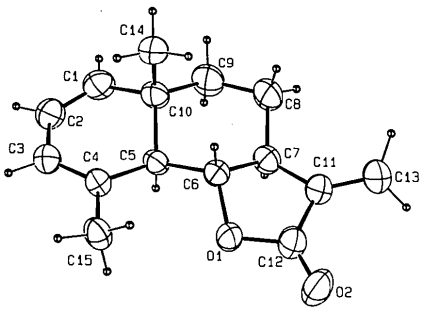

Fig. 1. The molecule of gazaniolide, with thermal ellipsoids drawn at the 40% probability level, and hydrogen atoms with arbitrary radius.

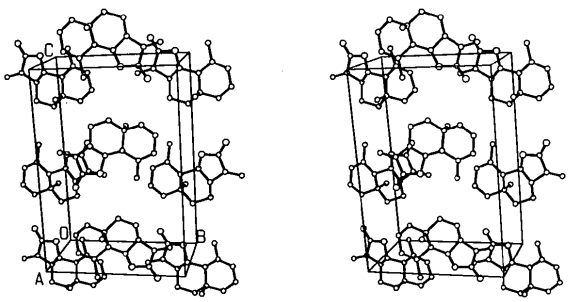

Fig. 2. Stereoview of the unit cell.

Related literature. Isolation of gazaniolide from the roots of *Rudbeckia subtomentosa*: Vasquez, Quijano, Fronczek, Macias, Urbatsch, Cox & Fischer (1990), occurrence of gazaniolide in *Gazania krebsiana*: Bohlmann & Zdero (1979), crystal structure of isodrimenin: Escobar & Wittke (1988), crystal structure of irazunolide: Hasbun, Calvo, Poveda, Malcolm, Delord, Watkins, Fronczek & Fischer (1982), crystal structure of tauremisin: Tavanaipour, Watson, Miski, Gage & Mabry (1987), crystal structure of *O*-(bromoacetyl)-tetrahydrodouglanine: Ul-Haque, Caughlan, Emerson, Geissman & Matsueda (1970).

We are grateful to Dr Leo Quijano and Dr Klaus Fischer for providing the crystals.

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Acta Cryst. (1990). C**46**, 2476–2478

trans-3,6-Dinonyl-1,2,4,5-tetroxane

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(*Received* 3 *April* 1990; *accepted* 23 *May* 1990)

Abstract. *trans*-3,6-Dinonyl-1,2,4,5-tetroxane, $C_{20}H_{14}O_4$, m.p. 340 K, $M_r = 172{\cdot}3$, triclinic, $P\bar{1}$, $a = 4{\cdot}517\ (2)$, $b = 5{\cdot}513\ (1)$, $c = 22{\cdot}225(3)$ Å, $\alpha = 89{\cdot}46\ (1)$, $\beta = 84{\cdot}07\ (1)$, $\gamma = 84{\cdot}34\ (1)°$, $V = 547{\cdot}8\ (3)$ Å^3, $Z = 1$, $D_x = 1{\cdot}04$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}066$ mm^{-1}, $F(000) = 192$, room

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Table 1. *Fractional coordinates and equivalent isotropic displacement parameters with e.s.d.'s in parentheses*

U_{eq} is the average of eigenvalues of U.

	x	y	z	U_{eq} (Å^2)
O(1)	0·2916 (7)	0·6799 (6)	0·5228 (2)	0·046 (3)
O(2)	0·6502 (7)	0·5527 (6)	0·4455 (1)	0·046 (3)
C(1)	0·348 (1)	0·630 (1)	0·4609 (3)	0·041 (5)
C(2)	0·274 (1)	0·861 (1)	0·4269 (3)	0·048 (5)
C(3)	0·251 (1)	0·841 (1)	0·3610 (3)	0·054 (5)
C(4)	0·153 (1)	1·081 (1)	0·3326 (3)	0·060 (6)
C(5)	0·098 (1)	1·070 (1)	0·2677 (3)	0·066 (6)
C(6)	−0·006 (1)	1·310 (1)	0·2395 (4)	0·071 (6)
C(7)	−0·074 (1)	1·303 (1)	0·1756 (3)	0·073 (6)
C(8)	−0·176 (2)	1·543 (1)	0·1487 (3)	0·079 (7)
C(9)	−0·248 (2)	1·541 (1)	0·0854 (4)	0·100 (7)
C(10)	−0·352 (3)	1·773 (2)	0·0571 (4)	0·13 (1)

Table 2. *Selected bond lengths* (Å), *bond angles and torsional angles* (°) *with e.s.d.'s in parentheses*

The primed atoms are obtained by the symmetry operation $1-x$, $1-y$, $1-z$.

