General Procedure for the Preparation of Biscyclododecylidene Cycloalkylidene Triperoxides. To a cold (5–10 °C), vigorously stirred solution of the ketone (100 mmol) in 20–25 ml of PhH containing 30 drops of 10% $HClO_4$ in glacial acetic acid, a $CHCl_3$ solution (1 g, 50 ml) of 1,1′-dihydroperoxydicyclododecyl peroxide was added from a buret (approximately 1 ml/min). After an additional 30–72 h in the cold, most of the solvent was removed at room temperature. On trituration of the residue with methanol, solid material appeared. Filteration and recrystallization from a 2:3 mixture of CH_3COOEt and $CHCl_3$ gave the desired product in reasonably pure condition.

Thermolysis of the Peroxide. Details of the procedure for pyrolysis and identification of the products of pyrolysis are given in the earlier papers.[7]

Ir and NMR Data for Compounds 1–12 (See Table I). Compound 1. Infrared (CCl_4) 2933 (vs), 2860 (w), 2850 (s), 1470 (vs), 1448 (s), 1370 (vw), 1350 (w), 1300 (vw), 1285 (w), 1250 (m), 1220 (m), 1205 (vw), 1192 (w), 1178 (m), 1160 (vw) 1110 (vw), 1080 (s), 1069 (s), 1010 (s), 992 (w), 980 (vw), 955 (m), 915 (w), 880 cm^{-1} (m).

Compound 2. NMR ($CDCl_3$, Me_4Si) δ 1.28 (singlet, sharp) 1.53 (shoulder, broad); Infrared (CCl_4) 2925 (vs), 2860 (vw), 2850 (s), 1470 (vs), 1448 (s), 1360 (w), 1335 (vw), 1325 (w), 1300 (vw), 1285 (vw), 1278 (m), 1265 (vw), 1250 (m), 1220 (m), 1205 (vw), 1192 (vw), 1178 (m), 1170 (m), 1160 (w), 1250 (vw), 1110 (vw), 1092 (w), 1070 (s), 1010 (s), 995 (w), 970 (w), 952 (m), 915 (m), 880 cm^{-1} (m). (m).

Compounds 3. Infrared (CCl_4) 2925 (vs), 2860 (w), 2850 (s), 1470 (vw), 1450 (s), 1370 (w), 1325 (w), 1300 (vw), 1285 (w), 1250 (m), 1220 (m), 1210 (vw), 1195 (vw), 1175 (m), 1160 (vw), 1110 (w), 1080 (v 1078 (s), 1040 (vw), 1015 (s), 970 (vw), 955 (w), 915 (w), 880 cm^{-1} (m).

Compound 4. Infrared (CCl_4) 2925 (vs), 2850 (s, sh at 2860), 1472 (vs), 1448 (s), 1370 (vw), 1352 (w), 1325 (w), 1300 (w), 1285 (w), 1270 (vw), 1250 (m), 1235 (vw), 1220 (m), 1195 (vw), 1178 (m), 1160 (w), 1124 (w), 1110 (w), 1095 (vw), 1070 (s), 1010 (s), 995 (m), 925 (m), 905 (m), 915 (m), 880 cm^{-1} (m).

Compound 5. NMR ($CDCl_3$, Me_4Si) δ 1.37 (shoulder, sharp), 1.50 (shoulder, broad); infrared (CCl_4) 2930 (vs), 2865 (s), 1470 (s), 1440 (s), 1330 (vw), 1255 (m), 1228 (m), 1115 (vw), 1075 (s), 1015 (s), 960 (m), 920 (m), 880 cm^{-1} (m).

Compound 6. NMR ($CDCl_3$, Me_4Si) δ 1.27 (singlet, sharp); infrared (CCl_4) 2930 (vs), 2860 (s), 1468 (s), 1445 (s), 1365 (vw), 13 (vw), 1320 (w), 1245 (m), 1218 (m), 1175 (w), 1165 (m), 1080 (w), 1065 (s), 1005 (s), 968 (w), 950 (m), 910 (w), 870 cm^{-1} (m).

