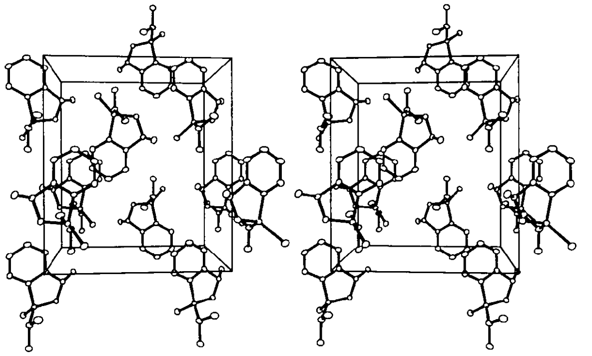

Fig. 2. A stereo pair illustrating the packing in the crystal viewed in the direction of the *c* axis.

The torsion angles S—P—N—C(1) and S—P—N—C(2) are −179·50 (11) and −36·03 (14)° respectively. This difference could be caused by steric interactions between S and C(2).

A stereo pair illustrating the packing in the crystal is shown in Fig. 2. All intermolecular contacts are larger than the sum of the van der Waals radii.

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Structure of a Sulfone that Reveals the Direction of Cleavage of a Camphor-based Thiirane 1,1-Dioxide by a Thiol*

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Abstract. 7,7-Dimethyl-1-[1-(2,4-dinitrophenylsulfonyl)-2-(*p*-tolylthio)ethyl]bicyclo[2.2.1]heptan-2-one, (V), $C_{24}H_{26}N_2O_7S_2$, $M_r = 518{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}436\,(2)$, $b = 27{\cdot}267\,(3)$, $c = 6{\cdot}987\,(1)$ Å, $V = 2369{\cdot}3$ Å^3, $Z = 4$, $D_m = 1{\cdot}459$, $D_x = 1{\cdot}454$ g cm^{-3}, Cu $K\beta$, $\lambda = 1{\cdot}39217$ Å (cell constants), Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å (intensity data), $\mu = 2{\cdot}61$ cm^{-1}, $F(000) = 1088$, $T = 293$ K, $R = 0{\cdot}061$ for 3926 independent reflections. Full-matrix least-squares refinement included isotropic hydrogen atoms. Bond distances ($\sigma = 0{\cdot}004$ Å) and angles ($\sigma = 0{\cdot}3°$) are given for the camphor moiety. The two phenyl rings in the molecule are parallel and in contact. Phenyl rings in adjacent molecules are also parallel and in contact. This structure shows that in the reaction of 2-(2-oxo-7,7-dimethyl-1-bicyclo[2.2.1]heptyl)thiirane 1,1-dioxide (I), *p*-toluenethiolate ion attacked the dioxide at the CH_2 site of the thiirane 1,1-dioxide ring.

Introduction. Protection of animals against ionizing radiation is afforded by salts of sulfinic acids (RSO_2H) of the type $—SS—C_4—SO_2Na$, where C_4 represents carbon atoms that either are in an acyclic chain or are wholly or partly in a benzenoid system (Srivastava, Field & Grenan, 1975; Bowman, Clement, Davidson, Eswarakrishnan, Field, Hoch, Musallam, Pick, Ravichandran & Srivastava, 1986). For chemical or biological details of the present report see Harmon

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* Paper 17 in the series 'Sulfinic acids and related compounds'. Paper 16: Chandra, R. & Field, L. (1986). *Phosphorus Sulfur*, **27**, 247–251.

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(1985); see Porras (1984) for crystallographic details. In a search for structures of optimum radioprotective capability, examination of C_2 counterparts has become important. Since thiirane 1,1-dioxide (I of Fig. 1, but with R=H) can be cleaved to give structures of the type $-S(CH_2)_2SO_2^-$ by sulphur-containing species such as RS^-, S^{2-}, and thiourea (Vilsmaier & Becker, 1975), such reactions seemed likely to afford routes to the desired new types of sulfinates, *i.e.* $-SS-C_2-SO_2Na$. A number of monosubstituted thiirane 1,1-dioxides are known (*cf.* Fig. 1, structure I, and Zoller, 1983), and a model reaction was desirable to assure that cleavage would occur by attack at CH_2 to give III, rather than at CH to give VI, or at sulfur to give VII or VIII (*cf.* Fig. 1). The camphor-based thiirane dioxide I therefore was used as a model substrate and sodium *p*-toluenethiolate (II) as a model attacking species typical of those needed to synthesize the desired C_2 compounds. The chirality of I was an attractive feature since it could afford a means of following reactions, as well as of assessing the possible biological importance of chirality in the C_2 products. Since sulfinate salts such as the expected product (III) or its alternative (VI) often are easily hydrated and oxidized, a suitable derivative was sought. The ester proved to be an oil (IX; see *Experimental*), but the dinitrophenyl sulfone derivative (V) proved to be quite suitable. As Fig. 1 shows, V was prepared from the sulfinate salt (III *vs* VI) by use of 2,4-dinitrochlorobenzene (IV).

