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## Presence of Conformational Rotamers in the Structures of Two Diaxially Substituted Dimeric Methylcyclohexanols

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**Abstract.** The structures of (1*S*,3*R*)-1-[(1*S*)-1-hydroxy-3-methylcyclohexyl]-3-methyl-1-cyclohexanol (referred to as 3-*M* hereinafter) and *trans*-1-(*trans*-1-hydroxy-4-methylcyclohexyl)-4-methyl-1-cyclohexanol (referred to as 4-*M* hereinafter) have been determined so as to provide unequivocal support for results of an ongoing study of metal-catalyzed pinacol coupling reactions. Both compounds have the chemical formula C<sub>14</sub>H<sub>26</sub>O<sub>2</sub>. Compound 3-*M*:  $M_r = 226.358$ , space group  $P1$ ,  $a = 10.453$  (3),  $b = 10.616$  (3),  $c = 11.057$  (3) Å,  $\alpha = 97.47$  (3),  $\beta = 104.23$  (3),  $\gamma = 118.72$  (3)°,  $V = 997.6$  (10) Å<sup>3</sup>,  $Z = 3$ ,  $D_m = 1.14$  (2),  $D_x = 1.131$  g cm<sup>-3</sup>,  $\lambda = 0.71069$  Å (graphite-monochromatized Mo  $K\alpha$  radiation),  $\mu = 0.068$  mm<sup>-1</sup>,  $F(000) = 378$ ,  $T = \sim 297$  K,  $R = 0.064$  for 2579 observed data ( $F > 2.5\sigma_F$ ) of 3508 unique reflections. Compound 4-*M*:  $M_r = 226.358$ , space group  $P2_1/n$ ,  $a = 10.349$  (2),  $b = 18.992$  (6),  $c = 11.143$  (3) Å,  $\beta = 108.86$  (2)°,  $V = 2067.9$  (8) Å<sup>3</sup>,  $Z = 6$ ,  $D_m = 1.11$  (2),  $D_x = 1.089$  g cm<sup>-3</sup>,  $\lambda = 0.71069$  Å (graphite-monochromatized Mo  $K\alpha$  radiation),  $\mu = 0.066$  mm<sup>-1</sup>,  $F(000) = 756$ ,  $T = \sim 297$  K,  $R = 0.064$  for 1457 ob-

served data ( $F > 4.0\sigma_F$ ) of 3643 unique reflections. The structural results reported here verify the NMR-derived conclusions as to the stereochemistry of pinacol coupling products. The pinacol coupling reactions were carried out in the presence of Al–Hg amalgam; the products are all axial although this is the less energetically favored configuration compared to the equatorial product. The lower-yield product (4-*M*) (produced in 64.6% yield of the diol as compared to the 75.8% yield of the 3-*M* diol) appears to be the energetically more favored molecule as there are fewer possible diaxial interactions. This evidence strongly suggests that the steric interactions in the intermediates formed from the Al–Hg methylcyclohexanone reaction are the determining factors with respect to product formation.

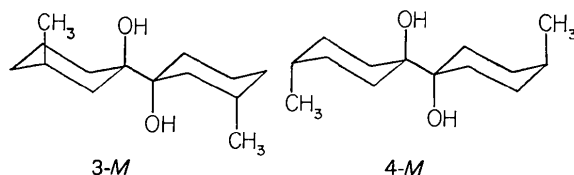
**Introduction.** The structural work on these two compounds was undertaken to verify the three-dimensional structures of two molecules produced in an ongoing study of pinacol coupling reactions (Mundy, Srinivasa, Kim, Dolph & Warnet, 1982; Mundy, Bruss, Kim, Larsen & Warnet, 1985). Nuclear magnetic resonance studies on these two compounds (3-*M*: Mundy *et al.*,

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1982; 4-*M*: Mundy *et al.*, 1985) indicated that both products contained the methyl group and the hydroxyl group in axial ring positions. The coupling reaction evidently forced the two methylcyclohexanol ring systems into this configuration. The X-ray work was carried out to substantiate the NMR results.

