Its *2,4-dinitrophenylhydrazone* melted at 254.5–255.5° when crystallized from ethyl acetate.

Anal. Calcd. for $C_{27}H_{27}O_5N_5$: C, 60.78; H, 5.10. Found: C, 60.73; H, 5.23.

5,8-Dihydroxy-7-[1-(o-carboxamidophenyl)ethyl]-1-tetralone (XIII). A solution of 5.25 g. of the tetralone XII in 30 ml. of glacial acetic acid was treated with 30 ml. of 48% hydrobromic acid and 30 ml. of 57% hydroiodic acid and heated under reflux for 2 hr. Dilution with ice and water gave a bright yellow precipitate, 4.20 g. (87%), m.p. 246.5–247.5°. Several recrystallizations from alcohol did not raise the melting point. Infrared maxima 2.96, 3.08, 5.96 μ.

Anal. Calcd. for $C_{19}H_{19}O_4N$: C, 70.14; H, 5.89. Found: C, 70.04; H, 5.99.

5,8-Dihydroxy-7-[1-(o-carboxyphenyl)ethyl]-1-tetralone (XIV). A solution of 0.820 g. (0.0025 mole) of 5,8-dihydroxy-7-1-(*o*-carboxamidophenyl)ethyl-1-tetralone (XIII) in 15 ml. of 10% sodium hydroxide solution through which a stream of nitrogen had been passed to remove the oxygen was heated under nitrogen on a steam bath for 11 hr. The solution was carbonated and filtered rapidly, and the filtrate was immediately acidified with 3*N* hydrochloric acid. The precipitate was collected and washed with ether to give 0.650 g. (79%) of a dark yellow solid that did not melt, but decomposed at 255.0–260°. This was recrystallized from ethanol to give 0.450 g. of bright yellow material that decomposed at 265–267°. Infrared maxima broad, 5.96 to 6.00 μ.

Anal. Calcd. for $C_{19}H_{18}O_5$: C, 69.92; H, 5.56. Found: C, 69.92; H, 6.03.

5,12-Dihydroxy-1,6-dioxo-1,2,3,4,6,11-hexahydro-11-methylnaphthacene (XV). A mixture of 1.00 g. (0.00307 mole) of crude XIV and 25 g. of freshly opened polyphosphoric acid was heated at 55–60° with stirring for 2 hr., diluted with water, and extracted with three 30-ml. portions of benzene. The benzene solution was extracted with 50 ml. of 5% sodium bicarbonate solution, washed with water, dried, and concentrated to yield 0.800 g. (85%) of dark orange crystalline material, m.p. 139.0–142.0°. Recrystallization from alcohol gave 0.600 g. (63%), m.p. 142.0–144.0°. The analytical sample was recrystallized several times from a very small amount of ethyl acetate and melted at 148.5–149.0°. Infrared max. 6.08, 6.13, 6.16 μ. No absorption in the 3.0 μ region.

Anal. Calcd. for $C_{19}H_{16}O_4$: C, 74.01; H, 5.23. Found: C, 73.89; H, 5.23.

5,12-Diacetoxy-1,6-dioxo-1,2,3,4,6,11-hexahydro-11-methylnaphthacene. A mixture of 0.100 g. (0.000325 mole) of 5,12-dihydroxy-1,6-dioxo-1,2,3,4,6,11-hexahydro-11-methylnaphthacene and 0.5 ml. of acetic anhydride was treated with a drop of concentrated sulfuric acid. The mixture was stirred for 10 min., during which a yellow solid separated, and then diluted with water. After several recrystallizations from alcohol, the precipitated material melted at 194.0–195.0°. Infrared max. 6.04, 6.13 μ.

Anal. Calcd. for $C_{23}H_{20}O_6$: C, 70.40; H, 5.14. Found: C, 70.55; H, 5.10.

5,12-Dihydroxy-1,6-dioxo-1,2,3,4,6,11-hexahydro-11-methylnaphthacene-1-oxime (XXX). A solution of 0.100 g. (0.000325 mole) of XV and 0.025 g. (0.00036 mole) of hydroxylamine hydrochloride in 1 ml. of a 50–50 pyridine-alcohol was heated on a steam bath for 2 hr., and the solvent was then removed with a stream of nitrogen. The product was washed with water, collected, and recrystallized from benzene to give a solid of m.p. 196.0–196.5°. Although the melting point was sharp, two crystalline forms, one pale orange and the other dark orange, persisted through several further recrystallizations.

