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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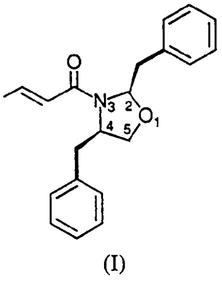
Abstract. $C_{21}H_{23}NO_2$, $M_r = 321.40$, monoclinic, $P2_1$, $a = 9.752\ (5)$, $b = 9.442\ (6)$, $c = 9.894\ (7)$ Å, $\beta = 99.24\ (5)°$, $V = 899\ (7)$ Å^3, $Z = 2$, $D_m = 1.16\ (2)$, $D_x = 1.19$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.68$ cm^{-1}, $F(000) = 344$, $T = 183\ (2)$ 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-1-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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Table 1. *Fractional positional parameters and equivalent isotropic thermal parameters* (Å^2) *for non*-H *atoms with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta).$$

	x	*y*	*z*	B_{eq}
O(1)	0.4371 (2)	0.2373*	0.4158 (2)	4.2
C(2)	0.3089 (3)	0.2813 (3)	0.3388 (2)	3.2
N(3)	0.2113 (2)	0.1660 (3)	0.3570 (2)	3.0
C(4)	0.2849 (3)	0.0539 (3)	0.4425 (2)	3.3
C(5)	0.4050 (3)	0.1406 (4)	0.5179 (3)	4.4
C(6)	0.3266 (3)	0.3004 (3)	0.1899 (2)	3.4
C(7)	0.4328 (2)	0.4111 (3)	0.1697 (2)	2.9
C(8)	0.3938 (3)	0.5451 (3)	0.1225 (3)	3.8
C(9)	0.4939 (3)	0.6447 (4)	0.1032 (3)	4.4
C(10)	0.6336 (3)	0.6110 (4)	0.1320 (3)	4.2
C(11)	0.6734 (3)	0.4778 (4)	0.1791 (3)	4.0
C(12)	0.5734 (3)	0.3793 (3)	0.1980 (3)	3.4
C(13)	0.3382 (3)	−0.0655 (3)	0.3601 (3)	3.8
C(14)	0.2252 (2)	−0.1514 (3)	0.2751 (3)	3.2
C(15)	0.1809 (3)	−0.1207 (3)	0.1388 (3)	3.7
C(16)	0.0778 (3)	−0.1985 (4)	0.0610 (3)	4.9
C(17)	0.0178 (3)	−0.3106 (4)	0.1192 (4)	5.5
C(18)	0.0612 (3)	−0.3436 (4)	0.2555 (4)	5.6
C(19)	0.1639 (3)	−0.2644 (4)	0.3336 (3)	4.3
C(20)	0.0722 (3)	0.1911 (3)	0.3297 (3)	3.2
O(21)	0.0293 (2)	0.3058 (3)	0.2815 (2)	4.5
C(22)	−0.0227 (3)	0.0749 (3)	0.3518 (3)	3.9
C(23)	−0.1563 (3)	0.0839 (4)	0.2987 (4)	5.1
C(24)	−0.2605 (4)	−0.0236 (5)	0.3039 (5)	7.2

*Coordinate fixed to define origin for structure with non-centrosymmetric space group.

