

Table 2. Bond lengths (Å) and angles (°)

C(1)—N	1.387 (7)	C(1)—O(1)	1.201 (7)
C(1)—O(2)	1.312 (8)	C(2)—C(3)	1.518 (8)
C(2)—O(2)	1.447 (8)	C(3)—C(16)	1.512 (7)
C(3)—N	1.487 (6)	C(4)—C(5)	1.524 (8)
C(4)—C(8)	1.510 (10)	C(4)—O(4)	1.442 (8)
C(4)—O(5)	1.418 (7)	C(5)—C(6)	1.507 (7)
C(5)—N	1.455 (6)	C(6)—C(7)	1.518 (11)
C(7)—O(3)	1.187 (9)	C(7)—O(4)	1.322 (9)
C(9)—C(10)	1.451 (12)	C(9)—O(5)	1.438 (8)
C(10)—C(11)	1.401 (12)	C(10)—C(15)	1.383 (11)
C(11)—C(12)	1.373 (14)	C(12)—C(13)	1.402 (13)
C(13)—C(14)	1.329 (17)	C(14)—C(15)	1.444 (16)
C(16)—C(17)	1.359 (9)	C(16)—C(21)	1.363 (9)
C(17)—C(18)	1.400 (10)	C(18)—C(19)	1.354 (14)
C(19)—C(20)	1.341 (13)	C(20)—C(21)	1.371 (12)
N—C(1)—O(1)	124.8 (6)	N—C(1)—O(2)	111.6 (5)
O(1)—C(1)—O(2)	123.6 (6)	C(3)—C(2)—O(2)	106.0 (4)
C(2)—C(3)—C(16)	111.9 (4)	C(2)—C(3)—N	100.6 (4)
C(16)—C(3)—N	113.3 (4)	C(5)—C(4)—C(8)	119.6 (5)
C(5)—C(4)—O(4)	105.6 (5)	C(8)—C(4)—O(4)	106.1 (5)
C(5)—C(4)—O(5)	103.2 (4)	C(8)—C(4)—O(5)	112.9 (5)
O(4)—C(4)—O(5)	109.0 (5)	C(4)—C(5)—C(6)	102.7 (4)
C(4)—C(5)—N	115.3 (4)	C(6)—C(5)—N	114.0 (4)
C(5)—C(6)—C(7)	103.3 (5)	C(6)—C(7)—O(3)	128.9 (8)
C(6)—C(7)—O(4)	110.2 (6)	O(3)—C(7)—O(4)	120.8 (8)
C(10)—C(9)—O(5)	108.3 (7)	C(9)—C(10)—C(11)	122.9 (7)
C(9)—C(10)—C(15)	117.4 (8)	C(11)—C(10)—C(15)	119.7 (8)
C(10)—C(11)—C(12)	119.8 (7)	C(11)—C(12)—C(13)	120.6 (9)
C(12)—C(13)—C(14)	120.6 (10)	C(13)—C(14)—C(15)	120.2 (9)
C(10)—C(15)—C(14)	119.1 (9)	C(3)—C(16)—C(17)	122.8 (5)
C(3)—C(16)—C(21)	119.1 (5)	C(17)—C(16)—C(21)	118.0 (6)
C(16)—C(17)—C(18)	119.7 (7)	C(17)—C(18)—C(19)	121.0 (8)
C(18)—C(19)—C(20)	118.8 (7)	C(19)—C(20)—C(21)	120.7 (9)
C(16)—C(21)—C(20)	121.6 (8)	C(1)—N—C(3)	108.8 (4)
C(1)—N—C(5)	122.5 (4)	C(3)—N—C(5)	120.4 (4)
C(1)—O(2)—C(2)	109.7 (5)	C(4)—O(4)—C(7)	111.1 (5)
C(4)—O(5)—C(9)	117.7 (5)		

**Related literature.** Two compounds have been structurally characterized that contain a 4-phenyl-1,3-oxazolidin-2-one substructure: 8-chloro-5,6-dihydro-3-(*o*-fluorophenyl)-6-(4-phenyl-2-oxo-oxazolidin-3-yl-carbonyl)furo[3,2-*f*]-1,2-benzisoxazole (Plattner *et al.*, 1984) and (*R*)-3-[(*R*)-(6,7-dichloro-2,3-dihydrobenzofuran-2-yl)carbonyl]-4-phenyl-2-oxazolidone (Nakai, 1988). The title compound (1) differs from these related compounds in that the N atom is bound to the C atom of a five-membered ring rather than a carbonyl C atom.

