

Table 3. *Relevant hydrogen bonds*

No.	D—H...A	D...A (Å)	H...A (Å)	DH...A (°)
1	N(2)—H...O(1 ^b)	2.961 (2)	2.23 (3)	139 (2)
2	N(1)—H...N(3 ^b)	2.754 (2)	2.27 (3)	118 (2)
3	N(3)—H...O(2 ^b)	2.593 (2)	2.09 (2)	113 (2)
4	C(17)—H...N(1 ^b)	3.370 (3)	2.85 (2)	117 (2)
5	C(22)—H...O(1 ^b)	3.491 (2)	2.77 (2)	137 (2)

Symmetry code: (i) *x*, *y*, *z*; (ii) \bar{x} , $1 - y$, $2 - z$.

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***rel*-(3*R*,3*aR*,8*S*,8*aR*)-3*a*-Hydroxy-1-oxo-6-daucen-8-yl *p*-Anisate (1) and
rel-(1*S*,3*R*,3*aS*,4*S*,8*S*,8*aR*)-1,8-Diacetoxy-3-hydroxy-6-daucen-4-yl *p*-Anisate (2)***

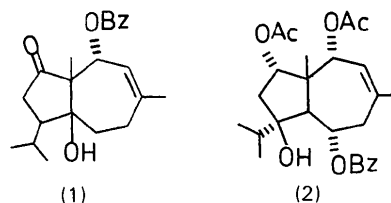
BY WILLIAM H. WATSON, RAM P. KASHYAP AND IRAJ TAVANAIEPOUR

FASTBIOS Laboratory, Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, USA

(Received 15 May 1985; accepted 16 July 1985)

Abstract. (1), C₂₃H₃₀O₅, *M_r* = 386.49, orthorhombic, *P*2₁2₁2₁, *a* = 11.723 (6), *b* = 17.261 (8), *c* = 10.515 (3) Å, *V* = 2128 (2) Å³, *Z* = 4, *D_x* = 1.206 g cm⁻³, Mo *Kα*, λ = 0.71069 Å, μ = 0.90 cm⁻¹, *F*(000) = 832, *T* = 295 K, *R* = 0.044 for 1448 independent reflections (*θ*:2*θ* scan). (2), C₂₇H₃₆O₈, *M_r* = 488.59, orthorhombic, *P*2₁2₁2₁, *a* = 11.733 (3), *b* = 26.654 (9), *c* = 8.644 (4) Å, *V* = 2703 (2) Å³, *Z* = 4, *D_x* = 1.200 g cm⁻³, Mo *Kα*, λ = 0.71069 Å, μ = 0.94 cm⁻¹, *F*(000) = 1048, *T* = 295 K, *R* = 0.052 for 1927 independent reflections (*θ*:2*θ* scan). The carotane-class sesquiterpenes were isolated from *Ferula communis* (Compositae). The five- and seven-membered rings are *cis* fused in compound (1) but *trans* fused in (2), which leads to conformational differences in the two compounds. The five-membered rings in both compounds exhibit envelope conformations with C(4) the flap in (1) and C(1) the flap in (2). The seven-membered ring in (2) is in an almost perfect chair conformation while that in (1) exhibits a distorted twist-chair conformation.

Introduction. *Ferula communis* L. subsp. *Communis* has been known as a medicinal since ancient times (Gunther, 1959) and has been used as an antihysterical and for the treatment of dysentery (French, 1971). *Ferula communis* was collected near Istanbul, Turkey, and the dried powdered roots were extracted with benzene. Chromatography led to the isolation of fourteen daucene-type sesquiterpene esters (Miski & Mabry, 1985). Because of the difficulty in determining relative stereochemistry and ester substitution patterns, the structures of two sesquiterpenes were determined by X-ray diffraction techniques. The absolute configuration can be assigned by comparison with known structures (Miski & Mabry, 1985).



* IUPAC names: (1) *rel*-(3*R*,3*aR*,8*S*,8*aR*)-3*a*-hydroxy-3-isopropyl-6,8*a*-dimethyl-1-oxo-1,2,3,3*a*,4,5,8,8*a*-octahydro-8-azulenyl *p*-anisate; (2) *rel*-(1*S*,3*R*,3*aS*,4*S*,8*S*,8*aR*)-1,8-diacetoxy-3-hydroxy-3-isopropyl-6,8*a*-dimethyl-1,2,3,3*a*,4,5,8,8*a*-octahydro-4-azulenyl *p*-anisate.

