

X-Ray Structure of a Partially Saturated Pentacyclic Methylene-bridged Isoindolo[2,1-*a*][3,1]benzoxazinone

Géza Stájer^{a*}, Reijo Sillanpää^b and Kalevi Pihlaja^{b*}

^a Institute of Pharmaceutical Chemistry, Albert Szent-Györgyi Medical University P. O. B. 121, H-6701 Szeged, Hungary and ^bDepartment of Chemistry, University of Turku, SF-20500 Turku, Finland

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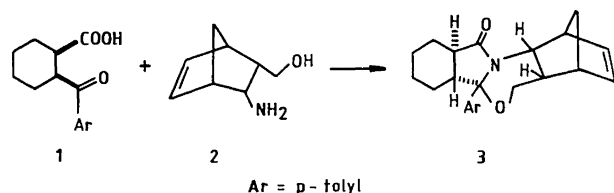
6a-*p*-Tolyl-1,4,4a,5,6a,6b,7,8,9,10,10a,12a-dodecahydro-1,4-methanoisoindolo[2,1-*a*][3,1]benzoxazin-11-one (**3**) has been synthesized from *cis*-2-(*p*-methylbenzoyl)cyclohexanecarboxylic acid (**1**) and 3-*endo*-hydroxymethylbicyclo[2.2.1]hept-5-enyl-2-*endo*-amine (**2**). The structure of the new compound was established by X-ray crystallography by using direct methods. The crystals of C₂₃H₂₇NO₂ are triclinic, space group P $\bar{1}$, $a = 9.471(5)$, $b = 11.851(4)$, $c = 9.385(4)$ Å, $\alpha = 107.84(3)$, $\beta = 108.94(4)$, $\gamma = 91.75(4)^\circ$, $V = 938.4(8)$ Å³, $Z = 2$, $D_c = 1.237$ g cm⁻³.

The syntheses and X-ray studies of a great number of structurally related condensed bicyclic heterocycles: dihydro- and tetrahydro-1,3-oxazines, tetrahydro-1,3-oxazin-4-ones, etc. have recently been reported.^{1,2} The aim of these structural studies has been to establish the configurations and conformations of these saturated heterocycles and elucidate the mutual effects of the carbocyclic and heterocyclic rings.

The present work deals with an extension of the X-ray analysis to a pentacyclic, partially saturated 3,1-benzoxazin[2,1-*a*]isoindolone derivative. The NMR structural analysis yielded only partial results owing to the severe overlap of the various signals.

Experimental

For the preparation, *cis*-2-(*p*-methylbenzoyl)cyclohexane-1-carboxylic acid (**1**)^{3,4} and 3-*endo*-hydroxymethylbicyclo[2.2.1]hept-5-enyl-2-*endo*-amine (**2**) were refluxed in toluene with *p*-toluenesulfonic acid as a catalyst. After evaporation of the mixture and purification of the residue by column chromatography (Kieselgel) crystallization from ethanol yielded 6a-*p*-tolyl-1,4,4a,5,6a,6b,7,8,9,10,10a,12a-dodecahydro-1,4-methanoisoindolo[2,1-*a*][3,1]benzoxazin-11-one (**3**) (Scheme 1), m.p. 210–212°C.



Scheme 1.

Crystal data for 3. C₂₃H₂₇NO₂, $M_r = 349.38$, triclinic, space group P $\bar{1}$, $a = 9.471(5)$, $b = 11.851(4)$, $c = 9.385(4)$ Å, $\alpha = 107.84(3)$, $\beta = 108.94(4)$, $\gamma = 91.75(4)^\circ$, $V = 938.4(8)$ Å³, $Z = 2$, $D_c = 1.237$ g cm⁻³, $\mu(\text{Mo } K_\alpha) = 0.73$ cm⁻¹, $T = 296(1)$ K; colourless cubes, crystal dimensions 0.2 × 0.28 × 0.30 mm.

