Final values of the extinction coefficient, residual densities in final ΔF maps, R factors and other details of the refinements are given in Table 1.

Atomic parameters for non-H atoms are given in Table 1, the molecular structures are shown in Figs. 1 and 2, and selected bond distances, bond angles and torsion angles are given in Table 3.*

Related literature. Compound (I) is isomorphous with the organometallic species $[(Me_3Si)_3C]_2M$ where M = Hg (Glockling, Hosmane, Mahale, Magos & King, 1977), Mn (Buttrus, Eaborn, Hitchcock, Smith & Sullivan, 1985) and Mg (Al-Juaid, Eaborn, Hitchcock, McGeary & Smith, 1989), and is similar in structure to the anions in $[Li(thf)_4][\{(Me_3Si)_3C\}_2M]$ where M = Li (Eaborn, Hitchcock, Smith & Sullivan, 1983), Ag (Eaborn, Hitchcock, Smith & Sullivan, 1984*a*) or Cu (Eaborn, Hitchcock, Smith & Sullivan, 1984*b*). Compound (II) is similar in geometry to $^tBu_3SiSi^tBu_3$ (Wiberg, Schuster, Simon & Peters, 1986) which, however, has a significantly longer central Si—Si bond of 2.696 Å. The related compounds $[(Me_3Si)_3Si]_2M$ (M = Hg, Cd and Zn) have been prepared and the structure of the Zn compound determined (Arnold, Tilley, Rheingold & Geib, 1987).

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* Lists of H-atom parameters, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55265 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HE0040]

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Structures of Benzo[*a*]phenothiazine and Benzo[*c*]phenothiazine

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Abstract. Benzo[*a*]phenothiazine (I), $C_{16}H_{11}NS$, $M_r = 249.3$, monoclinic, $P2_1/a$, $a = 25.269\,(2)$, $b = 23.347\,(2)$, $c = 8.306\,(2)$ Å, $\beta = 91.71\,(1)°$, $V = 4898\,(1)$ Å^3, $Z = 16$, $D_m = 1.35$, $D_x = 1.35$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.23$ mm^{-1}, $F(000) = 2080$, $T = 293$ K, $R = 0.069$ for 4248 observed reflections. Benzo[*c*]phenothiazine (II), $C_{16}H_{11}NS$, $M_r = 249.3$, monoclinic, $P2_1/a$, $a = 19.703\,(3)$, $b = 5.365\,(2)$, $c = 11.523\,(1)$ Å, $\beta = 95.48\,(1)°$, $V = 1212.5\,(4)$ Å^3, $Z = 4$, $D_m = 1.39$, $D_x = 1.37$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.23$ mm^{-1}, $F(000) = 520$, $T = 293$ K, $R = 0.047$ for 2685 observed reflections. Four crystallographically independent molecules of (I) are essentially identical in bond distances and angles. The molecules of both (I) and (II) are bent at the N–S axis, and dihedral angles

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between benzene and naphthalene planes are 151.5–165.4° for (I), and 141.6° for (II).

Experimental. The materials were synthesized by the method of Knoevenagel (1914). D_m was measured by flotation in an aqueous solution of K_2HgI_4. Data were collected on a Rigaku AFC-5 four-circle diffractometer, using $\theta/2\theta$ scans with θ-scan rate 4° $\min^{-1}$, to $2\theta_{\max} = 60°$. Standard reflections were monitored every 100 measurements. Corrections were made for Lorentz and polarization effects, but not for absorption. Structures were solved by direct methods using *MULTAN*84 (Main, Germain & Woolfson, 1984), and refined by block-diagonal least-squares methods. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$. Initial positions of H atoms were calculated. Atomic coordinates for all atoms and anisotropic temperature factors for non-H atoms were refined. Isotropic temperature factors for H atoms were fixed at $B_{\rm iso} = 4.0$ Å^2. Scattering factors including anomalous-dispersion factors for the S atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were carried out using a Panafacom U-1200II computer with the Rigaku *RASA*-5P program package and a HITAC M-680H at the Computer Centre of the University of Tokyo with the *UNICS* (Sakurai, 1967) program system.