Experimental. Syntex *P*2₁ diffractometer, *θ*:2*θ* scan, variable scan rate, 2*θ*_{max} = 50°, graphite-monochromated Mo *Kα* radiation; lattice parameters from

least-squares refinement of 15 reflections [$4.18 \leq 2\theta \leq 13.56^\circ$ for (1) and $6.0 \leq 2\theta \leq 23.0^\circ$ for (2)] with angles measured by a centering routine associated with the diffractometer system; after data collection all accessible equivalent reflections collected for selected reflections differing in 2θ ; Lorentz and polarization corrections, empirical absorption correction [transmission factors 0.9704 to 0.9725 for (1) and 0.9612 to 0.9750 for (2)]; direct methods (*MULTAN78*: Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); full-matrix least-squares refinement, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma^2(F_o)$ [derived from counting statistics evaluated by Syntex (1976) routine].

(1) Transparent prismatic crystal of dimensions $0.35 \times 0.38 \times 0.68$ mm with crystal oriented along *c* axis (long dimension), systematic absences ($h00$ $h = 2n + 1$, $0k0$ $k = 2n + 1$, $00l$ $l = 2n + 1$) consistent with space group $P2_12_12_1$ (222), monitored reflection (102) showed no change in intensity greater than $2\sigma(I)$; 2059 independent reflections ($0 \leq h \leq 12$, $0 \leq k \leq 18$, $0 \leq l \leq 11$), equivalent reflections averaged ($R_{\text{int}} = 0.031$), 1448 had intensities greater than $3\sigma(I)$; all except three H atoms located in difference Fourier map and refined with fixed thermal parameters, positions for missing H atoms calculated, after final refinement H-atom positions scaled to C–H distance of 1.08 \AA ; $R = 0.044$, $wR = 0.049$, 334 parameters refined,

$(\Delta/\sigma)_{\text{max}} = 0.4$, $S = 2.16$, $|0.14| e \text{ \AA}^{-3}$ largest peak in final difference Fourier map.

(2) Transparent prismatic crystal of dimensions $0.39 \times 0.61 \times 0.75$ mm with crystal oriented along *c* axis (long dimension), systematic absences consistent with space group $P2_12_12_1$ (222), monitored reflection (032) showed no change in intensity greater than $2\sigma(I)$; 2595 independent reflections ($0 \leq h \leq 12$, $0 \leq k \leq 27$, $0 \leq l \leq 9$), 1927 had intensities greater than $3\sigma(I)$; all H atoms located in difference Fourier maps, H-atom positions constrained during final cycles of refinement, after final refinement C–H distances scaled to 1.08 \AA ; $R = 0.052$, $wR = 0.063$, 316 parameters refined; $(\Delta/\sigma)_{\text{av}} = 0.031$, $(\Delta/\sigma)_{\text{max}} = 0.44$, $S = 2.88$, $|0.22| e \text{ \AA}^{-3}$ largest peak in final difference Fourier map.

Locally written programs used for data reduction, *MULTAN78* for direct-methods calculations, *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) for all others; atomic scattering factors for C and O from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965), anomalous dispersion from *International Tables for X-ray Crystallography* (1974). Tables 1 and 2 give atomic positional parameters and U_{eq} values for (1) and (2) while Table 3

