

Table 4. Selected torsion angles ($^{\circ}$), with e.s.d.'s in parentheses

	(I)	(II)
C(5A)–C(5)–C(12)–C(13)	65.5 (9)	74.5 (6)
C(14)–C(15)–N(15)–S(1)	135.4 (7)	183.6 (4)
C(15)–N(15)–S(1)–C(18)	–69.5 (7)	–72.2 (4)

and 3,6-diaminoacridine (Jones & Neidle, 1975). The acridine bonding geometry in (I) is as expected, subtly different from that of a normal acridine, with differences in bond lengths being most apparent in the central ring. These differences, though not large, do indicate that the carbonyl group at C(9) has reduced the delocalization in the ring. The ring nitrogen atom N(10) is sp^2 -hybridized as in normal acridines. Its low pK_a excludes it from being protonated, which would not in any case be feasible in structural terms as ring buckling would necessarily be produced. Thus, the single H atom attached to N(10) in (I) does not carry a positive charge, in contrast to that in (II), which exists as a salt.

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Structure of Neoandrographolide Monohydrate

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Abstract. [(1R)-(1 α ,4 α ,5 α ,8 α)]-3-(2-{5-[(β -D-Glucopyranosyloxy)methyl]decahydro-5,8a-dimethyl-2-methylene-1-naphthalenyl}ethyl)-2(5H)-furanone monohydrate, C₂₆H₄₀O₈·H₂O, M_r = 498.62, monoclinic, $P2_1$, a = 7.377 (3), b = 6.235 (9), c = 28.569 (8) Å, β = 95.73 (3)°, V = 1307 (2) Å³, Z = 2, D_x = 1.266 g cm⁻³, λ (Mo K α) = 0.71073 Å, μ = 0.9 cm⁻¹, $F(000)$ = 540, T = 294 K, R = 0.0537 for 1697 observed reflections with $I > 2\sigma(I)$. The crystal structure consists of two-dimensional hydrogen-bonded layers, involving the β -D-glucopyranose moiety and the water

molecule of crystallization, separated by layers consisting of a stacking of the neoandrographolide–glucan moieties with no short intermolecular contacts.

Introduction. Within the framework of our immunopharmacognostic studies we isolated a sugar-bound diterpenoid from herb material of *Andrographis paniculata*. Since this constituent showed significant inhibitory action on both pathways of human complement *in vitro* it was decided to study its molecular structure. The compound was identified as neoandrographolide. Among other diterpenoids from *Andrographis paniculata* that are being used against bacillary dysentery in

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China, neoandrographolide is considered to be the most active one. In addition the neoandrographolide we isolated showed promising activity in the haemagglutination test, showing its ability to interact with the formation of immune complexes, *i.e.* antibody-antigen reactions. These properties of neoandrographolide prompted us to study the conformational details of its molecular structure with an X-ray analysis. Earlier work on the structure and stereochemistry of neoandrographolide has been described (Chan, Taylor, Willis, Bodden & Fehlhaber, 1971); this concludes that neoandrographolide is the β -D-glucoside of *ent*-19-hydroxy-8(17),13-labdadien-16,15-olide. In a previous paper we reported the hydrogen-bonding scheme in the crystal structure of the related andrographolide (Spek, Duisenberg, Labadie, Ratnayake, Abeysekera & De Silva, 1987).

Experimental. The title compound was isolated from a methanolic extract of *Andrographis paniculata* collected in Sri Lanka. Subsequent fractionation using animal cole and a silica gel column led to an enriched neoandrographolide fraction. Crystals suitable for X-ray diffraction were obtained by recrystallization from methanol (m.p. 505 K). Transparent colourless plate-shaped crystal 0.90 × 0.25 × 0.10 mm glued on top of a glass fibre. Enraf-Nonius CAD-4F diffractometer, Zr-filtered Mo K α radiation, cell constants derived from the angular settings of 24 reflections with $9.6 < \theta < 13.2^\circ$. Intensity data of 2012 unique reflections were collected within a quarter of the reflection sphere: $-8 \leq h \leq 8$; $0 \leq k \leq 6$; $0 \leq l \leq 31$; $0.72 < \theta < 23.0^\circ$, $\omega/2\theta$ scan mode with $\Delta\omega = (0.70 + 0.35 \tan\theta)^\circ$. A reference reflection [111 (e.s.d. = 0.78%)], measured every hour showed a linear decay of 2% during the 43 h of X-ray exposure time. Intensities were corrected for this small decay and for Lp. Standard deviations in the intensities were increased according to an analysis of the excess variance of the reference reflection: $\sigma^2(I) = \sigma_{cs}^2(I) + (0.005I)^2$ (McCandlish, Stout & Andrews, 1975) resulting in 1967 reflections with $I > 2\sigma(I)$. Space group *P*2₁, derived from the systematic extinctions: $0k0$; $k = 2n+1$.