Data collection, analysis and refinement. A Rigaku AFC5S diffractometer was used, with graphite-mono-

Table 1. Positional parameters ($\times 10^4$) and B_{eq} (Å²) for non-hydrogen atoms of **3**.^a

| Atom | <i>x</i> | <i>y</i> | <i>z</i> | B_{eq} |
|--------|----------|----------|----------|-----------------|
| O(1) | 6248(2) | 1243(2) | 5229(2) | 3.25(6) |
| O(2) | 5219(2) | 3706(2) | 2263(2) | 4.70(8) |
| N | 5864(2) | 2846(2) | 4228(2) | 2.84(7) |
| C(1) | 5761(3) | 1821(3) | 6555(3) | 3.6(1) |
| C(1A) | 4742(3) | 2742(3) | 6290(3) | 3.7(1) |
| C(3) | 3025(3) | 2293(3) | 5627(4) | 4.3(1) |
| C(4) | 2667(3) | 1361(3) | 3981(4) | 4.7(1) |
| C(5) | 2765(3) | 1934(3) | 3003(4) | 4.3(1) |
| C(6) | 3210(3) | 3236(2) | 3973(4) | 3.6(1) |
| C(6A) | 4893(3) | 3378(2) | 5087(3) | 3.00(9) |
| C(8) | 5942(3) | 3058(2) | 2920(3) | 3.3(1) |
| C(8A) | 7131(3) | 2351(2) | 2440(3) | 3.1(1) |
| C(9) | 6930(4) | 1813(3) | 694(3) | 4.3(1) |
| C(10) | 8247(4) | 1131(3) | 568(4) | 5.1(1) |
| C(11) | 8430(4) | 216(3) | 1434(4) | 4.6(1) |
| C(12) | 8559(3) | 757(3) | 3207(4) | 3.7(1) |
| C(12A) | 7202(3) | 1403(2) | 3230(3) | 2.91(9) |
| C(12B) | 6958(3) | 2072(2) | 4782(3) | 2.42(8) |
| C(13) | 2412(4) | 3352(3) | 5171(4) | 4.9(1) |
| C(14) | 8378(3) | 2804(2) | 6138(3) | 2.50(8) |
| C(15) | 9358(3) | 2277(2) | 7120(3) | 3.4(1) |
| C(16) | 10669(3) | 2935(3) | 8319(3) | 4.0(1) |
| C(17) | 11070(3) | 4130(2) | 8588(3) | 3.5(1) |
| C(18) | 10110(3) | 4651(3) | 7613(3) | 3.7(1) |
| C(19) | 8792(3) | 4007(2) | 6413(3) | 3.09(9) |
| C(20) | 12519(4) | 4831(3) | 9892(5) | 5.7(1) |

* To whom correspondence should be addressed.

^a E.s.d.s are given in parentheses.

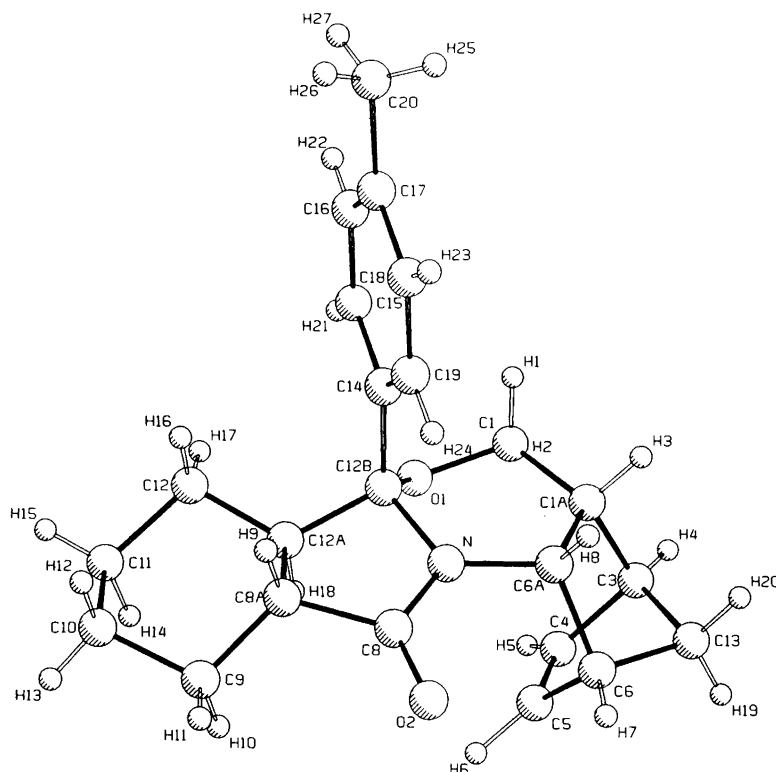


Fig. 1. The PLUTO perspective view of compound 3.