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters U_{eq} ($\text{\AA}^2 \times 10^3$) for (1)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^\dagger
C(1)	8008 (3)	3370 (2)	9191 (4)	41 (2)
C(2)	7158 (4)	3108 (3)	10212 (4)	49 (3)
O(2)	6516 (3)	3548 (2)	10773 (3)	61 (2)
C(3)	7219 (4)	2251 (2)	10392 (4)	51 (3)
C(4)	7618 (3)	1960 (2)	9082 (4)	44 (2)
C(5)	8511 (3)	2578 (2)	8692 (4)	42 (2)
O(5)	9532 (2)	2461 (2)	9422 (3)	55 (2)
C(6)	8776 (4)	2564 (2)	7280 (4)	50 (3)
C(7)	9543 (4)	3218 (3)	6779 (5)	60 (3)
C(8)	8997 (4)	3998 (2)	6613 (4)	53 (3)
C(9)	8043 (4)	4241 (2)	7164 (5)	54 (3)
C(10)	7356 (4)	3857 (2)	8215 (4)	46 (2)
O(10)	6426 (2)	3369 (1)	7697 (3)	48 (2)
C(11)	8010 (4)	1107 (2)	9025 (4)	54 (3)
C(12)	7688 (10)	745 (3)	7808 (6)	163 (7)
C(13)	7494 (6)	604 (3)	10063 (6)	100 (4)
C(14)	9609 (4)	4522 (3)	5690 (5)	73 (3)
C(15)	8883 (4)	3909 (2)	9832 (4)	55 (3)
C(16)	5516 (4)	3758 (3)	7229 (4)	57 (3)
O(16)	5488 (3)	4458 (2)	7154 (4)	83 (2)
C(17)	4582 (4)	3237 (3)	6813 (4)	50 (3)
C(18)	4543 (4)	2452 (2)	7055 (4)	52 (3)
C(19)	3623 (4)	2001 (3)	6698 (4)	56 (3)
C(20)	2703 (4)	2343 (3)	6091 (4)	59 (3)
O(20)	1743 (3)	1965 (2)	5716 (3)	72 (2)
C(21)	2735 (4)	3129 (3)	5857 (5)	66 (3)
C(22)	3653 (4)	3570 (3)	6197 (5)	62 (3)
C(23)	1628 (4)	1166 (3)	6000 (5)	79 (4)

$$\dagger U_{\text{eq}} = \frac{1}{3} [U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aba^*b^*\cos\gamma + 2U_{13}aca^*c^*\cos\beta + 2U_{23}bcb^*c^*\cos\alpha].$$

Table 2. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters U_{eq} ($\text{\AA}^2 \times 10^3$) for (2)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1)	1718 (4)	1182 (1)	3155 (5)	51 (2)
C(2)	2833 (4)	976 (2)	2476 (5)	60 (3)
O(2)	2669 (3)	885 (1)	834 (4)	64 (2)
C(3)	3693 (4)	1404 (2)	2674 (5)	59 (3)
C(4)	3003 (4)	1900 (2)	2745 (5)	35 (2)
O(4)	3091 (3)	2129 (1)	4262 (3)	58 (2)
C(5)	1738 (3)	1732 (1)	2571 (4)	43 (2)
C(6)	838 (3)	2071 (1)	3268 (5)	45 (2)
O(6)	987 (2)	2551 (1)	2446 (3)	49 (2)
C(7)	−400 (4)	1914 (2)	2958 (5)	56 (3)
C(8)	−836 (4)	1454 (2)	3815 (6)	65 (3)
C(9)	−377 (4)	1007 (2)	3670 (6)	64 (3)
C(10)	642 (4)	889 (1)	2687 (5)	57 (3)
O(10)	370 (3)	1007 (1)	1071 (4)	63 (2)
C(11)	3402 (4)	2291 (2)	1528 (6)	59 (3)
C(12)	3234 (5)	2110 (2)	−112 (6)	85 (4)
C(13)	4652 (4)	2445 (2)	1808 (7)	81 (4)
C(14)	−1849 (5)	1527 (2)	4799 (7)	97 (4)
C(15)	1848 (4)	1137 (2)	4937 (5)	68 (3)
C(16)	717 (3)	2983 (1)	3176 (5)	51 (2)
O(16)	328 (3)	2993 (1)	4470 (4)	69 (2)
C(17)	969 (3)	3425 (1)	2237 (6)	50 (3)
C(18)	733 (4)	3904 (2)	2848 (6)	71 (3)
C(19)	951 (5)	4329 (2)	2023 (10)	89 (4)
C(20)	1399 (5)	4290 (2)	567 (9)	87 (4)
O(20)	1613 (3)	4737 (1)	−199 (6)	124 (3)
C(21)	1648 (4)	3827 (2)	−118 (6)	72 (3)
C(22)	1430 (4)	3396 (1)	755 (6)	58 (3)
C(23)	1925 (6)	4730 (3)	−1738 (11)	158 (7)
C(24)	3175 (5)	511 (2)	156 (6)	69 (4)
O(24)	3792 (5)	227 (2)	873 (6)	127 (3)
C(25)	2958 (5)	501 (2)	−1528 (7)	85 (4)
C(26)	109 (5)	633 (3)	133 (7)	79 (4)
O(26)	121 (6)	207 (2)	494 (5)	155 (4)
C(27)	−220 (6)	823 (3)	−1425 (7)	85 (5)

lists interatomic distances, valence angles and torsion angles.*

Discussion. Figs. 1 and 2 are ORTEP (Johnson, 1971) drawings of (1) and (2). Both compounds belong to the carotane class of sesquiterpenes and are related biogenetically to daucene, which has a C(4)—C(5) double bond. Since addition to this bond may occur from either side, *cis* and *trans* ring fusions are possible.

