

Table 2. *Interatomic distances (Å) and angles (°)*

| | | | |
|-------------|------------|-------------|------------|
| C2—N1 | 1.392 (10) | C6—N1 | 1.374 (10) |
| C1'—N1 | 1.482 (11) | O2—C2 | 1.214 (10) |
| N3—C2 | 1.369 (10) | C4—N3 | 1.355 (10) |
| N4—C4 | 1.312 (11) | C5—C4 | 1.425 (12) |
| C6—C5 | 1.350 (12) | C2'—C1' | 1.492 (13) |
| O4'—C1' | 1.415 (11) | C3'—C2' | 1.517 (14) |
| N1'—C3' | 1.456 (14) | C4'—C3' | 1.513 (14) |
| N2'—N1' | 1.131 (16) | N3'—N2' | 1.097 (17) |
| C5'—C4' | 1.507 (11) | O4'—C4' | 1.433 (9) |
| O5'—C5' | 1.419 (10) | | |
| C6—N1—C2 | 120.7 (8) | C1'—N1—C2 | 117.8 (8) |
| C1'—N1—C6 | 121.4 (7) | O2—C2—N1 | 123.2 (8) |
| N3—C2—N1 | 114.9 (8) | N3—C2—O2 | 121.9 (8) |
| C4—N3—C2 | 126.0 (8) | N4—C4—N3 | 121.1 (8) |
| C5—C4—N3 | 117.7 (9) | C5—C4—N4 | 121.1 (9) |
| C6—C5—C4 | 117.0 (10) | C5—C6—N1 | 123.3 (9) |
| C2'—C1'—N1 | 115.3 (8) | O4'—C1'—N1 | 106.4 (7) |
| O4'—C1'—C2' | 105.1 (7) | C3'—C2'—C1' | 102.6 (8) |
| N1'—C3'—C2' | 113.7 (9) | C4'—C3'—C2' | 103.4 (8) |
| C4'—C3'—N1' | 113.7 (12) | N2'—N1'—C3' | 125.8 (14) |
| N3'—N2'—N1' | 170.8 (21) | C5'—C4'—C3' | 114.9 (8) |
| O4'—C4'—C3' | 107.7 (7) | O4'—C4'—C5' | 108.8 (6) |
| O5'—C5'—C4' | 111.6 (8) | C4'—O4'—C1' | 107.1 (7) |

dideoxycytidine (J. N. Low, unpublished work; Silverton, Quinn, Haugwitz & Todaro, 1988), in which $\chi = -156^\circ$ and $P = 205^\circ$. The χ and P values place the molecule at the lower P edge of the C2'-endo minimum, *i.e.* close to the potential barrier between the C2'-endo and C3'-endo conformations for 2'-deoxynucleoside molecules discussed by Low, Tollin & Wilson (1982), and to this extent the conformation is unusual. The azido group, as with the other azido-substituted nucleosides referred to above, is not involved in hydrogen bonding; it does

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A Novel β -Adrenergic Receptor Antagonist MY336-a: Structure of the Tetraacetylated Compound

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Abstract. (1*R**,3*S**)-1,2,3,4-Tetrahydro-8-acetoxy-1,3-bis(acetoxymethyl)-2-acetyl-7-methoxy-6-methylisoquinoline, C₂₁H₂₇NO₈, $M_r = 421.45$, monoclinic, $P2_1$, $a = 11.892$ (2), $b = 11.067$ (2), $c = 8.4866$ (8) Å, $\beta = 92.12$ (1)°, $V = 1116.1$ (5) Å³, $Z = 2$, $D_x = 1.255$ g cm⁻³, $Cu K\alpha$, $\lambda = 1.54184$ Å, $\mu = 7.702$ cm⁻¹, $F(000) = 448$, $T = 293$ K, $R = 0.048$, $wR = 0.072$ for 2178 observed reflections. The tetrahydropyridine ring adopts a half-boat conformation.