Table 2. Positional parameters ($\times 10^3$) and B_{eq} for the H atoms of 3.^a

| | x | y | z | B_{eq} |
|-------|---------|--------|---------|-----------------|
| H(1) | 666(3) | 220(2) | 750(3) | 4.5 |
| H(2) | 526(3) | 113(2) | 664(3) | 4.5 |
| H(3) | 495(3) | 339(2) | 735(3) | 4.6 |
| H(4) | 276(3) | 204(2) | 639(3) | 5.1 |
| H(5) | 253(3) | 47(3) | 377(3) | 5.6 |
| H(6) | 277(3) | 161(2) | 180(3) | 5.3 |
| H(7) | 304(3) | 381(2) | 341(3) | 4.5 |
| H(8) | 534(3) | 427(2) | 569(3) | 3.8 |
| H(9) | 807(3) | 291(2) | 296(3) | 3.7 |
| H(10) | 590(3) | 115(2) | 13(3) | 5.5 |
| H(11) | 688(3) | 243(2) | 24(3) | 5.5 |
| H(12) | 917(3) | 175(3) | 110(4) | 6.1 |
| H(13) | 807(3) | 77(3) | -51(4) | 6.1 |
| H(14) | 730(3) | -39(2) | 83(3) | 5.8 |
| H(15) | 930(3) | -13(3) | 140(3) | 5.8 |
| H(16) | 942(3) | 132(2) | 380(3) | 4.7 |
| H(17) | 856(3) | 18(2) | 377(3) | 4.7 |
| H(18) | 625(3) | 81(2) | 256(3) | 3.5 |
| H(19) | 134(3) | 318(3) | 466(3) | 5.9 |
| H(20) | 280(3) | 415(3) | 614(3) | 5.9 |
| H(21) | 910(3) | 146(2) | 695(3) | 4.1 |
| H(22) | 1124(3) | 255(2) | 893(3) | 4.7 |
| H(23) | 1032(3) | 549(2) | 774(3) | 4.3 |
| H(24) | 817(3) | 437(2) | 578(3) | 3.6 |
| H(25) | 1230(4) | 554(3) | 1047(4) | 6.7 |
| H(26) | 1316(4) | 518(3) | 948(4) | 6.7 |
| H(27) | 1314(4) | 436(3) | 1030(4) | 6.7 |

^a E.s.d.s are given in parentheses.

chromated Mo K_{α} radiation ($\lambda = 0.71069 \text{ \AA}$), in the ω -2 θ scan mode, with an ω -scan rate of $8.0^\circ \text{ min}^{-1}$ and a scan width of $(1.57 + 0.30 \tan \theta)$. The weak reflections [$I < 10\sigma(I)$] were rescanned up to two times. The data obtained were corrected for Lorentz and polarization effects. 3291 unique reflections were obtained ($2\theta_{\text{max}} = 50^\circ$). Direct methods and difference Fourier syntheses were used, with full-matrix least-squares refinement, with non-hydrogen atoms anisotropic and hydrogen atoms with fixed isotropic temperature parameters (1.2 times B_{eq} of the carrying atom). In the final cycles, the 2072 data with $I > 2\sigma(I)$ yielded an R value of 0.051 ($R_w = 0.054$, $w = [\sigma^2(F_o)]^{-1}$) for 316 parameters; maximum/minimum residual electron density = $0.37 / -0.18 \text{ e \AA}^{-3}$.

Neutral atomic scattering and dispersion factors were taken from *International Tables for X-Ray Crystallography*.⁵ All calculations were performed with *TEXSAN-89* software⁶ on a VAXSTATION 3520 computer. Figures were drawn with PLUTO [Ref. 7, cf. Fig. 1] software. The final atomic positional coordinates are listed in Tables 1 and 2. Lists of anisotropic thermal parameters and observed and calculated structure factors are available from the authors on request.