Experimental. All materials were commercial unless otherwise stated. IR and NMR spectra were consistent with the structures for I, III, V and IX proposed in Fig. 1 (see Harmon, 1985). For the synthesis of V, a solution of sodium (0·50 g) in methanol (15 ml) was added (10 min) to one of the camphor-based thiirane dioxide I (4·50 g; prepared from (+)-camphor-10-sulfonylchloride; Fischer & Opitz, 1968) and *p*-thiocresol (2·45 g) in 50 ml of methanol. After 19 h, removal of solvent *in vacuo* and rubbing of the residue with ether gave 6·3 g of hygroscopic white solid (85% yield calculated as III). Of this 6·3 g, 0·60 g was stirred under reflux with 0·26 g of 2,4-dinitrochlorobenzene (IV) in 35 ml of ethanol for 23 h. Centrifugation of the precipitate that resulted, washing (water, benzene) and drying gave 0·11 g (17%) of V, m.p. 453–459 K (decomp.). Recrystallization gave V with m.p. 468 K (decomp.). *Analysis*: Calculated for $C_{24}H_{26}N_2O_7S_2$: C, 55·58; H, 5·05; N, 5·40; S, 12·36%. Found: C, 55·36; H, 5·03; N, 5·26; S, 12·15%. A small amount of yellow material left in the mother liquor may have been a diastereomer of V.

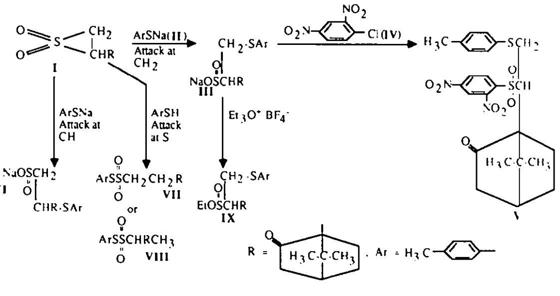

Fig. 1. Preparation of the title compound (V).

In the conversion of III to the ester IX, 0·51 g of triethyloxonium tetrafluoroborate in methylene chloride (15 ml) was added (5 min) to 1·00 g of III in methylene chloride (25 ml) at 273 K. After 2 h of vigorous stirring, the mixture was washed with water and dried (anhydrous $MgSO_4$). Removal of solvent *in vacuo* gave 0·87 g (86%) of IX, of which 0·5 g, when chromatographed on 15 g of silica gel with 15% ethyl acetate in hexane, gave 0·40 g (69%) of oily IX, indicated by NMR to be a mixture of two diastereomers: n_D^{25} 1·5572. *Analysis*: Calculated for $C_{20}H_{28}O_3S_2$: C, 63·12; H, 7·42; S, 16·85%. Found: C, 62·91; H, 7·23; S, 16·89%.