For (I), yellow transparent plates were obtained from 1,2-dichloroethane. The crystal used had dimensions 0.4 × 0.3 × 0.2 mm. Lattice parameters were determined from 20 reflections ($22.4 < 2\theta < 30.8°$). Scan width was $(1.0 + 0.5\tan\theta)°$ in θ. Range of *hkl* 0 to 35, 0 to 32, −11 to 11. Three standard reflections (14,3,7, 7,11,10 and 864) showed intensity variations in F of 3%. 14 927 reflections were measured, of which 13 328 were unique ($R_{\rm int} = 0.020$) and 4248 were observed with $F_o > 3\sigma(F)$. $R = 0.069$, $wR = 0.038$ [$w = 1/\sigma^2(F)$] and $S = 2.51$ for 781 variables; $(\Delta/\sigma)_{\max} = 0.257$ for non-H atoms; $\Delta\rho_{\min} = -0.30$, $\Delta\rho_{\max} = 0.40$ e Å^{-3} in the final difference synthesis.

For (II), yellow transparent plates were obtained from acetonitrile. The crystal used had dimensions 0.5 × 0.5 × 0.5 mm. Lattice parameters were determined from 20 reflections ($27.3 < 2\theta < 31.2°$). Scan width was $(1.0 + 0.35\tan\theta)°$ in θ. Range of *hkl* −27 to 27, 0 to 7, 0 to 16. Three standard reflections ($\bar{2}0\bar{8}$, $\overline{14}$,0,3 and $\overline{11}$,$\bar{1}$,5) showed intensity variations in F of 1.3%. 4084 reflections were measured, of which 3924 were unique ($R_{\rm int} = 0.019$) and 2685 were observed with $F_o > 3\sigma(F)$. $R = 0.047$, $wR = 0.049$ ($w = 1$) and $S = 0.74$ for 196 variables; $(\Delta/\sigma)_{\max} = 0.094$ for non-H atoms; $\Delta\rho_{\min} = -0.20$, $\Delta\rho_{\max} = 0.30$ e Å^{-3} in the final difference synthesis.

The final atomic positional parameters and equivalent isotropic thermal parameters of (I) and

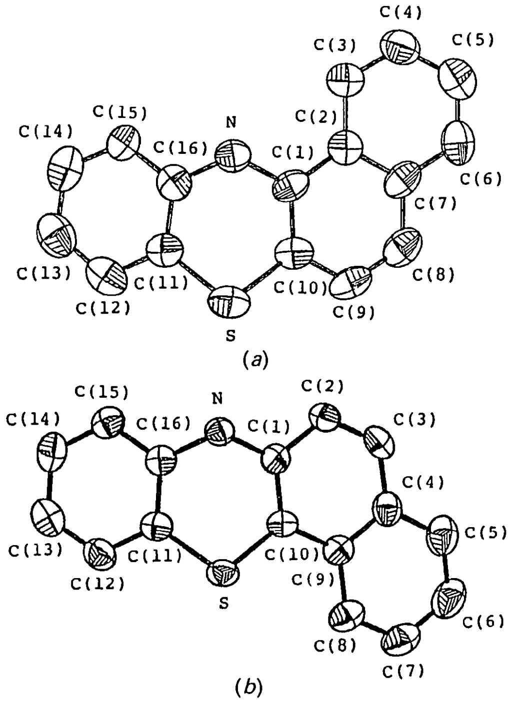
Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *for non*-H *atoms with e.s.d.'s in parentheses*

