

sponding R of 0.061.* The weighting scheme was $w = 1/[\sigma^2(F) + 0.001F^2]$, which gave a mean $w\Delta^2$ varying only slightly with $\sin \theta$ or $|F_o|$. A final difference map had no peaks $>0.26 \text{ e } \text{Å}^{-3}$.

Final atomic coordinates are given in Table 1, and bond distances and angles in Tables 2 and 3. Diagrams of the structure are given in Figs. 2 and 3.

Discussion. The stereochemistry at the asymmetric centres is established as (3a*RS*,6*RS*,6a*RS*,7*RS*,9a*SR*). The chemical significance of this is discussed elsewhere (Owens & Raphael, 1978). The two molecules in the asymmetric unit are very similar; the only bond-length differences $>0.02 \text{ Å}$ are for C(3a)–C(3) and C(3)–O(2), and the only bond-angle difference $>2^\circ$ is for C(3a)–C(3)–O(2). There are several small torsion-angle differences (up to 8°) but the major difference is in the orientation of the benzene rings, C(12)–C(11)–C(17)–C(7) being -119.6 , -137.9° and C(16)–C(11)–C(17)–C(7) 60.6 , 42.4° in molecules *A* and *B* respectively. The angles C(5)–C(4)–C(3a) and O(11)–C(1)–C(9a) are considerably greater than 120° in both molecules, which may be due to ring strain.

* Lists of structure factors, anisotropic thermal parameters and bond distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33374 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

In the cyclohexene rings, C(3a) lies 0.56 , 0.47 Å above and C(7a) 0.17 , 0.04 Å above the plane of the other four atoms in molecules *A* and *B* respectively. This corresponds to a flattened form of the sofa conformation (Bucourt & Hainaut, 1965).

The crystallographically independent molecules are linked in pairs by two hydrogen bonds connecting the N–H and O=C of the isoindole moieties (Fig. 3). Relevant distances (Å) are O(91)⋯N(8) 2.92 , O(91)⋯H(1) 2.05 Å and N(8)⋯O(91) 2.84 , H(1)⋯O(91) 1.99 Å (first atoms in molecule *B* at x, y, z ; second atoms in molecule *A* at $1 + x, -1 + y, z$).

We thank the MRC for financial support and the SRC for provision of the diffractometer. Figs. 2 and 3 were drawn with *PLUTO* written by Dr W. D. S. Motherwell. All other crystallographic programs were written by Dr G. M. Sheldrick.

References

- BUCOURT, R. & HAINAUT, D. (1965). *Bull. Soc. Chim. Fr.* pp. 1366–1378.
 JONES, P. G. & KENNARD, O. (1977). *Acta Cryst.* B33, 627–630.
 JONES, P. G. & KENNARD, O. (1978). *Acta Cryst.* B34, 2025–2027.
 OWENS, C. & RAPHAEL, R. A. (1978). *J. Chem. Soc. Perkin Trans.* 1. In the press.

Acta Cryst. (1978). B34, 2025–2027

(3a*SR*,4*RS*,5*SR*,7a*SR*)-4-(1-Oxo-2-phenylethyl)-5,6-dimethyl-1,4,5,7a-tetrahydroisobenzofuran-3(3a*H*)-one

BY PETER G. JONES AND OLGA KENNARD*

University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England

(Received 13 January 1978; accepted 28 January 1978)

Abstract. $\text{C}_{18}\text{H}_{20}\text{O}_3$, $M_r = 285.36$, monoclinic, $P2_1/c$, $a = 12.703(4)$, $b = 11.754(4)$, $c = 10.932(4) \text{ Å}$, $\beta = 109.34(2)^\circ$, $U = 1540 \text{ Å}^3$, $Z = 4$, $D_x = 1.230$, D_m (floatation) $= 1.23 \text{ g cm}^{-3}$, $\mu(\text{Cu } K\alpha) = 5.8 \text{ cm}^{-1}$. The structure was refined to $R = 0.056$ for 1862 unique reflexions. The relative configurations at the four asymmetric centres are established to be those given in the title.

Introduction. The title compound (Fig. 1) is a synthetic intermediate prepared during studies towards the synthesis of cytochalasan (Owens & Raphael, 1978). The structure determination was undertaken to establish the relative configurations at the four asymmetric centres. A related compound, obtained in the same synthetic pathway, has also been investigated crystallographically (Jones & Kennard, 1978).