As shown in the structural formulas below these molecules would be expected to have substantial numbers of diaxial interactions. The details of the synthetic procedure yielding molecules with axial substituents as opposed to the thermodynamically favored equatorially substituted products are of great interest to the synthetic organic chemist.

An interesting feature of these structures relating to the number of molecules in the asymmetric unit was noted. In the 3-*M* case, three conformational forms corresponding to rotational minima about the central bond are expected for the free molecule; all three of these are observed in the crystal structure. The molecular symmetry of 4-*M* reduces the expected conformers to two (as two of the three forms become equivalent); both of these forms are observed in the crystal structure.



**Experimental.** Intensity data for both compounds were collected on a Nicolet *R3mE* four-circle diffractometer using the  $\omega$ -scan technique. In each case, three standard reflections were measured periodically throughout the course of the data collection. The decrease in the average value of these standards was 2% over the course of the data collection. Lattice parameters for 3-*M* were obtained from the least-squares fit to 25 centered reflections for which  $18 < 2\theta < 24^\circ$ . For 4-*M* the same procedure was followed using 25 centered reflections with  $16 < 2\theta < 35^\circ$ . The data were reduced in the usual manner with appropriate Lorentz and polarization corrections. Scattering factors for C, O and H and anomalous-scattering parameters for C and O were taken from *International Tables for X-ray Crystallography* (1974); all atoms were assumed to be in the zero ionization state. Both structures were solved by direct methods (Sheldrick, 1983); atom C1A was used to fix the origin for compound 3-*M*. Initial attempts at refinement in  $P\bar{1}$  would not converge; compound 3-*M* was experimentally observed to be optically active. The choice of space group *P1* was based on the observed optical activity of compound 3-*M*. H atoms were located from difference electron density maps for both compounds. The  $\sigma_F$  cut-off value was varied for both compounds in

such a manner so as to achieve an over-determinancy of  $\sim 6.0$  assuming that the H-atom (except for hydroxyl H atoms) parameters would be calculated and not refined. While refinement of the H-atom parameters was possible, the final refinements were carried out with all H atoms attached to C atoms in calculated positions (0.96 Å C–H bond length,  $U_H = 1.2U_C$  for 4-*M*,  $U_H = 0.072 \text{ \AA}^2$  for 3-*M*). C and O atoms were refined with anisotropic thermal parameters by block cascade least squares, minimizing  $\sum w(|F_o| - |F_c|)^2$  with 101 parameters refined in each full-matrix block. Hydroxyl H-atom positions and isotropic thermal parameters were refined but the O–H distance was fixed at 0.800 Å for 3-*M*. The weighting used was  $w = k \times [\sigma^2(F_o) + 0.003F_o^2]^{-1}$ . The final refinement cycles of 4-*M* produced maximum shift over e.s.d. values of  $< 0.1$  and the final  $\Delta F$  map was essentially flat with a maximum peak height of  $0.23 \text{ e \AA}^{-3}$ . The final  $\Delta F$  map for 3-*M* exhibited no peaks greater than  $0.25 \text{ e \AA}^{-3}$  but the maximum shift over e.s.d. values for the last cycles of refinement varied from 0.29 to 0.45. The relatively small crystal volume for the 3-*M* compound caused some difficulties in terms of the numbers of intense reflections that could be measured.  $D_m$  was measured by flotation. Table 1 includes additional information pertaining to the experimental work on these two structures. No corrections for secondary extinction were made or refined.

The optically active compound 3-*M* was synthesized from (*R*)-(+)-3-methylcyclohexanone purchased from the Aldrich Chemical Company (please note that the choice of space group *P1* for compound 3-*M* is consistent with the observation that the compound is optically active). The configuration used in the structure determination was the one consistent with the configuration of the chiral starting material. Both compounds were synthesized and crystallized locally.