For the X-ray studies, elongated deep orange lath-shaped crystals of V were formed during *ca* 2 days when a saturated solution of V in 0·5 ml of benzene was introduced under 1 ml of pentane. Crystal density was determined by flotation in a $C_2H_2Cl_2/C_2H_2Cl_4$ mixture. Laue symmetry (*mmm*) and absent reflections (*h,k,l* odd for *h*00, 0*k*0 and 00*l*) on precession photographs require space group $P2_12_12_1$. Reported cell parameters are from a least-squares fit of 2θ, ω, χ for 12 reflections $(69 < 2\theta < 78°)$ measured at $\pm 2\theta$ with Cu $K\beta$ radiation. A crystal, 0·2 × 0·3 × 0·9 mm, mosaic spread 0·1° (FWHM), was mounted with the large dimension parallel to the diffractometer φ axis. The Picker FACS-I software (Lenhert, 1975) was used to measure 2θ step scans to $\sin\theta/\lambda$ of 0·7107 Å with Nb-filtered Mo $K\alpha$. For the 8910 reflections measured ($h\bar{k}l$ and $k\bar{k}\bar{l}$, 0–30° 2θ; hkl and $\bar{h}\bar{k}\bar{l}$, 0–60° 2θ), conditions were: 0·05° 2θ steps, 2 s each; scan width 1·2° plus dispersion; 20 s backgrounds at scan ends; coincidence loss correction. Four monitor reflections, measured at intervals, were used to correct the intensity decrease which accumulated to 2·7% in 320 h of X-ray exposure. Absorption was corrected with *ORABS* (Wehe, Busing & Levy, 1962) using an 8 × 8 × 8 Gaussian grid. Maximum, minimum and average transmission factors were 0·96, 0·91 and 0·94. Symmetry-related reflections were averaged ($R_{\rm int} = 0·045$) to give $3926F_o$; all (including 164 with zero intensity) were used for refinement.

A sharpened Patterson function showed the S—S vectors and other atoms were located with difference syntheses. H-atom positions were calculated, then confirmed with a difference synthesis. The rotationally disordered methyl group, C(40), was approximated with six equally spaced half hydrogen atoms which were not refined. Refinement was by full-matrix least squares; heavy atoms anisotropic, H atoms isotropic.

The quantity minimized was $\sum w(|F_o|-|F_c|)^2$ where $w = 1/\sigma^2(F_o)$ and the variance, σ^2, based on counting statistics included an instability term (3%). Atomic scattering factors were from Cromer & Mann (1968) except for Stewart's hydrogen values (Stewart, Davidson & Simpson, 1965). Anomalous-dispersion factors were those of Cromer & Liberman (1970). The final R (all reflections) was 0·061, wR was 0·046. If $I < 3\sigma(I)$ data are omitted, the values are 0·040 and 0·038, respectively. The maximum shift-to-e.s.d. for the final refinement cycle was 0·13, standard e.s.d. of an observation of unit weight, 1·88, maximum and minimum $\Delta\rho$ values were 0·30 and −0·35 e Å^{-3} near S(1). Atomic parameters are given in Table 1; Fig. 2 shows a stereoview of the molecule with atomic numbering (Johnson, 1976). The parameters in Table 1 were used before rounding to calculate the bond distances and angles given in Table 2.* Structure factors, electron density, bond distances and angles were calculated with the *XRAY*67 programs (Stewart, 1967) as implemented and updated on the Vanderbilt DEC-1099 computer.

Table 1. *Fractional atomic coordinates and isotropic* (B) *or equivalent isotropic* (B_{eq}) *thermal parameters* (Å^2)

$B_{eq} = \frac{8}{3}\pi^2(U_{11}^2 + U_{22}^2 + U_{33}^2)$ where the U_{ii} are the r.m.s. principal axis deviations.

