The evaluation of the $(xy\frac{1}{4})$ surface energies also indicates stability of the BPFB mixed crystal. In this paper the surface energy means the energy necessary to cleave the crystal along the given plane (namely to make two surfaces). Fig. 6 shows plots of the $(xy\frac{1}{4})$ surface energy *vs* molar ratio. The surface energy of BPFB does not significantly vary in the range 44–87% PFB, whereas the surface energies for BA and PFB structures are reduced by mixing a small amount of PFB or BA into the pure crystal. An increase of PFB in the BPFB crystal decreases the dispersion energy and increases the Coulomb energy. Since the Coulomb energy compensates the dispersion energy, the total surface energy keeps approximately the same value. The unchangeable surface energy of BPFB reflects the random disorder of BA and PFB acceptable for the intermolecular interactions across the $(xy\frac{1}{4})$ plane. Hence the surface energy also supports stability of the BPFB structure.

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Pitfalls of a Disordered Structure: Distinguishing the Compound Present, 4-Iodomethyl-1,7,7-trimethyl-3-oxabicyclo[2.2.1]heptan-2-one, from 5-Iodomethyl-1,2,2-trimethyl-4-oxabicyclo[3.2.0]heptan-3-one

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Abstract

4-Iodomethyl-1,7,7-trimethyl-3-oxabicyclo[2.2.1]-heptan-2-one, $C_{10}H_{15}IO_2$, $M_r = 294{\cdot}13$, orthorhombic, *Pnma*, $a = 18{\cdot}768\,(2)$, $b = 8{\cdot}029\,(1)$, $c = 7{\cdot}467\,(2)$ Å, $V = 1125{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}736$ g cm^{-3}, Mo $K\alpha$ radiation (graphite-monochromated), $\lambda = 0{\cdot}70926$ Å, $\mu = 26{\cdot}15$ cm^{-1}, $F(000) = 576$, $T = 295$ K, 1054 unique reflections, 766 with $I \geq 2\sigma(I)$, final $R = 0{\cdot}030$. The molecule sits on and is disordered about a crystallographic mirror plane. It is possible, by interchanging three atoms with their mirror-related partners, to generate a different compound, 5-iodomethyl-1,2,2-trimethyl-4-oxabicyclo[3.2.0]heptan-3-one, but it is unlikely that this is the species present, since refinement of this compound produced several

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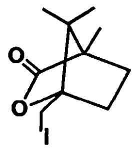
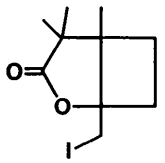
chemically unacceptable bond lengths and angles even with bond-length constraints applied. The title compound did not require any constraints during refinement and all bond lengths and angles were satisfactory. This is an important example of how disordered structures may lead to incorrect conclusions about the actual compound present, unless the results of refinement are inspected closely.

Introduction

Photolysis of 1-iodonorbornane in methanol gave 1-methoxynorbornane (Poindexter & Kropp, 1974, 1976). An analogous photolysis on 4-iodocamphor has been carried out as a possible route to 4-methoxycamphor. However, no 4-methoxycamphor was isolated. Instead, a 31% yield of the title compound, 4-iodomethyl-1,7,7-trimethyl-3-oxabicyclo[2.2.1]-heptan-2-one (1), was obtained. This compound, formed as a result of rearrangement (Yates & Loutfy, 1975), has acquired, from the air, an additional O atom. The structure of (1) has been confirmed by single-crystal X-ray analysis.



(1)



(2)

Experimental

Compound (1) was formed from the photolysis of 4-iodocamphor in methanol by using the procedure of Poindexter & Kropp (1974, 1976). It was obtained as crystals (from EtOAc), m.p. 380·5–381·5 K. 100 MHz ^{1}H NMR ($CDCl_3$, Me_4Si as internal standard): δ 0·95 (6H, *s*), 1·1 (3H, *s*), 1·5–2·2 (4H, *m*), 3·4 (2H, *s*). 25 MHz ^{13}C NMR ($CDCl_3$, Me_4Si): δ 51·59 [*s*, C(1), C(7)], 178·62 [*s*, C(2)], 91·42 [*s*, C(4)], 28·33, 33·48 [*t*, C(5), C(6)], 16·52 [*q*, C(8), C(9)], 10·64 [*q*, C(10)], 1·43 [*t*, C(11)]. Elemental analysis for $C_{10}H_{15}IO_2$, found: C 40·96, H 5·17, I 43·19%, calculated: C 40·84, H 5·14, I 43·15%.