Anal. Calcd. for $C_{19}H_{17}O_4N$: C, 70.57; H, 5.30. Found: C, 70.82; H, 5.45.

ROCHESTER, N. Y.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF IOWA STATE COLLEGE]

Alkylation and Related Reactions of Dibenzo-*p*-dioxin *

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A number of alkyl derivatives of dibenzo-*p*-dioxin(I) have been prepared. These include the 2-(II), 2,7(8)-di-*t*-butyldibenzo-*p*-dioxin(III); 2-(IV), 2,3-di-(V), 2,3,7,8-tetra-(VI), and a hexaisopropyldibenzo-*p*-dioxin(VII); and 2-benzyldibenzo-*p*-dioxin(VIII). Also obtained in the study were ethyl dibenzo-*p*-dioxin-2-dithiocarboxylate(IX), methyl dibenzo-*p*-dioxin-2-dithiocarboxylate(X), diethyl dibenzo-*p*-dioxin-2,7-bis(dithiocarboxylate)(XI), 2-chloromethyldibenzo-*p*-dioxin (XII), and the dibenzo-*p*-dioxin-2-carboxaldehyde(XIII). A 2,4-dinitrophenylhydrazone was formed from XIII yielding (XIV).

The acylation of dibenzo-*p*-dioxin (I) is known to proceed under normal conditions to a mixture of the 2, the 2,7 and, in one case, the 2,8 substituted compounds.[1,2] It is also fairly easy to obtain exclusively the mono or the di products by employing special conditions.[3]

In an extension of the above work, a study was made of direct alkylation of dibenzo-*p*-dioxin using mainly *t*-butyl chloride and isopropyl chloride since these reagents presented the least possibility of isomerization. Ethyl chloride, ethyl bromide, and methyl iodide were also used, but failed, as has already been reported[4–6] for other compounds, to give the normal alkylation products in carbon disulfide.

A number of solvents were tried before the choice of carbon disulfide was made. *s*-Tetrachloroethane could not be used since it apparently reacted with

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* This paper is a contribution in honor of Lyndon F. Small, former Editor of the Journal.

[1] German Patent 668,875 (December 13, 1938) [*Chem. Abstr.*, **33**, 5006 (1939)].

[2] M. Tomita, *J. Pharm. Soc. Japan*, **56**, 906 (1936).

[3] M. Tomita, *J. Pharm. Soc. Japan*, **57**, 131 (1937).

[4] H. Jorg, *Ber.*, **60**, 1466 (1927).

[5] H. Gilman and N. Calloway, *J. Am. Chem. Soc.*, **55**, 4197 (1933).

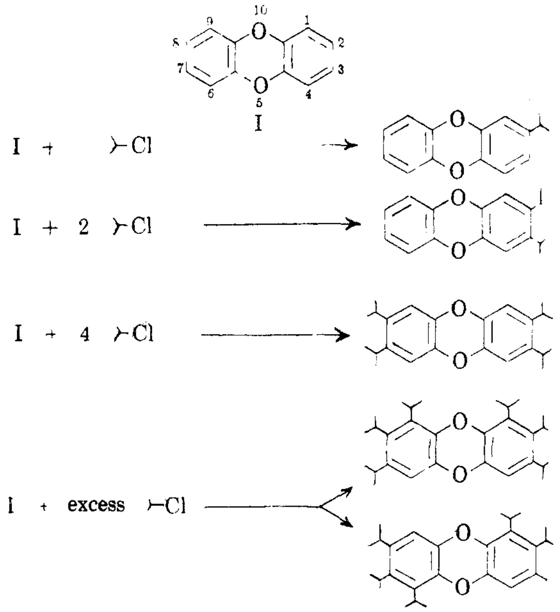
[6] P. George, Abstracts of Papers, 130th AMERICAN CHEMICAL SOCIETY Meeting, Atlantic City, N. J., Sept. 16–21, 1956, p. 54-O.

dibenzo-*p*-dioxin at room temperature in the presence of aluminum chloride to give a glossy brown, polymeric material. Likewise, chlorobenzene at 15° participated in the reaction to yield alkylated chlorobenzenes. No attempt was made to alkylate in nitrobenzene although this solvent has been frequently employed by others.[7,8]

