Chemistry of gem-Dihalocyclopropanes. X. Configuration Determination of 2,2,2',2'-Tetrahalobicyclopentyl Derivatives from Dipole Moment Measurements; Crystal Structure of meso-2,2,2',2'-Tetrachloro-3,3,3',3'-tetramethylbicyclopropyl

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The dipole moments of several alkylated 2,2,2',2'-tetrahalobicyclopentyl derivatives (2 and 3) and 1,1-dihalocyclopropanes (4–9) have been measured in benzene solution. These results are analysed to determine the configurations of the former compounds and it turns out that the isomers with the lower dipole moments (< 1.2 D) have a meso configuration.

In order to distinguish the meso-form with certainty from the racemate an X-ray structure determination of the lower-melting form of 2,2,2',2'-tetrachloro-3,3,3',3'-tetramethylbicyclopropyl at −160 °C was carried out. The crystals are triclinic, space group P1. Using 2244 reflections recorded by counter methods the structure was refined to an R-value of 0.05. The analysis proved the compound to be the meso-form; bond lengths and angles are discussed.

The addition of dihalocarbene to a diene may yield the corresponding bis-adduct as one of the products and when properly substituted this product may be a mixture of a meso-form and a racemate. In some cases the stereoisomers have been separated.

The molecular dipole moment has proved to be a useful tool to determine the configurations of molecules containing polar substituents. In the field of cyclopropanes Kumler et al. have established the configurations of several compounds containing the gem-dibromocyclopropane system by comparing the measured dipole moments with those calculated for different configurations and conformations of the molecules. In the bis-adducts studied here only the rotation around the inter-ring C–C bond will affect the magnitude of the dipole moment; consequently it is in principle possible to calculate the dipole moment of any conformation of both the meso- and the racemic form. The measured dipole moments will correspond to a number of calculated conformations and by

Scheme 1.

analyses of these with regard to interatomic interactions it should be possible to reject some of the solutions and draw a conclusion at least with respect to the configurations of the bis-adducts.

**RESULTS**

The addition of dibromocarbene, generated from bromoform and potassium tert-butoxide, to the dienes IA, Ib and Ic gave the corresponding bis-adducts in varying yields (Scheme 1). The dienes Ib and Ic were also reacted with dichlorocarbene and in the former case 2,2,2',2'-tetrachloro-3,3,3',3' -tetrachloromethylbicyclopropyl (3b) was isolated in good yield while in the latter case no bis-adduct was detected; the only isolable product was a liquid that consisted mainly of two isomeric chlorides of so far unknown constitutions in a ratio of approximately 3:2.

The conjugated dienes IA, Ib and Ic are all symmetrically substituted and the corresponding bis-adducts can therefore be mixtures of meso- and racemic forms. As shown by thin-layer chromatography (TLC) bicyclopropyl 2b was obtained as an isomeric mixture; this result is supported by the NMR spectrum of the bis-adduct according to which the compound is a mixture of 70% of one of the stereoisomers and 30% of the other (see Experimental). On the other hand adduct 2a appeared homogeneous by TLC as was observed also with 2c which is formed only in the meso configuration.16

The product 3b could not be analyzed by TLC due to volatility; however, it was shown by gas chromatography to be a mixture of two compounds in a ratio of 3:1. Separation by preparative GLC afforded pure samples of the two isomers, m.p. 90 °C (predominant) and 102 °C. The meso-form and the racemate of 2,2,2',2'-tetramromo-3,3,3',3' -tetrachloromethylbicyclopropyl (2b) were separated by column chromatography and after recrystallization the isomers were isolated as colourless needles, the predominant isomer melting at 121 – 122 °C and the other at 135 – 136 °C.

The dipole moments of the bis-adducts and of six gem-dihalocyclopropanes, measured to get the relevant CCl₄ and CBr₄ group moments, were measured in benzene at 25 °C; these results are summarized in Table 1. It is worth noting that replacing a cyclopropyl substituent with a methyl group hardly influences the dipole moment of a substituted gem-dihalo-cyclopropane.

**DISCUSSION**

The tetrahalides 2a, 2b, 2c and 3b studied here can be considered as dimers of gem-dihalo-

cyclopropane.

