88° in axillarine. However, the toxicity decreases in the order fulvine : monocrotaline : axillarine $(1:\frac{1}{2}:\frac{1}{7})$ (Schoental, 1976). Fulvine possesses only one hydroxyl substituent, monocrotaline two and axillarine three. Hence as suggested previously (Schoental, 1968), water solubility and hence increased rate of excretion of the alkaloids play an important role in the degree of toxicity.

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2-(*p*-Toluenesulfonyl)-3-(*p*-chlorophenyl)oxaziridine

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Abstract. $C_{14}H_{12}ClNO_3S$, $M_r = 309\cdot771$, triclinic, $P\bar{1}$, $a = 7\cdot504\,(3)$, $b = 8\cdot112\,(2)$, $c = 13\cdot041\,(3)$ Å, $\alpha = 105\cdot61\,(2)$, $\beta = 94\cdot52\,(2)$, $\gamma = 112\cdot33\,(2)°$, $V = 692\cdot54\,(2)$ Å^3, $Z = 2$, $D_c = 1\cdot485$ Mg m^{-3}, $\mu = 3\cdot69$ mm^{-1}, $\lambda(\mathrm{Cu}\ K\alpha) = 1\cdot54178$ Å, $R = 0\cdot069$ for all 1885 observed reflections. The *p*-toluenesulfonyl and *p*-chlorophenyl groups are *trans* with respect to the oxaziridine ring.

Introduction. Oxaziridines are of considerable interest because of their unusual heterocyclic ring system (Davis, Nadir & Kluger, 1977; Davis & Nadir, 1977), and the *p*-toluenesulfonyl derivatives are of particular interest because of the conformation about the S—N bond. The three-membered heterocyclic ring makes it energetically unfavorable for the N atom to achieve a pseudo-planar conformation which would favor lone-pair–*d*-orbital interactions, and it is postulated that electron-repulsion interactions are more significant in the determination of the conformation (Chen, Watson, Davis, Lamendola & Nadir, 1978).

The title compound was synthesized and recrystallized from toluene at room temperature yielding a white crystalline material. A crystal of dimensions 0·3 × 0·4 × 0·5 mm was used to collect all data. The unit cell was found to be triclinic and room-temperature cell dimensions were obtained by a least-squares fit to 15 medium-angle reflections. There were no systematic absences and statistics indicated space group $P\bar{1}$. Intensity data for $2\theta < 120°$ were collected on a Syntex $P2_1$ diffractometer by the $\theta{:}2\theta$ scan technique using graphite-monochromatized radiation and a variable scan speed. A periodically monitored reflection showed no significant crystal deterioration. Of the 1885 independent reflections measured, 1853 had intensities greater than $3\sigma(I)$ where $\sigma(I)$ was estimated from counting statistics. Negative measured intensities were assigned a value of zero. Lorentz and polarization corrections were applied but no absorption corrections were made.

The structure was solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971) to

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calculate phases for the 400 $|E|$ values greater than 1·27. The E map computed from the phase set with the largest combined figure of merit revealed all 20 non-hydrogen atoms. Full-matrix least-squares refinement with anisotropic thermal parameters yielded an R value of 0·075 where $R = [\sum \Delta F_{\rm meas}/\sum |F_{\rm meas}|]$. A difference map computed with the phases from the least-squares refinement revealed the positions of all H atoms. Least-squares refinement with isotropic H-atom thermal parameters yielded a final R of 0·069 for all 1885 reflections. The function minimized was $\sum w(\Delta F_{\rm meas})^2$ where $w = 1/\sigma^2(F)$. The scattering factors for all atoms were taken from *International Tables for X-ray Crystallography* (1974). The scattering factors for Cl were corrected for the real part of the anomalous dispersion.