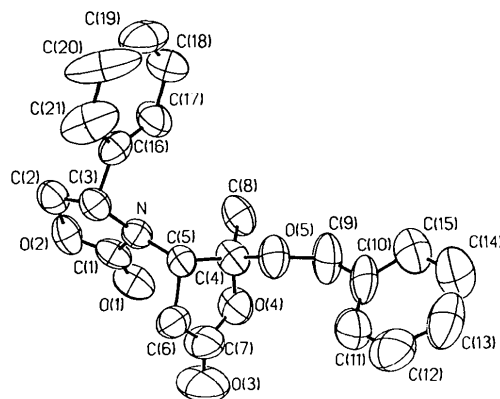


Fig. 1. The structure of (1) (50% probability thermal ellipsoids). H atoms have been omitted for clarity.

*al.*, 1984) and (*R*)-3-[(*R*)-(6,7-dichloro-2,3-dihydrobenzofuran-2-yl)carbonyl]-4-phenyl-2-oxazolidone (Nakai, 1988). The title compound (1) differs from these related compounds in that the N atom is bound to the C atom of a five-membered ring rather than a carbonyl C atom.

We acknowledge the support of the US National Science Foundation for the purchase of the Nicolet R3m diffractometer and computing system.

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## Structure of 2-Ethoxycarbonyl-4,6-diphenylpyridine 1-(4-Bromobenzoyl)imide

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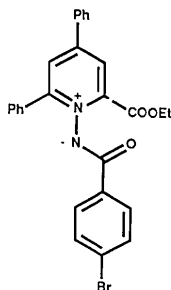
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**Abstract.** [N-(2-Ethoxycarbonyl-4,6-diphenyl)-pyridinio]-3-bromobenzamidate,  $C_{27}H_{21}BrN_2O_3$ ,  $M_r = 501.38$ , monoclinic,  $P2_1/c$ ,  $a = 13.1959$  (4),  $b = 8.0449$  (2),  $c = 22.0192$  (12) Å,  $\beta = 96.303$  (3)°,  $V =$

$2323.4$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.433$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 26.65$  cm<sup>-1</sup>,  $F(000) = 1024$ ,  $T = 293$  K,  $R = 0.055$  for 2068 observed reflexions. The structure includes a stacking zone, in which the

pyridinium rings somewhat overlap the two ends of the zone of charge delocalization. The spacing between the stacked molecules corresponds to half the *b* axis; the angle between near-neighbour pyridinium planes is 26.1° analogous to a  $\beta$ -tribenzopyrene type of packing (Desiraju, 1989). The present stacking, between molecules related by a helical axis, involves also a possible *T* interaction (Desiraju, 1989) between a phenyl ring and the Br-substituted one. Along *a* and around symmetry centers there are Br...H interactions that give rise to a type of dimer.

**Experimental.** To a suspension of 1-(*p*-bromo)-benzamido-2-ethoxycarbonyl-4,6-diphenylpyridinium tetrafluoroborate (Molina, Tarraga & Lorenzo Peña, 1986) (0.45 g, 0.76 mmol) in ethanol (15 ml), an equimolecular amount of triethylamine was added gradually. The resulting solution was stirred at room temperature for 18 h and the precipitated yellow solid was filtered off and recrystallized from methanol–diethyl ether (1:1, v/v) to give the title compound, as yellow needles, in 84% yield; m.p. 455–457 K.