The structure was solved by direct methods (*SHELX84*; Sheldrick, 1984); the solution with the best figure of merit showed all non-H atoms and a water molecule of crystallization. The non-H atoms were refined anisotropically (on *F*) by blocked full-matrix least-squares techniques. All hydrogen atoms except water, hydroxylic and methylenic [C(17)] H atoms were introduced on calculated positions (C-H = 0.98 Å). Two of the hydroxylic and both C(17) H atoms were located from difference maps; the remaining two hydroxylic and the water H atoms could not be identified unambiguously. H atoms were included in the final refinement cycles with one common isotropic

Table 1. *Positional and equivalent isotropic thermal parameters for the non-hydrogen atoms with e.s.d.'s in parentheses*

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Neoandrographolide-aglucon moiety				
O(1)	0.3192 (6)	0.177 (1)	0.4613 (2)	0.075 (2)
O(2)	0.0803 (8)	0.355 (1)	0.4290 (2)	0.094 (2)
C(1)	-0.2633 (7)	0.092 (1)	0.2818 (2)	0.041 (2)
C(2)	-0.3029 (7)	0.139 (1)	0.2292 (2)	0.044 (2)
C(3)	-0.5062 (7)	0.172 (1)	0.2161 (2)	0.040 (2)
C(4)	-0.6214 (6)	-0.0185 (9)	0.2285 (2)	0.035 (2)
C(5)	-0.5731 (6)	-0.0778 (9)	0.2812 (2)	0.033 (2)
C(6)	-0.6843 (7)	-0.261 (1)	0.2985 (2)	0.046 (2)
C(7)	-0.6571 (7)	-0.283 (1)	0.3521 (2)	0.051 (2)
C(8)	-0.4581 (7)	-0.300 (1)	0.3686 (2)	0.042 (2)
C(9)	-0.3475 (6)	-0.113 (1)	0.3530 (2)	0.033 (2)
C(10)	-0.3640 (6)	-0.1047 (9)	0.2979 (2)	0.030 (1)
C(11)	-0.1514 (7)	-0.105 (1)	0.3765 (2)	0.041 (2)
C(12)	-0.1380 (7)	-0.053 (1)	0.4283 (2)	0.046 (2)
C(13)	-0.0568 (7)	-0.016 (1)	0.4477 (2)	0.039 (2)
C(14)	0.1786 (8)	-0.148 (1)	0.4666 (2)	0.056 (2)
C(15)	0.3547 (8)	-0.038 (1)	0.4761 (2)	0.068 (3)
C(16)	0.1433 (8)	0.190 (1)	0.4434 (2)	0.055 (3)
C(17)	-0.3930 (9)	-0.471 (1)	0.3908 (2)	0.053 (2)
C(18)	-0.2771 (7)	-0.308 (1)	0.2801 (2)	0.040 (2)
C(19)	-0.5982 (7)	-0.209 (1)	0.1965 (2)	0.039 (2)
C(20)	-0.8230 (7)	0.053 (1)	0.2206 (2)	0.052 (2)
D-Glucopyranose moiety				
O(1')	-0.6474 (5)	-0.1487 (7)	0.1484 (1)	0.039 (1)
O(2')	-0.9419 (5)	-0.1133 (8)	0.0804 (1)	0.049 (1)
O(3')	-0.9209 (4)	-0.3567 (7)	-0.0040 (1)	0.034 (1)
O(4')	-0.7015 (5)	-0.7389 (7)	0.0057 (1)	0.049 (1)
O(5')	-0.5457 (4)	-0.4427 (7)	0.1126 (1)	0.038 (1)
O(6')	-0.2755 (5)	-0.6449 (9)	0.0626 (1)	0.057 (2)
C(1')	-0.7019 (6)	-0.316 (1)	0.1195 (2)	0.032 (2)
C(2')	-0.7872 (7)	-0.234 (1)	0.0723 (2)	0.034 (2)
C(3')	-0.8323 (6)	-0.4238 (9)	0.0400 (2)	0.030 (2)
C(4')	-0.6625 (6)	-0.5552 (9)	0.0351 (2)	0.032 (2)
C(5')	-0.5843 (6)	-0.629 (1)	0.0839 (2)	0.036 (2)
C(6')	-0.4105 (7)	-0.758 (1)	0.0836 (2)	0.046 (2)
Water molecule				
O(10)	0.8246 (7)	0.663 (1)	0.1332 (2)	0.108 (3)

thermal parameter [$U = 0.045$ (4) Å²]. Convergence was reached at $R = 0.0537$, $wR = 0.0705$, $w = [\sigma^2(F) + 0.0009F^2]^{-1}$, $S = 1.86$, $(\Delta/\sigma)_{av} = 0.031$. Residual densities in the final Fourier map are in the range 0.45 to -0.30 e Å⁻³.