Colourless crystals were obtained from diethyl ether in two forms. The first form, approximately equi-

* External Staff, Medical Research Council.

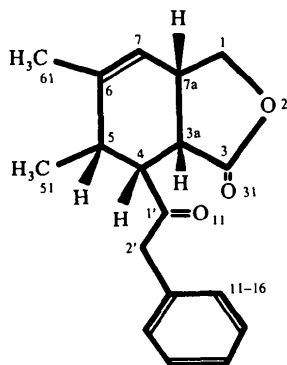


Fig. 1. The SRSS isomer of the title compound showing the numbering scheme for non-hydrogen atoms. [The H atoms are numbered such that H(*mn*) is the *n*th H on C(*m*).]

Table 1. Atom coordinates ($\times 10^4$) and isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

Anisotropic thermal parameters for C and O atoms have been deposited.

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>U</i> |
|--------|-----------|-----------|----------|----------|
| O(11) | 10542 (1) | 10139 (2) | 2583 (2) | |
| C(4) | 8916 (2) | 8983 (2) | 1764 (2) | |
| C(5) | 8235 (2) | 9911 (2) | 2147 (2) | |
| C(6) | 7004 (2) | 9636 (2) | 1619 (2) | |
| C(7) | 6620 (2) | 8606 (2) | 1251 (2) | |
| C(7a) | 7311 (2) | 7561 (2) | 1304 (2) | |
| C(1) | 7083 (2) | 6646 (2) | 2179 (3) | |
| O(2) | 7869 (2) | 6853 (2) | 3451 (2) | |
| C(3) | 8752 (2) | 7440 (2) | 3345 (2) | |
| C(3a) | 8561 (2) | 7771 (2) | 1966 (2) | |
| C(11) | 12151 (2) | 8473 (2) | 2681 (2) | |
| C(12) | 12801 (2) | 8526 (2) | 3964 (3) | |
| C(13) | 13958 (3) | 8729 (3) | 4289 (4) | |
| C(14) | 14412 (3) | 8858 (3) | 3330 (4) | |
| C(15) | 13769 (3) | 8803 (3) | 2106 (4) | |
| C(16) | 12633 (3) | 8606 (3) | 1760 (3) | |
| C(2') | 10925 (2) | 8188 (2) | 2329 (3) | |
| C(1') | 10172 (2) | 9196 (2) | 2277 (2) | |
| C(51) | 8582 (2) | 10167 (2) | 3601 (2) | |
| C(61) | 6223 (2) | 10614 (2) | 1594 (3) | |
| O(31) | 9546 (2) | 7630 (2) | 4284 (2) | |
| H(41) | 8702 | 9049 | 725 | 94 (2) |
| H(51) | 8414 | 10680 | 1711 | 94 (2) |
| H(71) | 5725 | 8504 | 875 | 94 (2) |
| H(7a1) | 7097 | 7318 | 299 | 94 (2) |
| H(11) | 7209 | 5809 | 1844 | 94 (2) |
| H(12) | 6239 | 6717 | 2192 | 94 (2) |
| H(3a1) | 9081 | 7277 | 1560 | 94 (2) |
| H(121) | 12432 | 8415 | 4716 | 94 (2) |
| H(131) | 14479 | 8781 | 5293 | 94 (2) |
| H(141) | 15296 | 9007 | 3572 | 94 (2) |
| H(151) | 14139 | 8911 | 1355 | 94 (2) |
| H(161) | 12132 | 8560 | 748 | 94 (2) |
| H(2'1) | 10657 | 7788 | 1386 | 94 (2) |
| H(2'2) | 10830 | 7597 | 3041 | 94 (2) |
| H(511) | 9453 | 10398 | 3995 | 121 (4) |
| H(512) | 8443 | 9387 | 4047 | 121 (4) |
| H(513) | 8078 | 10836 | 3797 | 121 (4) |
| H(611) | 6371 | 10939 | 2560 | 121 (4) |
| H(612) | 5347 | 10422 | 1150 | 121 (4) |
| H(613) | 6467 | 11245 | 1022 | 121 (4) |

dimensional with many well-defined faces, proved to be twinned. The second was tabular, elongated along *b*. A large crystal was cut to $0.9 \times 0.4 \times 0.2$ mm and used for data collection on an automated Syntex $P2_1$ diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were obtained by least squares from 15 strong reflexions. 2342 reflexions were measured in the range $0 < 2\theta < 116^\circ$; averaging equivalent reflexions (Lp corrections being applied) gave 1866 unique reflexions with $F > 4\sigma(F)$. Systematic absences were $0k0$, k odd and $h0l$, l odd, determining the space group as $P2_1/c$.