Results and discussion

The X-ray analysis of 3 yielded the PLUTO perspective view of the molecule (Fig. 1) computed from the final

Table 3. Relevant bond lengths (Å) for non-hydrogen atoms of **3**.^a

| | | | |
|---------|----------|-----------|----------|
| O1–C1 | 1.449(3) | C8A–C9 | 1.509(4) |
| O1–C12B | 1.410(3) | C8A–C12A | 1.516(4) |
| O2–C8 | 1.220(3) | C9–C10 | 1.523(5) |
| N–C6A | 1.448(3) | C10–C11 | 1.525(5) |
| N–C8 | 1.350(3) | C11–C12 | 1.550(4) |
| N–C12B | 1.479(3) | C12–C12A | 1.518(4) |
| C1–C1A | 1.504(4) | C12A–C12B | 1.526(4) |
| C1A–C3 | 1.550(4) | C12B–C14 | 1.524(3) |
| C1A–C6A | 1.576(4) | C14–C15 | 1.390(4) |
| C3–C4 | 1.521(4) | C14–C19 | 1.388(3) |
| C3–C13 | 1.519(4) | C15–C16 | 1.383(4) |
| C4–C5 | 1.321(5) | C16–C17 | 1.378(4) |
| C5–C6 | 1.496(4) | C17–C18 | 1.373(4) |
| C6–C6A | 1.567(4) | C17–C20 | 1.512(4) |
| C6–C13 | 1.522(4) | C18–C19 | 1.383(4) |
| C8–C8A | 1.526(4) | | |

^a E.s.d.s are given in parentheses.

fractional co-ordinates of non-hydrogen and hydrogen atoms listed in Tables 1 and 2, respectively. The relevant bond lengths for the non-hydrogen atoms are given in Table 3, bond angles for the non-hydrogen atoms in Table 4 and selected torsion angles in Table 5.

The conformation of the cyclohexane ring (C12A, ..., C12) is a chair (⁴C₁) which is slightly distorted toward a half-chair (²H₁). The puckering parameters⁸ are $Q = 0.583(1)$ Å, $\phi = 208(1)^\circ$ and $\theta = 175.2(1)^\circ$ [for the other enantiomer $\phi = 28(1)^\circ$ and $\theta = 4.7(1)^\circ$]. The torsion angles of the ring vary from 62.9(3) to 53.8(4)°. The conformation of the oxazine ring (O1, ..., C12B) is a

Table 4. Relevant bond angles (°) for the non-hydrogen atoms.^a

| | | | |
|-------------|----------|---------------|----------|
| C1–O1–C12B | 112.4(2) | C8A–C9–C10 | 108.2(3) |
| C6A–N–C8 | 124.4(2) | C9–C10–C11 | 112.3(3) |
| C6A–N–C12B | 122.4(2) | C10–C11–C12 | 113.6(3) |
| C8–N–C12B | 113.1(2) | C11–C12–C12A | 107.1(2) |
| O1–C1–C1A | 114.6(2) | C8A–C12A–C12 | 111.2(2) |
| C1–C1A–C3 | 116.1(2) | C8A–C12A–C12B | 102.7(2) |
| C1–C1A–C6A | 117.0(2) | C12–C12A–C12B | 122.2(2) |
| C3–C1A–C6A | 101.8(2) | O1–C12B–N | 109.1(2) |
| C1A–C3–C4 | 107.1(2) | O1–C12B–C12A | 108.5(2) |
| C1A–C3–C13 | 100.3(2) | O1–C12B–C14 | 111.8(2) |
| C4–C3–C13 | 100.6(3) | N–C12B–C12A | 100.4(2) |
| C3–C4–C5 | 107.4(3) | N–C12B–C14 | 111.7(2) |
| C4–C5–C6 | 107.1(3) | C12A–C12B–C14 | 114.6(2) |
| C5–C6–C6A | 106.1(2) | C3–C13–C6 | 93.6(2) |
| C5–C6–C13 | 101.4(3) | C12B–C14–C15 | 121.0(2) |
| C6A–C6–C13 | 100.6(2) | C12B–C14–C19 | 122.1(2) |
| N–C6A–C1A | 113.0(2) | C15–C14–C19 | 116.8(2) |
| N–C6A–C6 | 113.1(2) | C14–C15–C16 | 121.0(3) |
| C1A–C6A–C6 | 101.9(2) | C15–C16–C17 | 121.9(3) |
| O2–C8–N | 126.6(3) | C16–C17–C18 | 117.3(3) |
| O2–C8–C8A | 126.5(3) | C16–C17–C20 | 121.2(3) |
| N–C8–C8A | 106.9(2) | C18–C17–C20 | 121.5(3) |
| C8–C8A–C9 | 119.6(2) | C17–C18–C19 | 121.5(3) |
| C8–C8A–C12A | 102.4(2) | C14–C19–C18 | 121.5(3) |
| C9–C8A–C12A | 111.9(2) | | |