Using *t*-butyl chloride, it was possible to make two derivatives that analyzed correctly as a mono and as a di derivative respectively. On the basis of infrared spectral analysis, it was concluded that the mono derivative was the 2-*t*-butyldibenzo-*p*-dioxin (II) and the di derivative either the 2,7- or the 2,8-di-*t*-butyldibenzo-*p*-dioxin(III). The possibility of both groups being in one ring was ruled out by the absence of a 1,2 disubstitution band at 13.3μ and the presence of a strong 1,2,4 trisubstitution band at 12.4μ. The mono derivative had strong bands at 13.3μ and 12.4μ. An attempt was made to increase the yield of the 2-*t*-butyldibenzo-*p*-dioxin by alkylating with *n*-octadecyl bromide since it had been used earlier[9] to produce ethyl 4-*t*-butyl-5-bromo-2-furoate from ethyl 5-bromo-2-furoate in about 20% greater yield than the next best alkyl halide.

Attempts to make poly-*t*-butyl derivatives, *i.e.*, substitution directly into the 1 position after first blocking the 2,3 and the 7,8 positions sterically with two *t*-butyl groups, met only with the formation of glasses and very viscous oils which could not be purified. Only metalation has so far succeeded in attacking this seemingly inert position.[10]

Four derivatives were obtained when isopropyl chloride was used as the alkylating agent. The mono derivative had the customary infrared bands at 12.4μ and 13.3μ. It was therefore presumed to be the 2-isopropyldibenzo-*p*-dioxin(IV). In addition to the bands at 12.4μ and 13.3μ the di derivative had a new, strong band at 12.8μ. This band had previously been noticed in the infrared spectra of 2,3,7,8-tetrabromodibenzo-*p*-dioxin.[11] On this basis, the diisopropyl derivative was designated 2,3(V). No 1,2 disubstitution band was evident in the spectrum of the tetraisopropyl derivative, but the bands at 12.4μ and 12.8μ were present. It was thus assigned the 2,3,7,8(VI) configuration. Forced conditions yielded the final isopropyl derivative which contained six isopropyl groups and is designated only as a hexaisopropyldibenzo-*p*-dioxin(VII). Steric considerations lead to the conclusion that the six groups are divided equally between the two rings. This may lead to two different products although only one was isolated.



Benzyl chloride produced only polymeric material when aluminum chloride was used as a catalyst and failed to react when anhydrous zinc chloride was used. However, a catalytic amount of anhydrous ferric chloride promoted the formation of a mono benzyl derivative which is believed to be the 2-benzyldibenzo-*p*-dioxin(VIII) on the basis of infrared analysis and previous evidence on the position of electrophilic attack.

Ethyl bromide failed to yield the normal alkylated product in carbon disulfide, but produced the ethyl ester of the dithio acid(IX).[4–6] The reaction of this ester with alcoholic potassium hydroxide proceeded directly to the dibenzo-*p*-dioxin-2-carboxylic acid[12] upon acidification. Likewise, methyl iodide under more drastic conditions, refluxing carbon disulfide, yielded the methyl ester of the dithio acid(X). The diethyl ester of the bis-dithio acid(XI) was formed at room temperature by bubbling ethyl chloride into the carbon disulfide mixture. A proof of structure was effected by oxidizing the bis-dithio ester to the acid by means of hydrogen peroxide[5] and conversion of the acid to the dimethyl ester, a known compound,[13] in this case the 2,7 isomer.

From the work done here and that accomplished earlier,[4–6] it is concluded that dithio esters form only when the alkylating species has no possibility of rearranging to a secondary or tertiary carbonium ion. This has been checked[5] and was rechecked in this series by using *n*-propyl chloride, *n*-butyl chloride, and some other *n*-alkyl halides. In all instances, only alkylation products were formed which in some instances could not be identified or even purified.

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(10) H. Gilman and C. G. Stuckwisch, *J. Am. Chem. Soc.*, **65**, 1461 (1943).

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Chloromethylation proceeds smoothly to give the 2-chloromethyldibenzo-*p*-dioxin (XII) in fair yield. This material tends to polymerize so care must be taken to have very clean equipment during the vacuum distillation. The structure is based on the infrared spectrum and could be easily verified by oxidation to the corresponding acid. Treatment with silver nitrate readily causes the precipitation of silver chloride.

The dibenzo-*p*-dioxin-2-carboxaldehyde(XIII) was formed in very low yield *via* a Gattermann-Koch reaction. Identification was based on the infrared spectrum and the work-up used to obtain the final product. A 2,4-dinitrophenylhydrazone(XIV) derivative was also obtained from XIII and analyzed.