* Lists of structure factors, H-atom parameters and thermal parameters for (1) and (2) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42384 (49 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Interatomic distances (Å), valence angles (°) and torsion angles (°) for (1) and (2)

	(1)	(2)	(1)	(2)
C(1)—C(2)	1.534 (6)	1.535 (6)	C(2)C(1)C(5)	102.7 (3) 101.5 (3)
C(1)—C(5)	1.578 (5)	1.552 (6)	C(2)C(1)C(10)	107.9 (3) 114.7 (3)
C(1)—C(10)	1.530 (6)	1.538 (6)	C(2)C(1)C(15)	107.7 (3) 105.6 (7)
C(1)—C(15)	1.540 (6)	1.552 (6)	C(5)C(1)C(10)	116.0 (3) 113.9 (3)
C(2)—C(3)	1.493 (6)	1.533 (7)	C(5)C(1)C(15)	114.8 (3) 113.2 (3)
C(2)—O(2)	1.221 (5)	1.453 (5)	C(10)C(1)C(15)	107.1 (3) 107.6 (3)
C(3)—C(4)	1.539 (6)	1.551 (6)	C(1)C(2)C(3)	110.5 (3) 104.6 (3)
C(4)—C(5)	1.550 (6)	1.557 (6)	C(1)C(2)O(2)	123.7 (4) 108.7 (3)
C(4)—C(11)	1.544 (6)	1.552 (6)	C(3)C(2)O(2)	125.8 (4) 108.7 (3)
C(4)—O(4)	—	1.449 (5)	C(2)C(3)C(4)	102.9 (3) 107.2 (3)
C(5)—C(6)	1.517 (6)	1.515 (6)	C(3)C(4)C(5)	102.6 (3) 104.4 (3)
C(5)—O(5)	1.437 (5)	—	C(3)C(4)C(11)	115.9 (3) 112.7 (3)
C(6)—C(7)	1.537 (6)	1.537 (6)	C(3)C(4)O(4)	—
C(6)—O(6)	—	1.473 (5)	C(5)C(4)C(11)	116.4 (3) 114.5 (3)
C(7)—C(8)	1.500 (7)	1.521 (7)	C(5)C(4)O(4)	—
C(8)—C(9)	1.327 (7)	1.313 (7)	O(4)C(4)C(11)	—
C(8)—C(14)	1.505 (7)	1.475 (8)	C(1)C(5)C(4)	104.8 (3) 104.7 (3)
C(9)—C(10)	1.520 (6)	1.500 (7)	C(4)C(5)C(6)	112.7 (3) 117.0 (3)
C(10)—O(10)	1.482 (6)	1.467 (6)	C(4)C(5)O(5)	108.9 (3) —
C(11)—C(12)	1.473 (8)	1.510 (6)	C(1)C(5)C(6)	114.6 (3) 115.1 (3)
C(11)—C(13)	1.520 (8)	1.545 (7)	C(1)C(5)O(5)	104.8 (3) —
O(6)/O(10)—C(16)	1.354 (5)	1.350 (5)	C(6)C(5)O(5)	110.5 (3) —
C(16)—O(16)	1.211 (6)	1.209 (5)	C(5)C(6)C(7)	116.3 (4) 115.2 (3)
C(16)—C(17)	1.483 (6)	1.460 (6)	C(5)C(6)O(6)	—
C(17)—C(18)	1.380 (6)	1.410 (7)	C(7)C(6)O(6)	—
C(17)—C(22)	1.392 (6)	1.392 (7)	C(6)C(7)C(8)	116.8 (4) 117.0 (4)
