

1020. *Reactions of Cyclohexadienes. Part IV.* Some Transformations of Bisdihalogenocarbene Adducts.*

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The action of silver salts on the bisdibromocarbene adducts of some 1-methoxycyclohexa-1,4-diene derivatives results in initial conversion into bromocycloheptenone derivatives, and with the 1,4-dimethoxy-derivative finally into the benzocyclobutenone (III) in good yield. Reductive removal of bromine from the dibromocarbene adducts results in some formation of methoxycycloheptenone derivatives, although the major products contain methoxycyclopropane rings, which undergo acid fission to give 2-methylketones.

REACTION of dibromocarbene with cyclohexa-1,4-diene gives the adduct (I; $R = R' = H$),¹ and it has been briefly noted² that 1-methoxycyclohexa-1,4-diene gives the analogue (I; $R = H$, $R' = OMe$). Some reactions of these and similar adducts have been examined with two objectives: to see how far they can be used for expanding six-membered rings; and to see whether they can be used for ultimate introduction of methyl groups. Synthetic use of the latter process in the steroid series will be reported elsewhere.³ The dibromocarbene adducts were readily produced by the action of an excess of potassium *t*-butoxide and bromoform, and were usually employed because of their reactivity. A

* Part III, Birch, Butler, and Siddall, *J.*, 1964, 2941.

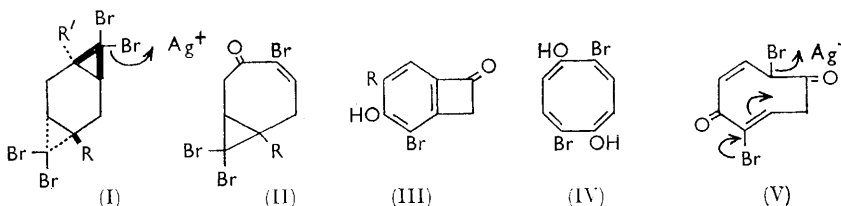
¹ Winstein and Boikess, *J. Amer. Chem. Soc.*, 1963, **85**, 343.

² Birch, Graves, and Stansfield, *Proc. Chem. Soc.*, 1962, 282.

³ Birch, Brown, and Subba Rao, *J.*, 1964, 3309.

dichlorocarbene adduct was made by the use of potassium *t*-butoxide and ethyl trichloroacetate.

The action of boiling aqueous-ethanolic silver nitrate or perchlorate on the adducts (I; R = H, R' = OMe; and R = R' = OMe) gave first the unsaturated ketones (II; R = H and OMe, respectively). The structures of the products were supported by analyses and spectra. The ketone (II; R = H) has λ_{max} 258 $\text{m}\mu$ similar to that recorded (256 $\text{m}\mu$) for other $\alpha\beta$ -unsaturated α -bromo ketones such as 2-bromocholest-1-en-3-one.⁴ The mechanism shown in (I) is presumed to be similar to that postulated for the first stage of our tropone synthesis involving the carbene monoadducts.^{2,5}



Reaction for a longer period resulted in the precipitation of up to three mol. of silver bromide. With the adduct (I; R, R' = OMe) silver perchlorate gave about a 50% yield of the crystalline phenolic ketone (III; R = H). Silver nitrate gave a lower yield (19%) of a nitro-derivative (III; R = NO₂), presumably formed by final nitration, since the same substance was readily obtained by the action of nitric acid in cold acetic acid on the compound (III; R = H). The production of the benzocyclobutenone system could involve an eight-membered ring-intermediate such as the conjugated enol (IV), which could collapse to a fused six-four-membered ring system in a way reminiscent of reactions of cyclo-octatetraene. Alternatively, a route such as (V) could be involved. Whatever the mechanism, it is a simple experimental method of preparing a compound containing the benzocyclobutenone (bicyclo[4,2,0]octa-1,2,5-trien-6-one) system.

The structure of the ketone (III; R = H) follows from its spectra and behaviour. It is a fairly strong acid, soluble in potassium hydrogen carbonate solution, indicative of the *para*-orientation of the carbonyl and the hydroxyl group. The presence of the cyclobutenone ring is indicated by the infrared spectra, particularly that of the methyl ether (ν_{max} 1750 cm^{-1}). The proton magnetic resonance of the ether shows the presence of two adjacent aromatic protons, OMe, and CH₂ adjacent to carbonyl (τ 6.10). The proton magnetic resonance of the nitro-compound (III; R = NO₂) shows the same CH₂ and a single aromatic proton at a position (τ 1.83) that indicates that it is probably adjacent to the NO₂. The substitution pattern of compounds (III) is also that to be expected from the structure of the starting material.