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have two short contacts less than 3.5 Å, N1'...N3'(- x , 0.5 + y , -2 - z), 3.07 (2) Å and O2...N3'(- x , -0.5 + y , -2 - z), 3.14 (2) Å. The atoms of the azido group have high thermal parameters, a common feature discussed by Low, Tollin, Howie & Wilson (1988).

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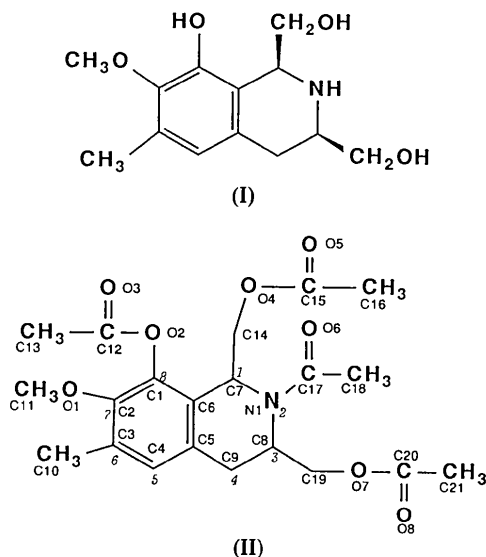
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The bond lengths and angles are within the normal ranges. There are no intermolecular contacts less than the sum of the van der Waals radii.

Introduction. MY336-a (I) was isolated from the culture broth of *Streptomyces gabonae* KY2234 and characterized as a new specific β -adrenergic antagonist based on the profile of the receptor binding assay and pharmacological properties (Kase, Fujita, Nakamura, Hashizume, Goto, Kubo & Shuto, 1986). (I) is the first microbial metabolite known to act on

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the β -adrenergic receptor. Usual chemical and spectroscopic methods could not determine the structure unequivocally. Although it was difficult to obtain good crystals of (I), the tetraacetylated compound (II) gave suitable crystals for X-ray analysis. The present X-ray analysis of (II) established the structure of (I).



Experimental. Colourless crystals from a mixture of ethyl acetate and cyclohexane (1:1), dimensions 0.50 × 0.40 × 0.40 mm. Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions from setting angles of 20 independent reflections with $36.25 \leq \theta \leq 49.14^\circ$. 2266 reflections surveyed in the range $4 \leq 2\theta \leq 150^\circ$; $-14 \leq h \leq 14$, $0 \leq k \leq 13$, $0 \leq l \leq 10$; 2178 independent reflections with $F_o > 3.0\sigma(F_o)$. Three reference reflections monitored periodically showed no significant variation in intensity. Absorption correction was not applied. A secondary-extinction correction (Zachariassen, 1963) was applied (refined coefficient 2.81×10^{-3}). Structure solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier-map recycling. Refinement using the *SDP* package (Frenz, 1985), full-matrix least-squares refinement on F , with non-H atoms having anisotropic temperature factors and H atoms (located from a difference Fourier synthesis) having isotropic temperature factors. The H atoms of the C(16) methyl group were included in the calculation of structure factors but not refined due to their high temperature factors. Weights $w = 4F_o^2/[\sigma(I_o)^2 + (0.04I_o)^2]^{1/2}/Lp$, final $R = 0.048$, $wR = 0.072$, $S = 2.64$, maximum shift/e.s.d. in the final least-squares cycle 0.95, maximum and minimum peaks in the final difference map 0.24 (3) and -0.31 (3) $e \text{ \AA}^{-3}$, respectively. The Bijvoet method

