

nearly parallel to the 2₁ or 6₅ axis [dihedral angle between adjacent base least-squares planes is 11.59 (7) (I) and 16.8 (1)° (II) respectively]. It thus appears that the base-stacking properties of nucleosides as described earlier by Bugg & Thomas (1971) are not only found in furanosyl nucleosides but also in pyranosyl nucleosides, although the latter consists of a larger and more rigid six-membered sugar ring instead of the smaller and more flexible five-membered ring. In both compounds only partial base overlap is found with a polar substituent like O(4) (I) or O(2) (II) positioned above the adjacent base (see Fig. 2). The result is that (II) is packed as a single stranded left-handed helix with six molecules per complete turn, an axial rise of 3.2 Å and a pitch of 19.48 (1) Å (for nomenclature see Saenger, 1984). This resembles more or less the structure of the 6₁ poly(2'-*O*-methylcytidine) single stranded helix (Leslie & Arnott, 1978), the latter with an axial rise of 3.2 Å and a pitch height of 18.9 Å. In contrast, however, the poly(2'-*O*-methylcytidine) helix is right-handed and the nucleosides are covalently bonded to each other by a sugar-phosphate backbone.

Many crystal structures in which solvent molecules are included are characterized by larger thermal parameters for those solvent molecules. The small thermal vibrations of the water molecule in (I) are a consequence of a dense hydrogen-bond network involving the water molecule and which consists of two extremely strong hydrogen bonds between N(3)—H(9) and the water O atom and from H(2*W*) towards O(44'), and a third somewhat weaker hydrogen bond between H(1*W*) and O(5'). The result is a neatly ordered water molecule with small thermal vibrations and normal geometry [mean O(*W*)—H(*W*) distance = 0.84 (4) Å and H(1*W*)—O(*W*)—H(2*W*) angle = 101 (3)°]. No intramolecular hydrogen bonds are present.

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N-[(1-Butyl-2-pyrrolidinyl)methyl]-2-methyl-5-sulfamoyl-2,3-dihydrobenzofuran-7-carboxamide

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Abstract. C₁₉H₂₇N₃O₄S, *M_r* = 393.5, monoclinic, *P*2₁, *a* = 12.955 (2), *b* = 35.050 (8), *c* = 9.3196 (7) Å, β = 94.548 (8)°, *V* = 4218 (1) Å³, *Z* = 8, *D_m* = 1.235, *D_x* = 1.239 Mg m⁻³, Cu *Kα*, λ = 1.5418 Å, μ = 1.58 mm⁻¹, *F*(000) = 1680, *T* = 298 K, *R* = 0.067 for

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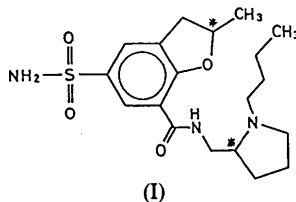
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4899 observed reflections. An intramolecular N⋯O hydrogen bond between the amide group and benzofuran ring makes these two parts of the molecule flat. These two parts and the sulfamoyl groups of the four unique molecules form a layer by two kinds of

intermolecular N...O hydrogen bonds. The layer is sandwiched by the other layers which consist of the butyl chains, pyrrolidine rings and methyl groups of the benzofuran rings.

Introduction. The title compound (I) shows atypical neuroleptic properties similar to sulpiride (Fukuda, Morimoto, Morimoto, Shoji, Murakami, Tahara & Setoguchi, 1989). The efficacies are varied by the stereospecific configurations of the two asymmetric carbons (Tahara, Hayano, Murakami, Fukuda, Setoguchi, Ikeda & Marubayashi, 1990; Murakami, Marubayashi, Fukuda, Takehara & Tahara, 1991). The *S,S* and *R,R* compounds among the four combinations could be crystallized. A crystallographic study has been carried out to determine their structures and absolute configurations.