The corresponding bisdichlorocarbene adduct reacted very much more slowly with silver nitrate in boiling aqueous ethanol than did the bromocarbene adduct, although some reaction occurred in boiling propan-1-ol.

The adduct (VI) from 1,5-dimethoxycyclohexa-1,4-diene reacted with silver nitrate in boiling aqueous acetone to give a ketone (VII), whose structure is supported by its ultraviolet and nuclear magnetic resonance spectra. Further reaction in boiling aqueous ethanol gave a gummy acid which seemed to be a mixture possibly containing a small proportion of a tropone.

The reduction of the adduct (I; R = R' = H) has already been reported, without experimental details,¹ to give a hydrocarbon (VIII; R = H). This was shown to be pure by gas-liquid chromatography and by its nuclear magnetic resonance. A similar *trans*-configuration of the rings in the other adducts is assumed, and is supported by examination of their nuclear magnetic resonance spectra.

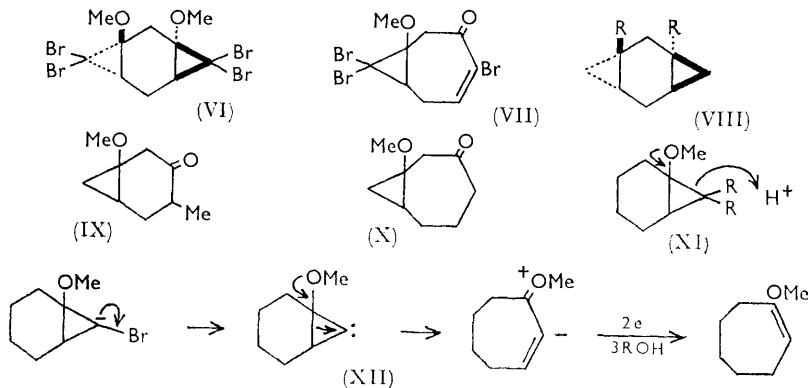
⁴ Fieser and Fieser, "Steroids," Reinhold Publ., Inc., New York, 1959, p. 287.

⁵ Birch, Graves, and Siddall, *J.*, 1963, 4234.

On the basis of this result, the lithium–ammonia reduction of the tetrabromide (VI) gave an unexpected result, since the product showed a weak enol-ether peak (ν_{\max} 1660 cm^{-1}) varying in intensity with the experimental details of the reduction. The enol-ether nature of the component was confirmed by mild hydrolysis with oxalic acid, and the resulting ketone was separated by chromatography in 5–25% yield; it could also be detected, and estimated approximately, by its infrared spectrum. The slower the rate of lithium addition and the higher the concentration of ethanol in the ammonia, the more ketone was eventually obtained. The ketone was a mixture and probably contained both compounds (IX) and (X) (ν_{\max} 1710 cm^{-1} with a shoulder at 1700 cm^{-1}).

In order to investigate a simpler case, the adduct (XI; R = Br) of methoxycyclohexene was examined. In this case, about 15% of the final product was ketonic and consisted mainly of cycloheptanone, identified by derivatives and spectra (ν_{\max} 1696 cm^{-1} with a shoulder at 1707 cm^{-1} probably due to some 2-methylcyclohexanone).

The non-ketonic material was shown in each case by analysis and by its nuclear magnetic



resonance spectrum to be the expected cyclopropane derivative: (VIII; R = OMe) from (VI), and (XI; R = H) from (XI; R = Br).

The rearrangement observed must be due to the presence of the methoxyl group, since there is no trace of it with the parent adduct (I; R = R' = H). The most likely mechanism therefore involves production of an electron-deficient carbene system by reductive removal of one bromine atom, followed by loss of the other carrying the negative charge. Rearrangement of such a carbene could be assisted as shown (XII) by the *p*-electrons of the oxygen. In accordance with this idea, the tetrachloro-analogue of (VI), which should give a carbene less readily, rearranged on reduction only to a minor extent (~2%).

The action of hydrogen bromide in chloroform on the adduct (XI; R = H) gave 2-methylcyclohexanone, as expected from protonation of the least substituted carbon and fission of the ring at the carbon carrying the methoxyl group, in accord with the ability of the latter to stabilise the cationic intermediate.

