

Fig. 2. Packing diagram.

A packing diagram (Johnson, 1965) is shown in Fig. 2. The two molecules are linked by two intermolecular hydrogen bonds [O(10A)···O(9B) 2.631 (2) Å and O(9A)···O(10B) ($x, y, z - 1$) 2.636 (2) Å].

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2-(*p*-Chlorophenyl)-3-nitro-2*H*-chromene

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Abstract. C₁₅H₁₀ClNO₃, $M_r = 287.70$, triclinic, $P\bar{1}$, $Z = 2$, $F(000) = 296$, $a = 5.422$ (1), $b = 9.624$ (1), $c = 12.636$ (2) Å, $\alpha = 76.66$ (2), $\beta = 78.67$ (2), $\gamma = 87.97$ (2)°, $V = 629.03$ Å³, $D_m = 1.507$ (3), $D_x = 1.519$ Mg m⁻³, $\lambda(\text{CuK}\alpha) = 1.5418$ Å, $\mu = 26.25$ mm⁻¹, $T = 413$ K, final $R = 0.0577$ for 1859 observed reflections [$I > 2.5\sigma(I)$]. Bond lengths [1.512 (5) Å] and angles [109.2 (3)°] at the phenyl substitution site are

comparable with those in other molecules. The bond angle at the nitro substitution site C(7)-C(8)-C(9) is 122.9 (3)° owing to the electron-withdrawing character of the nitro group. The pyran ring adapts a half-chair conformation.

Introduction. The title compound is of therapeutic value in the treatment of infectious diseases.

Experimental. Yellow crystals were obtained by slow evaporation of the saturated solution of the substance in ethanol. A crystal of dimensions 0.23 × 0.25 × 0.5 mm was mounted on a Nonius CAD-4 diffractometer and the cell parameters were refined by the least-squares method from the measurements of 24 well centred reflections lying in the θ range 16–54°. Density measured by the flotation method using a mixture of carbon tetrachloride and heptane led to two molecules

per unit cell. Data were collected at room temperature in the ω - 2θ scan mode with graphite-mono-chromatized Mo $K\alpha$ radiation, in the range $\sin\theta/\lambda$ from 0 to 0.63 Å⁻¹ with index range $h \pm 5$, $k \pm 10$, $l \pm 13$ and backgrounds measured for one-sixth of the total scan angle on either side of the Bragg peak. 1859 unique reflections were collected with $I > 2.5\sigma(I)$ and used for structure determination. Three standard reflections (117, 154 and 2 $\bar{1}\bar{8}$), well distributed in reciprocal space, were monitored every hour of data collection. Their intensities were corrected for Lorentz and polarization effects, but not for absorption [$\mu(\text{Cu } K\alpha) = 26.25 \text{ mm}^{-1}$].

Table 1. Atomic positional parameters ($\times 10^4$) and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$$U_{\text{eq}} = \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}(\text{\AA}^2)$
C(1)	11819 (8)	4630 (5)	8380 (3)	0.0505 (16)
C(2)	9906 (8)	5538 (5)	8111 (3)	0.0503 (16)
C(3)	8584 (8)	5321 (4)	7334 (3)	0.0432 (15)
C(4)	9132 (6)	4178 (4)	6842 (3)	0.0349 (12)
C(5)	11071 (6)	3264 (4)	7124 (3)	0.0354 (12)
C(6)	12454 (7)	3493 (4)	7895 (3)	0.0426 (14)
C(7)	7891 (7)	3919 (4)	5979 (3)	0.0375 (13)
C(8)	8316 (6)	2692 (3)	5673 (3)	0.0343 (12)
C(9)	9855 (7)	1528 (4)	6228 (3)	0.0366 (12)
C(10)	8289 (7)	463 (3)	7168 (3)	0.0354 (12)
C(11)	6349 (7)	-308 (4)	6970 (3)	0.0430 (14)
C(12)	4907 (8)	-1276 (4)	7790 (3)	0.0459 (15)
C(13)	5349 (7)	-1515 (4)	8862 (3)	0.0425 (13)
C(14)	7284 (8)	-798 (4)	9082 (3)	0.0509 (15)
C(15)	8704 (8)	172 (4)	8248 (3)	0.0470 (15)
O(1)	11782 (4)	2158 (2)	6620 (2)	0.0377 (8)
O(2)	5786 (6)	3315 (3)	4362 (2)	0.0582 (12)
O(3)	7878 (6)	1380 (3)	4417 (2)	0.0595 (12)
N(1)	7253 (6)	2451 (3)	4753 (2)	0.0422 (12)
Cl(1)	3469 (2)	-2725 (1)	9921 (1)	0.0602 (4)