Since the dibenzo-*p*-dioxin nucleus appeared to be easily alkylated, several attempts were made at direct Friedel-Crafts arylations. Chattaway[14] succeeded in condensing α-chloronaphthalene with benzene, and bromobenzene with naphthalene. In a similar manner, *p*-fluorophenol was condensed with benzene.[15] Both of the above reactions employed no solvent. 2-Chloroquinoline, 4,7-dichloroquinoline, and 2-chlorobenzothiazole react with resorcinol and 4-chlororesorcinol in nitrobenzene or dry quinoline, the better solvent, to give the expected Friedel-Crafts products.[16]

Both of these solvents were tried in unsuccessful attempts to condense *p*-chlorophenol, α-bromonaphthalene, and α-fluoronaphthalene with dibenzo-*p*-dioxin. The more promising solvent appeared to be quinoline since the dibenzo-*p*-dioxin nucleus is not destroyed at the temperatures used to effect reaction when this solvent is employed. No dibenzo-*p*-dioxin was recovered from the reactions employing nitrobenzene.

The compounds described at this time were prepared incidental to a study concerned with organic liquid solution scintillators. Evaluation of the compounds for this purpose is being made by Drs. Wright H. Langham, F. Newton Hayes, and Donald G. Ott of the Los Alamos Laboratories who will report the results later.

EXPERIMENTAL[17]

2-t-Butyldibenzo-p-dioxin (II). While a mixture of 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 22 g. (0.16 mole) of aluminum chloride, and 100 ml. of carbon disulfide was stirred at room temperature, 4.62 g. (0.05 mole) of *t*-butyl chloride was added over a period of 10 min. Stirring was continued for 1 hr. Hydrolysis was effected with ice–hydrochloric acid and the carbon disulfide layer was then washed with sodium carbonate solution followed by drying over anhydrous sodium sulfate. Evaporation of the carbon disulfide and vacuum distillation of the residue produced 1 g. of a product, b.p. 125–128° (0.25 mm.). This was recrystallized three times from methanol-water to yield 0.3 g. (2.5%) of white needles, m.p. 97–98.5°.

Anal. Calcd. for $C_{16}H_{16}O_2$: C, 80.00; H, 6.66. Found: C, 79.76, 79.74; H, 6.66, 6.58.

2,7(8)-Di-t-butyldibenzo-p-dioxin (III). The residue remaining in the flask after vacuum distillation of the 2-*t*-butyldibenzo-*p*-dioxin was recrystallized three times from ethanol to produce 0.5 g. (3.3%) of white plates, m.p. 226–228°.

Anal. Calcd. for $C_{20}H_{24}O_2$: C, 81.08; H, 8.10. Found: C 81.14, 81.15; H, 8.31, 8.26.

2-Isopropyldibenzo-p-dioxin (IV). To a stirred mixture of 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 22 g. (0.16 mole) of aluminum chloride and 100 ml. of carbon disulfide was slowly added 3.9 g. (0.05 mole) of isopropyl chloride at room temperature. The resulting mixture was stirred for 1.5 hr. at room temperature, hydrolyzed with ice–hydrochloric acid, and the resulting carbon disulfide layer washed with sodium carbonate solution before drying over anhydrous sodium sulfate. Evaporation of the carbon disulfide followed by vacuum distillation of the residue produced 1.5 g. of a product, b.p. 126–131° (0.5 mm.). Two recrystallizations of this material from methanol-water produced 1 g. (9%) of white needles, m.p. 87.5–89°.

Anal. Calcd. for $C_{15}H_{14}O_2$: C, 79.64; H, 6.19. Found: C, 79.46, 79.43; H, 6.30, 6.38.

n-Propyl chloride also produced the isopropyl derivative under the same conditions employed above.

2,3-Diisopropyldibenzo-p-dioxin (V). This reaction was conducted in the same manner as that used to obtain 2-isopropyldibenzo-*p*-dioxin except that two equivalents of isopropyl chloride were used. A 0.025 mole run produced 1 g. (8%) of the redistilled, colorless oil, b.p. 128–131° (0.25 mm.), n_D^{20} 1.5670.

Anal. Calcd. for $C_{18}H_{20}O_2$: C, 80.60; H, 7.46. Found: C, 80.47, 80.49; H, 7.61, 7.75.