Experimental. Colorless, spherical crystal 0.275 mm in diameter. Enraf-Nonius CAD-4 diffractometer, graphite-monochromatized Cu K α , ω -2 θ scan technique, $0 \leq 2\theta \leq 120^\circ$. 25 reflections ($16 \leq 2\theta \leq 44^\circ$) used to measure lattice parameters. Index range 0 to 14 (*h*), -39 to 0 (*k*), -10 to 10 (*l*). $\bar{3}22$, $\bar{2}32$, $\bar{2}01$ reflections used as intensity standards, average total count 83 923 with $\sigma = 1222$. 6719 reflections measured, 4899 unique reflections with $I > 3.0\sigma(I)$ used in the analysis ($R_{\text{int}} = 0.033$). No absorption corrections applied ($\mu r = 0.22$). Structure solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Subsequent least-squares refinements, Fourier and difference Fourier syntheses revealed all non-H atoms. Positional and anisotropic thermal parameters for all non-H atoms refined by block-diagonal least-squares methods, $\sum w(|F_o| - k|F_c|)^2$ minimized with $w = a_1 + b_1|F_o|$ for $|F_o| < F_1$, $w = c_2$ for $F_1 \leq |F_o| \leq F_2$ and $w = (a_3 + b_3|F_o| + c_3|F_o|^2)^{-1}$ for $|F_o| > F_2$ where $F_1 = 32.24$, $F_2 = 105.85$, $a_1 = 0.7611$, $b_1 = -0.0131$, $c_2 = 0.035$, $a_3 = 272.31$, $b_3 = -5.42$ and $c_3 = 0.0321$ at final cycle. Final $R = 0.0625$, $wR = 0.082$ and $S = 0.989$. Max. $\Delta/\sigma = 0.41$, max. $\Delta\rho = 0.3 \text{ e } \text{\AA}^{-3}$. H atoms not calculated. Absolute configuration decided by 38 Bijvoet pairs.* Atomic scattering factors involving

Table 1. Fractional atomic coordinates ($\times 10^4$) and isotropic thermal parameters with their *e.s.d.*'s in parentheses