![Fig. 1. Projections of the bicyclopropyl molecule showing quantities used for calculation of dipole moments.](image-url)

**Table 1.** The dipole moments of bis-adducts and gem-dihalocyclopropanes measured in benzene at 25 °C.

<table>
<thead>
<tr>
<th>Compound (m.p.)</th>
<th>Dipole moment (D)</th>
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<tbody>
<tr>
<td>2a (89.5 – 90 °C)</td>
<td>1.65</td>
</tr>
<tr>
<td>2b (102 – 102.5 °C)</td>
<td>3.74</td>
</tr>
<tr>
<td>3b (99.5 – 100 °C)</td>
<td>0.75</td>
</tr>
<tr>
<td>2,3-Dibromo-2,2,2'-tetrachlorocyclopropane (6)</td>
<td>2.49</td>
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<tr>
<td>2,3-Dibromo-2,2,2'-tetrachlorocyclopropane (7)</td>
<td>2.30</td>
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<tr>
<td>2,3-Dibromo-1,1'-dimethylbicyclopropyl (8)</td>
<td>2.34</td>
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<tr>
<td>1,1-Dichloro-2,2,2'-tetrachlorocyclopropane (9)</td>
<td>2.42</td>
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cyclopropanes; hence the dipole moment $\mu$ can be expressed approximately as a function of the torsional angle $\alpha$ about the central C–C bond (taken here as 0° for a trans configuration) and the dipole moment $\mu_1$ of a gem-dihalocyclopropane (Fig. 1). If the cyclopropane rings are assumed to be equilateral triangles, the assumption that fits experimental results well, and the dipole moment of the gem-dihalocyclopropane is directed along the normal from C2 to the C3-C1 bond as depicted (Fig. 1) simple vector addition gives the eqns. I and II for the dipole moment of a meso and a racemic bis-adduct, respectively.

$$\mu = \frac{2t/2}{(3 + \cos \phi)(1 - \cos \alpha)} I \mu_1,$$

$$\mu = \frac{2t/2}{(3 + \cos \phi)(1 + \cos(\alpha + 53.14°))} I \mu_1.$$

Among the bicyclic compounds studied the value of $\beta$ (see Fig. 1) is known only for one of the isomers of tetrachloride 3b ($127.3°$), crystal structure determination, see below). From the works of others, however, it seems as if the substituents have only a slight influence on the magnitude of this angle and an angle of $127.3°$ was therefore taken between the inter-ring C–C bond axis and the plane of each ring, i.e. $\phi = 37.3°$.

As a good approximation, the exact value of $\mu_1$ that is not known for any of the bis-adducts can be replaced by the dipole moment of an appropriate gem-dihalocyclopropane derivative. Among the measured dipole moments of compounds 4 to 9 (see Table 1) the moment of compound 7, viz. 2.30 D, was chosen as $\mu_1$ primarily because the structure best resembles each gem-dihalocyclopropyl group of the bis-adducts, but also because the magnitude of the moment is close to the average of the dipole moments of the six gem-dihalocyclopropanes recorded in Table 1. The variations in the dipole moment for meso and racemic bis-adducts as a function of $\alpha$ are shown in Fig. 2.

Agreement between calculated and measured dipole moments for the lower-melting form of bis-adduct 3b is achieved when $\alpha = 29$ and 331° (eqn. I) and $\alpha = 98$ and 155° (eqn. II). Examination of molecular models indicates that the latter values result in sterically impossible conformations whereas the first two are free of substantial steric strain; consequently the lower-melting isomer of 3b should be the meso-form. A complete X-ray analysis of this isomer proved this conclusion (see below). Hence, the higher-melting isomer of 3b must be a racemate and this is also borne out by the calculations; the conformations corresponding to both solutions of eqn. I ($\alpha = 116$ and 244°) and one of the solutions of eqn. II ($\alpha = 243°$) appear impossible because of considerable steric interactions whereas the second solution of eqn. II, $\alpha = 10°$, leads to a conformation that is sterically acceptable. It is worth noting that for this bis-adduct (and also for the tetrabromo analog 2b) the correspondence between configurations and melting points are opposite to that usually observed, i.e. the high-melting isomer is the meso form.

The values of $\alpha$ giving coincidence between calculated and observed dipole moments for the isomers of adducts 2a, 2b and 2c are compiled in Table 2. As for the stereoisomers of adduct 3b examination of scale molecular models of 2c and the isomers of 2b indicates their configurations. The lower-melting isomer of bicyclopropyl 2b must have a meso configuration with either $\alpha = 31$ or 329°, whereas for the higher-melting isomer a racemate with

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**Table 2. The solutions of eqns. I and II for the tetrabromobicyclopropyl derivatives.**