2,3,7,8-Tetraisopropyldibenzo-p-dioxin (VI). This reaction was run under the same conditions used for the preparation of 2-isopropyldibenzo-*p*-dioxin. A 0.05 mole run was made using a four to one ratio of isopropyl chloride to dibenzo-*p*-dioxin. The crude product obtained by vacuum distillation was redistilled to yield 9.5 g. (54%) of colorless oil, b.p. 158–161° (0.5 mm.), n_D^{20} 1.5475.

Anal. Calcd. for $C_{24}H_{32}O_2$: C, 81.81; H, 9.09. Found: C, 81.74, 81.83; H, 9.06, 9.04.

Hexaisopropyldibenzo-p-dioxin (VII). The stirred mixture of 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 11 g. (0.08 mole) of aluminum chloride, and 100 ml. of carbon disulfide was kept at room temperature while 31.4 g. (0.46 mole) of isopropyl chloride was added over a period of 15 min. After addition, a calcium chloride drying tube was attached to the condenser and the mixture refluxed for 1 hr. Work-up produced 1.7 g. of material melting from 93–170°. Three recrystallizations of this material from petroleum ether (b.p. 60–70°) yielded 0.3 g. (1.4%) of white needles, m.p. 251–252°.

Anal. Calcd. for $C_{30}H_{44}O_2$: C, 82.57; H, 10.09. Found: C, 82.68, 82.74; H, 10.18, 10.14.

2-Benzyldibenzo-p-dioxin (VIII). To a stirred mixture of 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 1 g. of anhydrous ferric chloride and 100 ml. of carbon disulfide was slowly added 6.2 g. (0.05 mole) of benzyl chloride at room temperature. The resulting reaction mixture was then worked up in the usual manner. After evaporation of the dry carbon disulfide layer, the residue was vacuum-distilled to produce 2 g. of material, b.p. 178–180° (0.25 mm.). This material was recrystallized twice from ethanol to produce 1.2 g. (9%) of white needles, m.p. 106–108°.

Anal. Calcd. for $C_{19}H_{14}O_2$: C, 83.21; H, 5.11. Found: C, 82.96, 83.05; H, 4.95, 5.12.

Ethyl dibenzo-p-dioxin-2-dithiocarboxylate (IX). To a stirred mixture of 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 22 g. (0.16 mole) of aluminum chloride and 100 ml. of carbon

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[14] F. Chattaway, *J. Chem. Soc.*, **63**, 1185 (1893).

[15] A. Weston and C. Suter, *J. Am. Chem. Soc.*, **61**, 2556 (1939).

[16] G. Illuminati and H. Gilman, *J. Am. Chem. Soc.*, **74**, 2896 (1952).

[17] All melting points are uncorrected.

disulfide at room temperature was added 5.5 g. (0.05 mole) of ethyl bromide. Stirring was continued for 1.5 hr. and then the reaction mixture was worked up. The crude product was dissolved in benzene and chromatographed on alumina. Distillation of the benzene followed by two recrystallizations of the product from absolute ethanol resulted in 2.2 g. (16%) of orange needles, m.p. 104–106°.

Anal. Calcd. for $C_{15}H_{12}O_2S_2$: C, 62.50; H, 4.17; S, 22.22. Found: C, 62.37, 62.50; H, 4.37, 4.21; S, 22.33, 22.22.

Methyl dibenzo-p-dioxin-2-dithiocarboxylate (X). To a stirred mixture of 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 22 g. (0.16 mole) of aluminum chloride and 100 ml. of carbon disulfide was added 7.1 g. (0.05 mole) of methyl iodide. The mixture was then refluxed for 6.5 hr. and then worked up in the usual manner. After evaporation of the carbon disulfide, the residue was dissolved in benzene and chromatographed on alumina. This did not effect purification, so the crude material was subjected to vacuum sublimation. The unreacted dibenzo-*p*-dioxin sublimed away from the product which was recrystallized twice from ethanol to yield 0.2 g. (1.4%) of orange needles, m.p. 121–123°.

Anal. Calcd. for $C_{14}H_{10}O_2S_2$: S, 23.36. Found: S, 23.73, 23.51.

Diethyl dibenzo-p-dioxin-bis(dithiocarboxylate) (XI). Ethyl chloride was slowly bubbled into 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 22 g. (0.16 mole) of aluminum chloride, and 200 ml. of carbon disulfide over a period of 1.5 hr. at room temperature. The usual work-up produced crude material which was recrystallized three times from benzene-ethanol to form 1.7 g. (7%) of dark orange needles, m.p. 174–176°.