$$B_{\rm eq} = (4/3)\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
(I*A*)				
S	0.7171 (1)	0.2328 (1)	0.5654 (2)	5.59 (5)
N	0.8231 (2)	0.2192 (2)	0.7380 (4)	4.7 (1)
C(1)	0.8070 (2)	0.1712 (2)	0.6442 (5)	3.9 (3)
C(2)	0.8384 (2)	0.1200 (2)	0.6468 (5)	3.8 (2)
C(3)	0.8860 (2)	0.1146 (2)	0.7407 (5)	4.6 (2)
C(4)	0.9124 (2)	0.0634 (2)	0.7465 (6)	5.4 (2)
C(5)	0.8931 (2)	0.0153 (2)	0.6608 (6)	6.1 (2)
C(6)	0.8480 (2)	0.0197 (2)	0.5678 (6)	5.6 (2)
C(7)	0.8195 (2)	0.0718 (2)	0.5581 (5)	4.5 (2)
C(8)	0.7724 (2)	0.0770 (2)	0.4637 (6)	5.3 (2)
C(9)	0.7439 (2)	0.1258 (2)	0.4616 (5)	5.0 (2)
C(10)	0.7598 (2)	0.1728 (2)	0.5554 (5)	4.2 (2)
C(11)	0.7641 (2)	0.2883 (2)	0.6019 (5)	4.2 (2)
C(12)	0.7525 (2)	0.3438 (2)	0.5527 (5)	5.1 (2)
C(13)	0.7882 (2)	0.3873 (2)	0.5875 (6)	5.9 (2)
C(14)	0.8356 (2)	0.3745 (2)	0.6680 (6)	5.9 (2)
C(15)	0.8476 (2)	0.3194 (2)	0.7125 (6)	5.1 (2)
C(16)	0.8115 (2)	0.2752 (2)	0.6844 (5)	4.2 (2)
(I*B*)				
S	0.8622 (1)	0.1897 (1)	1.2160 (2)	7.72 (6)
N	0.9547 (2)	0.2313 (2)	1.0199 (5)	6.2 (2)
C(1)	0.9241 (2)	0.2741 (2)	1.0872 (5)	4.6 (2)
C(2)	0.9349 (2)	0.3335 (2)	1.0509 (5)	4.2 (2)
C(3)	0.9772 (2)	0.3507 (2)	0.9562 (5)	4.5 (2)
C(4)	0.9838 (2)	0.4073 (2)	0.9162 (5)	5.2 (2)
C(5)	0.9485 (2)	0.4495 (2)	0.9707 (6)	5.8 (2)
C(6)	0.9086 (2)	0.4340 (2)	1.0671 (6)	5.6 (2)
C(7)	0.9000 (2)	0.3767 (2)	1.1099 (5)	4.9 (2)
C(8)	0.8586 (2)	0.3598 (3)	1.2099 (6)	6.0 (3)
C(9)	0.8497 (2)	0.3052 (3)	1.2437 (6)	6.1 (2)
C(10)	0.8822 (2)	0.2610 (2)	1.1824 (5)	5.2 (2)
C(11)	0.9214 (2)	0.1507 (2)	1.1776 (5)	5.6 (2)
C(12)	0.9279 (2)	0.0958 (3)	1.2330 (6)	7.4 (2)
C(13)	0.9713 (2)	0.0628 (2)	1.1929 (7)	7.7 (2)
C(14)	1.0097 (2)	0.0877 (2)	1.0987 (6)	6.5 (2)
C(15)	1.0044 (2)	0.1434 (2)	1.0447 (6)	5.4 (2)
C(16)	0.9607 (2)	0.1758 (2)	1.0830 (5)	5.1 (2)
(I*C*)				
S	0.9738 (1)	0.2369 (1)	0.5823 (2)	5.86 (5)
N	1.0795 (1)	0.2746 (2)	0.7253 (4)	4.6 (1)
C(1)	1.0538 (2)	0.3158 (2)	0.6265 (5)	3.9 (2)
C(2)	1.0773 (2)	0.3710 (2)	0.6048 (5)	3.9 (2)
C(3)	1.1265 (2)	0.3875 (2)	0.6778 (5)	4.6 (2)
C(4)	1.1460 (2)	0.4417 (2)	0.6558 (6)	5.9 (2)
C(5)	1.1183 (2)	0.4817 (2)	0.5609 (6)	6.4 (2)
C(6)	1.0713 (2)	0.4668 (2)	0.4891 (6)	5.9 (2)
C(7)	1.0489 (2)	0.4124 (2)	0.5100 (5)	4.8 (2)
C(8)	0.9995 (2)	0.3970 (2)	0.4361 (5)	5.3 (2)
C(9)	0.9784 (2)	0.3450 (2)	0.4577 (5)	4.9 (2)
C(10)	1.0050 (2)	0.3040 (2)	0.5520 (5)	4.2 (2)
C(11)	1.0298 (2)	0.1922 (2)	0.6194 (5)	4.4 (2)
C(12)	1.0269 (2)	0.1342 (2)	0.5855 (6)	5.5 (2)
C(13)	1.0681 (2)	0.0980 (2)	0.6299 (6)	6.3 (2)
C(14)	1.1128 (2)	0.1210 (2)	0.7060 (6)	6.0 (2)
C(15)	1.1168 (2)	0.1791 (2)	0.7352 (5)	4.8 (2)
C(16)	1.0755 (2)	0.2156 (2)	0.6927 (5)	4.2 (2)
(I*D*)				
S	1.1223 (1)	0.4061 (1)	1.1072 (2)	6.79 (6)
N	1.2153 (2)	0.3351 (2)	0.9977 (4)	5.4 (1)
C(1)	1.1765 (2)	0.3045 (2)	1.0782 (5)	4.5 (2)
C(2)	1.1820 (2)	0.2437 (2)	1.0991 (5)	4.6 (2)
C(3)	1.2251 (2)	0.2120 (2)	1.0429 (6)	5.1 (2)
C(4)	1.2272 (2)	0.1543 (2)	1.0634 (6)	6.1 (2)
C(5)	1.1879 (2)	0.1238 (2)	1.1422 (6)	6.8 (2)
C(6)	1.1456 (2)	0.1535 (2)	1.2000 (6)	6.2 (2)
C(7)	1.1406 (2)	0.2133 (2)	1.1790 (5)	5.1 (2)
C(8)	1.0964 (2)	0.2451 (2)	1.2348 (6)	5.9 (2)
C(9)	1.0932 (2)	0.3016 (2)	1.2155 (6)	5.8 (2)
C(10)	1.1326 (2)	0.3324 (2)	1.1352 (5)	4.9 (2)
C(11)	1.1881 (2)	0.4307 (2)	1.0871 (5)	5.1 (2)
C(12)	1.2009 (2)	0.4865 (2)	1.1191 (6)	6.0 (2)
C(13)	1.2508 (2)	0.5071 (2)	1.0919 (6)	6.9 (2)
C(14)	1.2889 (2)	0.4710 (2)	1.0355 (6)	6.6 (2)
C(15)	1.2774 (2)	0.4137 (2)	1.0061 (6)	5.7 (2)
C(16)	1.2267 (2)	0.3923 (2)	1.0314 (5)	4.8 (2)