Table 1. *Positional parameters and equivalent isotropic temperature factors of non-H atoms with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i a_j$$

| | x | y | z | $B_{eq}(\text{\AA}^2)$ |
|-------|------------|------------|------------|------------------------|
| O(1) | 0.7541 (2) | 0.330 | 0.8807 (3) | 7.08 (4) |
| O(2) | 0.9826 (1) | 0.3358 (1) | 0.9522 (2) | 5.26 (3) |
| O(3) | 0.9714 (2) | 0.1390 (2) | 0.8988 (2) | 7.84 (5) |
| O(4) | 1.3118 (2) | 0.2708 (2) | 0.7038 (2) | 6.66 (4) |
| O(5) | 1.2729 (2) | 0.2180 (2) | 0.9501 (2) | 7.67 (5) |
| O(6) | 1.2869 (2) | 0.6830 (2) | 0.6944 (3) | 8.44 (5) |
| O(7) | 1.2956 (2) | 0.4159 (3) | 0.3757 (3) | 8.90 (5) |
| O(8) | 1.4517 (3) | 0.4595 (5) | 0.2538 (4) | 14.0 (1) |
| N(1) | 1.1955 (2) | 0.5071 (2) | 0.6585 (2) | 5.37 (3) |
| C(1) | 0.9420 (2) | 0.3653 (2) | 0.8012 (2) | 5.08 (4) |
| C(2) | 0.8266 (2) | 0.3695 (2) | 0.7689 (3) | 6.07 (5) |
| C(3) | 0.7847 (2) | 0.4082 (3) | 0.6215 (4) | 6.75 (5) |
| C(4) | 0.8620 (3) | 0.4429 (3) | 0.5128 (3) | 6.79 (5) |
| C(5) | 0.9768 (2) | 0.4410 (2) | 0.5436 (2) | 5.77 (4) |
| C(6) | 1.0186 (2) | 0.4005 (2) | 0.6906 (2) | 5.05 (4) |
| C(7) | 1.1437 (2) | 0.3990 (2) | 0.7241 (2) | 4.92 (3) |
| C(8) | 1.1634 (3) | 0.5464 (3) | 0.4964 (2) | 6.46 (5) |
| C(9) | 1.0628 (3) | 0.4752 (3) | 0.4254 (2) | 6.85 (6) |
| C(10) | 0.6600 (3) | 0.4078 (4) | 0.5814 (5) | 8.84 (9) |
| C(11) | 0.7347 (3) | 0.4148 (4) | 1.0030 (5) | 8.81 (8) |
| C(12) | 0.9926 (2) | 0.2176 (2) | 0.9902 (3) | 5.48 (4) |
| C(13) | 1.0367 (3) | 0.2023 (3) | 1.1561 (3) | 7.01 (6) |
| C(14) | 1.1927 (2) | 0.2804 (3) | 0.6654 (3) | 5.84 (4) |
| C(15) | 1.3406 (2) | 0.2372 (3) | 0.8521 (4) | 6.83 (6) |
| C(16) | 1.4633 (3) | 0.2268 (5) | 0.8798 (6) | 9.9 (1) |
| C(17) | 1.2572 (2) | 0.5862 (2) | 0.7503 (3) | 6.15 (5) |
| C(18) | 1.2869 (3) | 0.5549 (3) | 0.9183 (3) | 7.11 (6) |
| C(19) | 1.2626 (3) | 0.5422 (4) | 0.3886 (3) | 8.16 (7) |
| C(20) | 1.3950 (3) | 0.3868 (5) | 0.3149 (4) | 9.2 (1) |
| C(21) | 1.4188 (4) | 0.2567 (6) | 0.3236 (5) | 10.1 (1) |

(Bijvoet, Peerdeman & van Bommel, 1951) using the anomalous-dispersion term of O atoms failed to determine the absolute configuration. Scattering factors from *International Tables for X-ray Crystallography* (1974). Final fractional coordinates and equivalent B values are listed in Table 1.*

Discussion. Bond lengths, bond angles and selected torsion angles are listed in Table 2. An *ORTEPII* drawing (Johnson, 1976) of the molecule is shown in Fig. 1. The IUPAC atomic numbering for the isoquinoline ring is given in italics in the chemical structure of (II) together with the numbering scheme used in the text. The tetrahydropyridine ring adopts a half-boat conformation with C(8) and N(1) deviating by 0.890 (3) and 0.923 (2) \AA , respectively, from the least-squares plane [± 0.013 (3) \AA] defined by C(5), C(6), C(7) and C(9). Although the stabler half-chair conformation seems to be unacceptable in (II) due to the large steric hindrance between the bulky substituents, (I) may adopt a half-chair conformation. The benzene ring is nearly regular with mean C—C bond