## Discussion.

### (a) Derived parameters

Tables 2 and 3 contain the refined parameters for the two compounds.\* All bond lengths are reasonable for both compounds. The C1–C1 distances for the two compounds are 1.56 (1) and 1.553 (7) Å for 3-*M* and 4-*M*, respectively; this difference in bond lengths is not significant. When these values are compared to literature values for similar compounds they are very close to values that would be predicted where similar amounts of steric interactions occur (Burnett & Rossmann, 1971; Faber & Altona, 1974).

\* Tables of structure factors, anisotropic thermal parameters, bond lengths, bond angles, dihedral angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43926 (53 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Bond angles are all close to the theoretical value of 109° 28' for tetrahedrally hybridized carbon (Pauling, 1960, p. 114).

Table 1. *Experimental details*

	3-M	4-M
Crystal dimensions	~0.8 × 0.4 × 0.04 mm	~0.75 × 0.5 × 0.4 mm
(sinθ)/λ <sub>max</sub> (Å <sup>-1</sup> )	0.595	0.595
Range h	0 to ±10	0 to ±13
k	0 to ±12	0 to 23
l	0 to 13	0 to 14
Unique reflections measured	3508	3643
Observed reflections	2579 (2.5σ <sub>p</sub> )	1457 (4.0σ <sub>p</sub> )
Number of parameters refined	449	229
R	0.064	0.063
wR	0.070	0.052
S	1.070	1.432

Table 2. *Atomic coordinates (×10<sup>4</sup>) and equivalent isotropic thermal parameters (Å<sup>2</sup> × 10<sup>3</sup>) for compound 3-M*

	x	y	z	U <sub>eq</sub> /U <sub>iso</sub>
C1A	1355	7623	9396	45 (3)*
C2A	-221 (6)	7164 (6)	8432 (5)	55 (3)*
C3A	-393 (8)	6952 (7)	7010 (7)	72 (4)*
C4A	1021 (7)	8362 (7)	6861 (7)	72 (4)*
C5A	2556 (6)	8684 (7)	7761 (7)	73 (3)*
C6A	2691 (6)	8939 (6)	9186 (5)	51 (3)*
C7A	-583 (9)	5497 (8)	6325 (7)	88 (4)*
OA	1632 (4)	6416 (4)	9172 (4)	62 (2)*
HOA	935	5671	9226	72 (2)
C1B	1382 (5)	7926 (5)	10842 (5)	44 (2)*
C2B	2894 (6)	8203 (6)	11788 (5)	56 (3)*
C3B	3183 (7)	8700 (8)	13238 (6)	72 (4)*
C4B	3006 (7)	10018 (7)	13579 (6)	74 (3)*
C5B	1450 (8)	9718 (8)	12689 (7)	72 (4)*
C6B	1294 (8)	9342 (6)	11261 (7)	68 (4)*
C7B	2245 (9)	7427 (9)	13770 (7)	87 (5)*
OB	134 (4)	6650 (4)	10926 (4)	54 (2)*
HOB	-631	6690	10713	72 (2)
C1C	8641 (5)	2371 (5)	10584 (5)	40 (2)*
C6C	10242 (6)	2794 (6)	11533 (5)	52 (3)*
C5C	10332 (7)	3029 (9)	12947 (6)	68 (4)*
C4C	9125 (8)	1718 (9)	13127 (6)	78 (4)*
C3C	7481 (7)	1274 (6)	12304 (5)	57 (3)*
C2C	7370 (6)	1095 (6)	10885 (6)	61 (3)*
C7C	6982 (8)	2310 (7)	12760 (7)	73 (4)*
OC	8489 (4)	3632 (4)	10879 (4)	53 (2)*
HOC	9278	4382	10951	72 (2)
C1D	8536 (5)	2018 (5)	9134 (5)	40 (2)*
C6D	7065 (7)	1852 (7)	8223 (6)	66 (3)*
C5D	6819 (7)	1371 (7)	6769 (6)	75 (4)*
C4D	6854 (8)	-37 (7)	6389 (7)	76 (4)*
C3D	8381 (6)	196 (5)	7199 (5)	53 (3)*
C2D	8725 (5)	718 (5)	8691 (5)	42 (2)*
C7D	9724 (7)	1190 (6)	6817 (6)	63 (3)*
OD	9824 (4)	3389 (4)	9031 (4)	56 (2)*
HOD	10567	3315	9124	72 (2)
C1E	5411 (5)	5194 (5)	10765 (5)	45 (2)*
C6E	4467 (6)	3990 (6)	11315 (5)	43 (3)*
C5E	4901 (9)	4340 (8)	12725 (8)	73 (5)*
C4E	4990 (7)	5771 (6)	13333 (5)	55 (3)*
C3E	6134 (6)	7085 (5)	12955 (5)	53 (3)*
C2E	5738 (5)	6729 (5)	11488 (4)	43 (2)*
C7E	7823 (7)	7626 (7)	13699 (6)	65 (3)*
OE	6956 (4)	5403 (4)	11043 (3)	52 (2)*
HOE	6876	4606	10976	72 (2)
C1F	4610 (5)	4801 (5)	9236 (4)	35 (2)*
C2F	5670 (7)	6115 (6)	8777 (6)	49 (3)*
C3F	5170 (7)	5694 (6)	7223 (5)	48 (3)*
C4F	5019 (8)	4231 (8)	6691 (7)	72 (4)*
C5F	3867 (8)	2958 (8)	7055 (6)	59 (4)*
C6F	4388 (7)	3321 (5)	8561 (5)	54 (3)*
C7F	3764 (8)	5757 (8)	6564 (6)	73 (4)*
OF	3111 (4)	4587 (4)	8972 (3)	52 (2)*
HOF	3200	5361	9286	72 (2)

\* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized *U*<sub>ij</sub> tensor.

Table 3. *Atomic coordinates (×10<sup>4</sup>) and equivalent isotropic thermal parameters (Å<sup>2</sup> × 10<sup>3</sup>) for compound 4-M*

	x	y	z	U <sub>eq</sub> /U <sub>iso</sub>
C1A	5539 (4)	8697 (2)	544 (5)	50 (2)*
C2A	4380 (5)	8494 (3)	-645 (4)	78 (3)*
C3A	4887 (5)	8287 (3)	-1748 (4)	104 (3)*
C4A	5934 (6)	7689 (3)	-1382 (5)	83 (3)*
C5A	7089 (4)	7888 (2)	-200 (4)	66 (2)*
C6A	6590 (4)	8105 (2)	889 (4)	50 (2)*
C1B	5024 (4)	8904 (2)	1657 (4)	46 (2)*
C2B	4242 (4)	8323 (2)	2053 (4)	50 (2)*
C3B	3789 (5)	8515 (2)	3178 (4)	74 (3)*
C4B	4961 (6)	8760 (3)	4335 (5)	91 (3)*
C5B	5679 (6)	9362 (3)	3919 (5)	94 (3)*
C6B	6172 (4)	9166 (2)	2810 (4)	69 (2)*
C7A	5291 (5)	6987 (3)	-1247 (5)	118 (3)*
C7B	5917 (6)	8163 (3)	4969 (5)	123 (4)*
OA	6153 (3)	9326 (2)	269 (3)	97 (2)*
HOA	6742 (40)	9293 (23)	131 (41)	46 (16)
OB	4047 (3)	9467 (1)	1216 (3)	77 (2)*
HOB	4389 (42)	9722 (22)	881 (40)	63 (17)
C1E	359 (4)	178 (2)	650 (3)	39 (2)*
C2E	-250 (4)	899 (2)	757 (4)	52 (2)*
C3E	377 (4)	1240 (2)	2054 (4)	63 (2)*
C4E	316 (4)	777 (2)	3151 (4)	64 (2)*
C5E	971 (4)	70 (2)	3050 (4)	67 (2)*
C6E	330 (4)	-285 (2)	1760 (4)	53 (2)*
C7E	-1138 (4)	698 (3)	3196 (4)	89 (3)*
OE	1765 (3)	316 (2)	742 (3)	58 (2)*
HOE	2184 (44)	-2 (22)	984 (44)	70 (20)

\* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized *U*<sub>ij</sub> tensor.