Compound 7. NMR ($CDCl_3$, Me_4Si) δ 0.88, 1.02 (doublet, sharp) 1.23 (singlet, sharp), 1.48 (shoulder, broad); infrared (CCl_4) 2930 (vs), 2860 (s), 1468 (s), 1445 (s), 1365 (vw) 1345 (vw), 1320 (w), 1245 (m), 1218 (m), 1175 (w), 1165 (m), 1105 (vw), 1080 (w), 1065 (s), 1005 (s), 968 (w), 950 (m), 910 (w), 870 cm^{-1} (m).

Compound 8. NMR ($CDCl_3$, Me_4Si) δ 0.82, 0.90 (doublet?, broad), 1.28 (singlet, sharp) 1.52 (shoulder, broad); infrared (CCl_4) 2930 (vs), 2850 (s), 1470 (s), 1445 (s), 1365 (vw), 1350 (w), 1290 (vw), 1250 (m), 1220 (w), 1190 (vw), 1165 (s), 1150 (m), 1078 (vw), 1062 (s), 1045 (w), 1010 (s), 990 (w), 980 (w), 950 (m), 910 (w), 900 (w), 870 cm^{-1} (m).

Compound 9. NMR ($CDCl_3$, Me_4Si) δ 0.80 (singlet, broad), 0.90 (singlet, broad) 1.28 (singlet, sharp), 1.52 (shoulder, broad); infrared (CCl_4) 2930 (vw), 2855 (s), 1470 (s), 1445 (s), 1350 (w), 1325 (w), 1280 (vw), 1245 (w), 1190 (w), 1165 (s), 1110 (w), 1080 (vw), 1062 (s), 1065 (m), 990 (vw), 970 (w), 950 (s), 905 (vw), 870 cm^{-1} (m).

Compound 10. NMR ($CDCl_3$, Me_4Si) δ 0.80 (singlet, sharp), 1.22 (singlet, sharp), 1.50 (shoulder, broad); infrared (CCl_4), 2940 (vs), 2860 (s), 1470 (s), 1445 (m-s), 1365 (m), 1350 (vw), 1320 (vw), 1280 (w), 1240 (w), 1215 (w), 1190 (w), 1172 (w), 1155 (w), 1065 (s), 1005 (m, sh at 990), 950 (m-w, sh at 930), 910 (m-w), 872 (m-w).

Compound 11. NMR ($CDCl_3$, Me_4Si) δ 1.23 (singlet, sharp), 1.50 (shoulder, broad), 3.11 (singlet, sharp); infrared (CCl_4) 2430 (vs), 2860 (s), 2820 (vw), 1470 (vs), 1450 (s), 1370 (w), 1350 (vw), 1325 (w), 1280 (w), 1245 (m), 1320 (w), 1190 (vw), 1175 (vw), 1160 (m), 1145 (w), 1105 (s), 1100 (s), 1080 (vw), 1065 (vs), 1005 (m), 990 (w), 965 (w), 950 (m), 915 (m), 872 cm^{-1} (m).

Compound 12. NMR ($CDCl_3$, Me_4Si) δ 1.23 (singlet, sharp), 1.40–2.30 (complex); infrared (CCl_4) 2930 (vs), 2860 (s), 1470 (s), 1450 (s), 1320 (w), 1285 (w), 1110 (s), 1065 (s), 1015 (s), 1000 (s), 960 (m), 925 (m), 970 cm^{-1} (m).

Registry No.—**1**, 36079-74-0; **2**, 33525-84-7; **3**, 36079-75-1; **4**, 53783-78-1; **5**, 57951-51-6; **6**, 57951-52-7; **7**, 53783-75-8; **8**, 53783-73-6; **9**, 53783-72-5; **10**, 53783-71-4; **11**, 32616-65-2; **12**, 57951-53-8; III (n = 11), 50782-53-1; cyclopentanone, 120-92-3; cyclohexanone, 108-94-1; cycloheptanone, 502-42-1; cyclooctanone, 502-49-8; cycloundecanone, 878-13-7; cyclopentadecanone, 502-72-7; 2-methylcyclohexanone, 583-60-8; 4-methylcyclohexanone, 589-92-4; 4-ethylcyclohexanone, 5441-51-0; 4-*tert*-butylcyclohexanone, 98-53-3; 4-methoxycyclohexanone, 13482-23-0; 2-adamantanone, 700-58-3; cyclohaxacosane, 297-16-5; cycloheptacosane, 297-23-4; cyclooctacosane, 297-24-5; cycloheptacosanolide, 57951-54-9; cyclooctacosanolide, 57951-55-0; cyclononacosanolide, 57951-56-1.