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(2)	1.418 (3)	C(11)—C(12)	1.382 (4)
O(1)—C(5)	1.432 (4)	C(13)—C(14)	1.511 (4)
C(2)—N(3)	1.475 (4)	C(14)—C(15)	1.379 (4)
C(2)—C(6)	1.522 (4)	C(14)—C(19)	1.393 (4)
N(3)—C(4)	1.469 (4)	C(15)—C(16)	1.376 (4)
C(4)—C(5)	1.523 (4)	C(16)—C(17)	1.380 (5)
C(4)—C(13)	1.530 (4)	C(17)—C(18)	1.382 (6)
C(6)—C(7)	1.507 (4)	C(18)—C(19)	1.383 (5)
C(7)—C(8)	1.380 (4)	N(3)—C(20)	1.361 (3)
C(7)—C(12)	1.388 (3)	C(20)—O(21)	1.229 (4)
C(8)—C(9)	1.390 (5)	C(20)—C(22)	1.474 (4)
C(9)—C(10)	1.383 (4)	C(22)—C(23)	1.328 (4)
C(10)—C(11)	1.375 (5)	C(23)—C(24)	1.442 (6)
C(2)—O(1)—C(5)	107.0 (2)	C(9)—C(10)—C(11)	119.7 (3)
O(1)—C(2)—N(3)	104.4 (2)	C(10)—C(11)—C(12)	119.6 (3)
O(1)—C(2)—C(6)	109.1 (2)	C(11)—C(12)—C(7)	121.5 (3)
N(3)—C(2)—C(6)	112.4 (2)	C(4)—C(13)—C(14)	114.3 (2)
C(2)—N(3)—C(4)	109.7 (2)	C(13)—C(14)—C(15)	121.1 (3)
C(2)—N(3)—C(20)	119.3 (2)	C(13)—C(14)—C(19)	120.2 (2)
C(4)—N(3)—C(20)	128.3 (2)	C(15)—C(14)—C(19)	118.6 (2)
N(3)—C(4)—C(5)	99.2 (2)	C(14)—C(15)—C(16)	121.4 (8)
N(3)—C(4)—C(13)	113.6 (2)	C(15)—C(16)—C(17)	119.8 (3)
C(5)—C(4)—C(13)	110.8 (2)	C(16)—C(17)—C(18)	119.7 (3)
O(1)—C(5)—C(4)	103.6 (2)	C(17)—C(18)—C(19)	120.3 (3)
C(2)—C(6)—C(7)	113.5 (2)	C(18)—C(19)—C(14)	120.2 (3)
C(6)—C(7)—C(8)	121.5 (2)	N(3)—C(20)—O(21)	119.8 (3)
C(6)—C(7)—C(12)	120.0 (3)	N(3)—C(20)—C(22)	118.0 (3)
C(8)—C(7)—C(12)	118.4 (3)	O(21)—C(20)—C(22)	122.1 (3)
C(7)—C(8)—C(9)	120.4 (3)	C(20)—C(22)—C(23)	119.5 (3)
C(8)—C(9)—C(10)	120.4 (3)	C(22)—C(23)—C(24)	126.4 (4)

The phase-problem solution used *SHELXS*86 (Sheldrick, 1985). Full-matrix least-squares refinements on F_{obs} used *SHELX*76 (Sheldrick 1976). H atoms were found from an $F_o - F_c$ synthesis and refined isotropically, except for the H atoms on the C(24) methyl group which were refined as a rigid body. C, N and O atoms were refined anisotropically. $R(F) = 0.062$, $wR = 0.084$ $\{w = 1.000/[\sigma^2(F) + 0.0075(F^*F)]\}$, $S = 0.849$. $\Delta/\sigma_{max} = 0.09$ for non-H-atom parameters and 0.42 for H-atom parameters; $\Delta\rho_{max} = 0.27$, $\Delta\rho_{min} = -0.34$ e Å^{-3}. Atomic scattering factors for non-H atoms were taken from *International Tables for X-ray Crystallography* (1968, Vol. III) and for H atoms from Stewart, Davidson & Simpson (1965). Anomalous-dispersion contributions were included in the calculated structure factors.

The atomic coordinates of C, O and N atoms are listed in Table 1* and the bond distances and angles are listed in Table 2. Fig. 1 shows an *ORTEP* (Johnson, 1965) drawing with the numbering of the atoms.

Related literature. This structure determination was undertaken to determine the substituent stereochemistry of the oxazolidine ring and to verify structural analyses made from NMR spectroscopy. The bond angles and distances of the oxazolidine ring compare favorably with related structures reported by Neelakantan & Molin-Case (1971) and Bernardi, Cardani, Pilati, Poli, Scolastico & Villa (1988). Torsional angles of the ring are close to those reported by Bernardi, Cardani, Pilati, Poli, Scolastico & Villa (1988) for a ring in which the substituents at C(2) and C(4) are *cis*, but deviate from those reported by Neelakantan & Molin-Case (1971) for a structure with a *trans* arrangement at these C atoms.