EXPERIMENTAL

4,4,8,8-Tetrabromo-1-methoxytricyclo[5,1,0,0^{3,5}]octane (I; R = OMe, R' = H).—Dry sodium *t*-butoxide (from sodium, 3.5 g., 3 atom-equiv.), suspended in a solution of 2,5-dihydroanisole (5.5 g., 1 mol.) in dry benzene (70 ml.) and dry pentane (70 ml.), was stirred and cooled in ice-salt whilst a solution of bromoform (38 g., 3 mol.) in benzene (40 ml.) and pentane (40 ml.) was added dropwise in 45 min. Stirring and cooling were continued for 1 hr. more, the ice-salt bath was removed, and the mixture stirred until it reached room temperature (1 hr.). It was then shaken with water (250 ml.) and the organic layer separated, dried, and evaporated *in vacuo* at 40° (bath). Ethanol (20 ml.) was added and the mixture left overnight in the refrigerator. 4,4,8,8-Tetrabromo-1-methoxytricyclo[5,1,0,0^{3,5}]octane (7.2 g.) was filtered off,

washed with ethanol, dried, and crystallised from ethanol (charcoal) as colourless crystals, m. p. 138° (6·7 g.), no infrared peaks in the 1500—2000 cm.⁻¹ region (Found: C, 23·9; H, 2·3; Br, 70·1. C₉H₁₀Br₄O requires C, 23·8; H, 2·2; Br, 70·5%).

4,8,8-Tribromobicyclo[5,1,0]oct-4-en-3-one.—Silver nitrate (2 g.) was dissolved in warm water (1 ml.), and 95% ethanol (25 ml.) was added, followed by the preceding di-adduct (0·70 g.). The mixture was boiled under reflux for 2 hr. and the precipitated silver bromide (0·47 g., 1·62 mol.) filtered off and washed several times with small amounts of ethanol. The combined filtrate and washings were evaporated on the steam-bath to about 15 ml. and left to crystallise overnight in the refrigerator. The 4,8,8-tribromobicyclo[5,1,0]oct-4-en-3-one was filtered off, washed with small amounts of cold ethanol and dried, giving colourless crystals (0·30 g.), m. p. 130 raised to 132° by crystallisation from alcohol or by sublimation *in vacuo* (Found: C, 26·95; H, 2·0; Br, 66·55. C₈H₇Br₃O requires C, 26·8; H, 2·0; Br, 66·8%), λ_{\max} . 258 m μ (ϵ_{\max} . 5600) in ethanol, ν_{\max} . 1665 cm.⁻¹ (Nujol mull).

The 2,4-dinitrophenylhydrazone formed orange-red cubes, m. p. 197°, from boiling ethyl acetate, in which it was very sparingly soluble (Found: C, 31·6; H, 2·4. C₁₄H₁₁Br₃N₄O₄ requires C, 31·2; H, 2·05%).

4,4,8,8-Tetrabromo-1,5-dimethoxytricyclo[5,1,0,0^{3,5}]octane (I; R = R' = OMe).—Dry sodium t-butoxide (from 3·5 g. of sodium), suspended in dry pentane and dry benzene (80 ml. each) containing 1,4-dimethoxycyclohexa-1,4-diene (7·0 g., 1 mol.), was stirred and cooled in ice-salt whilst bromoform (38 g., 3 mol.) in pentane-benzene (1:1; 60 ml.) was added dropwise in 45 min. The mixture was stirred for an hour more, the ice-salt bath removed, and stirring continued for a further 1 hr. whilst the mixture attained room temperature. The product was then shaken with water (300 ml.) and the solid, insoluble in both layers, was filtered off, washed with a little water and then with ethanol, and dried (20·0 g.). The pentane-benzene layer was separated from the filtrate and dried, and the solvent distilled *in vacuo*, leaving a pale brown oil consisting mainly of the mono-adduct, which rapidly darkened. When left overnight in the refrigerator, more crystals of the di-adduct were deposited and were filtered off, washed with ethanol, and dried (0·7 g.). The 4,4,8,8-tetrabromo-1,5-dimethoxytricyclo[5,1,0,0^{3,5}]octane (total yield 20·7 g.), m. p. 178° (decomp.), gave large colourless rhombs, m. p. 180° (decomp.) (from benzene), having no infrared absorption between 1500 and 1700 cm.⁻¹ (Found: C, 25·0; H, 2·55; Br, 66·05. C₁₀H₁₂Br₄O₂ requires C, 24·8; H, 2·5; Br, 66·1%).