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Bromination of Nitroalkanes with Alkyl Hypobromites

Victor L. Heasley,* Don R. Titterington, and Tracy L. Rold

Department of Chemistry, Point Loma College, San Diego, California 92106

Gene E. Heasley

Department of Chemistry, Bethany Nazarene College, Bethany, Oklahoma 73008

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Recently we reported that methyl hypobromite adds to olefins by an ionic mechanism if the solvent is polar, such as methylene chloride, and in the presence of a radical inhibitor.[1] In another publication we showed that alkyl hypochlorites react with olefins in nitromethane by chlorinating the solvent to give chloronitromethane rather than adding to the olefin.[2] In this reaction the olefin functions as a catalyst. Certain aromatics also catalyze chlorination of nitromethane (naphthalene and the xylenes) while other aromatics react with alkyl hypochlorites in nitromethane to give chloroaromatics.[3]

On the basis of these observations we became interested in determining what type of reaction would occur when alkyl hypobromites are added to olefins in nitromethane. Methyl hypobromite could add to the olefin as occurred with the less polar solvent methylene chloride, or bromonitromethane might be the product, by analogy with the reaction of alkyl hypochlorites in nitromethane. Finally, we considered that it was possible that the polar solvent nitromethane might cause even *tert*-butyl hypobromite to add to olefins by an ionic mechanism.

When *tert*-butyl hypobromite was added to 1-hexene in nitromethane we were surprised to find that in addition to bromonitromethane (**1**), dibromonitromethane (**2**) and tribromonitromethane (**3**) were also formed.

$$CH_2{=}CH(CH_2)_3CH_3 + (CH_3)_3COBr \xrightarrow{CH_3NO_2} \underset{\mathbf{1}}{BrCH_2NO_2} + \underset{\mathbf{2}}{Br_2CHNO_2}$$

$$\underset{\mathbf{3}}{Br_3CNO_2} + (CH_3)_3COH$$

The catalytic role of the olefin, 1-hexene, was established by the fact that no reaction occurred between *tert*-butyl

hypobromite and nitromethane unless 1-hexene was present. We tried several other olefins, such as 1-butene, 1-propene, styrene, cyclohexene, α-methylstyrene, and 1,2-dichloroethylene, and all of them functioned as catalysts except 1,2-dichloroethylene, which has strong electron-withdrawing groups. The relative amounts of **1**, **2**, and **3** did not depend on the structure of the olefin.

The amounts of **1**, **2** and **3** that are formed depend on the method of addition. For example, if *tert*-butyl hypobromite is added instantly to a solution of 1-hexene in nitromethane the products are formed in the following amounts (yield, 85%); 1 (68%), 2 (10%), and 3 (22%). On the other hand, if the hypobromite is added dropwise the following product mixture is formed (yield, 88%): 1 (24%), 2 (5%), and 3 (71%). Furthermore, when the dropwise addition was followed by VPC, we observed that initially 1 is formed, but as more hypobromite is added increasing amounts of 2 and 3 are formed. The concentrations of 1 and 2 soon reach a constant level and addition of more hypobromite results in formation of 3. These results suggest that 3 is formed from 2, and 2 in turn is formed from 1, and that there is an increase in reactivity toward the hypobromite in the order of $2 > 1 > CH_3NO_2$. In a separate experiment we established that 2 reacts with *tert*-butyl hypobromite to give 3. Using the two different addition procedures and an appropriate olefin as a catalyst offers a unique method for synthesizing large quantities of 1 or 3: the olefin can be removed very easily if it is low boiling (1-butene); the reactions are "clean", with very few by-products; 1 can be made in large quantities simply by carrying out the rapid addition procedure on a large scale; large quantities of 3 can be produced by adding the alkyl hypobromite dropwise to the nitromethane–olefin solution until most of the nitromethane has been converted to 3; and the 1 and 3 can be separated easily by distillation, since both are relatively free of the other. Methyl hypobromite also reacted with olefins in nitromethane to give results which were essentially identical with those of *tert*-butyl hypobromite.