<table>
<thead>
<tr>
<th>Bis-adduct (m.p.)</th>
<th>$\alpha$ (°)</th>
<th>Eqn. I</th>
<th>Eqn. II</th>
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<tr>
<td>2a</td>
<td>9</td>
<td>351</td>
<td>117</td>
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<tr>
<td>2b (121–122°C)</td>
<td>31</td>
<td>329</td>
<td>96</td>
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<tr>
<td>2b (135–136°C)</td>
<td>103</td>
<td>257</td>
<td>23</td>
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<tr>
<td>2c</td>
<td>20</td>
<td>340</td>
<td>107</td>
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α = 23° is the only acceptable solution of eqns. I and II. Finally, bis-adduct 2c is a *meso*-form with either α = 20 or 340°, a result that is confirmed by the observed crystallographic symmetry. For the adduct 2a, however, the analyses of molecular models with respect to steric interactions are less conclusive because all four values of α correspond to sterically acceptable conformations. Since a deviation of 9° from the *s-trans* position is much more likely than more than 115°, the only isolated isomer of 2,2,2',2'-tetabromo-1,1'-dimethylbicyclopropyl is probably a *meso*-form.

Crystal structure of 2,2,2',2'-tetrachloro-3,3,3',3'-tetramethylbicyclopropyl, lower-melting form. The X-ray crystal structure analysis showed

Table 3. Selected interatomic distances, bond angles, and dihedral angles. For numbering of atoms, see Fig. 3.

<table>
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<tr>
<th>Bond distances (Å)</th>
<th>C1 – C1'</th>
<th>1.481(4)</th>
<th>C2 – C1</th>
<th>1.763(2)</th>
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<td>C2 – C12</td>
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<td></td>
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<td>C2 – C3 – C5</td>
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<td></td>
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<td>C11 – C2 – C12</td>
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<td>C4 – C3 – C5</td>
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<td></td>
<td>C11 – C2 – C3</td>
<td>119.9(1)</td>
<td></td>
<td></td>
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| Dihedral angles (°) (positive for a right-hand screw) | C1’ – C1 – C2 – C11 | −137.4(2) | C11 – C2 – C3 – C4 | −1.3(2) |
|                                                        | C1’ – C1 – C2 – C12 | 1.5(3)    | C11 – C2 – C3 – C5 | 142.6(1) |
|                                                        | C1’ – C1 – C3 – C4  | 142.4(2)  | C12 – C2 – C3 – C4 | −144.1(1) |
|                                                        | C1’ – C1 – C3 – C5  | −1.4(3)   | C12 – C2 – C3 – C5 | −0.1(1)  |

Fig. 3. Perspective view of the molecule showing bond lengths (Å) corrected for thermal libration and angles (°). Nonhydrogen atoms are represented by thermal ellipsoids defined by the principal axes of thermal vibration and scaled to include 50% probability.

the compound to be the *meso*-form, the molecular centres of symmetry being required by the crystallographic symmetry. Selected interatomic distances, bond angles and dihedral angles are listed in Table 3; bond lengths corrected for thermal libration are given in Fig. 3 which also shows the conformation, thermal ellipsoids and the numbering of the atoms. The C-H distances were found in the range 0.94(3) to 1.00(3) Å, mean value 0.96 Å.

The central C-C bond is found to be of normal length for a bicyclopropyl derivative, 1.481(4) Å, as compared to 1.487(4) Å in bicyclopropyl\(^{14}\) and 1.49(2) Å in 2,2'-dibromo-bicyclopropyl\(^{15}\); this value corresponds to that given for a single bond between \(sp^3\) hybridized carbon atoms (1.478 Å). The central C-C bond forms angles (\(\beta\)) of 127.3° with the planes of the cyclopropene rings.

The \(C_{\text{methy1}}-C_{\text{ring}}\) distance (mean value 1.511 Å) corresponds to the sum of single bond radii for \(sp^2\) and \(sp^3\) hybridized carbon atoms (1.505 Å). The Cl-C ring bond length (mean value 1.763 Å) is found to be slightly longer than the C-Cl bond found in 1,1-dichlorocyclopropane (1.754 Å)\(^{16}\), but within the accuracy of the determinations the same as in 1,1-dichloro-2,2-diphenylcyclopropane (1.754 Å)\(^{17}\). The \(C_{\text{methy1}}-C_{\text{ring}}-C_{\text{methy1}}\) angle is 113.1(2)° and the Cl-C-Cl angle 110.2(1)°; the corresponding \(C_{\text{phenyl}}-C_{\text{ring}}-C_{\text{phenyl}}\) and Cl-C-Cl angles in 1,1-dichloro-2,2-diphenyl-cyclopropane are 113.9 and 110.9°, respectively.