Crystal dimensions 0·3 x 0·3 x 0·2 mm, Enraf–Nonius CAD-4 diffractometer, lattice parameters from 25 reflections with $\theta = 13$–16°. Intensities for $1 \leq \theta \leq 25°$, *hkl*: 0 to 22, 0 to 9, −1 to 8, ω–2θ scan, ω-scan width $(0{\cdot}7 + 0{\cdot}35\tan\theta)°$ at 0·7–4·0° min^{-1}, extended 25% on each side for background measurement, three standard reflections (no decay), intensities reduced to a standard scale (Cameron & Cordes, 1979), Lp corrections applied, no corrections for extinction. Absorption corrections were calculated (Walker & Stuart, 1983), but made no significant difference to the final structure. 1370 reflections measured, 1054 unique ($R_{\rm int} = 0{\cdot}019$) and 766 considered observed with $I \geq 2\sigma(I)$. The structure was solved by Patterson and Fourier methods, and refined by using the *SHELX*76 system (Sheldrick, 1976). Scattering factors for neutral atoms were obtained from *International Tables for X-ray Crystallography* (1974) and were corrected for the real part of anomalous dispersion. H atoms were placed by geometric calculation [d(C—H) = 1·08 Å]. The final refinements were with anisotropic temperature factors on the non-H atoms and individual isotropic temperature factors on the H atoms. A two-block-matrix least-squares method was employed, minimizing $\sum w(|F_o| - |F_c|)^2$ where $w = 0{\cdot}161/[\sigma^2(F_o) + 0{\cdot}00277|F_o|^2]$. The refinement converged at $R = 0{\cdot}030$, $wR = 0{\cdot}030$ for 764 observed reflections, 133 parameters, $R = 0{\cdot}047$ for all 1054 reflections, max. shift/e.s.d. = 0·4, max. residual electron density in the final difference Fourier map 0·58 e Å^{-3} within 1·0 Å of the I atom and 0·36 e Å^{-3} elsewhere. Reflections 020 and 011 were omitted because of suspected extinction. A damping factor was applied to the least-squares shifts during refinement. Although the refinement had converged, this procedure virtually fixes the $U_{\rm iso}$ for the H atoms close to the initially set value of 0·05 and produces standard deviations for these parameters which must be considered to be unrealistically low.

Discussion

Table 1 contains the final positional parameters of the non-H atoms of (1). Interatomic distances and interbond angles are listed in Table 2 and torsion angles around the bicyclic atoms are given in Table 3.* A view of the molecule is shown in Fig. 1.

The systematic absences indicated that the space group of (1) was either *Pnma* or *Pn*2$_1$*a*. Synthetic, racemic camphor was used in the preparation of (1). Subsequently the product is also racemic and does not exhibit any optical rotation. Furthermore there is no detectable piezoelectric effect with the crystals. This is consistent with both of the above space groups which possess glide planes and require that both enantiomers of a chiral compound be present.

When dealing with related centrosymmetric and noncentrosymmetric space groups, such as *Pn*2$_1$*a* and *Pnma*, it is often difficult to distinguish which is the correct space group to use for the analysis, particularly if the molecule sits across the mirror plane of the centrosymmetric space group but the molecule itself

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51352 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic positional parameters and equivalent isotropic thermal parameters* (Å^2) *for the non*-H *atoms of* (1) *with e.s.d.'s in parentheses*