Table 1 (*cont.*)

(II)				
S	0.70359 (3)	0.0703 (1)	0.7625 (1)	3.33 (1)
N	0.7369 (1)	0.5088 (4)	0.9159 (2)	2.88 (4)
C(1)	0.7823 (1)	0.4720 (4)	0.8291 (2)	2.65 (5)
C(2)	0.8392 (1)	0.6320 (5)	0.8262 (2)	3.36 (6)
C(3)	0.8834 (1)	0.5994 (5)	0.7429 (2)	3.70 (6)
C(4)	0.8731 (1)	0.4096 (5)	0.6577 (2)	3.30 (6)
C(5)	0.9175 (1)	0.3769 (6)	0.5690 (2)	4.52 (8)
C(6)	0.9069 (2)	0.1919 (7)	0.4884 (2)	4.86 (8)
C(7)	0.8510 (2)	0.0313 (6)	0.4910 (2)	4.56 (8)
C(8)	0.8063 (1)	0.0564 (5)	0.5744 (2)	3.73 (6)
C(9)	0.8162 (1)	0.2472 (4)	0.6600 (2)	2.86 (5)
C(10)	0.7718 (1)	0.2808 (4)	0.7497 (2)	2.66 (5)
C(11)	0.6437 (1)	0.2825 (4)	0.8106 (2)	2.93 (5)
C(12)	0.5741 (1)	0.2504 (5)	0.7789 (2)	3.86 (6)
C(13)	0.5273 (1)	0.4070 (6)	0.8228 (3)	4.43 (7)
C(14)	0.5495 (1)	0.6021 (6)	0.8958 (2)	4.15 (7)
C(15)	0.6187 (1)	0.6392 (5)	0.9264 (2)	3.38 (6)
C(16)	0.6661 (1)	0.4788 (4)	0.8842 (2)	2.73 (5)



(*b*)

Fig. 1. *ORTEP*II (Johnson, 1976) view of the molecules showing the atomic numbering of (*a*) (I*A*), (I*B*), (I*C*) and (I*D*), and (*b*) (II). Thermal ellipsoids are drawn at the 50% probability level.