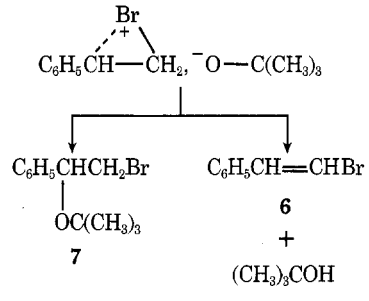
A careful study was made on the addition of *tert*-butyl hypobromite to styrene in nitroethane (rapid addition, see the following equation) to determine whether addition of hypobromite to the double bond of styrene had occurred.

$$C_6H_5CH{=}CH_2 + (CH_3)_3COBr \xrightarrow{CH_3CH_2NO_2} CH_3\underset{\displaystyle Br}{\underset{|}{C}}HNO_2 + CH_3\overset{\displaystyle Br}{\overset{|}{\underset{|}{C}}}{-}NO_2 + (CH_3)_3COH$$

4 **5**

Besides 4 (50%) and 5 (18%), small amounts of β-bromostyrene (**6**, <1%) and the ionic addition product 2-bromo-1-*tert*-butoxy-1-phenylethane (**7**, 5%) were also detected.[4] Apparently 6 and 7 are formed by the following mechanism:

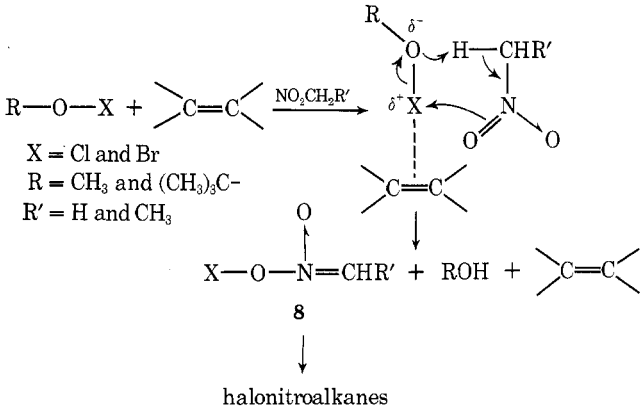
$$C_6H_5CH{=}CH_2 + (CH_3)_3OBr \xrightarrow{CH_3CH_2NO_2}$$



Dibromostyrene (15%) was also formed in the above reaction. In fact, we always observe the formation of dibromides when alkyl hypobromites are allowed to react with olefins, even when no bromine was detectable in the hypobromite solution. We have no explanation for their formation.[6] An analogous study on the addition of *tert*-butyl hypobromite to styrene in nitromethane gave approximately the same amount of addition product (**7**); of course, **1**, **2**, and **3** were also formed. We anticipated an increase in the amount of **6** and **7** in nitromethane since it is more polar than nitroethane but this did not occur. Methyl hypobromite reacted with styrene in nitromethane and nitroethane to give similar amounts of substitution (β-bromostyrene) and addition (2-bromo-1-methoxy-1-phenylethane) products to that of *tert*-butyl hypobromite.

The one striking difference between the reactions of alkyl hypobromites and hypochlorites with olefins in nitromethane involves the formation of tribromonitromethane (**3**). Under no conditions was the formation of trichloronitromethane (Cl_3CNO_2) observed even when *tert*-butyl hypochlorite was added dropwise until a very concentrated solution of chloronitromethane ($ClCH_2NO_2$) was formed. Apparently the reactivity of chloronitromethane toward hypochlorite is not greater than nitromethane itself (it may even be less reactive). The order of reactivity of $2 > 1 > CH_3NO_2$ would appear to follow the expected acidities of the hydrogens in the bromonitromethanes (**1** and **2**). However, if the acidities of the hydrogens in 1 and 2 do account for the order of reactivity with the bromonitromethanes we have no explanation why this also should not be a factor in the chloronitromethanes since the same order of acidities would be expected here.

