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# Structure of an Oxazepinone Derivative

BY JOCHEN ANTEL AND GEORGE M. SHELDRICK

*Institut für Anorganische Chemie der Universität, Tammannstrasse* 4, *D*-3400 *Göttingen, Federal Republic of Germany*

AND THOMAS PFEIFFER AND LUTZ-F. TIETZE

*Institut für Organische Chemie der Universität, Tammannstrasse* 2, *D*-3400 *Göttingen, Federal Republic of Germany*

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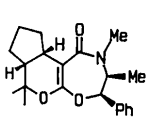
**Abstract.** (3*S*,4*R*,7a*R*,10a*S*)-1,2,3,4,7,7a,8,9,10,10a-Decahydro-2,3,7,7-tetramethyl-4-phenylcyclopenta[*d*]-pyrano[3,2-*f*][1,4]oxazepin-1-one, $C_{21}H_{27}NO_3$, $M_r$ = 341·26, orthorhombic, $P2_12_12_1$, $a$ = 7·379 (1), $b$ = 12·993 (1), $c$ = 20·094 (2) Å, $V$ = 1926·49 Å$^3$, $Z$ = 4, $D_x$ = 1·177 Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu$ = 0·07 mm$^{-1}$, $F(000)$ = 736, $T$ = 298 K, $R$ = 0·043 for 3254 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. The seven-membered ring adopts a half-chair conformation. A thermal-ellipsoid plot shows apparent high thermal motion for C(9), suggesting disorder of this atom.

**Experimental.** (I): crystal size 0·8 × 0·7 × 0·5 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 3960 reflections measured, $2\theta_{max}$ 50°, $+h+k+l$, three check reflections with no significant intensity change. 3392 unique reflections ($R_{int}$ = 0·0086), of which 3254 with $F > 4\sigma(F)$ were used for all calculations (*SHELXS*86, Sheldrick, 1985; *SHELX*76, Sheldrick, 1976). Cell constants refined from $\pm 2\theta$ values of 56 reflections in the range 20–25°. Absorption correction was not necessary. Extinction correction was applied yielding a value of 0·018 for the secondary-extinction coefficient $x$, where $F_c^* = F_c(1 + 0{\cdot}002xF_c^2/\sin 2\theta)^{-0.25}$. Structure solution by direct methods. Refinement on $F$ to $R$ = 0·043, $wR$ = 0·065; all non-H atoms anisotropic, H atoms were included using a riding model [C–H 0·96 Å, $U$(H) = 0·08 Å$^2$, except for methyl protons $U$(H) = 0·1 Å$^2$]. We refined 227 parameters, $S$ = 2·607, weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0004F^2$ which led to a featureless analysis of variance in terms of $\sin\theta$ and $F_o$, max. $\Delta/\sigma$ = 0·007, max. and min. height in final $\Delta\rho$ map 0·66 and −0·36 e Å$^{-3}$ respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).




(I)

Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51203 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (× 10$^4$) *and equivalent isotropic displacement parameters* (Å$^2$ × 10$^3$)