O(1)—O(2)′	1·473 (5)	O(2)′—O(1)—C(1)	106·8 (3)
O(1)—C(1)	1·398 (8)	O(1)′—O(2)—C(1)	106·1 (3)
O(2)—C(1)	1·400 (6)	O(1)—C(1)—O(2)	110·6 (5)

C(1)—O(1)—O(2)′—C(1)′	62·0 (5)	C(2)—C(3)—C(4)—C(5)	−174·7 (5)
O(2)′—O(1)—C(1)—O(2)	−64·9 (5)	C(3)—C(4)—C(5)—C(6)	178·8 (5)
O(1)′—O(2)—C(1)—O(1)	64·5 (5)	C(4)—C(5)—C(6)—C(7)	−177·5 (6)
O(2)′—O(1)—C(1)—C(2)	176·9 (4)	C(5)—C(6)—C(7)—C(8)	−179·9 (6)
O(1)—C(1)—C(2)—C(3)	−166·5 (5)	C(6)—C(7)—C(8)—C(9)	−179·4 (6)
C(1)—C(2)—C(3)—C(4)	175·6 (5)	C(7)—C(8)—C(9)—C(10)	179·7 (8)

temperature, $R = 0{\cdot}063$, $wR = 0{\cdot}042$ for 1759 reflections with $|F_o| > 4\sigma(F_o)$. The tetroxane ring adopts a perfect chair conformation and is located on a crystallographic centre of inversion. The *n*-nonyl substituents are *trans* disposed in equatorial positions. The alkyl chain exhibits an all-*trans* conformation with a maximum deviation of 0·142 (8) Å [C(1)] from the mean plane passing through C(1) to C(10). No intermolecular distances significantly shorter than normal values were found.

Experimental. Single crystals were grown at 269 K from $CHCl_3/CH_3OH$ (1/1) solution; crystal size 0·10 × 0·18 × 0·20 mm; Philips PW1100 diffractometer; graphite-monochromated Mo$K\alpha$ radiation; $\omega/2\theta$ scans; scan speed 0·05° s^{-1}; $4 < 2\theta < 42°$, h −4 to 4, k −5 to 5, l 0 to 22, 1185 unique reflections collected from which 759 are considered as observed [$|F_o| > 4\sigma(F_o)$]. Lattice parameters from 20 reflections ($23 < 2\theta < 39°$). Two standard reflections every 60 min [variation < $2{\cdot}5\sigma(I)$]. Lorentz–polarization correction; no absorption or secondary-extinction corrections; structure solved by *MULTAN*87 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1987). Scattering factors from Cromer & Mann (1968). H atoms were located in a difference map and refined with a fixed value ($U = 0{\cdot}05$ Å^2) for isotropic atomic displacement parameters. All calculations were performed with the *XTAL*2.6 program (Hall & Stewart, 1989). Full-matrix least squares using $|F|$ values, 169 variables, 759 contributing reflections, $R = 0{\cdot}063$, $wR = 0{\cdot}042$ [$w = 1/\sigma^2(F_o)$], $S = 2{\cdot}96$, max. Δ/σ: 0·324 (for H atom), max. and min. $\Delta\rho$: 0·32, −0·42 e Å^{-3}. It should be noted that the metric of the unit cell is close to a monoclinic *C* system (delta = 0·11; Le Page, 1982) but the intensities of symmetry equivalent reflections are clearly inconsistent with such a system. Atomic coordinates and selected structural data are listed in Tables 1 and 2 respectively.* Plots of the molecule and unit cell are given in Figs. 1 and 2 respectively.

Related literature. Synthesis of 1,2,4,5-tetroxanes from bis(trimethylsilyl) peroxide and carbonyl compounds was recently developed in our laboratories (Jefford, Jaber & Boukouvalas, 1988). Surprisingly,

* Lists of structure factors, atomic positional and anisotropic displacement parameters for all atoms and other information in the printed form of the Standard Crystallographic File Structure of Brown (1985) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53184 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

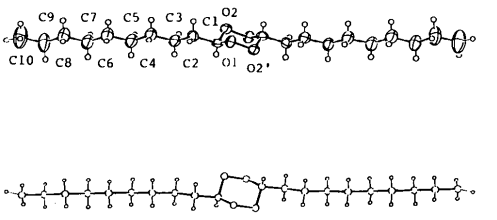

Fig. 1. Perspective views of *trans*-3,6-dinonyl-1,2,4,5-tetroxane with atom numbering.

