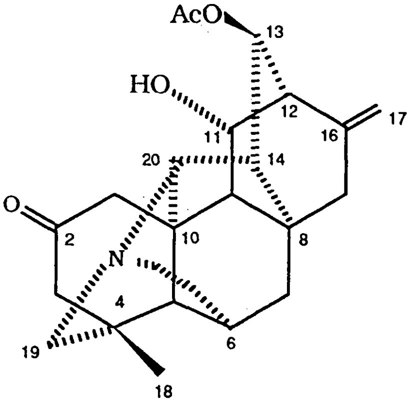

Fig. 1. Chemical numbering scheme.

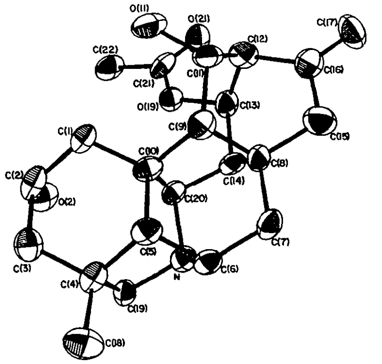

Fig. 2. *ORTEP* plot (Johnson, 1965) of hetisine 13-*O*-acetate. Thermal ellipsoids are drawn at the 50% probability level.

angles are given in Table 2.* Fig. 1 shows the chemical numbering scheme and an *ORTEP* plot is presented in Fig. 2. Relative stereochemistry and skeletal framework determined in this study confirm preliminary identification of the new diterpenoid alkaloid as the 13-*O*-acetyl derivative of hetisine.

Related literature. A preliminary report of this work and the isolation and spectroscopic identification of the title compound have been published (Benn, Richardson & Majek, 1986). The absolute configuration of the structure reported herein was not determined but the stereochemistry presented is in agreement with that determined for the parent alkaloid, hetisine (Przybylska, 1962).

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* 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 52307 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). **C46**, 346–348

Structure of 3-Oxa-1,5-pentanediyl Bis(*p*-toluenesulfonate)

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Abstract. $C_{18}H_{22}O_7S_2$, $M_r = 414{\cdot}5$, monoclinic, $C2/c$, $a = 23{\cdot}772\ (8)$, $b = 5{\cdot}472\ (5)$, $c = 15{\cdot}284\ (6)$ Å, $\beta = 89{\cdot}61\ (3)°$, $V = 1987{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}385$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 29{\cdot}06$ cm^{-1}, $F(000) = 872$, $T = 294$ K, $R = 0{\cdot}045$ for 538 unique observed reflections [$I > 3\sigma(I)$]. This linear polyether

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Table 1. *Crystallographic data-collection and structure-refinement details*

Crystal	Colourless
Crystal size (mm)	0·45 × 0·07 × 0·07
Diffractometer	CAD-4 Enraf–Nonius
Monochromator	Graphite
Reflections used for measuring lattice parameters	25
θ range (°)	7–13
Corrections	Background, Lorentz, polarization
Scan type	$\omega/2\theta$
Scan range (°)	$1 + 0·35 \tan\theta$
2θ range (°)	4–50
Standard reflections	002, 20$\bar{4}$, $\bar{5}\bar{1}\bar{1}$; measured every hour
Variation of standards	< 1%
Reflections measured	Two octants, *hkl*, *hk*$\bar{l}$ $0 \leq h \leq 28, 0 \leq k \leq 6, -18 \leq l \leq 18$
Unique reflections	1829
Reflections kept for refinement	538 $I > 3\sigma(I)$
Computing program	*SDP* (B. A. Frenz & Associates Inc., 1982)
Function minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Weighting scheme	$w = 1/\sigma^2(F)$
S	2·68
Max. shift/e.s.d. (last cycle)	< 0·03
R	0·045
wR	0·043
$\Delta\rho$(e Å^{-3})	0·055

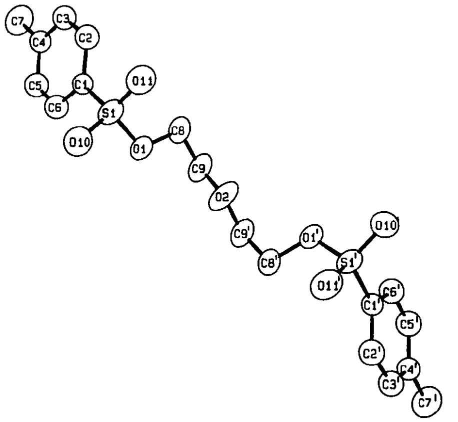

Fig. 1. *ORTEP* (Johnson, 1976) drawing of the molecule. Thermal ellipsoids are scaled to enclose 50% probability.

in *gauche–trans–trans–gauche* (*gttg*) conformation represents an important intermediate in the synthesis of crown ethers.

Experimental. Crystals were obtained from dichloroethane. A summary of data-collection parameters and structural refinement is given in Table 1.

The structure was solved using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). All non-H atoms were located from an E map. H atoms placed and fixed in calculated positions. Refinement by full-matrix least squares. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The final cycle of refinement, with thermal parameters for H atoms fixed at $B = 5$ Å^2, included 92 variable parameters and converged to $R = 0·045$.