### (b) H bonding and crystal structure

The H-bonding schemes for these two compounds are shown in Figs. 1(a) and 1(b). It is immediately obvious that there are great similarities in the two structures in terms of H bonding and, hence, the crystal structure. A 'tetramer' appears to be the basis for the H-bonding scheme and is responsible for the long chains of H-bonded molecules that make up the crystal structure. The arrangement of the methyl substituents in compound 3-M (*anti* form)\* apparently causes the dicyclohexyl portion of the *anti* form to be rotated relative to the analogous molecule in the 4-M structure. This rotation caused by steric effects results in elongation of the H bonds that, while probably not significant in a statistical sense, certainly seems to be substantial. The distances and e.s.d.'s for the atoms involved in the H-bonding scheme are included in Figs. 1(a) and 1(b). Two of the H atoms in both structures are involved in bifurcated H bonds (Hamilton & Ibers, 1968, p. 166). The bond lengths and angles for the H atoms involved in bifurcated bonding arrangements are within experimental error of those observed in the MgSO<sub>4</sub>·4H<sub>2</sub>O structure (Baur, 1964, 1965). The H atoms that would be expected to take part in internal H bonds in the *gauche* conformers are also the atoms that are involved in the bifurcated H bonds in the crystal structure.

\* We have utilized the terms *gauche* and *anti* to describe the relative positions of the two O atoms in the various conformers found in these structures.

It should be noted that the optical activity of 3-*M* allows three conformational rotamers corresponding to energy minima; all three of these occur in the crystal structure. Comparing the two structures, one finds the same H-bonding scheme in 4-*M* but, owing to the symmetry in the 4-*M* molecule, the higher-symmetry space group  $P2_1/n$  reduces the asymmetric unit to one and one half molecules. The *anti* conformer is described by half a molecule with the second half being generated by a center of symmetry. The 4-*M* *gauche* conformer is really equivalent to both of the 3-*M* *gauche* conformers as the 'two' *gauche* conformers in 4-*M* are related by a center of symmetry as seen in Fig. 1(b).

The crystal structure and packing arrangements for these two compounds are shown in Figs. 2(a) and 2(b). Some study of the stereoview of the 3-*M* packing diagram will indicate to the reader how all three possible low-energy rotational conformers are present in the crystal structure and are necessary for the existence of the H bonding that is of primary importance to the stability of this crystal structure.

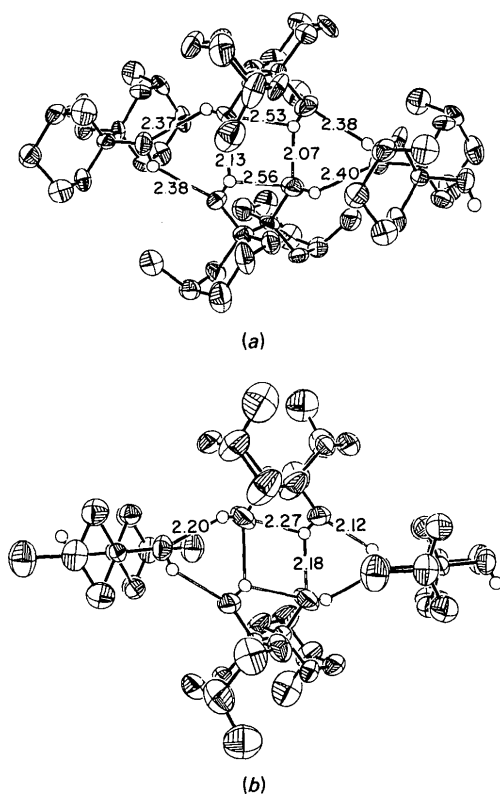


Fig. 1. (a) Thermal-ellipsoid plot of the 'tetramer' involved in the H-bonding scheme for compound 3-*M*. H atoms have been assigned isotropic  $B$  values of  $1.0 \text{ \AA}^2$  for clarity. (b) Thermal-ellipsoid plot of the 'tetramer' involved in the H-bonding scheme for compound 4-*M*. H atoms have been assigned isotropic  $B$  values of  $1.0 \text{ \AA}^2$  for clarity. E.s.d.'s are  $\sim 0.01\text{--}0.02 \text{ \AA}$ .