4,8,8-Tribromo-7-methoxybicyclo[5,1,0]oct-4-en-3-one (II, R = OMe).—The above dimethoxybis-adduct (3·0 g.) and acetone (150 ml.) were added to a solution of silver perchlorate (6·0 g.) in water (6 ml.), and the mixture was boiled under reflux for 22 hr. Silver bromide (1·34 g., 1·15 mol.) was filtered from the hot solution, washed several times with hot acetone, and dried. The combined filtrate and washings were evaporated to about 30 ml. and cooled, and the starting material that crystallised was filtered off and dried (0·75 g.). The filtrate was poured into water (200 ml.) and left for 2 days, and the solid that separated was filtered off, washed with water, and dried (0·82 g.). The 4,8,8-tribromo-7-methoxybicyclo[5,1,0]oct-4-en-3-one, ν_{\max} . 1667 cm.⁻¹, crystallised successively from ethanol, carbon tetrachloride, and ethanol, giving colourless crystals, m. p. 109—111° (Found: C, 27·5; H, 2·2. C₉H₉Br₃O₂ requires C, 27·8; H, 2·35%). With Brady's reagent it gave a 2,4-dinitrophenylhydrazone, which was sparingly soluble in most organic solvents but gave orange-red plates, m. p. 171° (decomp.) from hot pyridine (Found: C, 32·1; H, 2·4; N, 10·4. C₁₅H₁₃Br₃N₄O₄ requires C, 32·55; H, 2·35; N, 10·1%).

2-Bromo-3-hydroxybicyclo[4,2,0]octa-1,3,5-trien-7-one (3-Bromo-4-hydroxybenzocyclobuten-1-one) (III; R = H).—Silver perchlorate (6·0 g.) was dissolved in water (6 ml.) and 95% ethanol (150 ml.) was added, followed by the dimethoxy di-adduct above (3·0 g.), and the mixture was boiled under reflux for 24 hr. with magnetic stirring to avoid bumping. The precipitated silver bromide was filtered from the hot solution, washed several times with hot ethanol, dried, and weighed (3·12 g., 2·68 mol. per mol. of di-adduct). To the combined filtrate and washings was added a solution of sodium chloride (4·0 g.) in water (15 ml.) to remove the excess of silver salts, the precipitated silver chloride was filtered off, and the filtrate evaporated to small volume under reduced pressure. Water (200 ml.) was added and the product extracted four times with ether (total 150 ml.). The combined extracts were washed with water (25 ml.) and extracted four times with potassium carbonate solution (total of 30 g. in 150 ml. of water). As saturation of the combined carbonate extracts with carbon dioxide gave no solid, the solution was acidified with concentrated hydrochloric acid. The mixture was cooled in ice, and the

product was filtered off, washed with water, and dried (0.70 g., 53%). It crystallised from boiling water containing a little ethanol (charcoal) as colourless needles, m. p. 195° (Found: C, 45.0; H, 2.5; Br, 37.75. $C_8H_7BrO_2$ requires C, 45.1; H, 2.35; Br, 37.55%), λ_{\max} . (EtOH) $m\mu$ (ϵ in parentheses): 235 (17,900), 275 (10,000), 297 (10,100), and 335 (1940). The infrared spectrum of a Nujol mull showed a broad peak at ν_{\max} 1730 cm^{-1} .

2-Bromo-3-methoxybicyclo[4,2,0]octa-1,3,5-trien-7-one.—The hydroxy-compound (0.22 g.) was suspended in ether and dissolved by addition of the minimum amount of methanol (*ca.* 1 ml.). A solution of diazomethane in light petroleum (b. p. 40–60°) was added in portions until the yellow colour was permanent and the solution was then evaporated. The residue was dissolved in ether, shaken three times with potassium carbonate solution, then with water, and dried (Na_2SO_4). After evaporation the almost colourless residue crystallised from light petroleum (b. p. 60–80°) as colourless needles of the *methyl ether*, m. p. 106° (Found: C, 47.9; H, 3.3. $C_9H_7BrO_2$ requires C, 47.6; H, 3.1). The proton magnetic resonance spectrum in $CDCl_3$ showed two doublets centred at τ 2.66 and 3.05 with identical coupling constants (one aromatic proton each), a singlet at 6.00 (3 protons, OMe group), and a singlet at 6.10 (2 protons, CH_2 group). The compound had ν_{\max} 1750 cm^{-1} in a Nujol mull, and in ethanol solution, had absorption peaks $m\mu$ (ϵ in parentheses) at 235 (21,000), 275 (11,300), and 290 (10,200).