Anal. Calcd. for $C_{18}H_{16}O_2S_4$: S, 32.65. Found: S, 32.65, 32.74.

2-Chloromethyldibenzo-p-dioxin (XII). A mixture of 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 4 g. (0.13 mole) of paraformaldehyde, 50 ml. of glacial acetic acid, 40 ml. of concentrated hydrochloric acid, and 15 ml. of 85% phosphoric acid was heated at 80° for 10 hr. with stirring. Dilution of the reaction mixture with water followed by suction filtration produced 12.7 g. of crude material. Vacuum-distillation of the crude product yielded 3 g., b.p. 143–144° (0.15 mm.). This material was recrystallized three times from ethanol to form 1.5 g. (13%) of white needles, m.p. 111–113°.

Anal. Calcd. for $C_{13}H_9O_2Cl$: Cl, 15.28. Found: Cl, 15.41, 15.31.

Dibenzo-p-dioxin-2-carboxaldehyde (XIII). An excess of carbon monoxide and hydrogen chloride, obtained by dropping chlorosulfonic acid into formic acid, was bubbled into a mixture of 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 23 g. (0.175 mole) of aluminum chloride, 3 g. of cuprous chloride and 50 ml. of benzene for 8 hr. at 40–50°. After hydrolysis and distillation of the benzene layer, the residue was dissolved in methanol and treated with saturated sodium bisulfite solution. The precipitate which formed was washed with ethanol and ether. Treatment of the precipitate with dilute hydrochloric acid produced the product which melted 87–90°. Two recrystallizations from ethanol-water yielded 0.2 g. (2%) of white crystals, m.p. 91–93°.

Anal. Calcd. for $C_{13}H_8O_3$: C, 73.58; H, 3.77. Found: C, 74.40, 74.31; H, 3.54, 3.62.

The 2,4-dinitrophenylhydrazone recrystallized from pyridine-ethanol melted 300–301°.

Anal. Calcd. for $C_{19}H_{12}O_6N_4$: N, 14.29. Found: N, 13.89, 14.00.

Attempted preparation of 2-(α-naphthyl)dibenzo-p-dioxin. A mixture of 11 g. (0.08 mole) of aluminum chloride, 9.2 g. (0.05 mole) of dibenzo-*p*-dioxin, 10 g. (0.08 mole) of quinoline, and 7.3 g. (0.05 mole) of α-fluoronaphthalene were stirred at 150° for 2 hr. Then 7 g. (0.055 mole) more of aluminum chloride was added and the temperature raised to 180° for 2 hr. The purple-black mixture was allowed to cool and hydrolyzed with ice–hydrochloric acid. This was extracted with ether and dried over anhydrous sodium sulfate. The residue, obtained by evaporation of the ether, was vacuum distilled to yield a small amount of α-fluoronaphthalene. By chromatographing the material remaining in the flask after vacuum distillation, 2.5 g. (28%) of starting material was obtained. No product has been isolated.

The other runs using α-bromonaphthalene and *p*-chlorophenol were very similar to that described above except for variations in the solvent.

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AMES, IOWA

[CONTRIBUTION FROM DEPARTMENT OF CHEMISTRY, ISRAEL INSTITUTE OF TECHNOLOGY]

Alicyclic Studies. XIII.[1] Preparation and Reactions of 1,1'-Dicycloalkenyls*

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The preparation of 1,1'-dicyclopentenyl, 1,1'-dicyclohexenyl, 1,1'-dicycloheptenyl, and 1,1'-dicycloöctenyl has been improved. Diels-Alder reactions of these dienes are described.

Diene reactions of 1,1'-dicylopentenyl[1] (Ia) and 1,1'dicylohexenyl[2,3] (Ib) have been described. However, it is clear that particularly in the former case, the diene has not heretofore been obtained pure. The dienes are prepared by dehydration under acidic conditions of the pinacols resulting from pinacol-reduction of cyclopentanone and cyclohexanone, respectively. It has been shown that acidic dehydration of 1,1'-dihydroxy-1,1'-dicyclopentyl gives primarily 2-spirocyclopentanocyclohexanone (II).[4] The yields of Diels-Alder adducts form

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* This paper is a contribution in honor of Lyndon F. Small, former Editor of the Journal.

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