### (c) Conformers, conformational analysis

Thermal-ellipsoid plots of the various conformers of 3-*M* and 4-*M* are shown in Figs. 3(a)–3(e). Both *gauche* forms and the one possible *anti* form of 3-*M* are found to exist in the crystal structure. Figs. 3(c) and 3(d) also illustrate the axial–axial substitution pattern for these two compounds. The number of diaxial interactions between the methyl and hydroxyl groups in 4-*M* is substantially reduced relative to 3-*M*; this is particularly easy to see in the *anti* forms of the two molecules. Apparently, these three forms are so similar in terms of steric interactions that they are found in equal amounts in the crystal structure (and presumably in solution, although the H bonding necessary for the crystal structure requires the presence of all three forms as previously mentioned). Internal H bonds are found in both *gauche* forms as might be expected when compared to other substituted diols (Eliel, 1962, pp. 132–133 and references therein). Since all six groups on each C1–C1 are of about the same size and are significant in terms of steric interactions, the effects of internal H bonds appear to be negligible but the long-range H bonding involved in the crystal structure may be significant in terms of observing the presence of all three possible rotamers.

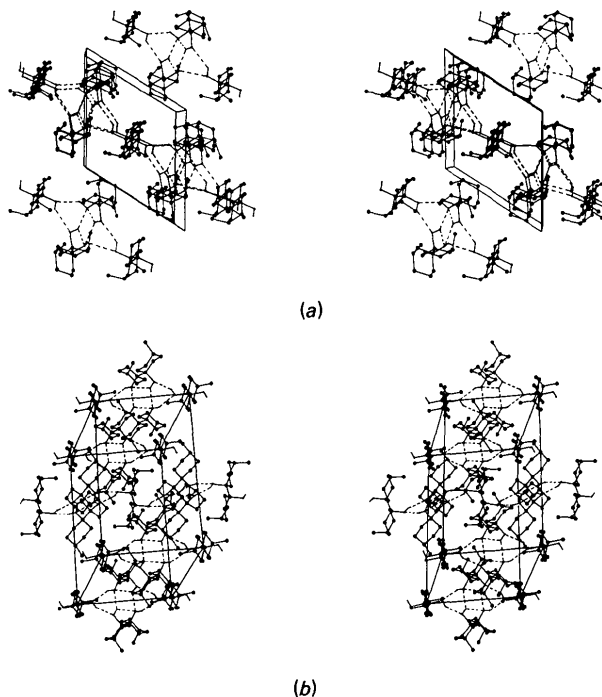


Fig. 2. (a) Stereoscopic view illustrating packing and H bonding in the crystal structure of compound 3-*M*. (b) Stereoscopic view illustrating packing and H bonding in the crystal structure of compound 4-*M*.