Composition: Found: C, 64.70; H, 4.18; N, 5.41%. Calc. for C<sub>27</sub>H<sub>21</sub>BrN<sub>2</sub>O<sub>3</sub>: C, 64.68; H, 4.22; N, 5.59%.  $V_{\text{max}}$ : (Nujol) 1747, 1618, 1587, 1541, 1462, 1392, 1371, 1331, 1292, 1263, 1246, 1238, 1194, 1170, 1084, 1010, 1001, 912, 889, 844, 763, 752, 740 and 704 cm<sup>-1</sup>;  $\delta_{\text{H}}$  (CDCl<sub>3</sub>) 8.11 (*d*, 1H, *J* = 2.4), 7.93 (*d*, 1H, *J* = 2.4), 7.78 (*d*, 2H, *J* = 8.2), 7.70 (*m*, 6H), 7.53 (*m*, 3H), 7.42 (*m*, 3H), 4.38 (*q*, 2H, *J* = 7.0), 1.24 (*t*, 3H, *J* = 7.0);  $\delta_{\text{C}}$  (CDCl<sub>3</sub>) 169.9 (C=O), 161.1 (COOEt), 154.6 (*s*), 149.5 (*s*), 146.4 (*s*), 137.1 (*s*), 135.8 (*s*), 133.0 (*s*), 130.9 (CH), 130.1 (CH), 129.6 (CH), 129.3 (CH), 128.2 (CH), 128.1 (CH), 127.2 (CH), 126.5 (CH), 124.6 (*s*), 122.8 (CH), 63.1 (CH<sub>2</sub>), 13.8 (CH<sub>3</sub>); *m/z* (%) 502 (*M*<sup>+</sup> + 2,2), 500 (*M*<sup>+</sup>, 2), 429 (100), 427 (98), 303 (7), 231 (43), 230 (16), 199 (4), 197 (4), 185 (2), 183 (2), 157 (2), 155 (2), 77 (2). Melting point was determined on a Kofler hot-stage apparatus and is uncorrected. IR spectra were recorded on a Nicolet-FT 5DX spectrometer. <sup>1</sup>H NMR data were obtained using a Bruker AC200 instrument at 200.13 MHz, using SiMe<sub>4</sub> as internal standard. <sup>13</sup>C NMR spectrum was recorded on a

Bruker AC200 at 50.32 MHz. Electron-impact mass spectrum was recorded on a Hewlett Packard 5993 C spectrometer at an ionization potential of 70 eV. Elemental determination was performed with a Perkin–Elmer 240 C analyzer.

A transparent yellow prism sample (0.33 × 0.07 × 0.07 mm) was used for the determination of lattice constants (72 reflexions, up to  $\theta = 45^\circ$ ) and for data collection on a Philips PW 1100 diffractometer, with Cu *K* $\alpha$  radiation, graphite  $\omega/2\theta$  scans, bisecting geometry, 1 × 1° detector apertures, 1.5° scan width, one reflection per minute, +*h*, +*k*, ±*l* range of 14, 9, 24 respectively. Good stability for the sample, checked every 90 min. Of 3457 independent reflexions,  $\theta_{\text{max}} = 60^\circ$ , 2068 were considered observed [ $3\sigma(I)$  criterion]. The structure was solved by Patterson methods and refined by full-matrix least-squares procedures (on *F*) for 382 parameters. No absorption correction was applied. All the H atoms were located on a difference synthesis and they were isotropically included in the last cycles of refinement. An empirical weighting scheme, so as to give no trends in  $\langle w\Delta^2F \rangle$  vs  $\langle |F_o| \rangle$  and  $\langle (\sin\theta)/\lambda \rangle$ , was introduced. The final shift/e.s.d. was 0.04, goodness of fit *S* = 1.15, max. peak in the final  $\Delta F$  of 0.26 e Å<sup>-3</sup> and max. thermal factor was  $U_{33}(\text{Br}) = 0.128(1) \text{ \AA}^2$ . The 114 reflexion was affected by secondary extinction and was omitted in the last cycles of refinement. The final *R* and *wR* values were 0.055 and 0.051 respectively. Almost all the calculations were performed using the XRAY76 system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) on a VAX 11/750 computer. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final positional and the equivalent isotropic vibrational parameters are listed in Table 1.\* The main geometrical parameters are displayed in Table 2 according to the numbering system given in Fig. 1 (Motherwell & Clegg, 1978). The crystal packing along the *b* and *a* axes are shown in Figs. 1 and 2, respectively.