A survey of ring carbon-carbon bond lengths observed in various cyclopropane derivatives is given in Ref. 17. The bond lengths seem to be influenced by the type of substituents, the observations being in the range 1.44-1.56 Å with an average of 1.511 Å; standard deviation from the mean is 0.024 Å. In the present investigation two of the C-C bonds are of the same length (1.512 and 1.507 Å) as in cyclopropane itself (1.510 Å)\(^{18}\), whereas the C-C bond across the ring relative to the carbon atom with chlorine attached was found as long as 1.541 Å.

Intermolecular distances were found to be as expected for a crystal in which the packing forces are of the van der Waals type.

**EXPERIMENTAL**

**General.** NMR spectra were measured on Varian Associates A-60A and HA-100-15D spectrometers. Mass spectral data were obtained using an A.E.I. MS902 mass spectrometer. The IR spectra were recorded on a Perkin-Elmer model 457 spectrophotometer. Gas chromatographic analyses were performed with Varian Aerograph Models 90E and 711. All reactions were carried out under pure nitrogen.

1,2-Dimethyl-1,3-butadiene (1a) and 2,3-di-methyl-2,4-hexadiene (1b) are commercial products and were used without further purification, whereas 2,3-Di-ethyl-1,3-butadiene (1e) and 2,3-di-methyl-2,4-hexadiene (1b) were prepared as previously described.\(^{1}\) The purity as determined by GLC was in no case less than 98%.

**Preparation of bis-adducts.** The bis-adducts were prepared from the dienes, potassium tert-butoxide and halofluoride following the procedure of Kleveland and Skattebol.\(^{2}\)

2,2',2'-Tetrabromo-1,1'-dimethylbicyclopropyl (2a). Bis-adduct 2a was isolated in 31% yield, m.p. 99 °C from ethanol (lit.\(^{2}\) m.p. 96-99 °C). The product was shown to be homogeneous by TLC [silica gel; light petroleum (40-65 °C), carbon tetrachloride, benzene, ether, ethyl acetate and methanol], a conclusion supported by its NMR spectra in different solvents.\(^{2}\)

2,2',2'-Tetrabromo-3,3',3'-tetrabromomethylbicyclopropyl (2b). From the reaction of diene 1b and dibromocarbene adduct 2b was isolated in 65% yield, m.p. 109 °C from ethanol (lit.\(^{2}\) m.p. 109 °C). This product was shown to be a mixture of isomers by TLC [silicagel, light petroleum (40-65 °C); \(R_F\) values 0.39 and 0.55 (predominant)] and these were separated by column chromatography [silica gel (40-60 mesh), light petroleum (40-65 °C)]. After recrystallization from benzene the compounds gave sharp melting points, 121-122 °C for the predominant form and 135-136 °C for the other.\(^{1}\) H NMR (60 MHz, CCl\(_4\): the lower-melting compound): \(\delta\) 1.19 (2 H, s), 1.43 (6 H, s), 1.52 (6 H, s).\(^{1}\) H NMR (60 MHz, CCl\(_4\): the higher-melting compound): \(\delta\) 1.33 (2 H, s), 1.32 (6 H, s), 1.48 (6 H, s), 1.52 (6 H, s). The IR spectra of the two compounds as solids are almost identical but different in carbon tetrachloride. Their mass spectra did not show any significant differences; in no case was the molecular ion observed and the highest observable mass was a result of extensive fragmentation.

According to its NMR spectrum (100 MHz, CS\(_2\)) bis-adduct 2b is composed of 70% of the lower-melting and 30% of the higher-melting form.

2,2',2'-Tetrabromo-3,3',3'-di-tert-butyl-3,3'-dimethylbicyclopropyl (2c). Bicyclopropyl 2c was isolated in a yield of 25%, m.p. 142 °C from ethanol (lit.\(^{2}\) m.p. 143 °C).

2,2',2'-Tetrachloro-3,3',3'-tetrabromomethylbicyclopropyl (3b). From the reaction of diene 1b with dichlorocarbene adduct 3b was isolated

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in 80% yield, m.p. 79–80 °C from light petroleum (40–65 °C) (lit.18 m.p. 78 °C). The product could not be analyzed by TLC because no compound was visible by any means even after a short distance of migration. However, GLC analysis (30% Apiezol L, 170 °C) of an acetone solution of the bis-adduct disclosed that the compound was an isomeric mixture; the isomer with the shorter retention time amounts to 75% and the other to 25% of the mixture. The isomers were separated by preparative GLC (30% Apiezol L, 170 °C) and after recrystallization from acetone, the isomer with the shorter retention time melted at 89.5–90 °C, whereas the other melted at 102–105 °C. The IR spectra of the isomers as solids are different as well as those in carbon tetrachloride. 1H NMR spectra (60 MHz, CS₂; the lower-melting form): δ 1.03 (2 H, s), 1.32 (6 H, s), 1.41 (6 H, s). 1H NMR (60 MHz, CS₂; the higher-melting form): δ 1.11 (2 H, s), 1.19 (6 H, s), 1.37 (6 H, s). The NMR spectrum (100 MHz, CS₂) of the bis-adduct 36 confirms the ratio between the stereoisomers (3:1).