$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (\AA^2)
SA	8348 (1)	1243 (1)	4867 (2)	4.4 (0)
O(1) <i>A</i>	8000 (5)	1062 (2)	3543 (6)	5.9 (2)
O(2) <i>A</i>	9422 (4)	1164 (2)	5464 (7)	5.9 (2)
O(3) <i>A</i>	7881 (4)	2877 (2)	3948 (6)	5.3 (1)
O(4) <i>A</i>	10176 (4)	2476 (2)	7092 (6)	5.6 (1)
N(1) <i>A</i>	7611 (4)	1139 (2)	6048 (6)	4.5 (2)
N(2) <i>A</i>	9609 (6)	2989 (2)	5800 (8)	6.1 (2)
N(3) <i>A</i>	9435 (7)	3727 (3)	4876 (11)	8.5 (3)
C(1) <i>A</i>	8296 (5)	1738 (2)	4564 (7)	4.1 (2)
C(2) <i>A</i>	8963 (5)	1979 (2)	5357 (7)	4.0 (2)
C(3) <i>A</i>	8873 (5)	2372 (2)	5199 (7)	3.9 (2)
C(4) <i>A</i>	8085 (5)	2508 (2)	4207 (7)	4.2 (2)
C(5) <i>A</i>	6910 (7)	2900 (3)	3005 (10)	6.1 (2)
C(6) <i>A</i>	7106 (8)	3200 (3)	1837 (11)	7.1 (3)
C(7) <i>A</i>	6715 (7)	2498 (3)	2391 (11)	6.9 (3)
C(8) <i>A</i>	7423 (5)	2263 (2)	3426 (8)	4.5 (2)
C(9) <i>A</i>	7520 (6)	1871 (2)	3574 (8)	4.8 (2)
C(10) <i>A</i>	9620 (5)	2618 (2)	6117 (7)	4.3 (2)
C(11) <i>A</i>	10286 (8)	3268 (3)	6563 (12)	7.8 (3)
C(12) <i>A</i>	9728 (10)	3654 (3)	6412 (13)	8.5 (4)
C(13) <i>A</i>	8771 (10)	3695 (4)	7193 (15)	9.7 (4)
C(14) <i>A</i>	7944 (14)	3891 (8)	6086 (19)	16.3 (9)
C(15) <i>A</i>	8563 (13)	4005 (4)	4849 (19)	11.9 (6)
C(16) <i>A</i>	10231 (12)	3824 (5)	3981 (20)	12.2 (6)
C(17) <i>A</i>	9923 (16)	3813 (7)	2410 (19)	15.3 (8)
C(18) <i>A</i>	10888 (19)	3803 (8)	1577 (29)	19.9 (12)
C(19) <i>A</i>	10837 (21)	3874 (12)	118 (25)	24.4 (18)
SB	4865 (1)	1092 (1)	1030 (2)	4.7 (0)
O(1) <i>B</i>	3907 (4)	1100 (2)	1667 (7)	6.6 (2)
O(2) <i>B</i>	5070 (5)	804 (2)	-16 (6)	6.5 (2)
O(3) <i>B</i>	5445 (4)	2556 (2)	-1757 (6)	5.4 (1)
O(4) <i>B</i>	2942 (4)	2422 (2)	814 (6)	5.7 (2)
N(1) <i>B</i>	5758 (5)	1052 (2)	2318 (7)	5.8 (2)
N(2) <i>B</i>	3845 (5)	2840 (2)	-388 (9)	6.4 (2)
N(3) <i>B</i>	4624 (6)	3588 (2)	711 (8)	6.7 (2)
C(1) <i>B</i>	5006 (5)	1542 (2)	212 (7)	4.2 (2)
C(2) <i>B</i>	4317 (5)	1831 (2)	455 (7)	4.0 (2)
C(3) <i>B</i>	4434 (5)	2195 (2)	-193 (7)	3.9 (2)
C(4) <i>B</i>	5238 (5)	2235 (2)	-1065 (7)	4.1 (2)
C(5) <i>B</i>	6328 (5)	2492 (2)	-2650 (8)	4.9 (2)
C(6) <i>B</i>	7084 (8)	2802 (3)	-2262 (14)	8.6 (4)
C(7) <i>B</i>	6686 (7)	2081 (3)	-2372 (11)	6.7 (3)
C(8) <i>B</i>	5916 (5)	1938 (2)	-1317 (7)	4.4 (2)
C(9) <i>B</i>	5829 (6)	1595 (2)	-695 (8)	4.6 (2)
C(10) <i>B</i>	3687 (5)	2492 (2)	125 (7)	4.2 (2)
C(11) <i>B</i>	3148 (7)	3168 (3)	-212 (12)	6.7 (3)
C(12) <i>B</i>	3727 (8)	3546 (3)	-294 (10)	6.8 (3)
C(13) <i>B</i>	4201 (11)	3613 (4)	-1819 (12)	9.8 (4)
C(14) <i>B</i>	5155 (13)	3842 (5)	-1445 (16)	12.2 (6)
C(15) <i>B</i>	5280 (9)	3902 (3)	220 (15)	8.7 (4)
C(16) <i>B</i>	4557 (20)	3600 (5)	2279 (18)	15.5 (9)
C(17) <i>B</i>	3897 (22)	3909 (7)	2820 (25)	20.0 (12)
C(18) <i>B</i>	4044 (18)	3910 (6)	4487 (16)	14.5 (8)
C(19) <i>B</i>	3344 (18)	4200 (9)	4901 (22)	19.1 (12)
SC	3509 (2)	2676 (1)	5052 (2)	5.3 (1)
O(1) <i>C</i>	3201 (6)	2945 (2)	6080 (7)	8.0 (2)
O(2) <i>C</i>	4508 (4)	2694 (2)	4573 (8)	7.8 (2)
O(3) <i>C</i>	2759 (3)	1171 (1)	7429 (5)	4.8 (1)
O(4) <i>C</i>	5456 (3)	1352 (2)	5194 (5)	4.9 (1)
N(1) <i>C</i>	2704 (5)	2735 (2)	3654 (7)	5.7 (2)
N(2) <i>C</i>	4483 (5)	914 (2)	6285 (7)	5.3 (2)
N(3) <i>C</i>	3879 (5)	124 (2)	6184 (7)	5.4 (2)
C(1) <i>C</i>	3321 (5)	2218 (2)	5767 (8)	4.7 (2)
C(2) <i>C</i>	4003 (5)	1921 (2)	5506 (7)	4.3 (2)
C(3) <i>C</i>	3879 (5)	1562 (2)	6061 (7)	3.9 (2)
C(4) <i>C</i>	3036 (5)	1508 (2)	6853 (7)	4.2 (2)
C(5) <i>C</i>	1720 (6)	1214 (3)	7906 (9)	5.6 (2)
C(6) <i>C</i>	1625 (10)	965 (4)	9240 (11)	8.6 (4)
C(7) <i>C</i>	1592 (6)	1644 (3)	8138 (9)	5.5 (2)
C(8) <i>C</i>	2367 (5)	1799 (2)	7158 (7)	4.4 (2)
C(9) <i>C</i>	2495 (6)	2166 (2)	6619 (8)	4.9 (2)
C(10) <i>C</i>	4675 (5)	1257 (2)	5814 (7)	4.0 (2)
C(11) <i>C</i>	5271 (7)	610 (3)	6379 (12)	6.6 (3)
C(12) <i>C</i>	4896 (7)	239 (3)	5724 (9)	5.8 (2)
C(13) <i>C</i>	4685 (9)	245 (3)	4097 (10)	7.4 (3)
C(14) <i>C</i>	3965 (10)	-105 (3)	3778 (11)	8.0 (4)
C(15) <i>C</i>	3537 (9)	-193 (3)	5231 (11)	7.5 (3)
C(16) <i>C</i>	3917 (8)	11 (3)	7698 (10)	7.3 (3)
C(17) <i>C</i>	2858 (9)	-94 (4)	8157 (11)	8.0 (3)