All parameter shifts during the final cycle were less than 0·05σ, and a final difference map showed a peak of 0·46 e Å^{-3} associated with the S atom with the remainder of the peaks less than 0·35 e Å^{-3}. Atomic positional parameters are given in Table 1 while bond lengths and angles are indicated in Figs. 1 and 2.*

Table 1. *Atomic positional parameters* $\times 10^4$ (*hydrogen parameters* $\times 10^3$) *for* 2-(*p-toluenesulfonyl*)-3-(*p-chlorophenyl*)*oxaziridine*

	x	y	z
Cl(1)	1614 (2)	8467 (2)	6329 (1)
S(1)	8645 (1)	6781 (1)	1903 (1)
O(1)	8260 (5)	7970 (4)	1379 (2)
O(2)	10439 (4)	7455 (4)	2647 (2)
O(3)	6924 (4)	4922 (4)	3159 (2)
N(1)	6661 (4)	6149 (4)	2542 (2)
C(1)	7237 (5)	6796 (5)	3718 (3)
C(2)	5798 (5)	7127 (5)	4361 (3)
C(3)	6489 (6)	8534 (5)	5344 (3)
C(4)	5199 (6)	8950 (6)	5956 (3)
C(5)	3244 (6)	7911 (5)	5570 (3)
C(6)	2492 (6)	6470 (6)	4601 (3)
C(7)	3795 (6)	6071 (6)	3985 (3)
C(8)	8178 (5)	4596 (5)	963 (3)
C(9)	8903 (6)	3436 (6)	1271 (3)
C(10)	8401 (6)	1667 (6)	553 (3)
C(11)	7254 (5)	1047 (6)	−459 (3)
C(12)	6574 (6)	2266 (6)	−746 (3)
C(13)	7022 (6)	4024 (6)	−47 (3)
C(14)	6728 (7)	−881 (6)	−1237 (4)
H(1)	855 (5)	762 (5)	400 (3)
H(3)	787 (6)	927 (6)	555 (3)
H(4)	574 (5)	983 (5)	658 (3)
H(6)	102 (6)	569 (6)	428 (3)
H(7)	314 (6)	497 (6)	320 (3)
H(9)	955 (6)	372 (5)	200 (3)
H(10)	882 (5)	103 (5)	81 (3)
H(12)	592 (6)	203 (6)	−144 (3)
H(13)	669 (7)	489 (6)	−23 (3)
H(14*a*)	518 (9)	842 (8)	−180 (4)
H(14*b*)	673 (6)	−140 (6)	−97 (3)
H(14*c*)	775 (8)	−106 (7)	−188 (4)

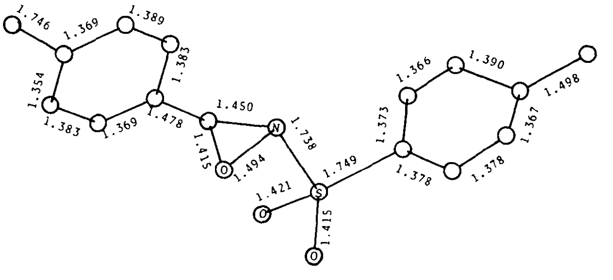

Fig. 1. Bond lengths (Å). Standard deviations range from 0·003 to 0·008 Å.

Discussion. Fig. 3 is an *ORTEP* drawing (Johnson, 1965) of the 2-(*p*-toluenesulfonyl)-3-(*p*-chlorophenyl)-oxaziridine molecule. The *p*-toluenesulfonyl and *p*-

Table 2. *Comparison of torsion angles* (°) *and interplanar angles* (°) *for* 2-(*p-toluenesulfonyl*)-3-(*p-chlorophenyl*)*oxaziridine* (I) *and the m-nitrophenyl analogue* (II)

Torsion angles	I	II
O(2)S(1)N(1)O(3)	−49	−36
O(1)S(1)N(1)O(3)	−178	−163
O(2)S(1)N(1)C(1)	12	27
O(1)S(1)N(1)C(1)	−117	−101
O(1)S(1)C(8)C(9)	164	160
O(1)S(1)C(8)C(13)	−19	−24
O(2)S(1)C(8)C(9)	29	26
O(2)S(1)C(8)C(13)	−154	−159
S(1)N(1)O(3)C(1)	107	107
S(1)N(1)C(1)O(3)	−96	−100
S(1)N(1)C(1)C(2)	153	149
N(1)C(1)C(2)C(3)	−147	−136
N(1)C(1)C(2)C(7)	32	39
N(1)S(1)C(8)C(9)	−88	−93
N(1)S(1)C(8)C(13)	89	83
C(1)N(1)S(1)C(8)	128	146
O(3)N(1)S(1)C(8)	67	83
O(3)N(1)C(1)C(2)	−110	−111
Interplanar angles		
O(3)N(1)C(2)–C(2)···C(7)	85	86
O(3)N(1)C(2)–C(8)···C(13)	21	28
C(2)···C(7)–C(8)···C(13)	66	59