2-Bromo-3-hydroxy-4-nitrobicyclo[4,2,0]octa-1,3,5-trien-7-one (III; R = NO_2).—(a) Silver nitrate (10.2 g., 8 mol.) was dissolved in warm water (5 ml.), and 95% ethanol (75 ml.) was added, followed by the dimethoxy-di-adduct above (3.63 g., 1 mol.), and the mixture was boiled under reflux for 18 hr. The precipitated silver bromide was filtered off from the hot solution, washed with hot ethanol, and dried (4.60 g., 3.25 mol.). The combined ethanolic filtrate and washings were evaporated to *ca.* 25 ml., then cooled in ice, and the yellow *nitro-compound* was filtered off, washed with water, then with ethanol, dried (0.37 g., 19%), and crystallised from ethanol; it had m. p. 199–200° (Found: C, 37.1; H, 1.4; Br, 31.3; N, 5.3. $C_8H_4BrNO_4$ requires C, 37.2; H, 1.55; Br, 31.0; N, 5.4%), λ_{\max} . (EtOH) 258 $m\mu$ (ϵ 23,000), ν_{\max} . (Nujol) 1770 cm^{-1} . The proton magnetic resonance spectrum in $CDCl_3$ showed peaks at τ –2.05 (1 proton, OH), 1.83 (1 proton, aromatic CH) and 5.93 (2 protons, CH_2 group).

(b) 2-Bromo-3-hydroxybicyclo[4,2,0]octa-1,3,5-trien-7-one (66 mg.) was dissolved in warm glacial acetic acid (5 ml.), water (1 ml.) was added, and the solution cooled rapidly in ice–salt. Nitric acid (d 1.42; 3 ml.) was added, and the almost colourless solution shaken at room temperature for 3 hr. It rapidly became deep yellow and the nitro-compound gradually crystallised. Finally water (20 ml.) was added and, after being cooled in ice, the product was filtered off, washed with water, and dried (37 mg.). The pale yellow crystals, m. p. 200°, did not depress the m. p. of the nitro-compound prepared by the silver nitrate reaction, and had an identical infrared spectrum.

4,4,8,8-Tetrachloro-1,5-dimethoxytricyclo[5,1,0,0^{3,5}]octane.—Dry sodium methoxide (from sodium, 5.6 g., 3 atom-equiv.) was suspended in a solution of 1,4-dimethoxycyclohexa-1,4-diene (11.3 g., 1 mol.) in dry cyclohexane (200 ml.), and the mixture was stirred and cooled in ice whilst a solution of ethyl trichloroacetate (46.4 g., 3 mol.) in cyclohexane (80 ml.) was added dropwise in 50 min. The reaction mixture was stirred for 1 hr. more, the bath was removed, and the mixture stirred until it reached room temperature (30 min.). The mixture was then shaken with water (300 ml.) and the 4,4,8,8-tetrachloro-1,5-dimethoxytricyclo[5,1,0,0^{3,5}]octane collected, washed with water, and dried (8.6 g.). A further amount (2.3 g.) was obtained by separating the cyclohexane layer from the filtrate, drying it, and evaporating it to small volume (*ca.* 15 ml.) *in vacuo*, then cooling it in ice. The total yield was 10.9 g. The compound crystallised from boiling ethanol as colourless needles (9.2 g.), m. p. 155° (Found: C, 39.25; H, 4.0; Cl, 46.25. $C_{10}H_{12}Cl_4O_2$ requires C, 39.25; H, 3.92; Cl, 46.35%). It had no infrared absorption between 1500 and 2000 cm^{-1} .

4,4,8,8-Tetrabromo-1,3-dimethoxytricyclo[5,1,0,0^{3,5}]octane (VI).—This compound was prepared from 1,5-dimethoxycyclohexa-1,4-diene in 52% yield in essentially the same manner as its isomer above. Crystallised from ethanol it had m. p. 139° (Found: C, 25.15; H, 2.55. $C_{10}H_{12}Br_4O_2$ requires C, 24.85; H, 2.5%). 4,4,8,8-Tetrachloro-1,3-dimethoxytricyclo[5,1,0,0^{3,5}]octane was prepared similarly, by using chloroform instead of bromoform and had m. p. 114° (Found: C, 39.2; H, 3.9. $C_{10}H_{12}Cl_4O_2$ requires C, 39.4; H, 4.0%).

