Discussion. The final positional parameters are listed in Table 1,* and bond lengths and bond angles are given in Table 2. Fig. 1 shows the structure of the molecule with the numbering scheme. The geometry of the molecule was calculated using *FFE*3 (Busing, Martin & Levy, 1971).

The thirteen-membered ring is in a twist conformation. The ring is approximately symmetrical with respect to a pseudo-twofold axis running through O8 and the middle of the C3—C4 bond; the asymmetry coefficient $\Delta_2 = 0.35°$, $\Delta_2 = [\Sum(\varphi_i - \varphi_i')^2/n(n-1)]^{1/2}$,

where φ_i and φ_i' are torsional angles of approximately symmetrical atoms.

The fused ring planes form a dihedral angle of 46.9 (1)°, while the tosyl substituent planes (excluding the O atoms) form a dihedral angle of 11.2 (1)°. There are van der Waals molecular contacts only.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44075 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structure of 2,2'-Bis(tosylmethylphenyl) Ether

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Abstract. $C_{28}H_{26}O_5S_2$, $M_r = 506.64$, triclinic, $P\bar{1}$, $a = 9.243$ (3), $b = 10.439$ (3), $c = 13.646$ (3) Å, $\alpha = 92.79$ (4), $\beta = 99.36$ (4), $\gamma = 101.39$ (4)°, $V = 1269.1$ (7) Å^3, $Z = 2$, $D_x = 1.326$ g cm^{-3}, $\mu = 20.87$ cm^{-1}. Diffractometer data collected at room temperature [λ(Cu $K\alpha$) = 1.54178 Å] gave 4386 independent reflections with $I > 3\sigma(I)$, $F(000) = 532$, final $R = 0.0570$. The molecule is not a symmetrical one; the angles formed by the four six-membered planar rings are different.

Introduction. Condensation of 2,2'-bis(bromoethylphenyl) ether with *N,N'*-ditosylhydrazine produces *N,N'*-ditosyltetrahydrodibenzo[*b,h*][1,5,6]oxadiazonine. The structure of this product has been established by elementary and spectral (IR, NMR, MS) analyses. Its melting point is 416–417 K. The compound was found to be unstable. During its de-

composition with the evolution of nitrogen a product characterized by a melting point of 406–408 K was obtained. On the basis of elemental analysis which excluded the presence of nitrogen, IR and NMR studies, identification as 2,2'-bis(tosylmethylphenyl) ether was proposed (Glinka, 1981).

Experimental. Yellowish crystals grown from methanol at room temperature, $\mu r = 0.21$, ceil parameters and intensity data measured on a CAD-4 diffractometer using θ–2θ scan technique; lattice parameters refined by least-squares method using 25 reflections with $\theta_{max} = 50.8°$; total of 4390 independent reflections measured to (sin θ)/$\lambda = 0.63$ Å^{-1}, values of h,k,l were -11 to 11, -12 to 12, 0 to 15 respectively, data not corrected for absorption, standard reflection $\bar{2}\bar{2}2$, mean variation 0.6%; solution by direct method using *SHELX*76 (Sheldrick, 1976), all non-H atoms found on

Table 1. *Final fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\times 10^4$) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
C31	−44 (2)	2138 (2)	9650 (1)	387 (8)
C36	−1030 (2)	2784 (2)	9079 (1)	417 (8)
C41	−1859 (2)	3718 (2)	6689 (1)	400 (8)
C42	−2321 (2)	2950 (2)	7428 (1)	437 (9)
O5	−1208 (2)	2512 (2)	8063 (1)	536 (8)
C32	201 (2)	2370 (2)	10683 (1)	483 (10)
C33	−498 (3)	3232 (2)	11140 (2)	596 (12)
C34	−1441 (3)	3886 (2)	10571 (2)	588 (12)
C35	−1713 (2)	3661 (2)	9536 (2)	541 (11)
C43	−3813 (2)	2563 (2)	7503 (2)	560 (11)
C44	−4885 (2)	2949 (3)	6814 (2)	586 (12)
C45	−4458 (3)	3716 (3)	6072 (2)	574 (12)
C46	−2970 (2)	4093 (2)	6008 (2)	471 (9)
C1	732 (2)	1225 (2)	9149 (1)	426 (9)
C11	3681 (2)	2325 (2)	10122 (1)	434 (9)
C12	4038 (2)	3599 (2)	10560 (2)	522 (11)
C13	4938 (3)	3865 (3)	11496 (2)	630 (14)
C14	5456 (3)	2879 (3)	11985 (2)	652 (14)
C15	5067 (4)	1595 (4)	11532 (2)	830 (18)
C16	4182 (3)	1325 (3)	10605 (2)	648 (13)
C2	−240 (2)	4053 (2)	6581 (1)	407 (9)
C21	−537 (2)	1433 (2)	5832 (1)	406 (8)
C22	407 (2)	742 (2)	6363 (2)	463 (9)
C23	−175 (3)	−513 (2)	6591 (2)	519 (11)
C24	−1679 (3)	−1077 (2)	6302 (2)	498 (10)
C25	−2602 (3)	−373 (2)	5745 (2)	556 (12)
C26	−2046 (2)	882 (2)	5506 (2)	508 (11)
C141	6462 (5)	3201 (7)	12989 (3)	1061 (31)
C241	−2307 (4)	−2441 (3)	6563 (2)	676 (15)
S1	2552 (1)	1974 (0)	8931 (0)	422 (2)
O11	3081 (2)	982 (2)	8399 (1)	596 (9)
O12	2471 (2)	3189 (2)	8490 (1)	546 (8)
S2	169 (0)	3065 (0)	5595 (0)	386 (2)
O21	−683 (2)	3317 (1)	4668 (1)	500 (8)
O22	1777 (2)	3293 (1)	5690 (1)	515 (7)