	x	y	z	B or B_{eq}
S(1)	0·63235 (6)	0·19731 (2)	0·64933 (10)	2·89
S(2)	0·48269 (5)	0·25285 (2)	0·23078 (11)	3·00
O(1)	0·43818 (16)	0·12589 (8)	0·17497 (33)	4·69
O(2)	0·74577 (14)	0·19190 (7)	0·61626 (30)	4·07
O(3)	0·58686 (17)	0·17738 (7)	0·82099 (28)	4·20
O(4)	0·34797 (14)	0·28745 (7)	0·83840 (33)	4·26
O(5)	0·40117 (15)	0·22202 (7)	0·69680 (32)	4·25
O(6)	0·68198 (25)	0·44088 (9)	0·61590 (47)	7·42
O(7)	0·51292 (24)	0·43779 (8)	0·68076 (50)	7·69
N(1)	0·41428 (16)	0·26398 (8)	0·74877 (34)	2·96
N(2)	0·59938 (29)	0·41831 (10)	0·65157 (49)	5·49
C(1)	0·59278 (20)	0·12050 (8)	0·39198 (39)	2·62
C(2)	0·50666 (23)	0·10163 (10)	0·25108 (43)	3·31
C(3)	0·52111 (36)	0·04663 (11)	0·23762 (64)	4·83
C(4)	0·60957 (29)	0·03748 (10)	0·38238 (53)	4·49
C(5)	0·71407 (32)	0·05463 (12)	0·29080 (66)	4·81
C(6)	0·70095 (26)	0·11119 (10)	0·28938 (53)	3·61
C(7)	0·58898 (26)	0·07719 (10)	0·54055 (44)	3·74
C(8)	0·47971 (38)	0·07224 (14)	0·63778 (64)	5·10
C(9)	0·67800 (46)	0·07561 (15)	0·69071 (74)	5·72
C(10)	0·56480 (23)	0·17408 (9)	0·43974 (39)	2·37
C(11)	0·58807 (22)	0·20828 (9)	0·26996 (43)	2·64
C(20)	0·61041 (19)	0·26301 (8)	0·65046 (39)	2·62
C(21)	0·70179 (24)	0·29038 (10)	0·61159 (44)	3·42
C(22)	0·69925 (28)	0·34128 (12)	0·61266 (48)	3·98
C(23)	0·60495 (24)	0·36432 (9)	0·65615 (45)	3·54
C(24)	0·51332 (24)	0·33871 (10)	0·70462 (42)	3·21
C(25)	0·51660 (20)	0·28846 (9)	0·70165 (37)	2·51
C(30)	0·55192 (19)	0·30830 (9)	0·18774 (36)	2·54
C(31)	0·49316 (24)	0·35154 (10)	0·21171 (46)	3·41
C(32)	0·54200 (25)	0·39665 (10)	0·18090 (47)	3·93
C(33)	0·64940 (24)	0·40045 (10)	0·13035 (44)	3·61
C(34)	0·70576 (25)	0·35718 (11)	0·10503 (45)	3·50
C(35)	0·65854 (22)	0·31140 (10)	0·12912 (43)	2·99
C(40)	0·70401 (31)	0·44994 (11)	0·10608 (57)	5·38
H(031)	0·4526 (26)	0·0324 (11)	0·2707 (53)	6·03 (90)
H(032)	0·5320 (27)	0·0363 (11)	0·1121 (51)	5·33 (88)
H(4)	0·6053 (20)	0·0043 (10)	0·4463 (41)	4·47 (67)
H(51)	0·7791 (23)	0·0422 (11)	0·3529 (49)	4·94 (80)
H(52)	0·7211 (27)	0·0414 (13)	0·1509 (59)	6·81 (98)
H(61)	0·7021 (22)	0·1230 (10)	0·1522 (45)	4·16 (70)
H(62)	0·7575 (20)	0·1309 (9)	0·3501 (41)	3·46 (63)
H(81)	0·4784 (25)	0·0968 (11)	0·7286 (48)	5·36 (87)
H(82)	0·4182 (29)	0·0726 (14)	0·5557 (62)	8·35 (131)
H(83)	0·4805 (23)	0·0389 (11)	0·7056 (46)	5·52 (78)
H(91)	0·6596 (28)	0·0962 (12)	0·7944 (54)	6·65 (104)
H(92)	0·6818 (27)	0·0420 (13)	0·7417 (55)	7·18 (96)
H(93)	0·7396 (31)	0·0816 (15)	0·6317 (69)	8·97 (166)
H(10)	0·4935 (18)	0·1754 (7)	0·4662 (32)	1·46 (48)
H(111)	0·5940 (20)	0·1897 (9)	0·1600 (38)	3·29 (61)
H(112)	0·6542 (18)	0·2260 (8)	0·2834 (36)	2·04 (51)
H(21)	0·7680 (19)	0·2731 (8)	0·5840 (38)	2·65 (57)
H(22)	0·7678 (22)	0·3602 (11)	0·5877 (43)	4·66 (74)
H(24)	0·4461 (19)	0·3562 (9)	0·7396 (39)	2·98 (57)
H(31)	0·4191 (19)	0·3489 (9)	0·2238 (41)	3·37 (62)
H(32)	0·4965 (22)	0·4258 (10)	0·1868 (43)	5·31 (75)
H(34)	0·7736 (19)	0·3589 (9)	0·0709 (35)	2·18 (56)
H(35)	0·6930 (20)	0·2813 (9)	0·1200 (37)	2·99 (59)