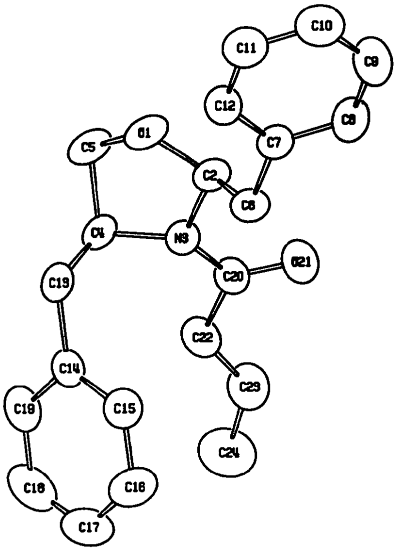

Fig. 1. Thermal ellipsoid plot (50% probability) of the molecule showing the numbering scheme.

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* Lists of H-atom positions, structure factors, anisotropic thermal parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55408 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR0191]

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Structure of 6-(3,3-Dimethyl-2-oxo-2,3-dihydro-5-furanyl)-2-pyridone at 145 K

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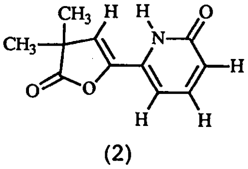
Abstract. $C_{11}H_{11}NO_3$, $M_r = 205.21$, triclinic, $P\bar{1}$, $a = 5.725\,(1)$, $b = 9.038\,(1)$, $c = 10.401\,(2)$ Å, $\alpha = 101.37\,(1)$, $\beta = 102.63\,(1)$, $\gamma = 102.83\,(1)°$, $V = 494.7\,(3)$ Å^3, $Z = 2$, $D_x = 1.38$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0.7107$ Å, $\mu = 0.62$ cm^{-1}, $F(000) = 216$, $T = 145$ K, $R = 0.049$, $wR = 0.063$ for 2386 unique observed reflections. The molecules, which are self-associating in solution, are arranged in the crystal in centrosymmetric dimers joined by N—H···O bonds between the amide functional groups. Analysis of displacement parameters indicates that the furanyl and pyridone groups are independently quite rigid, with the furanyl group librating with respect to the hydrogen-bonded pyridone.

Experimental. The title compound (2) was prepared *via* the methodology described by Ducharme & Wuest (1988) as part of our efforts to synthesize substituted pyridones. The crucial C—C bond formation linking pyridone and lactone precursors and the γ-lactone ring formation were accomplished in one step by the use of tetrakis(triphenylphosphine)palladium. Proton nuclear magnetic resonance studies indicated a self-associating system with a concentration dependence of the lactam N—H chemical shift. A mathematic extrapolation, as described by Chen & Rosenberger (1990), gave a dimerization constant $K = 8$ in chloroform at 294 K.

6-(3,3-Dimethyl-2-oxo-2,3-dihydro-5-furanyl)-2-benzyloxypyridine (1), the precursor to (2), was prepared by stirring a mixture of 2-benzyloxy-6-bromopyridine (Duggan, Grabowski & Russ, 1980) (2.5 g, 0.01 mol), 2,2-dimethylbut-3-ynoic acid (Schexnayder & Engel, 1975) (1.13 g, 0.01 mol), dry triethylamine (15 mL), tetrakis(triphenylphosphine)palladium (0.35 g, 0.3 mmol) and copper(I) iodide (0.1 g, 0.5 mmol) in a sealed thick-walled pressure tube at 353 K, under an atmosphere of argon, for 5 h. The resultant cooled suspension was added to water (120 mL) and extracted with diethyl ether (2 × 70 mL). The combined ethereal extracts were dried (anhydrous magnesium sulfate) and evaporated *in vacuo* to a yellow oil (2.77 g, 94%). Radial chromatography gave (1) in the second fraction with dichloromethane/hexane (1:1) as eluent, as a pale yellow oil (2.12 g, 72%). Crystallization from benzene/hexane gave (1) as white stubby needles (2.0 g, 70%), m.p. 350.6–351.1 K. IR ($CHCl_3$): ν 1803 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 1.43 (*s*, 6H); 5.41 (*s*, 2H); 6.27 (*s*, 2H); 6.78 (*d*, 1H, *J* 8 Hz); 7.31–7.46



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