Table 2. *Positional and isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}/B_{iso}(Å^2)
S(1)	0·3842 (1)	0·0573 (4)	0·5632 (1)	4·26 (5)
O(10)	0·3367 (2)	0·148 (1)	0·6103 (3)	5·5 (2)
O(11)	0·4260 (2)	0·223 (1)	0·5296 (3)	5·4 (2)
C(1)	0·3621 (3)	−0·135 (1)	0·4798 (4)	3·1 (2)*
C(2)	0·3808 (3)	−0·096 (2)	0·3947 (5)	4·4 (2)*
C(3)	0·3606 (3)	−0·247 (2)	0·3293 (5)	4·3 (2)*
C(4)	0·3243 (3)	−0·436 (2)	0·3455 (4)	3·8 (2)*
C(5)	0·3062 (3)	−0·465 (5)	0·4295 (5)	4·7 (2)*
C(6)	0·3238 (3)	−0·319 (1)	0·4968 (5)	4·3 (2)*
C(7)	0·3023 (2)	−0·5954 (9)	0·2723 (2)	6·3 (3)
O(1)	0·4131 (3)	−0·1223 (9)	0·6303 (5)	4·5 (1)
C(8)	0·4671 (3)	−0·228 (2)	0·6052 (5)	4·9 (2)
C(9)	0·4845 (3)	−0·388 (2)	0·6779 (5)	5·2 (2)
O(2)	0·500	−0·250	0·750	5·0 (2)

* Atoms refined isotropically.

Table 3. *Interatomic distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

S(1)	O(10)	1·424 (5)	C(3)	C(4)	1·369 (11)
S(1)	O(11)	1·436 (6)	C(4)	C(5)	1·361 (10)
S(1)	C(1)	1·738 (7)	C(4)	C(7)	1·514 (8)
S(1)	O(1)	1·582 (2)	C(5)	C(6)	1·368 (11)
C(1)	C(2)	1·389 (10)	O(1)	C(8)	1·457 (8)
C(1)	C(6)	1·380 (11)	C(8)	C(9)	1·475 (11)
C(2)	C(3)	1·382 (11)	C(9)	O(2)	1·386 (8)

O(10)	S(1)	O(11)	120·3 (4)	C(2)	C(3)	C(4)	122·7 (7)
O(10)	S(1)	C(1)	109·9 (3)	C(3)	C(4)	C(5)	116·9 (7)
O(10)	S(1)	O(1)	103·6 (2)	C(3)	C(4)	C(7)	121·5 (6)
O(11)	S(1)	C(1)	109·4 (3)	C(5)	C(4)	C(7)	121·5 (7)
O(11)	S(1)	O(1)	108·7 (3)	C(4)	C(5)	C(6)	123·1 (8)
C(1)	S(1)	O(1)	103·5 (3)	C(1)	C(6)	C(5)	119·2 (7)
S(1)	C(1)	C(2)	119·8 (6)	S(1)	O(1)	C(8)	117·5 (3)
S(1)	C(1)	C(6)	120·5 (5)	O(1)	C(8)	C(9)	106·7 (6)
C(2)	C(1)	C(6)	119·6 (7)	C(8)	C(9)	O(2)	110·8 (7)
C(1)	C(2)	C(3)	118·4 (7)	C(9)	O(2)	C(9′)	114·2 (5)

S(1)	O(1)	C(8)	C(9)	−179·9 (7)	C(9)	O(2)	C(9′)	C(8′)	176·7 (5)
O(1)	C(8)	C(9)	O(2)	−71·2 (8)	O(2)	C(9′)	C(8′)	O(1′)	−71·2 (8)
C(8)	C(9)	O(2)	C(9′)	176·7 (5)	C(9′)	C(8′)	O(1′)	S(1′)	−179·9 (7)

Fig. 1 shows a projection of the molecule and the atomic numbering scheme. Final positional and isotropic thermal parameters for all non-H atoms are given in Table 2.* Bond distances, bond and torsion angles are listed in Table 3. The intermolecular packing is governed by C···O contacts which are of the order of the sum of the van der Waals radii [the shortest intermolecular distance is O(11)···C(8^{i}) = 3·27 Å, (i) = $\bar{x}$, $\bar{y}$, $\bar{z}$].

Related literature. The torsion angles from S(1) to S(1′) clearly show a *tgttgt* conformation in contrast

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* 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 52284 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

to all uncomplexed 18-crown-6 crown ethers (Dobler, 1981, and references therein). The ranges of distance and angle values for the tosyl group are similar to values found in related compounds (James & McConnell, 1971; Sim, 1987). The low-temperature (143 K) structure of the title compound was reported by Groth (1985).

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SHORT COMMUNICATION

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Acta Cryst. (1990). **C46**, 348

Structure of recinine. Erratum.

By M. Soriano-García, M. Jímenez E., R. Reyes Vaca and R. A. Toscano, *Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán* 04510, *Mexico DF*

(*Received* 23 *November* 1989)

Abstract

In the paper by Soriano-García, Jímenez E., Reyes Vaca & Toscano [*Acta Cryst.* (1989). **C45**, 957–959] the name of the title compound should be ricinine and the name of the plant from which it is derived is *Ricinus communis*.

All relevant information is given in the *Abstract*.

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