^a E.s.d.s are given in parentheses.

distorted envelope (E_1). The puckering parameters are $Q = 0.355(1)$ Å, $\phi = 179.2(3)^\circ$ and $\theta = 135.9(3)^\circ$ [for the other enantiomer $\phi = 359.3(3)^\circ$ and $\theta = 44.1(3)^\circ$]. The ring torsion angles, starting clockwise from O1–C1–C1A–C6A, are $-26.4(3)$, $8.4(3)$, $-18.4(3)$, $45.5(3)$, $-62.6(2)$ and $55.5(3)^\circ$, respectively. Both the puckering parameters and the torsion angles given above are close to those reported earlier for a comparable molecule.⁹ The pyrrolidine ring (C12B, ..., C12A) exists in a distorted envelope conformation with puckering parameters $Q = 0.276(1)$ Å and $\phi = 309(1)^\circ$.

In accordance with Fig. 1 the cyclohexane and pyrrolidone rings are fused *trans*-diequatorially, i.e., the configuration of the initial reactant **1** has been inverted. Similar *cis* → *trans* epimerizations have already been observed for cyclohexane-condensed dihydrouracils.¹⁰ However, according to our earlier observation these compounds can also exhibit the reverse *trans* → *cis* isomerization.¹¹ The results show that the mutual orientation of the cycles in the condensed systems often varies, depending on the structure of the reactants and the reaction conditions. In all cases, the *trans* fusion of the six-membered hetero rings condensed with cyclohexane is the more stable. The intramolecular transacylation of cyclohexane *cis*-condensed azetidones to the stable *trans*-fused pyrimidinediones by polyphosphoric acid supports this conclusion.¹²

It should be noted, however, that *cis* fusion is advantageous for the six-membered cyclopentane-condensed heterocycles containing two hetero atoms, as the *trans*-condensed analogues can be prepared only in exceptional cases.^{13,14} Compound **3** contains a cyclohexane-fused five-membered heterocycle with one hetero atom, which is further fused *via* the N–C12B bond (1.479 Å) with another six-membered heterocycle. This may be the reason for the formation of the saturated *trans*-fused isoindole instead of the expected *cis*-fusion.

It is interesting that no inversion of configuration takes place at the bicyclic norbornene-1,3-oxazine fusion: the starting *di-endo*-amino alcohol (**2**) moiety can be found unchanged in the pentacycle (**3**) formed. A similar retention of configuration was experienced by the norbornane-fused azetidones analogous to the cyclohexane derivatives, when the ring-transformation of the methylene-bridged derivatives took place *via* transacylation without inversion.¹⁵ The inversion-resistant configuration of the bicyclic compounds results from the bias caused by the methylene bridge.

An oxazole-condensed benzo analogue of **3** containing only one carbocyclic ring is already known.¹⁶ For structural reasons, however, the mutual positions of the two heterocyclic rings fused at the nitrogen–quaternary carbon bond, i.e., the stereoposition of the *p*-tolyl group relative to the fusion hydrogens on both terminal rings, have to be taken into consideration in this case. The X-ray results indicate that the aryl substituent of **3** and the axial fusion hydrogen adjacent to the carbonyl group are *cis* relative to the pyrrolidine ring. On the other hand, the

Table 5. Torsion angles (°) for 3.