* 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 52181 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å), bond angles (°) and selected torsion angles (°)

| | | | | | |
|-------------|-----------|-------------|-----------|-------------|-----------|
| O(1)—C(2) | 1.376 (3) | O(1)—C(11) | 1.425 (4) | O(2)—C(1) | 1.392 (2) |
| O(2)—C(12) | 1.352 (3) | O(3)—C(12) | 1.186 (3) | O(4)—C(14) | 1.445 (3) |
| O(4)—C(15) | 1.344 (4) | O(5)—C(15) | 1.197 (4) | O(6)—C(17) | 1.229 (4) |
| O(7)—C(19) | 1.457 (5) | O(7)—C(20) | 1.345 (5) | O(8)—C(20) | 1.182 (6) |
| N(1)—C(7) | 1.464 (3) | N(1)—C(8) | 1.479 (3) | N(1)—C(17) | 1.366 (3) |
| C(1)—C(2) | 1.390 (3) | C(1)—C(6) | 1.386 (3) | C(2)—C(3) | 1.397 (4) |
| C(3)—C(4) | 1.380 (4) | C(3)—C(10) | 1.508 (4) | C(4)—C(5) | 1.380 (4) |
| C(5)—C(6) | 1.399 (3) | C(5)—C(9) | 1.507 (4) | C(6)—C(7) | 1.504 (3) |
| C(7)—C(14) | 1.527 (3) | C(8)—C(9) | 1.537 (4) | C(8)—C(19) | 1.519 (5) |
| C(12)—C(13) | 1.494 (4) | C(15)—C(16) | 1.473 (4) | C(17)—C(18) | 1.496 (4) |
| C(20)—C(21) | 1.469 (8) | | | | |

| | | | |
|------------------|-----------|------------------|-----------|
| C(2)—O(1)—C(11) | 114.5 (2) | C(1)—O(2)—C(12) | 118.1 (2) |
| C(14)—O(4)—C(15) | 116.4 (2) | C(19)—O(7)—C(20) | 120.0 (3) |
| C(7)—N(1)—C(8) | 119.7 (2) | C(7)—N(1)—C(17) | 122.1 (2) |
| C(8)—N(1)—C(17) | 117.1 (2) | O(2)—C(1)—C(2) | 119.7 (2) |
| O(2)—C(1)—C(6) | 118.4 (2) | C(2)—C(1)—C(2) | 121.6 (2) |
| O(1)—C(2)—C(1) | 119.5 (2) | O(1)—C(2)—C(3) | 120.2 (2) |
| C(1)—C(2)—C(3) | 120.2 (2) | C(2)—C(3)—C(4) | 117.4 (2) |
| C(2)—C(3)—C(10) | 121.1 (3) | C(4)—C(3)—C(10) | 121.5 (3) |
| C(3)—C(4)—C(5) | 123.2 (2) | C(4)—C(5)—C(6) | 119.3 (2) |
| C(4)—C(5)—C(9) | 124.1 (2) | C(6)—C(5)—C(9) | 116.6 (2) |
| C(1)—C(6)—C(5) | 118.3 (2) | C(1)—C(6)—C(7) | 122.5 (2) |
| C(5)—C(6)—C(7) | 119.2 (2) | N(1)—C(7)—C(6) | 110.2 (2) |
| N(1)—C(7)—C(14) | 114.1 (2) | C(6)—C(7)—C(14) | 109.5 (2) |
| N(1)—C(8)—C(9) | 112.6 (2) | N(1)—C(8)—C(19) | 111.8 (2) |
| C(9)—C(8)—C(19) | 111.0 (2) | C(5)—C(9)—C(8) | 113.9 (2) |
| O(2)—C(12)—O(3) | 122.6 (2) | O(2)—C(12)—C(13) | 111.0 (2) |
| O(3)—C(12)—C(13) | 126.3 (2) | O(4)—C(14)—C(7) | 111.8 (2) |
| O(4)—C(15)—O(5) | 123.0 (3) | O(4)—C(15)—C(16) | 112.8 (3) |
| O(5)—C(15)—C(16) | 124.2 (3) | O(6)—C(17)—N(1) | 119.6 (2) |
| O(6)—C(17)—C(18) | 120.5 (3) | N(1)—C(17)—C(18) | 119.9 (2) |
| O(7)—C(19)—C(8) | 106.9 (3) | O(7)—C(20)—O(8) | 121.9 (5) |
| O(7)—C(20)—C(21) | 112.7 (4) | O(8)—C(20)—C(21) | 125.3 (4) |