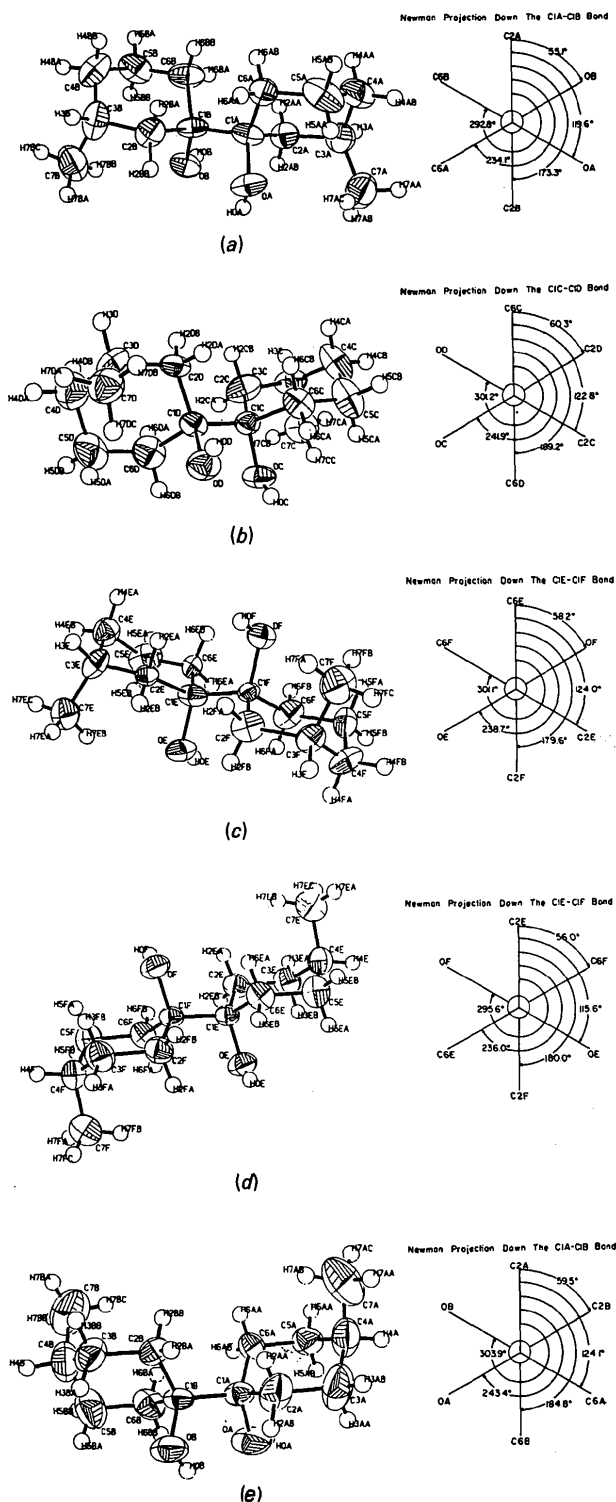


Fig. 3. Thermal-ellipsoid plots illustrating labeling in the different conformers of compounds 3-*M* and 4-*M*. H atoms have been assigned isotropic  $B$  values of  $1.0 \text{ \AA}^2$  for clarity. Newman projections of each molecule along the central C—C bond are also included. E.s.d.'s of torsion angles  $\sim 0.4^\circ$ . (a) *gauche* form of 3-*M*; (b) second *gauche* form of 3-*M*; (c) *anti* form of 3-*M*; (d) *anti* form of 4-*M*; (e) *gauche* form of 4-*M*.

Newman projections of all forms of 3-*M* are also shown in Figs. 3(a)–3(c) and of 4-*M* in Figs. 3(d)–3(e). For 3-*M* none of the dihedral angles are more than  $8^\circ$  from predicted values for the conformational-energy minima. The dihedral angles for 4-*M* do not differ from expected values for conformational-energy minima by more than  $4^\circ$ . Dihedral angles in the cyclohexyl rings are smaller, particularly at the C1—C1 junction, than for cyclohexane itself (Geise, Buys & Mijlhoff, 1971) reflecting a flattening of the ring system. For the portion of the cyclohexyl rings at the junction, the slight flattening from the  $55.9^\circ$  observed in cyclohexane to the  $52.1^\circ$  average value for dihedral angles involving C1, C2 and C6 observed for 4-*M* appears to be significant and apparently allows the compound to relieve some of the stress introduced in these highly substituted systems.

These two structures validate the structural conclusions obtained through NMR spectroscopy work on these compounds. One can also conclude that since all three rotamers exist in the crystal structure they must all be of about equal energy. It is expected that the *gauche* forms may attain additional stabilization through internal H-bond formation; indeed, these internal H bonds are contained in the crystal structure and the H atoms involved form bifurcated H bonds suggesting that the internal H bonds are fairly important energetically. Alternately, one can argue that the crystal structure itself forces the formation of the bifurcated H bonds and the existence of the three rotamers as a result of the long-range H-bonding stabilization of the crystal structure. While there may be some validity to this argument, certainly one does not expect to see a conformation appearing in the crystal that is substantially higher in energy than other conformations that exist in solution.

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