Scattering factors from Cromer & Mann (1968). Calculations performed with *SHELX76* (Sheldrick, 1976) and the *EUCLID* package (geometry calculations and illustrations) (Spek, 1982) on the CDC CYBER 855 of the University of Utrecht.

Discussion. Fractional atomic coordinates and equivalent isotropic thermal parameters of the non-hydrogen atoms are listed in Table 1.* Bond distances and angles are given in Table 2. The molecular

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and geometrical data concerning H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44115 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

structure and the adopted atom numbering are presented in Fig. 1. For the structure determination of this natural product, the enantiomer with the *D*-glucopyranose moiety was chosen. The title compound consists of a slightly modified andrographolide molecule with its C(19) atom bonded to the O(1') atom of a β -*D*-glucopyranose group. The present grapholide moiety resembles that in the structure of andrographolide (Smith, Toder, Carroll & Donohue, 1982; Spek, Duisenberg, Labadie, Ratnayake, Abeysekera &

De Silva, 1987), the most important difference being the absence (in the present structure) of the hydroxyl groups at C(3) and C(14) and the planarity (within 0.02 Å) of the five-membered lactone ring in view of the C(13)–C(14) double bond. The orientation of this five-membered ring is also different as is illustrated by the torsion angles C(10)–C(9)–C(11)–C(12), C(9)–C(11)–C(12)–C(13) and C(11)–C(12)–C(13)–C(16), which are 164.3 (5), –171.8 (5), 83.3 (7)° in the present structure and 170.6 (2), 158.2 (2), 175.9 (2)° in andrographolide (Spek *et al.*, 1987).

The two central six-membered rings of the grapholide skeleton adopt the chair conformation, as can be seen from the puckering parameters [corresponding values in andrographolide (Spek *et al.*, 1987) in square brackets]: $Q=0.548(6)$ [0.553(2) Å, $\theta=175.4(6)$ [175.7(3)]°, $\varphi=234(8)$ [230(3)]° for ring [C(1), C(2), ...C(10)] and $Q=0.599(6)$ [0.597(2) Å, $\theta=174.3(6)$ [175.8(2)]°, $\varphi=101(6)$ [121(3)]° for ring [C(5), C(6), ...C(10)] (Cremer & Pople, 1975).

The geometry of the glucopyranose moiety strongly resembles that of methyl β -*D*-glucopyranoside (Jeffrey & Takagi, 1977), the largest differences between corresponding bond distances and angles being 0.014 Å and 3.3° respectively. The six-membered O(5')...C(5') glucoside ring has a chair conformation as is illustrated by the puckering parameters $Q=0.593(6)$ Å, $\theta=1.7(6)$ °, $\varphi=240(18)$ °; the endocyclic torsion angles are in the range: 56.5 (5)–62.0 (5)°.

Table 2. Bond distances (Å) and bond angles (°) for the non-hydrogen atoms with e.s.d.'s in parentheses