Although we are uncertain of the mechanisms in the reactions of alkyl hypohalites with olefins in nitromethane, these reactions (alkyl hypochlorites and hypobromites) do appear to have several areas in common. Both react with olefins in nitroalkanes in the presence of the radical inhibitor oxygen, suggesting that ionic reactions are involved; and the rates of reaction of both hypohalites are dependent on the basicity of the olefins. Taking into consideration these similarities and the catalytic role of the olefin, the mechanism is probably best described as we did previously with hypochlorites,[2] and modified here to include the hypobromites:



halonitroalkanes

An explanation for the difference in product formation between rapid and slow addition may involve the fact that the reaction between nitromethane, hypobromite, and the olefin to give 8 (where R′ = H) is considerably more rapid than decomposition of 8 to 1. Therefore, under the conditions of rapid addition most of the hypobromite would be converted to 8 before a substantial amount of monobromonitromethane (**1**) was formed. Consequently, the simultaneous concentrations of hypobromite and 1 would be low

and little dibromonitromethane (2) would be formed. In the case of slow addition there would be ample time for 8 to give 1 and the hypobromite would preferentially react with the more reactive 1 than nitromethane.[7]

Experimental Section

Reaction Conditions. To a stirred solution of 0.004 mol of olefin in 11.2 ml of nitroalkane maintained at ice-bath temperatures was added 2 ml (1.2 M) of *tert*-butyl hypobromite–carbon tetrachloride solution. (The concentrations of the alkyl hypobromites solutions varied somewhat depending on the preparation.) The reactions were essentially instantaneous but stirring was continued for a short time. Slow addition of the hypobromite was done with a dropping funnel or a dropping pipet. Rapid addition simply involved letting the hypobromite solution run in directly from a volumetric pipet. The reaction products were analyzed directly by VPC. The synthesis of methyl hypobromite has been described previously;[1] we used this same procedure to make *tert*-butyl hypobromite. As was the case with the alkyl hypochlorites, no reaction occurred between the alkyl hypobromites and the nitroalkanes unless the olefins were present.

Identification of Products. Bromonitromethane (1) and tribromonitromethane (3) were synthesized unambiguously by addition of the appropriate amount of bromine to a solution of nitromethane and base, and were identified by comparison of their infrared spectra with the reported spectra for these compounds.[8] Dibromonitromethane (2) was prepared as described for 1 and 3, and its structure was confirmed from its infrared spectrum (absorption bands, cm^{-1}), C–H, 2400; $-NO_2$, 1325 and 1575; C–Br, 600 and 675; and from its boiling point; reported,[9] 58–60 °C (13 mm) [175 °C (760 mm)]; found, 50 °C (5.5 mm) [180 °C (760 mm)]. 1-Bromonitroethane (4) and 1,1-dibromonitroethane (5) were synthesized unambiguously as previously described.[10] The ir spectrum of 4 also compared favorably with the reported spectrum.[7] The compound responsible for peak 4 was isolated from the reaction product (hypobromite, olefin, and nitroethane) by preparative VPC; the ir spectrum of the collected compound was identical with that of the unambiguously synthesized 4, with the exception of a small carbonyl absorption (contaminant) in the later. Compounds 1, 2, 3, and 5 were confirmed as products by comparisons of the retention times of the peaks assigned to them with the retention times of the authentic compounds. Styrene dibromide was prepared by addition of bromine to styrene. β-Bromostyrene was synthesized unambiguously by the decarboxylation of 2,3-dibromocinnamic acid.[11] The synthesis of 2-bromo-1-methoxy-1-phenylethane has been described previously.[12] 2-Bromo-1-*tert*-butoxy-1-phenylethane was isolated by preparative VPC and identified by its NMR spectrum:[13] δ 1.20 [s, 9, $C(CH_3)_3$], 3.35 (d, 2, CH_2), 4.65 (t, 1, CH), 7.35 (s, 5, C_6H_5).