**Related literature.** The crystal structure is built with zwitterionic molecules, that is, a pyridinium ring and a C—O<sup>δ-</sup> part. Structural details for this zwitterionic moiety have been searched in the Cambridge Structural Database (Allen, Kennard & Taylor, 1983). Besulpamide, a diuretic compound (CSD code KEFROC) includes this moiety with analogous

\* Lists of structure factors, anisotropic thermal parameters, full bond distances, bond angles and torsion angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54205 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic coordinates and thermal parameters

$$U_{eq} = (1/3)\sum[U_{ij}a_i^*a_j^*a_i a_j \cos(\alpha, \alpha_j)]$$

	x	y	z	$U_{eq}$ (Å <sup>2</sup> )
Br	1.07031 (6)	0.20635 (13)	0.03821 (5)	0.0976 (4)
N1	0.5400 (3)	0.2543 (6)	0.2007 (2)	0.040 (2)
C6	0.5269 (4)	0.2786 (8)	0.2604 (3)	0.041 (2)
C5	0.4294 (4)	0.3025 (8)	0.2765 (3)	0.043 (2)
C4	0.3448 (4)	0.3015 (8)	0.2333 (3)	0.042 (2)
C3	0.3616 (5)	0.2790 (8)	0.1732 (3)	0.045 (2)
C2	0.4574 (4)	0.2531 (7)	0.1569 (3)	0.041 (2)
N7	0.6382 (3)	0.2202 (7)	0.1839 (2)	0.044 (2)
C8	0.6652 (4)	0.3346 (8)	0.1445 (3)	0.044 (2)
O9	0.6158 (3)	0.4610 (6)	0.1272 (2)	0.061 (2)
C10	0.4712 (4)	0.1982 (10)	0.0935 (3)	0.050 (2)
O11	0.5126 (4)	0.0727 (6)	0.0816 (2)	0.071 (2)
O12	0.4213 (3)	0.3009 (6)	0.0533 (2)	0.058 (2)
C13	0.4168 (6)	0.2517 (11)	-0.0111 (3)	0.062 (3)
C14	0.3236 (7)	0.1475 (13)	-0.0284 (4)	0.075 (4)
C15	0.2396 (4)	0.3136 (8)	0.2498 (3)	0.048 (2)
C16	0.2122 (5)	0.2421 (9)	0.3027 (3)	0.052 (3)
C17	0.1117 (6)	0.2410 (10)	0.3143 (4)	0.070 (3)
C18	0.0380 (6)	0.3131 (11)	0.2748 (5)	0.077 (4)
C19	0.0633 (6)	0.3887 (12)	0.2226 (5)	0.077 (4)
C20	0.1645 (5)	0.3913 (9)	0.2105 (4)	0.062 (3)
C21	0.6165 (4)	0.2813 (8)	0.3079 (3)	0.040 (2)
C22	0.7017 (5)	0.3786 (9)	0.3014 (3)	0.052 (3)
C23	0.7793 (6)	0.3888 (11)	0.3490 (4)	0.067 (3)
C24	0.7716 (6)	0.3019 (12)	0.4022 (4)	0.075 (3)
C25	0.6893 (6)	0.2039 (11)	0.4079 (3)	0.068 (3)
C26	0.6103 (5)	0.1964 (10)	0.3612 (3)	0.055 (3)
C27	0.7650 (4)	0.2993 (8)	0.1204 (3)	0.042 (2)
C28	0.7970 (6)	0.4052 (11)	0.0777 (3)	0.063 (3)
C29	0.8876 (5)	0.3776 (10)	0.0523 (3)	0.064 (3)
C30	0.9460 (5)	0.2440 (9)	0.0716 (3)	0.055 (3)
C31	0.9153 (5)	0.1379 (11)	0.1137 (4)	0.068 (3)
C32	0.8251 (5)	0.1661 (9)	0.1391 (3)	0.057 (3)

Table 2. Main geometrical characteristics (Å, °)