* Lists of structure factors, anisotropic thermal parameters of the non-H atoms and a comparison of 38 Bijvoet pairs have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53855 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1 (cont.)

	x	y	z	$B_{eq}(\text{\AA}^2)$
C(18)C	2880 (12)	-157 (4)	9766 (13)	10.6 (5)
C(19)C	1758 (17)	-293 (6)	10129 (18)	14.9 (8)
<i>SD</i>	52 (1)	2562 (1)	1269 (2)	4.8 (0)
O(1)D	-961 (4)	2663 (2)	718 (7)	6.6 (2)
O(2)D	480 (4)	2735 (2)	2578 (6)	6.2 (2)
O(3)D	328 (4)	921 (1)	2242 (5)	4.8 (1)
O(4)D	-1756 (5)	1346 (2)	-1053 (7)	6.7 (2)
N(1)D	816 (5)	2663 (2)	43 (6)	5.1 (2)
N(2)D	-1434 (5)	853 (2)	386 (7)	5.9 (2)
N(3)D	-2043 (8)	119 (3)	1520 (10)	8.6 (3)
C(1)D	102 (5)	2060 (2)	1565 (7)	4.4 (2)
C(2)D	-590 (5)	1830 (2)	744 (7)	4.3 (2)
C(3)D	-563 (5)	1438 (2)	921 (7)	3.8 (2)
C(4)D	184 (5)	1294 (2)	1947 (7)	4.1 (2)
C(5)D	1133 (6)	890 (2)	3455 (8)	5.1 (2)
C(6)D	1850 (7)	574 (3)	3159 (11)	6.8 (3)
C(7)D	1620 (6)	1277 (3)	3639 (9)	5.9 (2)
C(8)D	899 (5)	1524 (2)	2740 (7)	4.6 (2)
C(9)D	852 (5)	1913 (2)	2547 (8)	4.6 (2)
C(10)D	-1287 (5)	1201 (2)	31 (7)	4.2 (2)
C(11)D	-2175 (8)	591 (2)	-438 (10)	6.7 (3)
C(12)D	-1961 (7)	184 (3)	-38 (10)	6.5 (3)
C(13)D	-2842 (12)	-72 (3)	-764 (15)	10.9 (5)
C(14)D	-3524 (12)	-125 (5)	397 (24)	14.4 (7)
C(15)D	-2891 (11)	-140 (5)	1709 (19)	12.9 (6)
C(16)D	-1074 (14)	-2 (5)	2291 (16)	13.2 (6)
C(17)D	-1315 (14)	94 (4)	4088 (16)	12.0 (6)
C(18)D	-474 (22)	16 (7)	4984 (30)	20.4 (12)
C(19)D	-553 (19)	65 (7)	6504 (17)	15.8 (9)