All atoms have site-occupation factors of 0·5. $U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}
I	−0·09671 (3)	0·7641 (6)	0·86589 (7)	0·0615
C(1)	0·1421 (4)	0·6980 (7)	0·5241 (9)	0·0512
C(2)	0·0767 (3)	0·7637 (12)	0·4131 (7)	0·0475
O(2)	0·0680 (3)	0·7676 (10)	0·2557 (5)	0·0594
O(3)	0·0235 (2)	0·7782 (5)	0·5315 (5)	0·0384
C(4)	0·0542 (2)	0·7561 (10)	0·7101 (6)	0·0413
C(5)	0·0639 (4)	0·5691 (8)	0·7392 (9)	0·0555
C(6)	0·1274 (5)	0·5288 (8)	0·6152 (10)	0·0708
C(7)	0·1308 (4)	0·8247 (9)	0·6798 (9)	0·0480
C(8)	0·1306 (5)	1·0053 (9)	0·6196 (10)	0·0670
C(9)	0·1812 (4)	0·7899 (8)	0·8413 (8)	0·0602
C(10)	0·2098 (4)	0·7050 (9)	0·4139 (10)	0·0737
C(11)	0·0096 (4)	0·8563 (8)	0·8414 (9)	0·0506

Table 2. *Interatomic distances* (Å) *and interbond angles* (°) *for* (1) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·572 (10)	C(1)–C(7)	1·559 (10)
C(2)–O(2)	1·187 (7)	C(4)–C(7)	1·557 (10)
C(2)–O(3)	1·339 (8)	C(7)–C(8)	1·518 (11)
O(3)–C(4)	1·463 (7)	C(7)–C(9)	1·558 (10)
C(4)–C(5)	1·528 (11)	C(1)–C(10)	1·515 (11)
C(5)–C(6)	1·542 (12)	C(4)–C(11)	1·557 (10)
C(6)–C(1)	1·544 (10)	I–C(11)	2·136 (8)
C(2)–C(1)–C(6)	112·8 (7)	C(5)–C(4)–C(7)	104·9 (6)
C(2)–C(1)–C(7)	93·9 (6)	C(5)–C(4)–C(11)	119·6 (6)
C(2)–C(1)–C(10)	110·9 (6)	C(7)–C(4)–C(11)	114·5 (6)
C(6)–C(1)–C(7)	102·8 (6)	C(4)–C(5)–C(6)	102·3 (6)
C(6)–C(1)–C(10)	115·0 (6)	C(1)–C(6)–C(5)	102·6 (6)
C(7)–C(1)–C(10)	119·6 (6)	C(1)–C(7)–C(4)	90·2 (5)
C(1)–C(2)–O(2)	129·7 (7)	C(1)–C(7)–C(8)	113·8 (6)
C(1)–C(2)–O(3)	105·3 (5)	C(1)–C(7)–C(9)	112·2 (6)
O(2)–C(2)–O(3)	123·3 (6)	C(4)–C(7)–C(8)	112·2 (7)
C(2)–O(3)–C(4)	107·3 (5)	C(4)–C(7)–C(9)	112·6 (6)
O(3)–C(4)–C(5)	107·2 (6)	C(8)–C(7)–C(9)	113·7 (6)
O(3)–C(4)–C(7)	100·8 (5)	I–C(11)–C(4)	112·7 (5)
O(3)–C(4)–C(11)	107·9 (5)		

Table 3. *Selected torsion angles* (°) *for* (1) *with e.s.d.'s in parentheses*

C(1)–C(2)–O(3)–C(4)	10·1 (8)	O(3)–C(2)–C(1)–C(7)	−46· (1)
C(1)–C(6)–C(5)–C(4)	−3·1 (9)	O(3)–C(2)–C(1)–C(10)	−170· (1)
C(1)–C(7)–C(4)–O(3)	−56·1 (9)	O(3)–C(4)–C(5)–C(6)	72·5 (9)
C(1)–C(7)–C(4)–C(5)	55· (1)	C(4)–C(7)–C(1)–C(6)	−56· (1)
C(1)–C(7)–C(4)–C(11)	−172· (1)	C(4)–C(7)–C(1)–C(10)	175· (1)
C(2)–C(1)–C(6)–C(5)	−61· (1)	C(5)–C(6)–C(1)–C(7)	40· (1)
C(2)–C(1)–C(7)–C(4)	58·2 (9)	C(5)–C(6)–C(1)–C(10)	171· (1)
C(2)–O(3)–C(4)–C(5)	−79·3 (9)	C(6)–C(5)–C(4)–C(7)	−34·1 (9)
C(2)–O(3)–C(4)–C(7)	30· (1)	C(6)–C(5)–C(4)–C(11)	−164· (1)
O(3)–C(2)–C(1)–C(6)	60· (1)		