4,8,8-Tribromo-1-methoxybicyclo[5,1,0]oct-4-en-3-one (VII).—The above tetrabromo-compound (2.15 g.) was refluxed for 16 hr. in aqueous acetone (20 ml.) containing silver perchlorate (2.35 g.). Silver bromide (1.52 g.) was filtered off, and the solvent removed under reduced

pressure. Trituration of the residual oil with carbon tetrachloride gave solid material (800 mg.), which was crystallised from carbon tetrachloride to give 4,8,8-tribromo-1-methoxybicyclo[5,1,0]oct-4-en-3-one (550 mg.), m. p. 154—156° (Found: C, 27.9; H, 2.4. $C_9H_9Br_3O_2$ requires C, 27.8; H, 2.3%), λ_{max} 256 (ϵ 6000), ν_{max} 1675 cm^{-1} .

1-Methoxybicyclo[4,1,0]heptane.—1-Methoxycyclohexene was prepared in 68% yield from 1,5-dimethoxycyclohexa-1,4-diene.⁶ A solution of the former (1.12 g.) was stirred in pentane (30 ml.) and potassium t-butoxide (from potassium, 0.62 g.) at -10° under nitrogen. Bromoform (3.42 g.) was added in 30 min., and the mixture stirred for a further hour, and washed with water; the solvent was removed, leaving an oil (3.2 g.) which could not be purified by distillation but showed no enol-ether band in its infrared spectrum. The crude adduct (3.0 g.) was added to liquid ammonia (60 ml.) and methanol (10 ml.). Lithium (0.7 g.) was added to the stirred solution during 30 min. After addition of water, the product was extracted with pentane, washed, and dried, and the solvent evaporated. The crude product showed a marked enol-ether band at 1660 cm^{-1} , which was converted into a band at 1695 cm^{-1} after the material had been allowed to stand in an aqueous methanolic solution of oxalic acid. The product was chromatographed on H-alumina (Spence) and eluted with pentane-ether 49:1, to give 1-methoxybicyclo[4,1,0]heptane (620 mg.). It was identified as a uniform compound by gas-liquid chromatography, and by its proton magnetic resonance spectrum, which showed the presence of 14 protons with characteristic peaks at τ 6.87 (OMe) and 9.9 (1 proton in cyclopropane CH_2); the infrared spectrum had bands at 1035, 1105, 1205, and 3035 cm^{-1} . Further elution with pentane-ether (10%) gave cycloheptanone (110 mg.), identified by infrared comparison with an authentic specimen and by its 2,4-dinitrophenylhydrazone, m. p. and mixed m. p. 135—137°.

Dry hydrobromic acid was passed into alcohol-free chloroform (3 ml.) containing 1-methoxybicyclo[5,1,0]heptane (110 mg.) for 20 min. at 0° . The liquid was left overnight at room temperature and washed with water, and the solvent was removed under reduced pressure. The residual oil had an infrared spectrum identical with that of 2-methylcyclohexanone and gave the 2,4-dinitrophenylhydrazone, m. p. and mixed m. p. 135—137°.

1,3-Dimethoxytricyclo[5,1,0,0^{3,5}]octane.—Tetrabromo-1,3-dimethoxytricyclo[5,1,0,0^{3,5}]octane (5.8 g.) was finely ground and suspended in liquid ammonia (200 ml.) and ethanol (40 ml.). Lithium (1.68 g.) was slowly added to the stirred solution. After addition of water, the mixture was extracted with pentane, washed, dried, and evaporated. The residue showed enol-ether absorption at 1660 cm^{-1} , which changed to 1710 cm^{-1} after the material had been hydrolysed with oxalic acid in aqueous methanol. The hydrolysed mixture (1540 mg.) was chromatographed on Florex. Elution with pentane-ether (19:1) gave 1,3-dimethoxytricyclo[5,1,0,0^{3,5}]octane (1.24 g.) (Found: C, 71.6; H, 9.6. $C_{10}H_{16}O_2$ requires C, 71.45; H, 9.55%). The infrared and nuclear magnetic resonance spectra were completely in accord with the assigned structure. Further elution with 4:1 pentane-ether gave a ketonic mixture (240 mg.), which could not be resolved by further chromatography.

Reduction of the bromo-compound was investigated under varying conditions, and the yield of rearranged product estimated by the intensity of the carbonyl band at 1710 cm^{-1} compared with that of a standard solution of 2-methylcyclohexanone in carbon disulphide. It was found to increase to a maximum of 25% with increasing time of addition of the lithium. Inverse addition of the adduct to lithium in ammonia gave no appreciable amount of rearranged product.

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⁶ Birch, *J.*, 1950, 1551.