Analysis Procedure. Compounds 1, 2, and 3 were separated on a 4 ft × 0.25 in. column packed with 2% DNP on Chromosorb W (60/80 mesh) DCMS at 65 °C (flow rate 60 ml/min He); under these conditions the retention times (min) were respectively 3.3, 6.5, and 13.0. The internal standard was *p*-bromochlorobenzene. Compounds 4 and 5 were separated as described for the bromonitromethanes with the exception that the column was 8 ft; the retention times (min) were respectively 4.8 and 9.1. The internal standard was *o*-bromotoluene. Compounds 6, 7, 2-bromo-1-methoxy-1-phenylethane, and styrene dibromide were separated on the same column as used for the bromonitromethanes (column temperature 100 °C) with the following retention times (min), respectively: 4.6, 11.7, 6.9, and 15.8.

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Registry No.—1, 563-70-2; 2, 598-91-4; 3, 464-10-8; 4, 563-97-3; 5, 7119-88-2; 6, 103-64-0; 7, 57951-57-2; nitromethane, 75-52-5; nitroethane, 79-24-3; methyl hypobromite, 28078-73-1; *tert*-butyl hypobromite, 1611-82-1; styrene dibromide, 7436-90-0; bromine, 7726-95-6; styrene, 100-42-5; 2-bromo-1-methoxy-1-phenylethane, 13685-00-2.

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(5) It is conceivable that the addition product could result from addition of Br_2 (formed by decomposition of the alkyl hypobromite) to styrene in the presence of *tert*-butyl alcohol (from the reaction of hypobromite with nitroethane) or by solvolysis of styrene dibromide in the presence of *tert*-butyl alcohol. We eliminated these possibilites by bromination of styrene in a mixture of nitroethane–*tert*-butyl alcohol and by solvolysis of styrene dibromide in the same solution, and determining that no addition product was formed.
(6) In our earlier study[1] on the addition of alkyl hypobromites to 1-hexene and styrene in dichloromethane, we also observed dibromide formation. At that time, we absolutely confirmed, using ultraviolet spectroscopy, that no more than a trace of molecular bromine was present in the hypobromite solutions. In the present study, more dibromides were formed when oxygen was passed through the reaction solution.
(7) At the moment of addition the concentration of hypobromite is high with rapid addition. It is conceivable that under these conditions the reaction occurs by a mechanism which is second order in hypobromite involving an anion of the structure $(R-O-Br-O-R)^-$; this would be analogous to bromination with molecular bromine in which the tribromide ion (Br_3^-) is involved. However, at this time we see no way of accounting for the difference in products between the two methods of addition on the basis of the structure of the anion.
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(13) The corresponding radical addition product (1-bromo-2-*tert*-butoxy-1-phenylethane) was prepared as described by Walling et al. [*J. Am. Chem. Soc.*, **87**, 1715 (1965)] and its spectrum was compared with that of **7**. The spectra were essentially identical except for differences in chemical shifts.

Chlorination of Cyclopentadiene and 1,3-Cyclohexadiene with Iodobenzene Dichloride and Trichloramine

Victor L. Heasley,* Kerry D. Rold, and David B. McKee

Department of Chemistry, Point Loma College, San Diego, California 92106

Gene E. Heasley

Department of Chemistry, Bethany Nazarene College, Bethany, Oklahoma 73008

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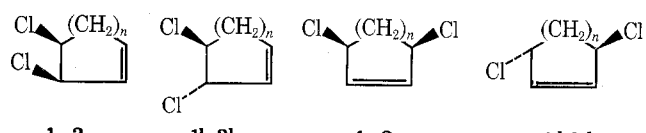
It has recently been shown that trichloramine reacts with olefins to give vicinal dichlorides by a radical mechanism,[1] and that iodobenzene dichloride and olefins also form vicinal dichlorides by either an ionic or radical mechanism depending on the conditions.[2] Since we had recently established the structures of the stereoisomeric dichlorides that result from chlorination of cyclopentadiene (1)[3] and 1,3-cyclohexadiene (2),[4] we felt that it would be of interest to



1a,2a 1b,2b 1c,2c 1d,2d

compare the product ratios from these chlorinating agents with those from molecular chlorine with the object being to obtain information on the bonding in the intermediate radicals and ion pairs. The products from reaction of the dienes with antimony pentachloride are also included for comparison purposes.[5]

The structures of the dichloride products are shown