does not contain that mirror plane. The use of the centrosymmetric space group then requires the inclusion of disorder in the model. Although the noncentrosymmetric space group does not require the inclusion of disorder, and might therefore appear to be the preferable choice, the centrosymmetric space group can only be excluded from consideration if there is some significant indication for its exclusion. Such an indication could be an improved refinement in the noncentrosymmetric space group, based on the Hamilton R test (Hamilton, 1965), for one direction of the polar axis in $Pn2_1a$ compared with the other direction, or refinement in *Pnma*. Accordingly refinement of the structure of (1) was carried out in *Pnma* and in $Pn2_1a$ using, separately and then together, both directions of the polar axis. Anisotropic refinement of the non-H atoms in $Pn2_1a$ led to the same R, 0·041, for each polar axis direction. Refinement in $Pn2_1a$ with both enantiomers present in 50% occupation led to a significant improvement in R to 0·033. This latter refinement was essentially the same as refining the disordered structure in *Pnma*, except without the constraint of a mirror plane. Nevertheless, in the $Pn2_1a$ refinement, the two enantiomers were related closely by a mirror plane. This strongly suggested that use of the space group *Pnma* with the atoms disordered across the mirror plane was satisfactory and was further supported when refinement gave $R = 0·034$.

Final refinements, therefore, were carried out in *Pnma* with all atoms having site occupation factors of 0·5. The molecule almost contains a mirror plane within itself. Atoms C(1), C(2), C(4), C(7), C(9), C(10), O(2) and O(3) all lie within 0·5 Å of the xz plane that contains the I atom, which itself lies only 0·1 Å from the crystallographic mirror plane. These atoms establish a 'false mirror' plane within the molecule, which is close to the crystallographic mirror plane. The C(8) atom is related almost exactly by the 'false mirror' to atom C(6), while the two remaining atoms, C(5) and C(11), are not related by this 'mirror' plane. The atom generated by the 'false mirror' from C(5) is 1·65 Å from C(11). It should be noted that because the I atom is very close to the crystallographic mirror plane, the electron densities from the two mirror-related I atoms overlap. This produces a significant increase in electron density at a point close

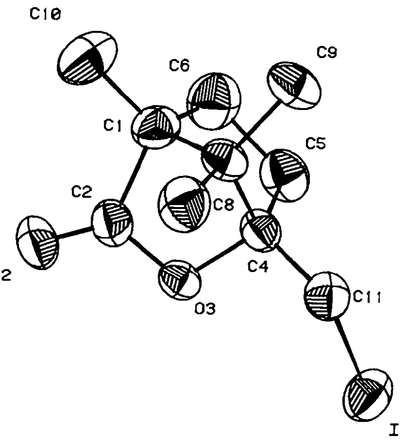

Fig. 1. A thermal ellipsoid plot of the molecule of (1) showing the atom-numbering scheme; H atoms have been omitted for clarity.

to the centres of the I atoms and on the mirror plane between them. The consequent uncertainty in the position of the I atom is reflected in the standard deviation for its *y* coordinate.

One drawback with the *Pnma* refinement is that because many of the *y* parameters are very close to 3/4, which is a mirror plane, many disordered pairs of atoms are very close together and consequently the least-squares matrix is nearly singular. The results therefore must be treated with caution. The angles C(2)—C(1)—C(7) and C(6)—C(1)—C(7) [93·9 (6) and 102·8 (6)° respectively] should be nearly equivalent because of the geometry of the molecule; however, the difference between these angles may reflect the problem of near singularity of the least-squares matrix. As discussed below, a discarded alternative configuration of the atoms led to even poorer C—C—C angles (*e.g.* 59°).