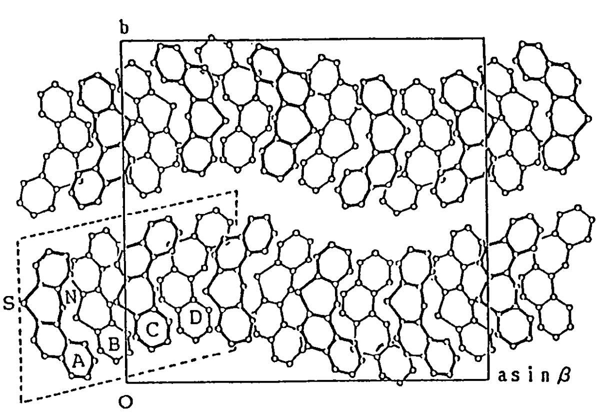


Fig. 2. Projection of the crystal structure of (I) along the *c* axis.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses* [*mean values are given for* (I)]

(I)			
S—C(10)	1.767 (3)	C(8)—C(9)	1.337 (3)
S—C(11)	1.778 (2)	C(9)—C(10)	1.406 (4)
N - C(1)	1.405 (4)	C(11) C(12)	1.378 (3)
N—C(16)	1.404 (2)	C(12)—C(13)	1.382 (2)
C(1)—C(2)	1.438 (2)	C(13)—C(14)	1.384 (3)
C(2)—C(3)	1.412 (3)	C(14)—C(15)	1.380 (3)
C(3)—C(4)	1.368 (2)	C(15)—C(16)	1.389 (2)
C(4)—C(5)	1.405 (2)	C(1)—C(10)	1.383 (2)
C(5)—C(6)	1.362 (3)	C(2)—C(7)	1.431 (3)
C(6)—C(7)	1.409 (2)	C(11)—C(16)	1.405 (3)
C(7)—C(8)	1.418 (3)		
C(10)—S—C(11)	100.9 (2)	C(2)—C(7)—C(6)	118.9 (1)
C(1)—N—C(16)	122.3 (6)	C(1)—C(2)—C(7)	118.34 (7)
N—C(1)—C(10)	120.5 (2)	C(2)—C(7)—C(8)	118.9 (1)
N—C(16)—C(11)	121.7 (2)	C(7)—C(8)—C(9)	121.44 (7)
S—C(10)—C(1)	121.4 (3)	C(8)—C(9)—C(10)	121.0 (1)
S—C(11)—C(16)	119.2 (1)	C(1)—C(10)—C(9)	120.6 (2)
C(3)—C(2)—C(7)	118.4 (1)	C(2)—C(1)—C(10)	119.6 (1)
C(2)—C(3)—C(4)	120.42 (7)	C(16)—C(11)—C(12)	120.5 (2)
C(3)—C(4)—C(5)	121.4 (2)	C(11)—C(12)—C(13)	120.6 (3)
C(4)—C(5)—C(6)	119.4 (2)	C(12)—C(13)—C(14)	119.1 (2)
C(5)—C(6)—C(7)	121.6 (2)	C(13)—C(14)—C(15)	120.80 (9)
C(14)—C(15)—C(16)	120.63 (6)	C(11)—C(16)—C(15)	118.2 (1)
(II)			
S—C(10)	1.772 (2)	C(8)—C(9)	1.422 (4)
S—C(11)	1.767 (2)	C(9)—C(10)	1.428 (3)
N—C(1)	1.418 (3)	C(11)—C(12)	1.397 (4)
N—C(16)	1.417 (3)	C(12)—C(13)	1.378 (4)
C(1)—C(2)	1.415 (3)	C(13)—C(14)	1.387 (4)
C(2)—C(3)	1.367 (4)	C(14)—C(15)	1.389 (4)
C(3)—C(4)	1.416 (4)	C(15)—C(16)	1.391 (3)
C(4)—C(5)	1.419 (4)	C(1)—C(10)	1.377 (3)
C(5)—C(6)	1.362 (5)	C(4)—C(9)	1.422 (4)
C(6)—C(7)	1.402 (5)	C(11)—C(16)	1.397 (3)
C(7)—C(8)	1.371 (4)		
C(10)—S—C(11)	98.6 (1)	C(5)—C(6)—C(7)	120.2 (3)
C(1)—N—C(16)	118.3 (2)	C(6)—C(7)—C(8)	120.9 (3)
N—C(1)—C(10)	120.5 (2)	C(7)—C(8)—C(9)	120.2 (3)
N—C(16)—C(11)	119.3 (2)	C(4)—C(9)—C(8)	119.0 (2)
S—C(10)—C(1)	119.2 (2)	C(4)—C(9)—C(10)	118.3 (2)
S—C(11)—C(16)	119.6 (2)	C(1)—C(10)—C(9)	120.8 (2)
C(2)—C(1)—C(10)	120.4 (2)	C(12)—C(11)—C(16)	119.9 (2)
C(1)—C(2)—C(3)	119.9 (2)	C(11)—C(12)—C(13)	120.2 (3)
C(2)—C(3)—C(4)	121.3 (3)	C(12)—C(13)—C(14)	120.0 (3)
C(3)—C(4)—C(9)	119.3 (2)	C(13)—C(14)—C(15)	120.5 (3)
C(5)—C(4)—C(9)	118.7 (3)	C(14)—C(15)—C(16)	119.9 (2)
C(4)—C(5)—C(6)	121.1 (3)	C(11)—C(16)—C(15)	119.6 (2)