| | $x$ | $y$ | $z$ | $U_{eq}$* |
|---|---|---|---|---|
| O(6) | 2685 (3) | 4763 (1) | 1689 (1) | 60 (1) |
| C(5*a*) | 2363 (3) | 5364 (1) | 1159 (1) | 45 (1) |
| O(5) | 1815 (2) | 4723 (1) | 680 (1) | 53 (1) |
| N(2) | 2319 (2) | 6843 (1) | −57 (1) | 43 (1) |
| O(1) | 2890 (2) | 8043 (1) | 707 (1) | 55 (1) |
| C(1) | 2590 (2) | 7130 (1) | 582 (1) | 39 (1) |
| C(10*b*) | 2578 (2) | 6400 (1) | 1154 (1) | 40 (1) |
| C(4) | 913 (3) | 5136 (1) | 113 (1) | 39 (1) |
| C(2*n*) | 2427 (3) | 7641 (2) | −562 (1) | 53 (1) |
| C(1*p*) | 162 (3) | 4245 (1) | −287 (1) | 43 (1) |
| C(10*a*) | 2955 (3) | 6946 (1) | 1816 (1) | 47 (1) |
| C(7) | 3900 (3) | 5157 (2) | 2200 (1) | 57 (1) |
| C(3) | 2176 (3) | 5790 (1) | −306 (1) | 44 (1) |
| C(2*p*) | −1297 (3) | 4422 (2) | −700 (1) | 52 (1) |
| C(6*p*) | 945 (3) | 3281 (1) | −277 (1) | 58 (1) |
| C(7'') | 3802 (5) | 4357 (2) | 2746 (1) | 81 (1) |
| C(7*a*) | 3185 (4) | 6203 (2) | 2407 (1) | 57 (1) |
| C(3*p*) | −1965 (3) | 3649 (2) | −1101 (1) | 61 (1) |
| C(5*p*) | 244 (4) | 2502 (2) | −683 (1) | 68 (1) |
| C(4*p*) | −1184 (3) | 2689 (2) | −1093 (1) | 61 (1) |
| C(10) | 1389 (4) | 7657 (2) | 2032 (1) | 66 (1) |
| C(8) | 1340 (5) | 6209 (3) | 2755 (2) | 99 (1) |
| C(3') | 4036 (3) | 5300 (2) | −406 (1) | 70 (1) |
| C(7') | 5793 (4) | 5217 (3) | 1920 (1) | 81 (1) |
| C(9*a*) | 781 (9) | 7307 (4) | 2694 (3) | 172 (3) |

* Equivalent isotropic $U$ defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

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Table 2. *Bond lengths* (Å) *and bond angles* (°)

| | | | |
|---|---|---|---|
| O(6)–C(5*a*) | 1·343 (2) | O(6)–C(7) | 1·456 (2) |
| C(5*a*)–O(5) | 1·336 (2) | C(5*a*)–C(10*b*) | 1·355 (2) |
| O(5)–C(4) | 1·424 (2) | N(2)–C(1) | 1·352 (2) |
| N(2)–C(2*n*) | 1·453 (2) | N(2)–C(3) | 1·460 (2) |
| O(1)–C(1) | 1·234 (2) | C(1)–C(10*b*) | 1·491 (2) |
| C(10*b*)–C(10*a*) | 1·532 (2) | C(4)–C(1*p*) | 1·515 (2) |
| C(4)–C(3) | 1·516 (2) | C(1*p*)–C(2*p*) | 1·378 (3) |
| C(1*p*)–C(6*p*) | 1·379 (3) | C(10*a*)–C(7*a*) | 1·541 (3) |
| C(10*a*)–C(10) | 1·542 (3) | C(7)–C(7″) | 1·513 (3) |
| C(7)–C(7*a*) | 1·515 (3) | C(7)–C(7′) | 1·509 (4) |
| C(3)–C(3′) | 1·526 (3) | C(2*p*)–C(3*p*) | 1·379 (3) |
| C(6*p*)–C(5*p*) | 1·400 (3) | C(7*a*)–C(8) | 1·530 (4) |
| C(3*p*)–C(4*p*) | 1·374 (3) | C(5*p*)–C(4*p*) | 1·359 (3) |
| C(10)–C(9*a*) | 1·477 (5) | C(8)–C(9*a*) | 1·491 (6) |