E map, refinement by full-matrix least squares (*F* magnitudes, 424 parameters), after initial anisotropic refinement *F* map revealed positions of all H atoms at $R = 0.102$, final $R = 0.0570$ for 4386 reflections with $I > 3\sigma(I)$, $wR = 0.0676$ where $w = 1.0/[\sigma^2(F) + 0.010199F^2]$, max. shift/e.s.d. $= 0.97$, largest peak on final difference map was 0.3 e Å^{-3}. Atomic scattering factors those of *SHELX*.

Discussion. The final positional parameters are listed in Table 1, bond lengths and bond angles in Table 2.*

The molecules of the title compound are in general positions (the numbering of the atoms is shown in Fig. 1). The benzene rings are planar within the limits of error, but not coplanar. The angle between these rings is 64.5 (3)°. The maximum distance of the benzene C atoms from the best planes is 0.010 (4) Å. The C1 and C2 atoms lie in benzene-ring planes [max. distances are 0.016 (4) and 0.036 (5)Å respectively]. The C—O

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distances (1.379 and 1.398 Å) and C—O—C angle (119.9°) are typical.

The angles between the benzene rings and the phenyl rings of the tosyl groups are 33.8 (3) and 40.9 (3)°. The distances between the centres of the tosyl rings are 3.84 and 4.15 Å. Hence, it seems, they can take part in intermolecular charge-transfer interaction.

The structure obtained confirms that proposed for the compound by Glinka.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

O5—C36	1·379 (2)	O5—C42	1·398 (2)
C31—C36	1·398 (3)	C41—C42	1·388 (3)
C31—C32	1·391 (3)	C41—C46	1·398 (3)
C32—C33	1·386 (4)	C45—C46	1·372 (3)
C33—C34	1·376 (4)	C44—C45	1·380 (4)
C34—C35	1·393 (3)	C43—C44	1·386 (3)
C35—C36	1·388 (3)	C42—C43	1·380 (3)
C1—C31	1·502 (3)	C2—C41	1·501 (3)
S1—C1	1·788 (2)	S2—C2	1·788 (2)
S1—O11	1·442 (2)	S2—O21	1·442 (1)
S1—O12	1·440 (2)	S2—O22	1·442 (1)
S1—C11	1·762 (2)	S2—C21	1·766 (2)
C11—C12	1·385 (3)	C21—C22	1·379 (3)
C12—C13	1·389 (3)	C22—C23	1·385 (3)
C13—C14	1·375 (4)	C23—C24	1·382 (3)
C14—C15	1·402 (5)	C24—C25	1·392 (3)
C15—C16	1·372 (4)	C25—C26	1·385 (3)
C11—C16	1·378 (3)	C21—C26	1·389 (3)
C14—C141	1·507 (5)	C24—C241	1·511 (3)
O5—C36—C31	115·1 (2)	O5—C42—C41	116·7 (2)
O5—C36—C35	124·5 (2)	O5—C42—C43	120·9 (2)
C31—C36—C35	120·4 (2)	C41—C42—C43	122·2 (2)
C34—C35—C36	120·1 (2)	C42—C43—C44	118·8 (2)
C33—C34—C35	119·8 (2)	C43—C44—C45	120·2 (2)
C32—C33—C34	120·1 (2)	C44—C45—C46	120·2 (2)
C31—C32—C33	121·2 (2)	C41—C46—C45	121·1 (2)
C32—C31—C36	118·4 (2)	C42—C41—C46	117·4 (2)
C1—C31—C36	120·2 (2)	C2—C41—C42	121·2 (2)
C1—C31—C32	121·4 (2)	C2—C41—C46	121·2 (2)
C31—C1—S1	114·3 (1)	C41—C2—S2	112·6 (1)
C11—S1—C1	105·2 (1)	C21—S2—C2	104·9 (1)
C11—S1—O11	107·9 (1)	C21—S2—O21	107·8 (1)
C11—S1—O12	108·7 (1)	C21—S2—O22	109·7 (1)
C1—S1—O11	106·5 (1)	C2—S2—O21	108·4 (1)
C1—S1—O12	109·0 (1)	C2—S2—O22	107·3 (1)
O11—S1—O12	118·7 (1)	O21—S2—O22	118·0 (1)
S1—C11—C12	119·5 (2)	S2—C21—C22	119·4 (1)
S1—C11—C16	119·4 (2)	S2—C21—C26	119·3 (2)
C12—C11—C16	121·1 (2)	C22—C21—C26	121·2 (2)
C11—C12—C13	118·9 (2)	C21—C22—C23	119·0 (2)
C12—C13—C14	120·6 (2)	C22—C23—C24	121·3 (2)
C13—C14—C15	119·5 (2)	C23—C24—C25	118·7 (2)
C14—C15—C16	120·3 (3)	C24—C25—C26	121·1 (2)
C11—C16—C15	119·6 (3)	C21—C26—C25	118·7 (2)
C141—C14—C13	119·5 (4)	C241—C24—C23	120·9 (2)
C141—C14—C15	121·0 (4)	C242—C24—C25	120·4 (2)
C36—O5—C42	119·9 (2)		
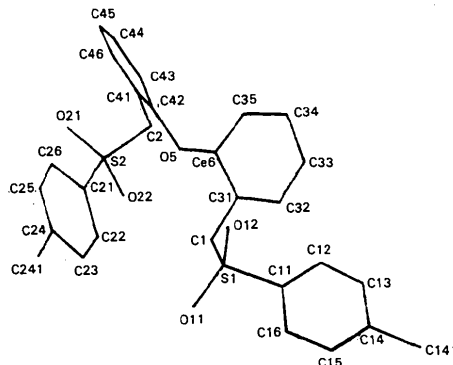
Fig. 1. Structure of the molecule with the atom-numbering scheme.