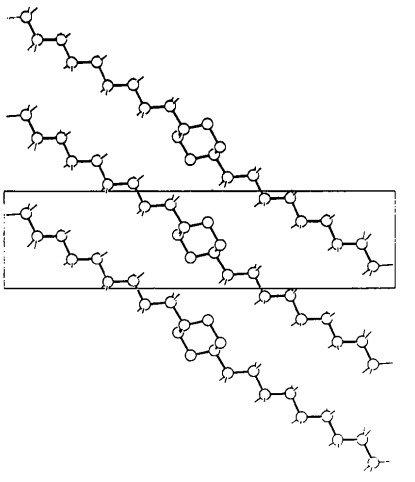

Fig. 2. Projection of the unit cell along the *a* axis.

the *trans*-3,6-dialkyl derivatives (*n*-pentyl and *n*-nonyl) prepared by this approach exhibit melting points and ^{1}H NMR data different from those recorded in the literature (Miura & Nojima, 1980; Miura, Ikegami, Nojima, Kusabayashi, McCullough & Walkinshaw, 1983). Consequently we undertook an X-ray study of the *n*-nonyl derivative obtained by our method in order to confirm its identity. Apart from two disubstituted derivatives, namely the 3,6-diphenyl- (Groth, 1967*a*) and 3,6-dimethoxy-1,2,4,5-tetroxanes (Chiang, Butler & Kuczkowski, 1988), most tetroxanes studied by X-ray crystallography are symmetrically tetrasubstituted (Groth, 1967*b*,*c*,*d*; Shulz, Kirsche & Höhne, 1967).

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Acta Cryst. (1990). C**46**, 2478–2480

Structure of 7-Hydroxy-1,6,12(*S*)-triacetoxyneoclerodane-4(18),13(14)-dien-15,16-olide (Grandifolide A): a Diterpene

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(*Received* 30 *November* 1989; *accepted* 16 *May* 1990)

Abstract. $C_{26}H_{36}O_9$, $M_r = 492{\cdot}5$, orthorhombic, $P2_12_12_1$, $a = 17{\cdot}467\ (3)$, $b = 17{\cdot}492\ (3)$, $c = 8{\cdot}568\ (2)$ Å, $V = 2616\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}25$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}741$ mm^{-1}, $F(000) = 1056$, $T = 293$ K, $R = 0{\cdot}047$ for 1711 observed reflections. The X-ray study confirms that in the solid state the structure of the title compound is similar to that inferred from chemical and spectroscopic evidence. The Cremer & Pople [*J. Am. Chem. Soc.* (1975), **97**, 1354–1358] ring-puckering parameters for the six-membered rings are $\theta = 7{\cdot}8\ (4)$, $\varphi = -127\ (3)°$, $Q = 0{\cdot}598\ (4)$ Å (ring *A*); $\theta = 6{\cdot}6\ (4)$, $\varphi = 79\ (4)°$, $Q = 0{\cdot}548\ (3)$ Å (ring *B*), indicating a distorted chair conformation in each case. The *A*/*B* junction is *cis*. The acetoxy, hydroxyl and C(8) methyl groups are equatorial whereas the methyl groups at C(5) and C(9) are axial. The crystal structure is stabilized by an intermolecular hydrogen bond between the O(3)—H hydroxyl group and the O(7) carbonyl group, O(3)—H···O(7) (x, y, $1 + z$) 2·956 (6) Å.

Experimental. Grandifolide *A* is a naturally occurring diterpene which was isolated from the aerial parts of the plant *Cormutia grandifolia* (Schl. et Cham) Schauer, Verbenacea family. The sample was collected in San Andrés Tuxtla (Estado de Veracruz, Mexico).

The title compound was crystallized from acetone–hexane and gave colourless crystals. Size of crystal 0·16 × 0·26 × 0·36 mm. Nicolet *R*3 four-circle diffractometer, Ni-filtered Cu $K\alpha$ radiation. Lattice parameters from 25 machine-centred reflections with $10{\cdot}1 < 2\theta < 21{\cdot}1°$. 1902 reflections with $3 < 2\theta < 110°$ for one octant, 1711 independent with $I > 2{\cdot}8\sigma(I)$, index range h 0→18, k 0→18, l 0→9, $2\theta/\theta$ scan mode, variable scan speed. Two standard reflections (111, 102) monitored every 50 measurements; no significant variation. Lp correction. Data

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† Contribution No. 1007 of the Instituo de Química, UNAM.

0108-2701/90/122478-03$03.00