(II) for non-H atoms are given in Table 1.* Fig. 1 shows *ORTEP*II (Johnson, 1976) drawings of the molecules with the atomic numbering scheme. Though an asymmetric unit of (I) contains four molecules, denoted by *A*, *B*, *C* and *D*, all the corresponding bond distances and angles of the four independent molecules agree within experimental e.s.d.'s. Bond lengths and angles of (I) (mean values) and (II) are given in Table 2. Dihedral angles between planar benzene and naphthalene rings are 151.5 (1) for (I*A*), 165.4 (2) for (I*B*), 152.1 (1) for (I*C*) and 154.7 (2)° for (I*D*), and 141.61 (8)° for (II). The arrangement of (I) in the unit cell projected down the *c* axis is shown in Fig. 2.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and full lists of bond lengths and angles of (I) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55559 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0593]

Related literature. Phenothiazine (PT) and its derivatives easily form solid charge-transfer (CT) complexes, in which they are present as stable cation radicals (Uchida, Ito & Kozawa, 1983; Someno, Hoshizaki, Kozawa, Uchida, Hayashi, Sugano & Kinoshita 1991). This work has been performed to compare the geometries of PT derivatives in their neutral and cation-radical states. For PT, the tilt structure of the thiazine ring in the neutral molecule (Bell, Blount, Briscoe & Freeman, 1968; Fritchie & Trus, 1968; McDowell 1976) changes to a planar structure in its cation-radical state (Kozawa & Uchida, 1990).

It has already been reported that, in CT complexes, the present chemical entities, (I) and (II), exist as cation radicals (Kozawa, Hoshizaki & Uchida, 1991), and that the PT moieties in them are almost planar (Kozawa, Hoshizaki & Uchida, 1991; Kozawa & Uchida, 1993). This work elucidates that both neutral (I) and (II) molecules possess tilt structures, similar to the neutral PT, and that their bond distances, bond angles, and dihedral angles are analogous to those of neutral PT.

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Structure of (*S*,*S*)-(*E*)-3-(2-Butenoyl)-2,4-bis(phenylmethyl)oxazolidine

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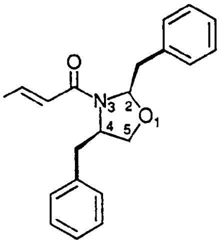
Abstract. $C_{21}H_{23}NO_2$, $M_r = 321.40$, monoclinic, $P2_1$, $a = 9.752\ (5)$, $b = 9.442\ (6)$, $c = 9.894\ (7)$ Å, $\beta = 99.24\ (5)°$, $V = 899\ (7)$ Å^3, $Z = 2$, $D_m = 1.16\ (2)$, $D_x = 1.19$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.68$ cm^{-1}, $F(000) = 344$, $T = 183\ (2)$ K, $R(F) = 0.062$ for 3125 independent reflections including Friedel pairs. The *cis* arrangement of the phenylmethyl groups indicates stereospecific closure of the oxazolidine ring. C(5) lies 0.576 (3) Å from the plane of O(1), C(2), N(3) and C(4).

Experimental. The title compound (I) was prepared by condensation of (*S*)-2-amino-3-phenyl-l-propanol and phenylacetaldehyde and subsequent acylation with crotonyl chloride. A crystal 0.30 × 0.35 × 0.45 mm was mounted on a glass fiber and cooled to 183 (2) K. 25 reflections were centered ($4 < \theta < 15°$) to obtain the unit-cell parameters. Intensities of 3125 independent reflections including Friedel pairs were measured by $\theta/2\theta$ scans ($R_{\text{int}} = 0.045$), for $0 < \theta < 25°$, $0 < h < 11$, $-11 < k < 11$, $-11 < l < 11$. Density was measured by flotation. No absorption corrections were made. Three standard reflections showed no significant deviations. 405 reflections with $F \leq 3\sigma(F)$ were considered unobserved. An Enraf–Nonius CAD-4 diffractometer was used.



(I)

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