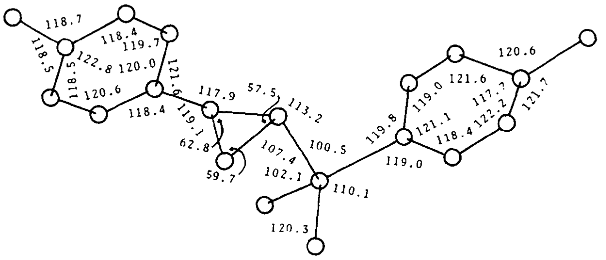

Fig. 2. Valence angles (°) with standard deviations either 0·2 or 0·3°.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33931 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

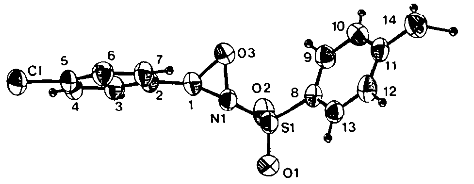

Fig. 3. *ORTEP* drawing of 2-(*p*-toluenesulfonyl)-3-(*p*-chlorophenyl)oxaziridine. Thermal ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

chlorophenyl groups are *trans* (*E* configuration) with respect to the oxaziridine ring. The two sulfonyl O atoms and N(1) are symmetrically oriented with respect to the toluene ring as indicated by the torsion angles of 29 and −19° for O(2)S(1)C(8)C(9) and O(1)S(1)C(8)C(13) and 89 and −88° for N(1)S(1)-C(8)C(13) and N(1)S(1)C(8)C(9). The N lone pair lies opposite the sulfonyl O atoms which is also the conformation adopted by 2-(*p*-toluenesulfonyl)-3-(*m*-nitrophenyl)oxaziridine (Chen, Watson, Davis, Lamendola & Nadir, 1978), Table 2. In *N*,*N*,*N*′,*N*′-tetramethylsulfamide (Jordan, Smith, Lohr & Lipscomb, 1963), *p*-methoxybenzenesulfon-*p*-anisidine (Pokrywiecki, Weeks & Duax, 1973*a*) and *p*-methoxybenzenesulfon-*N*-isopropyl-*p*-anisidine (Pokrywiecki, Weeks & Duax, 1973*b*) the lone pairs of the N atoms lie between the two sulfonyl O atoms. These compounds exhibit a flattened geometry around N and a shortening of the S—N bond to an average distance of 1·628 (4) Å which was interpreted in terms of lone-pair–*d*-orbital interactions. The N—S distance in the title compound is 1·738 (4) and 1·728 (4) Å in the *m*-nitrophenyl derivative. The C(1)N(1)O(3) angle of 57·5° makes it energetically unfavorable for the N atom to achieve a pseudo-planar conformation in which the angle approaches 120°. Lone-pair–*d*-orbital interactions in these systems are not significant.

The parameters for the oxaziridine ring in the title compound and in the *m*-nitrophenyl derivative compare quite well: C—O = 1·415 (5) and 1·414 (5) Å, C—N = 1·450 (5) and 1·443 (6) Å, O—N = 1·494 (5) and 1·487 (6) Å, C—O—N = 59·7 (2) and 59·6 (3)°, O—N—C = 57·5 (2) and 57·7 (3)° and N—C—O = 62·8 (2) and 62·7 (3)°. Torsion angles and interplanar angles are compared in Table 2.

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Structure of 15β-Acetoxy-(—)-kaur-16-en-19-oic Acid (Xylopic Acid)

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Abstract. $C_{22}H_{32}O_4$, orthorhombic, $P2_12_12_1$, $a = 14\cdot966$ (2), $b = 11\cdot885$ (2), $c = 11\cdot074$ (3) Å, $V = 1969\cdot7$ Å^3, $Z = 4$, $\rho_x = 1\cdot27$ Mg m^{-3}, μ(Cu $K\alpha$) = 0·711 mm^{-1}. The diterpene, xylopic acid, was isolated from the dried fruits of *Xylopia aethiopica*. The structure was solved by direct methods and refined to $R = 0\cdot045$. One hydrogen bond is observed.

Introduction. The dried fruits of the plant *Xylopia aethiopica* are used in parts of West Africa as spices and in folklore medicine for curing bronchial infections. The diterpene, xylopic acid, is obtained upon extraction of these fruits with petroleum ether 30/40 (Ekong &

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