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Structures of Two *N*-Boc Amino-Acid Phenacyl Esters

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Abstract. (I): *tert*-Butoxycarbonyl-L-phenylalanine phenacyl ester, $C_{22}H_{25}NO_5$, $M_r = 383.4$, orthorhombic, $P2_12_12_1$, $a = 9.277$ (3), $b = 14.379$ (3), $c = 15.874$ (3) Å, $V = 2117.5$ (5) Å^3, $Z = 4$, $D_x = 1.203$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.80$ cm^{-1}, $F(000) = 816$, room temperature, final $R = 0.033$ for 893 observed reflections. (II): *tert*-Butoxycarbonyl-glycine phenacyl ester, $C_{15}H_{19}NO_5$, $M_r = 293.3$, monoclinic, $P2_1/n$, $a = 5.105$ (2), $b = 26.593$ (3), $c = 11.406$ (2) Å, $\beta = 90.91$ (3)°, $V = 1548.3$ (5) Å^3, $Z = 4$, $D_x = 1.259$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.90$ cm^{-1}, $F(000) = 624$, room temperature, final $R = 0.0438$ for 1016 observed reflections. The values of bond lengths and angles of the Boc group and the urethane moiety, as well as the distances of the phenacyl and ester CO are normal. The urethane amide bonds [O(5)–C(6)–N(8)–C(9) = 174.4 (2)° in (I); −178.4 (2)° in (II)] have the *trans* conformation in both compounds. In (I) and (II) the molecules are interconnected by intermolecular hydrogen bonds of different types [O(22)···H(8) = 2.207(75)Å in (I); O(7)···H(8) = 2.114 (65) Å in (II)]. As a result of the *trans* conformation adopted by the amide bond of the urethane moiety in both molecules, O(5) and H(8) are forced to have short non-bonded contacts [O(5)–H(8) = 2.308 (77) Å in (I); 2.250 (64) Å in (II)].

Introduction. We recently reported a simple method for isolating free amino acids as their *N*-Boc (*N-tert*-butoxycarbonyl) amino-acid benzyl esters from

marine organisms (Matsoukas, Moharir & Findlay, 1983; Findlay, Daljeet, Matsoukas & Moharir, 1984). One limitation of this method is the oily nature of the non-polar amino-acid derivatives. In order to improve this method we have synthesized the phenacyl esters (Stelakatos, Panagou & Zervas, 1966) of several *N*-Boc amino acids which crystallize easily. This procedure was applied to isolate major free amino acids from the Mediterranean fruit fly *Ceratitis capitata*. The crystal structure determinations of the title compounds were undertaken to establish the conformation adopted by peptide molecules. Furthermore, we were interested in the position and the role of the phenacyl CO, in the *cis–trans* isomerism around the urethane —O(O=)C—NH— bond, and in information concerning possibly important non-bonded interactions.

Experimental. (I). Pure *N*-Boc-L-phenylalanine was prepared by the method of Nagasawa, Kuroiwa, Narita & Isowa (1973). Esterification was achieved, without racemization, with phenacyl bromide–triethylamine (Stelakatos, Panagou & Zervas, 1966). Recrystallization from ethyl acetate–petroleum ether, m.p. = 398 K. Prismatic crystal $0.22 \times 0.28 \times 0.72$ mm with prominent axis [$\bar{1}00$]. Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, lattice parameters determined from 50 reflections ($8 < \theta < 12°$), $\omega/2\theta$ scan technique up to $2\theta = 54°$, scan width $(0.8 + 0.3\tan\theta)°$, scan rate 1.27–$16.48°$ min^{-1}, background 1/4 of the scan time at each scan limit, max. scan time 60 s, aperture $(2.4 + 0.9\tan\theta)$ mm. 5198 measured reflections, 2628