anomalous-dispersion factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72-98). Calculations performed on a VAX11/750 using *Enraf-Nonius SDP-Plus* program package (Frenz, 1985) and *UNICSIII* program system (Sakurai & Kobayashi, 1979).

Discussion. The final atomic parameters are listed in Table 1.* The crystal has four unique molecules (*A*, *B*, *C* and *D*), and the mean bond distances and angles are listed in Table 2. The hydrogen-bond distances and selected torsion angles are also listed in Table 2. Fig. 1 shows one of the molecules with the atom-numbering scheme drawn by *ORTEP* (Johnson, 1965). Fig. 2 shows the stereo crystal structure along the *c* axis drawn by *PLOT80* (Sakurai & Kobayashi, 1979).

The title compound has two asymmetric C atoms in the dihydrofuran ring [C(5)] and in the pyrrolidine ring [C(12)]. Among the four combinations, the present results are for the *S,S* form. The *R,R* form is a complete antipode of the *S,S* form.

The benzofuran and amide groups of each molecule are connected by an O(3)⋯N(2) intramolecular hydrogen bond and are roughly planar. The pyrrolidine rings and butyl chains of the four unique molecules protrude in four directions respectively by rotations around the N(2)—C(11) and C(11)—C(12) bonds. The bond lengths are not significantly different from those of sulphiride (Ma, Camerman & Camerman, 1982).

The sulfamoyl, benzofuran and amide groups of molecules *A* and *D* are related by a pseudo twofold

Table 2. Bond lengths (\AA) and angles ($^\circ$) of the four unique molecules, hydrogen-bond distances (\AA) and selected torsion angles ($^\circ$)