Reaction of diene 1c with dichlorocarbene. Reaction of potassium tert-butoxide [from 4.3 g (0.11 mol) of potassium], 14.3 g (0.12 mol) of chloroform and 7.7 g (0.04 mol) of diene 1c in 100 ml of dry pentane gave, except 3.5 g (46%) of unreacted starting material, 3.1 g of a liquid, b.p. 64–68 °C (0.4 mmHg), which consisted essentially of two isomers with composition C₇H₁₈Cl in a ratio of 3:2 as shown by mass spectrometry and GLC; the structures of these isomers have not yet been elucidated.

Dipole moments. Dielectric constants were measured at 25 °C in a Weilhelm Dipolometer DM01 on four different solutions in benzene. Refractive indices were measured on the same solutions in a Brice-Phoenix Differential Refractometer. Calculation of dipole moments was performed according to Hedestrand,29 using no correction for atomic polarization.

X-Ray structure determination of 3b. Single crystals were prepared by recrystallization from pentane; a specimen of approximate dimensions 0.25 × 0.25 × 0.05 mm³ was used for the X-ray experiments.

Determination of unit cell dimensions and collection of intensity data were carried out on a SYNTES PI four-circle diffractometer at -160 °C using graphite crystal monochromated MoKα-radiation. The θ/2θ scanning mode was employed with scan speeds (2θ) of 2–8° min⁻¹ depending on the peak intensity. The scan range was from 1.5° below 2θ(α₁) to 1.5° above 2θ(α₂) and background counts were taken for 0.35 times the scan time at each of the scan limits. Three standard reflections were measured for every 40 reflections; they showed a systematic fluctuation and the data were accordingly adjusted. All unique reflections with 2θ < 45° were recorded; for reflections with 45° < 2θ < 75° the intensities were measured only if larger than a preset value according to a quick scan. The standard deviations were taken as σ(I) = [C_T + (0.05C_N)]¹/², where C_T is the total number of counts and C_N is the net count.

2244 reflections with sin θ/λ < 0.85 Å⁻¹ and intensities larger than 2σ(I) were used in the structure determination. They were corrected for Lorentz and polarization effects and for absorption.

The atomic form factors were those of Doyle and Turner 21 for chlorine and carbon atoms and of Stewart, Davidson and Simpson 22 for hydrogen. Computer programs used in the structure determination are described in Ref. 23. The full-matrix least-squares program employed minimizes the quantity \[ \sum w F^2 \] where w is the inverse of the variance of the observed

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<th>y</th>
<th>z</th>
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<td>H3C5</td>
<td>698(5)</td>
<td>682(4)</td>
<td>467(4)</td>
</tr>
<tr>
<td>H3C4</td>
<td>1033(5)</td>
<td>901(5)</td>
<td>309(4)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
structure factors.

Crystal data: 2,2,2′,2′-tetrachloro-3,3,3′,3′-tetramethylbicyclopropyl, C₁₉H₁₄Cl₄, triclinic. 
α = 6.352(3) Å; β = 8.743(4) Å; γ = 8.249(4) Å;
α = 93.63(4); β = 108.97(4); γ = 109.34(4); V = 309.4 Å³. (T = 110 K) Mol.wt. 276.03. Z = 2. Dcalc = 1.48 gcm⁻³, μ(MoKα) = 9.1 cm⁻¹. Space group P1 (No. 2).

The structure was solved by Patterson methods and refined by Fourier and least-squares methods. Final refinements including all positional parameters, anisotropic thermal parameters for the heavy atoms and a common isotropic thermal parameter for hydrogen atoms yielded a conventional R-factor of 0.051, Rw = 0.066 and goodness of fit (S = (ΣwΔF²)/(n−m)¹/²) = 1.92. The parameters with estimated standard deviations are given in Table 4; the structure factor listing is available from the authors. An analysis of the thermal parameters in terms of rigid-body motion of one half of the molecule (r.m.s. ΔU = 5 × 10⁻⁶ Å²) served as the basis for correction of bond lengths for libration.

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REFERENCES


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