Discussion. The bond distances and angles for the molecule are displayed in Table 2; those involving the refined H atoms are included with the deposited material. The geometry of the camphor moiety is of interest. The only other X-ray determination of comparable precision (Cullen, Mangion, Crist & Lightner, 1983) is the 3-hydrazone derivative. Comparison of the present work with the more precise of the two derivatives reported by Cullen *et al.* (1983), shows that, based on the reported standard deviations, the bonds in the two structures agree at the 0·01 confidence level except for C(2)—O(1), C(1)—C(2), C(1)—C(7), C(1)—C(10), C(2)—C(3) and C(4)—C(7). These differences can be understood as follows: O(1) is a hydrogen-bond acceptor in the 3-hydrazone derivative, C(1)—C(2) and C(2)—C(3) are affected by the 3-hydrazone attachment and the hydrogen-bond-lengthened C(2)—O(1), and C(10) is a methyl group in one case and a tertiary carbon in the other. This leaves unrationalized the C(7)—C(4) and C(7)—C(1) bonds, which average 1·571 Å in the present work.

Each of the two six-membered rings is planar with maximum atomic deviations of 0·02 Å as shown in Table 3 which is included with the deposited material.

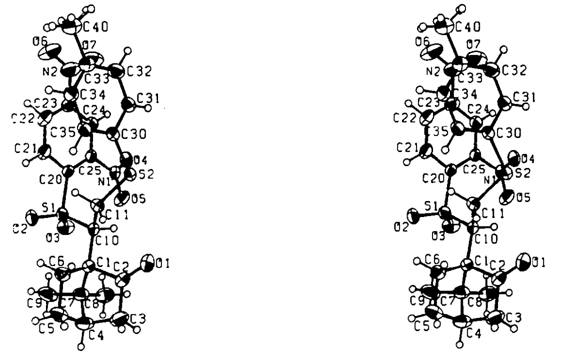

Fig. 2. Stereoview of V showing the atom numbering. Thermal ellipsoids at 40% probability.

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* A complete list of atomic parameters, bond distances, structure factors and selected least-squares planes has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43088 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The two rings are tipped at an angle of about 15° from the *ab* plane and are parallel to each other within 1·6°. They have an interplanar separation of 3·38 Å, which, projected on the *c* axis, is half the *c* translation of 6·99 Å. The N(1) nitro group is twisted out of the ring plane by 21° due to an intramolecular contact of 2·751 (3) Å between O(3) and O(5). The angles about S(1) between O(3) and C(10), C(20) are increased and those about S(1) between O(2) and C(20), C(10) are decreased. The longest dimension of all molecules lies nearly parallel or antiparallel to the *b* axis as shown in the stereo packing diagram included with the deposited material.

Two diastereomers of the dioxide I are possible, but only one was detected by ^{1}H and ^{13}C NMR spectra of the recrystallized material used for the reaction with the thiolate ion (II); presumably the other diastereomer was disfavored in the standard preparation used (Vilsmaier & Becker, 1975) and/or was removed in the recrystallization of the dioxide I. After the successive reactions of I with II and then of III with IV, as shown in Fig. 1, X-ray analysis showed the structure of the product to be that of V. The alternative attack of II at CH would give diastereomer VI; although a yellow compound seen in the purification of the deep orange V may have been VI, the amount was too small to handle. Attack on the dioxide VI thus *can* occur at the less hindered (CH_2) position rather than at the sulfur atom or the more hindered (CH) carbon. This mode of attack certainly seems likely to be preferred, but this conclusion is not unequivocal since V could be obtained in no more than 17% yield. Since (+)-camphor was converted to (+)-camphor-10-sulfonyl chloride by Eliel & Frazee (1979) and since the absolute configuration of (+)-camphor was determined by Allen & Rogers (1971), the absolute configuration of V could be determined from the relative configuration found *via* the X-ray studies and is that shown in Fig. 2.

Table 2. *Bond distances* (Å) *and angles* (°)