The structure was solved by multisolution \sum_2 sign expansion with $400 E > 1.2$, all non-hydrogen atoms being located. Isotropic least-squares refinement proceeded to $R = 0.19$, and anisotropic to 0.13. A difference synthesis then gave positions for all H atoms except two methyl H. In the final stages all H atoms were included, with C–H fixed at 1.08 Å and H–C–H at 109.5° . Four low-angle reflexions affected by extinction were omitted. Separate overall isotropic temperature factors were employed for methyl and non-methyl H. The final R was 0.056 with a corresponding $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o|$ of 0.076. The weighting scheme was $w = 1/[\sigma^2(F) + 0.001F^2]$. A final difference map had no peaks $> 0.28 e \text{ \AA}^{-3}$.

Table 2. Bond lengths (Å)

| | | | |
|-------------|-----------|-------------|-----------|
| C(4)–C(5) | 1.534 (5) | C(4)–C(3a) | 1.532 (5) |
| C(4)–C(1') | 1.527 (5) | C(5)–C(6) | 1.512 (5) |
| C(5)–C(51) | 1.533 (5) | C(6)–C(7) | 1.317 (4) |
| C(6)–C(61) | 1.513 (6) | C(7)–C(7a) | 1.499 (4) |
| C(7a)–C(1) | 1.529 (6) | C(7a)–C(3a) | 1.531 (5) |
| C(1)–O(2) | 1.439 (4) | O(2)–C(3) | 1.354 (5) |
| C(3)–C(3a) | 1.497 (4) | C(3)–O(31) | 1.198 (4) |
| C(11)–C(12) | 1.374 (4) | C(11)–C(16) | 1.350 (5) |
| C(11)–C(2') | 1.513 (5) | C(12)–C(13) | 1.413 (5) |
| C(13)–C(14) | 1.362 (7) | C(14)–C(15) | 1.318 (6) |
| C(15)–C(16) | 1.384 (6) | C(2')–C(1') | 1.512 (5) |
| C(1')–O(11) | 1.207 (4) | | |

Table 3. Bond angles ($^\circ$)

| | | | |
|-------------------|-----------|-------------------|-----------|
| C(5)–C(4)–C(3a) | 113.7 (3) | C(5)–C(4)–C(1') | 113.5 (3) |
| C(3a)–C(4)–C(1') | 115.0 (3) | C(4)–C(5)–C(6) | 110.5 (3) |
| C(4)–C(5)–C(51) | 115.3 (3) | C(6)–C(5)–C(51) | 110.1 (3) |
| C(5)–C(6)–C(7) | 123.0 (3) | C(5)–C(6)–C(61) | 115.7 (3) |
| C(7)–C(6)–C(61) | 121.3 (3) | C(6)–C(7)–C(7a) | 126.0 (3) |
| C(7)–C(7a)–C(1) | 111.7 (3) | C(7)–C(7a)–C(3a) | 112.9 (3) |
| C(1)–C(7a)–C(3a) | 101.5 (3) | C(7a)–C(1)–O(2) | 105.7 (3) |
| C(1)–O(2)–C(3) | 109.5 (3) | O(2)–C(3)–C(3a) | 110.8 (3) |
| O(2)–C(3)–O(31) | 120.6 (4) | C(3a)–C(3)–O(31) | 128.6 (4) |
| C(4)–C(3a)–C(7a) | 113.0 (3) | C(4)–C(3a)–C(3) | 115.4 (3) |
| C(7a)–C(3a)–C(3) | 103.4 (3) | C(12)–C(11)–C(16) | 119.3 (3) |
| C(12)–C(11)–C(2') | 119.3 (4) | C(16)–C(11)–C(2') | 121.3 (3) |
| C(11)–C(12)–C(13) | 119.2 (4) | C(12)–C(13)–C(14) | 119.7 (4) |
| C(13)–C(14)–C(15) | 119.9 (4) | C(14)–C(15)–C(16) | 121.7 (5) |
| C(11)–C(16)–C(15) | 120.3 (4) | C(11)–C(2')–C(1') | 114.9 (3) |
| O(11)–C(1')–C(4) | 121.0 (3) | O(11)–C(1')–C(2') | 121.8 (3) |
| C(4)–C(1')–C(2') | 117.1 (3) | | |