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

C(1)–C(2)	1.376 (6)	C(9)–C(10)	1.512 (5)
C(2)–C(3)	1.380 (5)	C(8)–N(1)	1.460 (4)
C(3)–C(4)	1.381 (5)	N(1)–O(2)	1.220 (4)
C(4)–C(5)	1.394 (5)	N(1)–O(3)	1.216 (4)
C(5)–C(6)	1.399 (5)	C(10)–C(11)	1.402 (5)
C(1)–C(6)	1.376 (6)	C(11)–C(12)	1.357 (5)
C(4)–C(7)	1.460 (5)	C(12)–C(13)	1.387 (5)
C(7)–C(8)	1.324 (5)	C(13)–C(14)	1.382 (5)
C(8)–C(9)	1.499 (5)	C(14)–C(15)	1.361 (6)
C(9)–O(1)	1.446 (4)	C(10)–C(15)	1.389 (5)
O(1)–C(5)	1.369 (4)	C(13)–Cl(1)	1.736 (4)
C(1)–H(1)	0.90 (5)	C(15)–H(15)	0.85 (5)
C(2)–H(2)	0.95 (3)	C(14)–H(14)	0.96 (5)
C(3)–H(3)	0.85 (4)	C(12)–H(12)	0.97 (5)
C(7)–H(7)	1.00 (4)	C(6)–H(6)	0.99 (4)
C(9)–H(9)	1.01 (4)	C(11)–H(11)	0.98 (5)
C(1)–C(2)–C(3)	120.0 (4)	C(9)–C(10)–C(11)	120.3 (3)
C(2)–C(3)–C(4)	120.3 (4)	C(9)–C(10)–C(15)	122.9 (3)
C(3)–C(4)–C(5)	119.2 (3)	C(11)–C(10)–C(15)	116.7 (3)
C(4)–C(5)–C(6)	120.9 (3)	C(10)–C(15)–C(14)	122.1 (4)
C(5)–C(6)–C(1)	118.1 (4)	C(10)–C(11)–C(12)	122.1 (3)
C(6)–C(1)–C(2)	121.5 (4)	C(12)–C(13)–C(14)	120.0 (3)
C(3)–C(4)–C(7)	123.3 (3)	C(11)–C(12)–C(13)	119.4 (4)
C(6)–C(5)–O(1)	117.4 (3)	C(15)–C(14)–C(13)	119.6 (4)
C(5)–C(4)–C(7)	117.4 (3)	C(12)–C(13)–Cl(1)	119.6 (3)
C(4)–C(5)–O(1)	121.6 (3)	C(14)–C(13)–Cl(1)	120.3 (3)
C(4)–C(7)–C(8)	118.0 (3)	C(8)–N(1)–O(3)	117.7 (3)
C(5)–O(1)–C(9)	116.8 (3)	C(8)–N(1)–O(2)	119.3 (3)
O(1)–C(9)–C(8)	109.2 (3)	O(2)–N(1)–O(3)	123.0 (3)
C(7)–C(8)–C(9)	122.9 (3)	C(7)–C(8)–N(1)	119.1 (3)
O(1)–C(9)–C(10)	110.6 (3)	C(9)–C(8)–N(1)	118.0 (3)
C(8)–C(9)–C(10)	112.9 (3)		

The structure was solved by direct methods using *MULTAN*, (Main, Woolfson & Germain, 1971). The $N(z)$ statistical test indicated a centre of symmetry in the unit cell. The structure was refined by *SHELX76* (Sheldrick, 1976) with isotropic temperature factors which led to an R value of 0.374 and the structure could not be refined further, even though the molecule generated through Fourier maps had a chemically acceptable geometry and the atomic coordinates gave a good relative agreement between the observed and calculated structure amplitudes. An attempt was made to modify the position of the molecule in the unit cell keeping its orientation the same. To do this a Fourier synthesis using one molecule conforming to the space group $P1$ was carried out. This generated a second molecule in the unit cell which was centrosymmetrically related to the first, through a point not coinciding with the origin. The origin was shifted to the centre of symmetry and successive least-squares refinement with anisotropic temperature factors reduced R to 0.0829. All hydrogen atoms were obtained by successive difference Fourier maps and refined isotropically to an R value of 0.0577. During the refinement, the temperature factors for hydrogen atoms were taken to be the same as those of the atoms to which the hydrogens are attached. The function minimized during the least-squares refinement is $(|F_o| - |F_c|)^2$, unit weights being shown to be satisfactory by a weight analysis. The atomic scattering factors given by Cromer & Mann (1968) were used for non-hydrogen atoms, while for hydrogen atoms those of Stewart, Davidson & Simpson (1965) were used. $(\Delta/\sigma)_{\text{max}} = 1.00$; $-0.20 < \Delta\rho < 0.22 \text{ e \AA}^{-3}$.