| | | | |
|---|---|---|---|
| C(7)–O(6)–C(5*a*) | 117·7 (1) | O(5)–C(5*a*)–O(6) | 105·2 (1) |
| C(10*b*)–C(5*a*)–O(6) | 124·2 (2) | C(10*b*)–C(5*a*)–O(5) | 130·6 (2) |
| C(4)–O(5)–C(5*a*) | 118·8 (1) | C(2*n*)–N(2)–C(1) | 117·3 (1) |
| C(3)–N(2)–C(1) | 126·4 (1) | C(3)–N(2)–C(2*n*) | 115·7 (1) |
| O(1)–C(1)–N(2) | 119·0 (2) | C(10*b*)–C(1)–N(2) | 123·8 (1) |
| C(10*b*)–C(1)–O(1) | 117·1 (2) | C(1)–C(10*b*)–C(5*a*) | 129·6 (1) |
| C(10*a*)–C(10*b*)–C(5*a*) | 118·4 (2) | C(10*a*)–C(10*b*)–C(1) | 111·9 (1) |
| C(1*p*)–C(4)–O(5) | 107·9 (1) | C(3)–C(4)–O(5) | 111·6 (2) |
| C(3)–C(4)–C(1*p*) | 111·0 (1) | C(2*p*)–C(1*p*)–C(4) | 118·5 (2) |
| C(6*p*)–C(1*p*)–C(4) | 122·1 (2) | C(6*p*)–C(1*p*)–C(2*p*) | 119·2 (2) |
| C(7*a*)–C(10*a*)–C(10*b*) | 113·5 (1) | C(10)–C(10*a*)–C(10*b*) | 112·7 (2) |
| C(10)–C(10*a*)–C(7*a*) | 103·9 (2) | C(7″)–C(7)–O(6) | 103·9 (2) |
| C(7*a*)–C(7)–O(6) | 107·2 (2) | C(7*a*)–C(7)–C(7″) | 113·6 (2) |
| C(7′)–C(7)–O(6) | 108·9 (2) | C(7′)–C(7)–C(7″) | 110·5 (2) |
| C(7′)–C(7)–C(7*a*) | 112·2 (2) | C(4)–C(3)–N(2) | 112·3 (1) |
| C(3′)–C(3)–N(2) | 111·8 (2) | C(3′)–C(3)–C(4) | 113·1 (2) |
| C(3*p*)–C(2*p*)–C(1*p*) | 120·6 (2) | C(5*p*)–C(6*p*)–C(1*p*) | 119·5 (2) |
| C(7)–C(7*a*)–C(10*a*) | 112·8 (2) | C(8)–C(7*a*)–C(10*a*) | 104·5 (2) |
| C(8)–C(7*a*)–C(7) | 116·1 (2) | C(4*p*)–C(3*p*)–C(2*p*) | 120·3 (2) |
| C(4*p*)–C(5*p*)–C(6*p*) | 120·7 (2) | C(5*p*)–C(4*p*)–C(3*p*) | 119·6 (2) |
| C(9*a*)–C(10)–C(10*a*) | 107·3 (2) | C(9*a*)–C(8)–C(7*a*) | 102·3 (3) |
| C(8)–C(9*a*)–C(10) | 106·6 (3) | | |

**Related literature.** For the preparation of the compound see Pfeiffer (1988). For the preparation of some related compounds see Tietze, Brand, Pfeiffer, Antel, Harms & Sheldrick (1987).

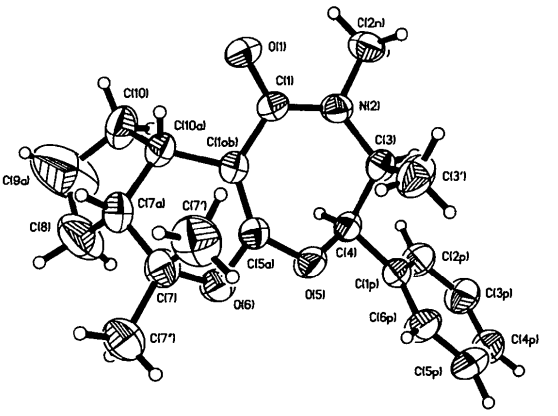


Fig. 1. The molecular structure showing atom-numbering scheme.

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# Structure of a Furo[3,4-*b*]azepine Derivative

BY JOCHEN ANTEL AND GEORGE M. SHELDRICK

*Institut für Anorganische Chemie der Universität, Tammannstrasse* 4, *D*-3400 *Göttingen, Federal Republic of Germany*

AND MATTHIAS BRATZ AND LUTZ-F. TIETZE

*Institut für Organische Chemie der Universität, Tammannstrasse* 2, *D*-3400 *Göttingen, Federal Republic of Germany*

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**Abstract.** (5a*RS*,8a*RS*)-Ethyl 6,6-dimethyl-8-oxo-perhydrofuro[3,4-*b*]azepine-8a-carboxylate, $C_{13}H_{21}NO_4$, $M_r = 255{\cdot}16$, monoclinic, $P2_1/c$, $a = 7{\cdot}189$ (1), $b = 15{\cdot}057$ (2), $c = 13{\cdot}101$ (2) Å, $\beta = 96{\cdot}62$ (3)°, $V = 1408{\cdot}65$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}204$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm$^{-1}$, $F(000) = 552$, $T =$

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