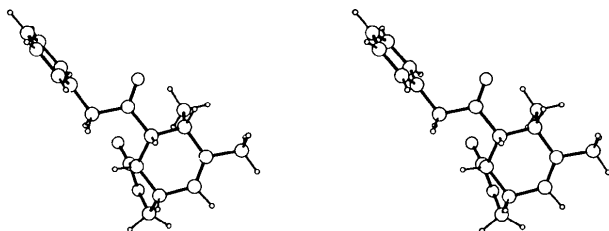


Fig. 2. Stereoview of a single molecule.

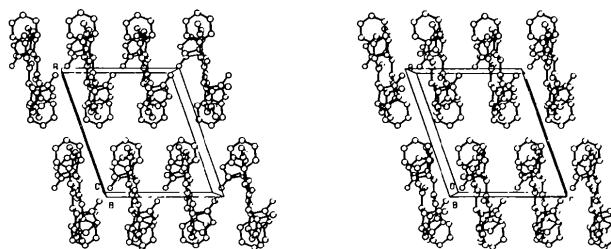


Fig. 3. Stereo packing diagram, viewed down *b*. H atoms are omitted.

Final atomic coordinates are given in Table 1, bond lengths and angles in Tables 2 and 3. Diagrams of the structure are given in Figs. 2 and 3.*

Discussion. The stereochemistry at the asymmetric centres is established as (3a*SR*,4*RS*,5*SR*,7a*SR*). The chemical significance of this is discussed elsewhere (Owens & Raphael, 1978).

Several bond lengths and angles show some deviation from ideal geometry, the most marked being C(6)–C(7)–C(7a), 126.0°, and C(3a)–C(3)–O(31), 128.6°. This may be due to strain at the ring junctions.

* Lists of structure factors, anisotropic thermal parameters and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33375 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

The cyclohexene ring adopts the sofa conformation with C(4) 0.49 Å above and C(3a) 0.15 Å below the plane of the other four atoms.

C(3) (at x, y, z) is 3.12 Å from O(11) (at $2 - x, -0.5 + y, 0.5 - z$). There are no other unusually short non-bonded distances.

We thank the MRC for financial support and the SRC for provision of the diffractometer. The figures were drawn with *PLUTO* written by Dr W. D. S. Motherwell. All other crystallographic programs were written by Dr G. M. Sheldrick.

References

- JONES, P. G. & KENNARD, O. (1978). *Acta Cryst.* **B34**, 2022–2025.
OWENS, C. & RAPHAEL, R. A. (1978). *J. Chem. Soc. Perkin Trans.* 1. In the press.

Acta Cryst. (1978). **B34**, 2027–2030

5-(3-Dimethylammonioprop-1-enylidene)-5*H*-dibenzo[*a,d*]cycloheptene Maleate, a Central Nervous System Depressant–Antidepressant

BY PETER G. JONES AND OLGA KENNARD*

University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England

AND ALAN S. HORN

Department of Pharmacy, University of Groningen, Groningen, The Netherlands

(Received 30 January 1978; accepted 21 February 1978)

Abstract. C₂₀H₂₀N⁺C₄H₃O₄⁻, $M_r = 389.46$, monoclinic, $P2_1/c$, $a = 9.554$ (2), $b = 9.656$ (2), $c = 22.799$ (6) Å, $\beta = 96.06$ (2)°, $U = 2092$ Å³, $Z = 4$, $D_x = 1.237$, D_m (floatation) = 1.24 g cm⁻³, $\mu = 6.0$ cm⁻¹ (Cu $K\alpha$). The structure was refined to $R = 0.048$ for 2553 diffractometer data. The cation and anion are

linked by a hydrogen bond. The structure is compared with that of the antidepressant imipramine.

Introduction. As part of our investigation of the structural and conformational properties of the tricyclic antidepressants (Post, Kennard & Horn, 1974, 1975; Rodgers, Kennard, Horn & Riva di Sanseverino, 1974; Rodgers, Horn & Kennard, 1975; Rodgers, Kennard,

* External Staff, Medical Research Council.