S(1)—O(2)	1·437 (2)	C(4)—C(5)	1·522 (5)
S(1)—O(3)	1·433 (2)	C(4)—C(7)	1·568 (4)
S(1)—C(10)	1·803 (3)	C(5)—C(6)	1·551 (4)
S(1)—C(20)	1·812 (2)	C(7)—C(8)	1·525 (6)
S(2)—C(11)	1·808 (3)	C(7)—C(9)	1·526 (6)
S(2)—C(30)	1·766 (2)	C(10)—C(11)	1·536 (4)
O(1)—C(2)	1·202 (4)	C(20)—C(21)	1·386 (4)
N(1)—O(4)	1·217 (3)	C(20)—C(25)	1·404 (3)
N(1)—O(5)	1·211 (3)	C(21)—C(22)	1·388 (4)
N(1)—C(25)	1·474 (3)	C(22)—C(23)	1·365 (4)
N(2)—O(6)	1·223 (4)	C(23)—C(24)	1·379 (4)
N(2)—O(7)	1·216 (4)	C(24)—C(25)	1·371 (4)
N(2)—C(23)	1·474 (4)	C(30)—C(31)	1·397 (4)
C(1)—C(2)	1·543 (4)	C(30)—C(35)	1·390 (4)
C(1)—C(6)	1·545 (4)	C(31)—C(32)	1·389 (4)
C(1)—C(7)	1·573 (4)	C(32)—C(33)	1·385 (4)
C(1)—C(10)	1·539 (3)	C(33)—C(34)	1·384 (4)
C(2)—C(3)	1·513 (4)	C(34)—C(35)	1·390 (4)
C(3)—C(4)	1·515 (6)	C(33)—C(40)	1·520 (4)
O(2)—S(1)—O(3)	118·90 (13)	C(1)—C(7)—C(4)	92·8 (2)
O(2)—S(1)—C(10)	106·90 (13)	C(1)—C(7)—C(8)	112·8 (3)
O(2)—S(1)—C(20)	104·47 (11)	C(1)—C(7)—C(9)	116·9 (3)
O(3)—S(1)—C(10)	111·26 (12)	C(4)—C(7)—C(8)	113·5 (3)
O(3)—S(1)—C(20)	108·16 (12)	C(4)—C(7)—C(9)	110·3 (3)
C(10)—S(1)—C(20)	106·30 (12)	C(8)—C(7)—C(9)	109·7 (3)
C(11)—S(2)—C(30)	104·36 (12)	S(1)—C(10)—C(1)	113·9 (2)
O(4)—N(1)—O(5)	124·0 (2)	S(1)—C(10)—C(11)	109·0 (2)
O(4)—N(1)—C(25)	117·5 (2)	C(1)—C(10)—C(11)	111·5 (2)
O(5)—N(1)—C(25)	118·5 (2)	S(2)—C(11)—C(10)	112·9 (2)
O(6)—N(2)—O(7)	123·8 (3)	S(1)—C(20)—C(21)	114·1 (2)
O(6)—N(2)—C(23)	117·9 (3)	S(1)—C(20)—C(25)	127·9 (2)
O(7)—N(2)—C(23)	118·3 (3)	C(21)—C(20)—C(25)	117·7 (2)
C(2)—C(1)—C(6)	104·7 (2)	C(20)—C(21)—C(22)	121·2 (3)
C(2)—C(1)—C(7)	98·6 (2)	N(2)—C(23)—C(22)	119·7 (3)
C(2)—C(1)—C(10)	107·3 (2)	N(2)—C(23)—C(24)	118·2 (3)
C(6)—C(1)—C(7)	102·1 (2)	C(22)—C(23)—C(24)	122·1 (3)
C(6)—C(1)—C(10)	117·0 (2)	C(23)—C(24)—C(25)	118·5 (3)
C(7)—C(1)—C(10)	124·3 (2)	N(1)—C(25)—C(20)	123·4 (2)
O(1)—C(2)—C(1)	126·2 (2)	N(1)—C(25)—C(24)	115·1 (2)
O(1)—C(2)—C(3)	127·0 (3)	C(20)—C(25)—C(24)	121·5 (2)
C(1)—C(2)—C(3)	106·7 (3)	S(2)—C(30)—C(31)	116·6 (2)
C(2)—C(3)—C(4)	102·0 (3)	S(2)—C(30)—C(35)	124·6 (2)
C(3)—C(4)—C(5)	106·8 (3)	C(31)—C(30)—C(35)	118·9 (2)
C(3)—C(4)—C(7)	103·8 (3)	C(30)—C(31)—C(32)	120·0 (3)
C(5)—C(4)—C(7)	102·9 (3)	C(31)—C(32)—C(33)	121·8 (3)
C(4)—C(5)—C(6)	102·6 (3)	C(32)—C(33)—C(34)	117·2 (3)
C(1)—C(6)—C(5)	104·6 (3)	C(32)—C(33)—C(40)	121·7 (3)
		C(34)—C(33)—C(40)	121·1 (3)
		C(33)—C(34)—C(35)	122·4 (3)
		C(30)—C(35)—C(34)	119·6 (3)

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