30 minutes the mixture was decomposed with ice. The benzene layer was extracted with sodium carbonate solution and this on acidification yielded 0.1 g. of solid which, after purification as described above, melted at 113–114°. A mixture with the material obtained in the preceding preparation melted at 111–113.5°. There was recovered from the benzene solution 445 mg. of starting material.

2-Acetoxy-1,2,3,4,4a,9,10,10a-octahydro-9-keto-4a-methylphenanthrene. To a solution of 0.235 g. of 5-acetoxy-2-methyl-2-phenylcyclohexaneacetic acid (Vc) in 0.6 ml. of dry benzene was added 0.22 g. of phosphorus pentachloride. Gas was vigorously evolved and the temperature rose to 40°. The solution (excess phosphorus pentachloride present) was chilled to 5° and 0.5 ml. of stannic chloride was added. The temperature rose to 22° and a heavy oil precipitated. This oil crystallized after one-half hour and the suspension then stood overnight at room temperature. It was decomposed with ice and the benzene layer was separated and washed with water and extracted with sodium carbonate solution. Removal of the benzene left an oil which crystallized, wt. 0.195 g. (89%), m.p. 92–99°. After purification from dilute alcohol it melted at 99–100.5°.

Anal. Calc'd for $C_{17}H_{20}O_3$: C, 74.97; H, 7.40.
Found: C, 74.82; H, 7.54.

A *semicarbazone* was recrystallized from alcohol, m.p. 216–217° (gas).

Anal. Calc'd for $C_{18}H_{23}N_3O_3$: N, 12.76. Found: N, 12.49.

A solution of 0.8 g. of the phenanthrone in 4 ml. of 3 *N* aluminum isopropoxide was refluxed for 1¼ hours. The reduction product was recovered as an oil and was mixed with 0.8 g. of 5% palladium charcoal. The mixture was heated from 260° to 325° over a period of 5 hours during which time the tube was swept with a slow stream of carbon dioxide and the inert gas was measured roughly in a nitrometer (10). The catalyst was extracted with benzene and the crude product was distilled onto a cold finger. The crystalline material was recovered with alcohol, wt. 0.082 g. (15.6%) m.p. 94–96°. Purified further it melted at 96–97° and the melting point was not depressed on admixture of the sample with phenanthrene. Further identification was made through the picrates which proved identical in melting point and mixture melting point determinations.

2-Benzoxy-1,2,3,4,4a,9,10,10a-octahydro-9-keto-4a-methylphenanthrene was made from Vb in the same way as was the acetoxy derivative from Vc, with the exception that the reaction time was only one hour. The substance was obtained in 95% yield, m.p. 171–174°. When purified from alcohol it melted at 173.5–175°.

Anal. Calc'd for $C_{22}H_{22}O_3$: C, 79.01; H, 6.63.
Found: C, 79.28; H, 6.74.

SUMMARY

Derivatives of isomeric 2,5-dihydroxycyclohexaneacetic acids have been investigated and the structural relationships of these substances have been presented. A derived lactone was condensed with benzene in a Friedel-Crafts reaction and the product was subsequently converted to a 9-octahydrophenanthrone.

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