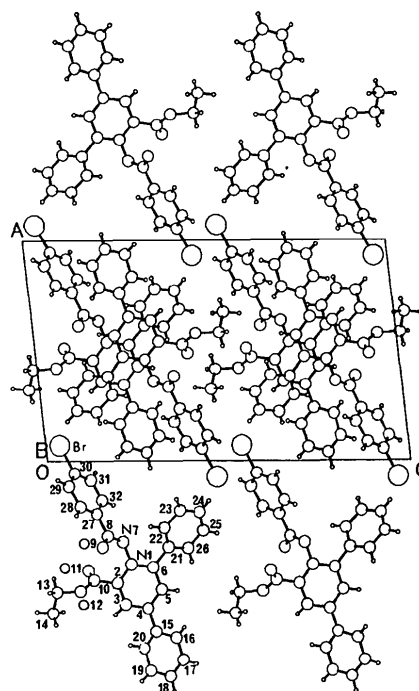
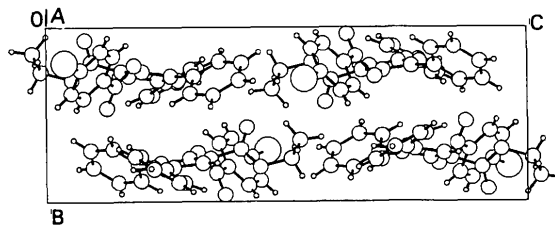
Br—C30	1.895 (6)	N1—C6	1.359 (7)
N1—C2	1.375 (7)	N1—N7	1.412 (6)
C2—C3	1.367 (9)	C2—C10	1.494 (8)
C3—C4	1.376 (9)	C4—C5	1.385 (8)
C5—C6	1.384 (8)	N7—C8	1.340 (8)
C8—O9	1.245 (8)	C8—C27	1.501 (8)
C10—O11	1.190 (9)	C10—O12	1.331 (8)
O12—C13	1.467 (8)	C13—C14	1.503 (12)
C6—N1—C7	119.7 (4)	C6—N1—N7	119.9 (4)
C6—N1—C2	120.3 (4)	N1—C2—C3	120.0 (5)
C3—C2—C10	120.2 (5)	N1—C2—C10	119.2 (5)
C4—C3—C2	121.6 (6)	C5—C4—C15	122.6 (5)
C5—C4—C3	117.2 (5)	C3—C4—C15	120.1 (5)
C6—C5—C4	121.7 (5)	N1—C6—C21	120.3 (5)
N1—C6—C5	119.2 (5)	C5—C6—C21	120.5 (5)
N1—N7—C8	110.7 (4)	N7—C8—C27	113.9 (5)
N7—C8—O9	126.3 (5)	O9—C8—C27	119.7 (5)
C2—C10—O12	109.6 (5)	C2—C10—O11	124.3 (6)
O11—C10—O12	125.8 (6)	C10—O12—C13	116.1 (5)
O12—C13—C14	109.9 (6)		
C2—N1—N7—C8	62.3 (6)	N1—N7—C8—O9	4.5 (8)
N1—N7—C8—C27	-175.2 (5)	N7—C8—C27—C28	177.2 (6)
N7—N1—C2—C3	177.2 (5)	N7—N1—C2—C10	6.4 (8)
N1—C2—C10—O11	50.2 (9)	C3—C2—C10—O12	52.9 (8)
C2—C10—O12—C13	-172.6 (5)	C10—O12—C13—C14	90.3 (7)
N1—C6—C21—C22	49.8 (8)	C5—C4—C15—C20	148.4 (7)

Pyridinium stacking: 4.480 (3) Å between centroids ( $1-x, \frac{1}{2}+y, \frac{1}{2}-z$ ); 18.8 (2)° between least-squares planes.

T-phenyl interaction: 2.83 (7) Å between Ph(15...20) and H32 ( $1-x, \frac{1}{2}+y, \frac{1}{2}-z$ ); 85.7 (2)° between Ph rings.

Br-interaction: 3.09 (8) Å between Br and H29 ( $2-x, 1-y, -z$ ); 152 (5)° for Br...H—C.

geometry, apart from N1—C6 and N2—C3 being somewhat longer in the present compound (Frigola, 1989, and references quoted therein).

Fig. 1. Crystal packing view as projected along the *b* axis together with the numbering system.Fig. 2. Crystal packing view as projected along the *a* axis showing the stacking of molecules.

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