| | | | |
|------------------|-----------|-------------------|-----------|
| O1-C1-C1A-C3 | 93.9(3) | C4-C3-C1A-C6A | -65.2(3) |
| O1-C1-C1A-C6A | -26.4(3) | C4-C3-C13-C6 | -48.9(3) |
| O1-C12B-N-C6A | 45.5(3) | C4-C5-C6-C6A | 70.8(3) |
| O1-C12B-N-C8 | -138.5(2) | C4-C5-C6-C13 | -33.9(3) |
| O1-C12B-C12A-C8A | 150.3(2) | C5-C4-C3-C13 | 32.2(3) |
| O1-C12B-C12A-C12 | -84.1(3) | C6-C6A-N-C8 | 51.0(3) |
| O1-C12B-C14-C15 | 39.8(3) | C6-C6A-N-C12B | -133.5(2) |
| O1-C12B-C14-C19 | -143.1(2) | C6A-N-C8-C8A | 178.3(2) |
| O2-C8-N-C6A | -0.6(4) | C6A-N-C12B-C12A | 159.4(2) |
| O2-C8-N-C12B | -176.5(2) | C6A-N-C12B-C14 | -78.6(3) |
| O2-C8-C8A-C9 | -35.6(4) | C6A-C1A-C3-C13 | -39.4(3) |
| O2-C8-C8A-C12A | -160.0(3) | C8-N-C12B-C12A | -24.6(2) |
| N-C6A-C1A-C1 | 8.4(3) | C8-N-C12B-C14 | 97.3(2) |
| N-C6A-C1A-C3 | -119.2(2) | C8-C8A-C9-C10 | -178.2(3) |
| N-C6A-C6-C5 | 51.5(3) | C8-C8A-C12A-C12 | -167.8(2) |
| N-C6A-C6-C13 | 156.6(2) | C8A-C8-N-C12B | 2.4(3) |
| N-C8-C8A-C9 | 145.6(2) | C8A-C9-C10-C11 | 53.9(4) |
| N-C8-C8A-C12A | 21.2(3) | C8A-C12A-C12-C11 | -57.2(3) |
| N-C12B-O1-C1 | -62.6(2) | C8A-C12A-C12B-C14 | -83.9(2) |
| N-C12B-C12A-C8A | 36.0(2) | C9-C8A-C12A-C12 | 62.9(3) |
| N-C12B-C12A-C12 | 161.5(2) | C9-C8A-C12A-C12B | -164.8(2) |
| N-C12B-C14-C15 | 162.4(2) | C9-C10-C11-C12 | -54.0(4) |
| N-C12B-C14-C19 | -20.5(3) | C10-C9-C8A-C12A | -58.5(3) |
| C1-O1-C12B-C12A | -171.2(2) | C10-C11-C12-C12A | 53.8(4) |
| C1-O1-C12B-C14 | 61.4(3) | C11-C12-C12A-C12B | -178.8(2) |
| C1-C1A-C3-C4 | -63.0(3) | C12-C12A-C12B-C14 | 41.7(3) |
| C1-C1A-C3-C13 | -167.6(2) | C12A-C12B-C14-C15 | -84.3(3) |
| C1-C1A-C6A-C6 | 130.1(2) | C12A-C12B-C14-C19 | 92.8(3) |
| C1A-C1-O1-C12B | 55.5(3) | C12B-C14-C15-C16 | 178.0(3) |
| C1A-C3-C4-C5 | -72.2(3) | C12B-C14-C19-C18 | -177.7(3) |
| C1A-C3-C13-C6 | 60.9(3) | C14-C15-C16-C17 | -0.5(5) |
| C1A-C6A-N-C8 | 166.1(2) | C14-C19-C18-C17 | 0.0(5) |
| C1A-C6A-N-C12B | -18.4(3) | C15-C14-C19-C18 | -0.5(4) |
| C1A-C6A-C6-C5 | -70.2(3) | C15-C16-C17-C18 | 0.0(5) |
| C1A-C6A-C6-C13 | 35.1(3) | C15-C16-C17-C20 | -179.2(3) |
| C3-C1A-C6A-C6 | 2.5(3) | C16-C15-C14-C19 | 0.7(4) |
| C3-C4-C5-C6 | 1.0(3) | C16-C17-C18-C19 | 0.2(4) |
| C3-C13-C6-C5 | 50.2(3) | C19-C18-C17-C20 | 179.4(3) |
| C3-C13-C6-C6A | -58.9(2) | C8-C8A-C12A-C12B | -35.4(2) |

p-tolyl group and the di-*exo* fusion hydrogens are also *cis* relative to each other.

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