O(1)–C(15)	1.422 (9)	C(5)–C(10)	1.577 (7)
O(1)–C(16)	1.349 (8)	C(6)–C(7)	1.531 (8)
O(2)–C(16)	1.186 (9)	C(7)–C(8)	1.500 (8)
O(1')–C(19)	1.436 (7)	C(8)–C(9)	1.515 (8)
O(1')–C(1')	1.366 (7)	C(8)–C(17)	1.307 (9)
O(2')–C(2')	1.405 (7)	C(9)–C(11)	1.534 (7)
O(3')–C(3')	1.421 (7)	C(9)–C(10)	1.568 (8)
O(4')–C(4')	1.432 (7)	C(10)–C(18)	1.530 (8)
O(5')–C(1')	1.427 (6)	C(11)–C(12)	1.509 (8)
O(5')–C(5')	1.434 (7)	C(12)–C(13)	1.505 (8)
O(6')–C(6')	1.403 (7)	C(13)–C(14)	1.296 (9)
C(1)–C(2)	1.530 (8)	C(13)–C(16)	1.445 (9)
C(1)–C(10)	1.528 (8)	C(14)–C(15)	1.470 (9)
C(2)–C(3)	1.523 (8)	C(1')–C(2')	1.519 (8)
C(3)–C(4)	1.523 (8)	C(2')–C(3')	1.517 (9)
C(4)–C(5)	1.557 (8)	C(3')–C(4')	1.515 (7)
C(4)–C(19)	1.519 (9)	C(4')–C(5')	1.526 (8)
C(4)–C(20)	1.547 (7)	C(5')–C(6')	1.514 (8)
C(5)–C(6)	1.518 (8)		
C(15)–O(1)–C(16)	108.1 (5)	C(9)–C(10)–C(18)	108.3 (4)
C(19)–O(1')–C(1')	114.2 (5)	C(9)–C(11)–C(12)	113.8 (4)
C(1')–O(5')–C(5')	114.2 (4)	C(11)–C(12)–C(13)	111.1 (4)
C(2)–C(1)–C(10)	113.5 (5)	C(14)–C(13)–C(16)	107.8 (5)
C(1)–C(2)–C(3)	110.7 (4)	C(12)–C(13)–C(14)	130.7 (6)
C(2)–C(3)–C(4)	113.3 (5)	C(12)–C(13)–C(16)	121.2 (5)
C(3)–C(4)–C(5)	109.3 (4)	C(13)–C(14)–C(15)	110.1 (5)
C(5)–C(4)–C(19)	111.6 (5)	O(1)–C(15)–C(14)	104.6 (5)
C(5)–C(4)–C(20)	109.5 (4)	O(1)–C(16)–C(13)	109.3 (5)
C(3)–C(4)–C(19)	111.8 (4)	O(1)–C(16)–O(2)	120.8 (6)
C(3)–C(4)–C(20)	107.0 (5)	O(2)–C(16)–C(13)	129.8 (6)
C(19)–C(4)–C(20)	107.6 (4)	O(1')–C(19)–C(4)	109.8 (5)
C(6)–C(5)–C(10)	111.4 (4)	O(1')–C(1')–O(5')	108.3 (4)
C(4)–C(5)–C(6)	114.5 (4)	O(1')–C(1')–C(2')	110.5 (5)
C(4)–C(5)–C(10)	116.1 (4)	O(5')–C(1')–C(2')	109.4 (4)
C(5)–C(6)–C(7)	111.8 (5)	O(2')–C(2')–C(1')	107.8 (4)
C(6)–C(7)–C(8)	110.3 (4)	C(1')–C(2')–C(3')	108.9 (5)
C(7)–C(8)–C(9)	113.1 (5)	O(2')–C(2')–C(3')	112.7 (4)
C(9)–C(8)–C(17)	126.0 (5)	O(3')–C(3')–C(2')	111.2 (5)
C(7)–C(8)–C(17)	120.7 (6)	C(2')–C(3')–C(4')	110.2 (4)
C(8)–C(9)–C(11)	114.1 (5)	C(3')–C(3')–C(4')	112.9 (4)
C(10)–C(9)–C(11)	114.4 (4)	C(3')–C(4')–C(5')	108.6 (4)
C(8)–C(9)–C(10)	109.3 (5)	O(4')–C(4')–C(3')	111.5 (4)
C(1)–C(10)–C(5)	108.3 (4)	O(4')–C(4')–C(5')	109.3 (4)
C(1)–C(10)–C(9)	109.7 (4)	O(5')–C(5')–C(4')	108.3 (5)
C(1)–C(10)–C(18)	109.4 (4)	O(5')–C(5')–C(6')	108.3 (4)
C(5)–C(10)–C(9)	106.4 (4)	C(4')–C(5')–C(6')	113.5 (4)
C(5)–C(10)–C(18)	114.6 (4)	O(6')–C(6')–C(5')	111.9 (5)

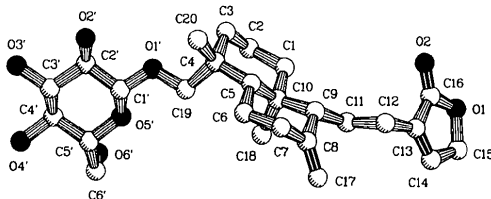


Fig. 1. PLUTO (EUCLID version; Spek, 1982) drawing of the title compound; H atoms and the water molecule were omitted for clarity. Double bonds are indicated.