Mean bond lengths			
S—O(1)	1.425 (3)	C(3)—C(4)	1.391 (5)
S—O(2)	1.431 (3)	C(3)—C(10)	1.489 (5)
S—N(1)	1.605 (3)	C(4)—C(8)	1.388 (5)
S—C(1)	1.768 (4)	C(5)—C(6)	1.514 (7)
O(3)—C(4)	1.344 (5)	C(5)—C(7)	1.524 (7)
O(3)—C(5)	1.475 (5)	C(7)—C(8)	1.513 (6)
O(4)—C(10)	1.235 (4)	C(8)—C(9)	1.375 (5)
N(2)—C(10)	1.314 (5)	C(11)—C(12)	1.514 (7)
N(2)—C(11)	1.477 (6)	C(12)—C(13)	1.546 (8)
N(3)—C(12)	1.468 (7)	C(13)—C(14)	1.523 (11)
N(3)—C(15)	1.473 (8)	C(14)—C(15)	1.506 (11)
N(3)—C(16)	1.453 (9)	C(16)—C(17)	1.564 (13)
C(1)—C(2)	1.387 (5)	C(17)—C(18)	1.484 (14)
C(1)—C(9)	1.397 (5)	C(18)—C(19)	1.461 (16)
C(2)—C(3)	1.393 (5)		
Mean bond angles			
O(1)—S—O(2)	119.9 (2)	O(3)—C(5)—C(6)	107.5 (4)
O(1)—S—N(1)	106.6 (2)	O(3)—C(5)—C(7)	106.1 (3)
O(1)—S—C(1)	107.3 (2)	C(6)—C(5)—C(7)	115.4 (4)
O(2)—S—N(1)	107.1 (2)	C(5)—C(7)—C(8)	101.6 (3)
O(2)—S—C(1)	107.5 (2)	C(4)—C(8)—C(7)	108.5 (4)
N(1)—S—C(1)	108.1 (2)	C(4)—C(8)—C(9)	120.5 (3)
C(4)—O(3)—C(5)	108.4 (3)	C(7)—C(8)—C(9)	130.9 (4)
C(10)—N(2)—C(11)	123.3 (3)	C(1)—C(9)—C(8)	117.4 (3)
C(12)—N(3)—C(15)	107.3 (4)	O(4)—C(10)—N(2)	123.2 (4)
C(12)—N(3)—C(16)	116.9 (5)	O(4)—C(10)—C(3)	119.6 (3)
C(15)—N(3)—C(16)	112.6 (5)	N(2)—C(10)—C(3)	117.3 (3)
S—C(1)—C(2)	119.4 (3)	N(2)—C(11)—C(12)	110.6 (4)
S—C(1)—C(9)	118.5 (3)	N(3)—C(12)—C(11)	112.2 (4)
C(2)—C(1)—C(9)	122.2 (3)	N(3)—C(12)—C(13)	103.6 (4)
C(1)—C(2)—C(3)	120.4 (3)	C(11)—C(12)—C(13)	113.4 (5)
C(2)—C(3)—C(4)	116.9 (3)	C(12)—C(13)—C(14)	103.9 (5)
C(2)—C(3)—C(10)	118.3 (3)	C(13)—C(14)—C(15)	106.1 (6)
C(4)—C(3)—C(10)	124.9 (3)	N(3)—C(15)—C(14)	104.3 (6)
O(3)—C(4)—C(3)	124.6 (3)	N(3)—C(16)—C(17)	111.2 (6)
O(3)—C(4)—C(8)	112.7 (3)	C(16)—C(17)—C(18)	110.1 (8)
C(3)—C(4)—C(8)	122.6 (3)	C(17)—C(18)—C(19)	112.9 (10)

Intramolecular hydrogen bonds [N(2)⋯O(3)]

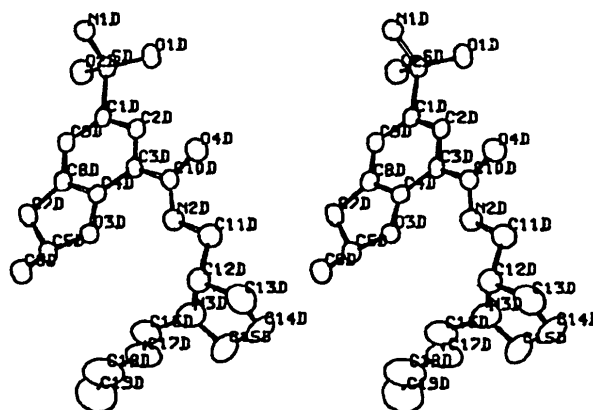
Molecule *A* 2.744 (9) Molecule *C* 2.704 (8)
Molecule *B* 2.708 (9) Molecule *D* 2.763 (8)