Discussion. Final positional parameters for all non-hydrogen atoms with their e.s.d.'s and U_{eq} values are listed in Table 1.* Bond lengths (Å) and bond angles (°) for the molecule are given in Table 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43328 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The average C—C bond length of the two planar phenyl rings *A* and *C* (Fig. 1) of the molecule is 1.381 Å. This value and also the individual C—C distances of the two phenyl rings compare well with the average and the individual C—C distances of similar phenyl rings in *rel*-(2*S*,3*S*)-3-hydroxy-7-methoxy-3',4'-methylenedioxy flavan (Kimura & Watson, 1979) and in epicatechin [(2*R*,3*R*)-2-(3,4-dihydroxyphenyl)-3,4-dihydro-2*H*-1-benzopyran-3,5,7-triol] (Spek & Kojić-Prodić, 1984). The angle C(8)—C(9)—O(1) [109.2 (3)°] at the site C(9) where the substitution of the phenyl group takes place is very similar to those found in the two references given above. The length of the C(9)—C(10) bond is 1.512 (5) Å. This is similar to the values of the single C—C bond [1.508 and 1.53 (1) Å] connecting the phenyl ring with the pyran ring in these molecules. This is also very close to the value of 1.51 Å given by Dunitz (1979). The C(7)—C(8)—C(9) angle is 122.9 (3)° which is higher than the usual C—C—C angles (120.0°). This is due to the strong electron-withdrawing NO₂ substituent at C(8). The C—N and N—O bonds have normal values and

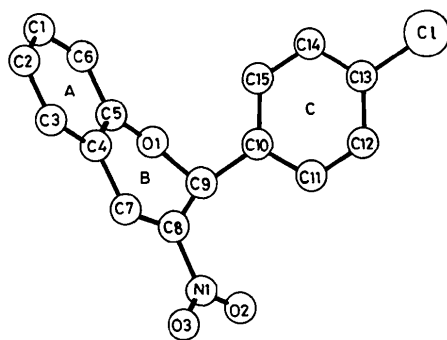


Fig. 1. Molecular conformation and atom-numbering scheme.

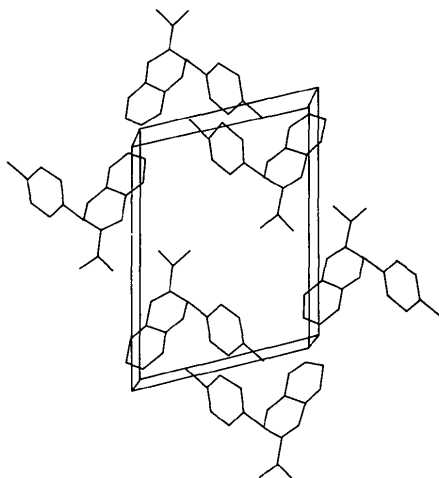


Fig. 2. Packing arrangement of molecules in the unit cell viewed down the *a* axis.

agree well with those observed in the case of *p*-nitrophenoxy acetic acid (Vijay Kumar & Rao, 1980) and *p*-nitrophenoxy acetamide (Lakshmi Rao, Seshadri & Rao, unpublished results). The Cl—C(13) bond is symmetrical in the plane of the phenyl ring (*C*) and does not undergo any bending as indicated by the angles Cl—C(13)—C(14) [120.3 (3)] and Cl—C(13)—C(12) [119.6 (3)°] and the distances Cl—C(14) [2.710 (5)] and Cl—C(12) [2.706 (6) Å]. Such symmetrical bonds without in-plane or out-of plane bending are found in *p*-chlorophenoxy acetic acid (Vijay Kumar & Rao, 1982*a*) and in *N*-(*n*-butyl)-*N'*-[(*p*-chlorophenoxy)acetyl]urea (Vijay Kumar & Rao, 1982*b*). The dihedral angle between the benzopyran ring and the phenyl ring is 81.48°, whereas the dihedral angle between the nitro group and the benzopyran ring is 9.89°. In the pyran ring (*B*), the average length of the C(*sp*²)—O(*sp*³) bond is 1.407 Å, which compares well with the average values for the corresponding bonds in the two molecules noted above (Kimura & Watson, 1979; Spek & Kojić-Prodić, 1984). The torsion angles in the pyran ring *B* are as follows: O(1)—C(5)—C(4)—C(7) —0.4 (5), C(4)—C(7)—C(8)—C(9) 4.6, C(4)—C(5)—O(1)—C(9) —29.6 (5), C(5)—C(4)—C(7)—C(8) 12.9 (5), C(7)—C(8)—C(9)—O(1) —31.7 (5) and C(8)—C(9)—O(1)—C(5) 42.9 (4)° and five atoms of the ring are in the same plane. Thus the pyran ring has the typical half-chair conformation.

The packing arrangement of the molecules in the unit cell is shown in Fig. 2. The molecules are held by normal van der Waals interactions.

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