C(18)—C(19)	1.382 (6)	1.363 (8)	C(7)C(8)C(9)	126.3 (4) 123.1 (4)
C(19)—C(20)	1.385 (7)	1.368 (11)	C(7)C(8)C(14)	114.2 (4) 116.5 (4)
C(20)—C(21)	1.380 (7)	1.399 (8)	C(9)C(8)C(14)	119.5 (4) 120.3 (5)
C(20)—O(20)	1.358 (6)	1.386 (8)	C(8)C(9)C(10)	120.8 (4) 124.8 (4)
C(21)—C(22)	1.366 (7)	1.399 (7)	C(1)C(10)C(9)	117.6 (3) 113.5 (4)
O(20)—C(23)	1.419 (7)	1.380 (11)	C(1)C(10)O(10)	107.6 (3) 108.7 (3)
O(2)—C(24)	—	1.300 (6)	C(9)C(10)O(10)	111.7 (3) 108.7 (4)
C(24)—O(24)	—	1.216 (7)	C(4)C(11)C(12)	111.2 (4) 112.5 (4)
C(24)—C(25)	—	1.478 (8)	C(4)C(11)C(13)	113.4 (4) 111.2 (4)
O(10)—C(26)	—	1.320 (7)	C(12)C(11)C(13)	106.3 (5) 111.0 (4)
C(26)—O(26)	—	1.178 (9)	C—O—C(16)	115.5 (4) 119.2 (3)
C(26)—C(27)	—	1.489 (9)	O—C(16)—O(16)	122.7 (4) 122.6 (4)
	(1)	(2)	O—C(16)—C(17)	112.8 (4) 112.4 (4)
C(1)C(2)C(3)C(4)	-27.9 (4)	-24.9 (4)	O(16)C(16)C(17)	124.5 (4) 124.9 (4)
C(2)C(3)C(4)C(5)	39.7 (4)	-0.3 (4)	C(16)C(17)C(18)	124.5 (4) 118.9 (4)
C(3)C(4)C(5)C(1)	-37.4 (4)	25.1 (4)	C(16)C(17)C(22)	117.6 (4) 123.0 (4)
C(4)C(5)C(1)C(2)	20.4 (4)	-40.2 (4)	C(18)C(17)C(22)	117.8 (4) 118.1 (4)
C(5)C(1)C(2)C(3)	4.6 (4)	39.8 (4)	C(17)C(18)C(19)	121.9 (4) 121.3 (5)
C(5)C(6)C(7)C(8)	76.4 (5)	72.1 (5)	C(18)C(19)C(20)	119.6 (4) 119.3 (5)
C(6)C(7)C(8)C(9)	19.9 (7)	-60.1 (6)	C(19)C(20)C(21)	118.7 (4) 122.5 (6)
C(7)C(8)C(9)C(10)	9.4 (8)	1.4 (8)	C(19)C(20)O(20)	125.1 (4) 116.3 (5)
C(8)C(9)C(10)C(1)	31.2 (7)	60.1 (6)	C(21)C(20)O(20)	116.2 (4) 121.2 (6)
C(9)C(10)C(1)C(5)	-70.5 (9)	-78.7 (5)	C(20)C(21)C(22)	121.5 (4) 117.3 (5)
C(10)C(1)C(5)C(6)	27.0 (5)	66.0 (4)	C(17)C(22)C(21)	120.5 (4) 121.5 (4)
C(1)C(5)C(6)C(7)	53.2 (5)	-61.0 (5)	C(20)O(20)C(23)	118.9 (4) 119.8 (5)
			C(2)O(2)C(24)	—
			O(2)C(24)C(25)	—
			O(2)C(24)O(24)	—
			O(24)C(24)C(25)	—
			C(10)O(10)C(26)	—
			O(10)C(26)O(26)	—
			O(10)C(26)C(27)	—
			O(26)C(26)C(27)	—