Intermolecular hydrogen bonds

N(1)A—O(4)C 2.938 (7) N(1)C—O(4)B 2.904 (9)
N(1)A—O(4)D 2.854 (9) N(1)C—O(2)D 2.975 (8)
N(1)B—O(1)A 3.036 (9) N(1)D—O(4)A 2.884 (8)
N(1)B—O(4)C 2.934 (8) N(1)D—O(4)B 2.916 (8)

Selected torsion angles

	Molecule <i>A</i>	Molecule <i>B</i>	Molecule <i>C</i>	Molecule <i>D</i>
N(1)—S—C(1)—C(2)	91.7 (6)	-105.9 (6)	101.9 (6)	-89.7 (6)
C(11)—N(2)—C(10)—C(3)	-179.3 (7)	177.7 (7)	-167.7 (7)	178.6 (7)
C(10)—N(2)—C(11)—C(12)	-154.0 (8)	154.9 (8)	-131.0 (8)	163.5 (7)
N(2)—C(11)—C(12)—N(3)	-52.8 (11)	-55.9 (11)	-47.4 (11)	59.1 (10)
N(2)—C(11)—C(12)—C(13)	68.5 (12)	61.7 (11)	68.3 (11)	172.3 (8)

Fig. 1. Stereoscopic view of molecule *D*.

* See deposition footnote.

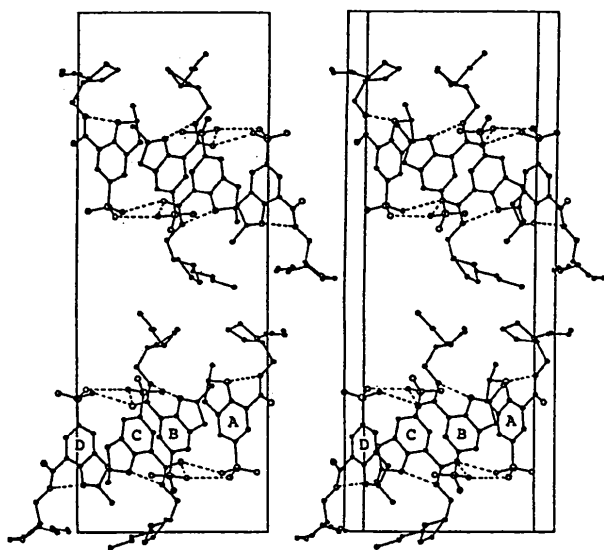


Fig. 2. Crystal structure along the *c* axis. The dotted lines indicate hydrogen bonds.

screw axis. Those of molecules *B* and *C* are also related by another pseudo twofold screw axis. The crystallographic twofold screw axis and the two pseudo screw axes are roughly perpendicular to each other. The four unique molecules are connected by the two types of N...O intermolecular hydrogen

bonds: one type is that between the sulfamoyl groups [N(1)...O(1) or N(1)...O(2)] and the other is that between the sulfamoyl and amide groups [N(1)...O(4)]. Thus, a layer structure of these three groups perpendicular to the *b* axis is formed by a network of the hydrogen bonds. The layer is sandwiched by the other two layers of the methyl, pyrrolidine and butyl groups. In those two layers the constituent groups are in contact *via* van der Waals forces.

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Structure of 7-(*N*-Formyl-*N*-phenyl)amino-4,6-dinitrobenzofurazan 1-Oxide

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Abstract. C₁₃H₇N₅O₇, *M_r* = 345.23, tetragonal, *I*4̄, *a* = 11.129 (3), *c* = 23.368 (6) Å, *V* = 2894 (1) Å³, *Z* = 8, *D_x* = 1.585 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 1.2 cm⁻¹, *F*(000) = 1616, *T* = 293 K, *R* = 0.056 for

1198 reflections with *F_o* > 4σ(*F_o*). The N—O distance in the nitro groups is found to be significantly smaller (1.172–1.218 Å) compared with a typical value of 1.23 Å. The formyl group at N(1) introduces