Table 3. Short intermolecular O...O contacts < 3 Å (Å)

O(2')...O(3')	(-x-2, y+1/2, -z)	2.812 (6)
O(2')...O(10)	(x-2, y-1, z)	2.776 (8)
O(3')...O(6')	(-x-1, y+1/2, -z)	2.670 (6)
O(3')...O(4')	(-x-2, y+1/2, -z)	2.876 (6)
O(5')...O(10)	(x-1, y-1, z)	2.815 (7)
O(6')...O(10)	(x-1, y-1, z)	2.829 (8)

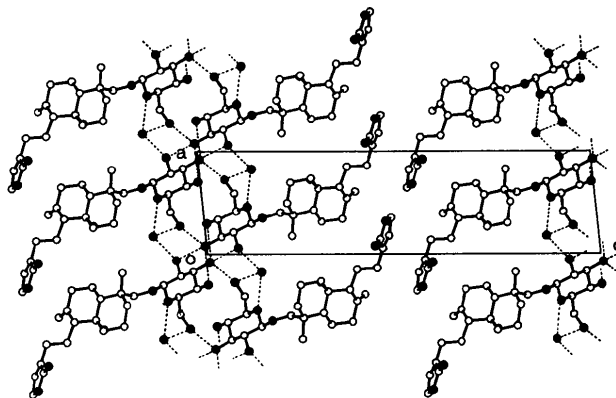


Fig. 2. Projection of the structure down *b*; short intermolecular O...O contacts < 3 Å are indicated by dashed lines.

The title molecule and the neighbouring water molecule are involved in six short intermolecular O...O contacts (< 3 Å) which are listed in Table 3. Although no detailed analysis of the hydrogen-bonding scheme is possible in view of the fact that only two of the six hydroxylic H atoms could be identified, it is clear that the crystal structure contains two-dimensional hydrogen-bonded bi-layers in the *ab* plane involving glucopyranose moieties and water molecules (Fig. 2). The hydrogen bonds which have been found [O(2')—H...O(3')(-*x*-2, $\frac{1}{2} + y$, -*z*) and O(3')—H...O(6')(-*x*-1, $\frac{1}{2} + y$, -*z*)] already form a two-dimensional network along the *ab* plane. The O(1) and O(2) atoms of the five-membered lactone are not involved in any significant short intermolecular contacts. The layers of hydrogen-bonded molecules are separated by layers with weak intermolecular interactions consisting of the lactonic groups of the title molecule (Fig. 2).

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Structure of 5-Hydroxy-6,7,8,3',4',5'-hexamethoxyflavone, Gardenin A*†

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Abstract. C₂₁H₂₂O₉, *M_r* = 418.4, orthorhombic, *Pbca*, *a* = 15.873 (2), *b* = 7.241 (1), *c* = 33.715 (5) Å, *V* = 3875 (2) Å³, *Z* = 8, *D_m* = 1.42 (2), *D_x* = 1.430 (1) Mg m⁻³, λ(Co *Kα*) = 1.7903 Å, μ(Co *Kα*) = 1.493 mm⁻¹, *F*(000) = 1760, *T* = 298 K, *R* = 0.060, *wR* = 0.057 for 1715 reflections. The phenyl ring is quite planar and makes an angle of 155.2° with the γ -pyrone ring.

Introduction. Gardenin A is the yellow crystalline component of the *Dikamali* gum, which is the resinous exudation of the leaf bud of *Gardenia lucida* Roxb. belonging to the N.O. Rubiaceae. The structure determination was undertaken to establish the structure–activity relationship and also to confirm the

structure predicted by chemical methods (Hegnauer, 1973).

Experimental. *D_m* by flotation, yellow needles (from methanol:acetone, 1:1 mixture) 0.3 × 0.5 × 0.2 mm, Picker four-circle manual diffractometer (at the Department of Crystallography and Biophysics, University of Madras), Co *Kα* radiation, cell parameters refined from 35 strong, accurately centered reflections in the range 25 ≤ 2θ ≤ 63°, θ/2θ scan technique with a symmetric range of ±1° in 2θ and a scan rate of 2° min⁻¹, stationary background counts for 10 s at each extreme of scan width, Lp correction, absorption ignored, 2096 unique reflections with 2θ ≤ 130°, 0 ≤ *h* ≤ 16, 0 ≤ *k* ≤ 7, 0 ≤ *l* ≤ 34, 1715 observed reflections with |*F_o*| ≥ 3.0σ(|*F_o*|), solution using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), |*E*| ≥ 1.30 for phase generation, all nonhydrogens except for the six methyl carbons located from the *E* map. The methyl carbons and hydrogens subsequently located from Δρ map. Full-matrix least-squares

* 5-Hydroxy-6,7,8-trimethoxy-2-(3,4,5-trimethoxyphenyl)-4*H*-1-benzopyran-4-one (*Chemical Abstracts* name).

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