Since the 'false mirror' is close to, but not coincident with, the true mirror of *Pnma*, the crystal structure has to be constructed from two closely interpenetrating disordered molecules.* The 13 non-H atoms of the molecule, with the mirror, produce 26 locations for the atoms of the two disordered molecules. The question then arises as to which 13 of the 26 atoms make up one of the two disordered molecules. In cases of this type, where many of the atoms are close to the mirror plane, it is possible to derive several theoretical molecules by selecting different combinations of the remaining atoms drawn from either side of the mirror. As a result, two or more chemically different molecules, each of which would fit the crystallographic data, could be derived and postulated as the actual compound present. Thus, in the case of compound (1), the presence of disorder across the mirror plane makes it necessary to consider that the data could correspond to an alternative compound.

In many cases of crystal structures which have this type of disorder, the choice of the correct combination of atomic positions would be obvious since none of the alternatives would be chemically sensible. In the present determination a possible alternative compound was found which could be derived from the disordered atoms and which, until close examination of the structure was carried out, appeared to be a chemically and structurally reasonable possibility.

5-Iodomethyl-1,2,2-trimethyl-4-oxabicyclo[3.2.0]-heptan-3-one (2) can be generated from (1) by moving just three atoms, C(5), C(11) and I, across the crystallographic mirror to the positions C(5)′, C(11)′ and I′, by using the space-group symmetry operation *x*, $1\frac{1}{2}-y$, *z*. Fig. 2 shows how compound (2) is derived from compound (1). Essentially, C(5) and C(11), which are not too far from being mirror-related, are exchanged across the *xz* plane. The I atom is moved also in order to maintain a reasonable bond length to C(11). Because C(5) and C(11) are attached to C(4), which lies very close to the mirror, this change is feasible without seriously disturbing the tetrahedral coordination of C(4). The structural change that results is that C(5)′ is brought close to C(8) and bonds with it, but C(11)′ is too far from C(6) to form a bond, thereby opening one five-membered ring of (1) and creating a new four-membered ring.

The overall comparison of the structures of (1) and (2) can be described as follows. The five-membered ring composed of C(1), C(2), O(3), C(4) and C(7) remains common to both (1) and (2). The methyl group, C(8), in (1) becomes part of the four-membered ring in (2) formed by C(4), C(5)′, C(8) and C(7). The second five-membered ring of (1) is not present in (2) because of the absence of C(5). In (2) there is still one dimethyl- and one monomethyl-substituted C atom; however, the C atoms on which these substituents occur are reversed from those in (1). In (2), C(1) has become dimethyl-substituted, since C(6) is no longer involved in a ring, and C(7) is monomethyl-substituted because C(8) has now become part of the four-membered ring.

That compound (2) is feasible chemically is supported by a report of the preparation and crystal structure of a similar bicyclic compound, *cis*-bicyclo[3.2.0]-hept-2-en-6-one *p*-nitrophenylhydrazone (Murray-Rust, Murray-Rust, Brown & Newton, 1979), which contains four- and five-membered rings. Compounds (1) and (2) cannot be easily distinguished by ^{1}H or ^{13}C NMR since both would give the same spin–spin coupling pattern and would be expected to have peaks with similar chemical shifts. Therefore it was necessary to consider the possibility that (2) was the true compound present. Because of the disorder, the same refinement and *R* factor can be obtained whether (1) or (2) is the true compound. However, upon close scrutiny, several

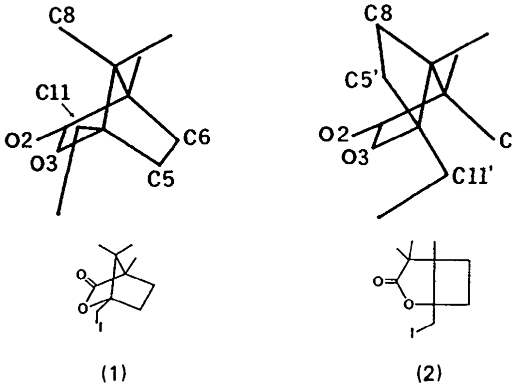

Fig. 2. A structural comparison of (1) and (2) plotted from the actual atomic coordinates. Compound (2) is generated from (1) by reflecting atoms C(5), C(11) and I in the (*x*, $\frac{3}{4}$, *z*) mirror plane.