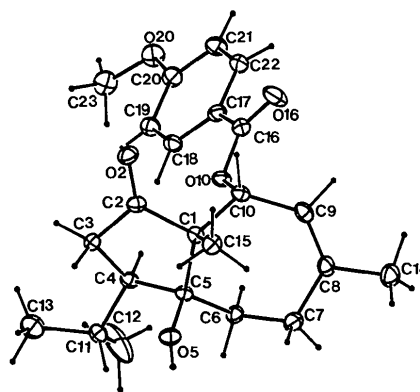


Fig. 1. ORTEP drawing of (1). Thermal ellipsoids are drawn at the 35% probability level. H atoms are drawn as spheres of arbitrary size.

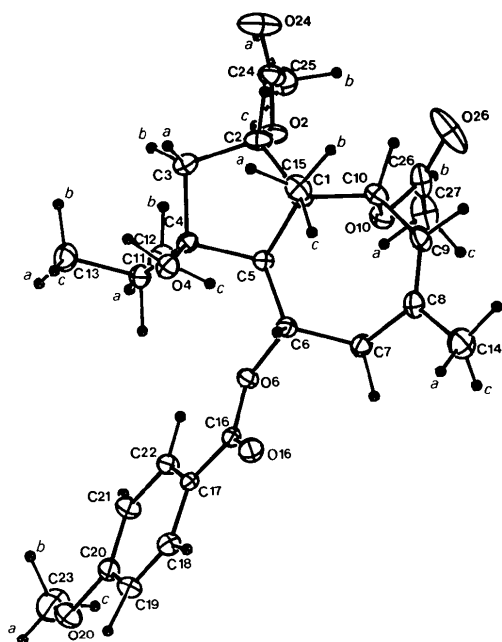


Fig. 2. ORTEP drawing of (2). Thermal ellipsoids are drawn at the 35% probability level. H atoms drawn as spheres of arbitrary size. Methyl-group hydrogen atoms are labeled a, b and c.

The conformations of (1) and (2) differ significantly owing to the differences in ring fusion. The five-membered ring in both compounds is in an envelope conformation; however, C(4) is the flap in (1) while C(1) is the flap in (2). The distances and angles around the ring cannot be compared owing to differences in functionality. The seven-membered heptene ring in (2) has an almost perfect chair conformation while that in (1) is a highly distorted twist chair (Toromanoff, 1980; Bucourt, 1974), see torsion angles in Table 3. The C(1)–C(5) bond common to both rings differs significantly, 1.578 (5) Å for (1) and 1.552 (6) Å for (2), as do a number of valence angles. The ester groups are not held tightly by packing interactions and the thermal parameters are relatively large as are the thermal parameters for the C(12) and C(13) methyl groups in (2).

We thank The Robert A. Welch Foundation (P-074) and the TCU Research Foundation for financial support.

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Structural Studies of Pyrimidine Cyclonucleoside Derivatives. IX.* Structure of 6,2'-Anhydro-6-ethyl-1-β-D-arabinofuranosyluracil

BY YURIKO YAMAGATA, NOBUO OKABE AND KEN-ICHI TOMITA

Faculty of Pharmaceutical Sciences, Osaka University, Yamadaoka, Suita, Osaka 565, Japan

AND SATOSHI SHUTO, HIDEO INOUE AND TOHRU UEDA

Faculty of Pharmaceutical Sciences, Hokkaido University, Kita-12, Nishi-6, Kitaku, Sapporo 060, Japan

(Received 13 November 1984; accepted 10 July 1985)

Abstract. C₁₁H₁₄N₂O₅, $M_r = 254.2$, orthorhombic, $P2_12_12_1$, $a = 6.400$ (1), $b = 9.734$ (2), $c = 17.797$ (5) Å, $V = 1108.7$ (4) Å³, $Z = 4$, $D_m = 1.506$ (1), $D_x = 1.523$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.69$ mm⁻¹, $F(000) = 536$, $T = 290$ K, $R = 0.057$ for 1030 independent observed reflections. The glycosidic torsion angle is in the *anti* range, $\chi_{\text{CN}}[\text{O}(4')\text{—C}(1')\text{—N}(1)\text{—C}(2)] = 268.1$ (3)°, and the sugar conformation is the unusual ^oT₄. The O atom O(5') takes two disordered orientations, *gauche*–*trans* and *trans*–*gauche*, but not *gauche*–*gauche* (*gauche* ≡ *synclinal*) as frequently found in nucleosides. No base stacking is observed.

Introduction. Cyclonucleosides have generally served as good model compounds for investigations of physicochemical properties of nucleosides and nucleotides in solution because they have conformational rigidity between the base and the sugar moieties by cyclization (Rogers & Ulbricht, 1970; Ikehara, Kaneko, Nakahara, Yamada & Uesugi, 1971; Ueda & Shibuya, 1974; Manor, Saenger, Davies, Jankowski & Rabcsenko, 1974; Yamagata, Fujii, Fujiwara, Tomita & Ueda, 1981). In particular, cyclonucleosides cyclized through a C atom are much better model nucleosides for spectroscopic studies because the bridging C atom has less effect on the physicochemical properties of the base or the sugar moiety than O or S bridges, and we recently synthesized several new types of C-bridged

* Part VIII: Yamagata, Fujiwara, Tomita, Shuto & Ueda (1984).