| | | | |
|----------------------|-----------|-----------------------|------------|
| C(8)—N(1)—C(7)—C(6) | -45.6 (3) | C(17)—N(1)—C(7)—C(14) | -114.3 (2) |
| C(7)—N(1)—C(8)—C(9) | 8.7 (3) | C(17)—N(1)—C(8)—C(19) | 74.7 (3) |
| C(9)—C(5)—C(6)—C(7) | 2.8 (3) | C(6)—C(5)—C(9)—C(8) | -41.6 (4) |
| C(5)—C(6)—C(7)—N(1) | 39.9 (3) | N(1)—C(8)—C(9)—C(5) | 35.1 (3) |
| C(19)—C(8)—C(9)—C(5) | 161.3 (3) | | |

length 1.389 Å and r.m.s. deviation 0.004 Å. The N(1)—C(7) bond which is *trans* to the O(6) carbonyl group is significantly shorter than N(1)—C(8). Asymmetry of the exocyclic bond angles around the N(1) and C(7) atoms is observed. The adjacent angles C(7)—N(1)—C(17) and N(1)—C(7)—C(14) are significantly larger than C(8)—N(1)—C(17) and

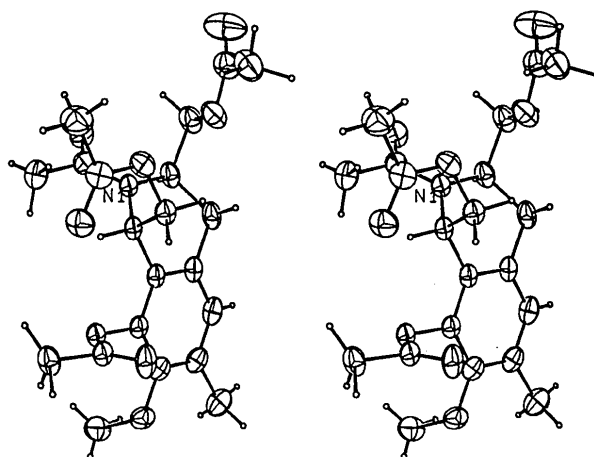


Fig. 1. Stereoview of the molecule with thermal ellipsoids at 30% probability.

C(6)—C(7)—C(14), respectively. The exocyclic bond angles around C(8), however, are almost symmetrical. In the packing there are no intermolecular contacts less than the sum of the van der Waals radii.

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Structure of *N*-Iodosuccinimide

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Abstract. C₄H₄INO₂, *M_r* = 224.99, tetragonal, *P4*₁ or *P4*₃, *a* = 6.339 (1), *c* = 15.455 (4) Å, *V* = 621.0 (2) Å³,

Z = 4, *D_x* = 2.406 g cm⁻³, *Mo Kα*, λ = 0.71073 Å, μ = 50.11 cm⁻¹, *F*(000) = 416, *T* = 295 K, *R* = 0.019 for 617 observed reflections [*I* > 2.58σ(*I*)] and 77 parameters for the structure refined in *P4*₁. The

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