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* The X-ray data were carefully checked photographically and no doubling of any cell length was detected.

difficulties with the reasonable chemical sense of the structure of (2) led to the conclusion that this was not the species present. The four-membered ring was distorted badly, with a short C(4)—C(5)′ distance of 1·42 Å and a very short diagonal contact for C(5)′··· C(7) of 1·67 Å. In addition, the C(5)′—C(8)—C(7) angle was 59° and the bonds around C(4) and C(7) formed severely distorted tetrahedra. Bond-length constraints were applied to the refinement in an attempt to meet the configurational requirements of the four-membered ring, but this still resulted in some angles being unacceptable and the thermal ellipsoids of C(5)′, C(6) and C(8) became thin and elongated. Furthermore, several peaks of residual electron density of approximately 1·0 e Å^{-3} remained in the region of the four-membered ring. These factors were taken as an indication that the constraints were pulling the atoms from their true positions and that the structural requirements of compound (2) were not sufficiently compatible with the data to enable (2) to be accepted as the actual compound present. Refinement based on the assumption that (1) was the true species present did not require constraints to ensure that the bond lengths and angles were reasonable chemically and all thermal ellipsoids were of an acceptable size and shape. Additionally, the maximum residual electron density was only 0·58 e Å^{-3} within 1·0 Å of the I atom and 0·36 e Å^{-3} elsewhere.

The above evidence strongly supports (1) as being the true species present in the crystal. In addition, (1) is related to the 4-iodocamphor starting material and can be derived by a known mechanism (Yates & Loutfy, 1975), while a much more unusual rearrangement would have taken place if (2) had been formed. Such a rearrangement, however, is not impossible. During the photolysis, no specific exclusion of air was attempted. It should be noted that the starting material, 4-iodocamphor, contains two chromophores and a broad-band mercury lamp was employed without a filter. It is entirely possible that an $n \rightarrow \sigma^*$ transition of the iodide could be brought about, although the $-CH_2-I$ grouping in the product is presumably photostable.

The C(11)—I distance [2·136 (8) Å] compares well with 2·134 (7) Å for triphenyliodomethyltin (Harrison & Molloy, 1978) and 2·12(4)Å for triiodomethane (Bastiansen, 1946)† obtained by electron diffraction of the vapour. The I—C(11)—C(4) angle makes the I···C(4) contact 3·062 (5) Å, which represents the closest intra- or intermolecular iodine contact to a non-H atom. There are no intermolecular distances shorter than the van der Waals distances. The shortest intermolecular contact between non-H atoms is 3·43 (1) Å for C(11)···O(2). There is no significant distortion of any part of the bicyclic rings of the molecule. Each of the wings that form the bicyclic rings are planar and each makes an angle of 115–125° with another. The backbone of the molecule, which includes atoms C(10), C(1), C(7), C(4), C(11) and I, is also planar. The maximum deviation from this plane is 0·12 Å at atom C(4).

The structural analysis presented here provides a clear example of the pitfalls which could be encountered when dealing with a disordered structure, particularly if the disorder is related by a symmetry element that passes through the molecule but the molecule itself does not possess that symmetry element. The solution to such a structure, provided either by direct methods or by Patterson and Fourier maps, might at first glance appear unambiguous and provide a satisfactory structure for a perhaps unexpected compound. Unless care is taken to examine closely all the possible structures which could be derived by taking different combinations of the disordered atoms, and to scrutinize critically any unusual bond lengths, contacts, angles or unaccountable residual electron density, the true structure might go unnoticed with the result that incorrect conclusions could be drawn.

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† Quoted in Hartl & Steidl (1980) in which are collected references that indicate a range of C—I bond lengths from 2·03 (5) to 2·24 (5) Å, depending on the crystal environment.