

Table 1. Positional parameters and their estimated standard deviations

$$B_{eq} = (4/3)[a^2 B_{1,1} + b^2 B_{2,2} + c^2 B_{3,3} + ab(\cos\gamma)B_{1,2} + ac(\cos\beta)B_{1,3} + bc(\cos\alpha)B_{2,3}]$$

	x	y	z	B _{eq} (Å ²)
S	0.1269 (2)	0.2019 (2)	0.1234 (1)	3.24 (3)
O1	0.6948 (6)	0.4927 (5)	0.4250 (4)	4.6 (1)
O2	0.9240 (6)	0.3854 (4)	0.3748 (4)	4.1 (1)
N1	0.2863 (6)	0.3845 (5)	0.2601 (4)	3.5 (1)
N2	0.4310 (6)	0.3181 (5)	0.1027 (4)	2.81 (9)
N3	0.5636 (6)	0.4026 (5)	0.1448 (4)	2.74 (9)
C1	0.8186 (8)	0.4710 (6)	0.3552 (5)	3.2 (1)
C2	0.8153 (8)	0.5643 (6)	0.2530 (6)	3.4 (1)
C3	0.8390 (8)	0.5024 (6)	0.1325 (5)	3.0 (1)
C4	0.6925 (7)	0.4129 (5)	0.0833 (5)	2.5 (1)
C5	0.7147 (8)	0.3431 (6)	-0.0301 (5)	3.6 (1)
C6	0.2920 (7)	0.3078 (5)	0.1660 (5)	2.7 (1)

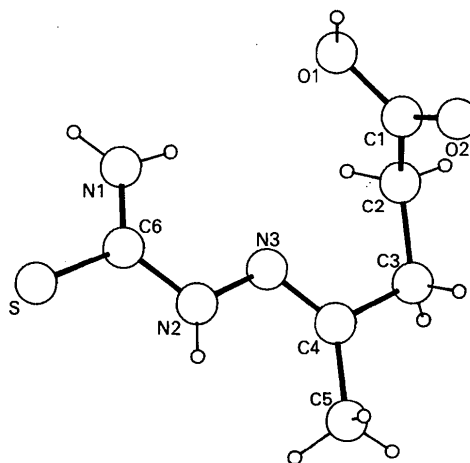


Fig. 1. Atom-numbering scheme.

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

S—C6	1.700 (6)	N3—C4	1.270 (7)
O1—C1	1.314 (8)	C1—C2	1.492 (9)
O2—C1	1.203 (7)	C2—C3	1.518 (9)
N1—C6	1.322 (8)	C3—C4	1.509 (8)
N2—N3	1.382 (7)	C4—C5	1.484 (9)
N2—C6	1.348 (7)		
S...O1 ⁱ	3.085 (5)	S...O1 ⁱⁱ	3.280 (5)
N3—N2—C6	117.8 (5)	N3—C4—C3	116.6 (5)
N2—N3—C4	117.2 (5)	N3—C4—C5	126.3 (6)
O1—C1—O2	122.3 (6)	C3—C4—C5	117.1 (5)
O1—C1—C2	113.0 (6)	S—C6—N1	121.2 (4)
O2—C1—C2	124.7 (6)	S—C6—N2	120.8 (5)
C1—C2—C3	113.9 (5)	N1—C6—N2	117.9 (5)
C2—C3—C4	114.8 (5)		

Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

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1,3-Dinitropyrrolo[1,2-*b*]isoquinoline-5,10-dione

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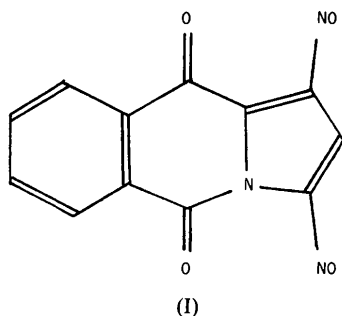
(Received 2 December 1991; accepted 3 March 1992)

Abstract. C₁₂H₅N₃O₆, *M_r* = 287.2, orthorhombic, *Pbca*, *a* = 11.336 (2), *b* = 10.134 (3), *c* = 20.275 (3) Å, *V* = 2329.2 Å³, *Z* = 8, *D_x* = 1.64 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 1.3 cm⁻¹, *F*(000) = 1168, *T* = 195 K, final *R* = 0.063 for 638 observed reflections. The fused ring system is planar and the molecules are disordered between two orientations such that the N

atom is distributed between two sites. The two nitro groups are in the 1,3 sites of the pyrrolo ring and make angles of 44 and 48° with the mean plane of the fused ring nucleus.

Experimental. The compound (I) was obtained by nitration of pyrrolo[1,2-*b*]isoquinoline-5,10-dione

(Cornforth & Du, 1990). NMR spectroscopy identified both nitro groups on the pyrrole ring but their positions could not be decided. The compound was



crystallized from ethyl acetate as extremely thin plates. For the structure determination a piece 0.2×0.2 mm was cut from a crystal 0.01 mm thick. Unit-cell parameters were determined by least-squares fit of 25 reflections with $9 < \theta < 10^\circ$; space group *Pbca* from systematic absences $0kl$ for k odd, $h0l$ for l odd, and $hk0$ for h odd. Data were collected on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation, using θ - 2θ scans with maximum scan time 1 min. 1292 unique reflections were measured (h $0 \rightarrow 9$, k $0 \rightarrow 10$, l $0 \rightarrow 19$, $2 < \theta < 20^\circ$), of which 638 were observed with $|F^2| > 2\sigma(F^2)$, where $\sigma(F^2) = [\sigma^2(I) + (0.04I)^2]^{1/2}/Lp$. Two standard reflections monitored each hour showed no significant change ($< 2\%$). Lorentz and polarization corrections were applied. Structure solution was by direct methods using *SHELXS86* (Sheldrick, 1985). Refinement on F was by full-matrix least squares using programs from the *SDP* package (Frenz, 1984). 50:50 N/C disorder was observed between sites labelled N(1) and C(9) and an averaged scattering factor was used for these sites. O atoms and

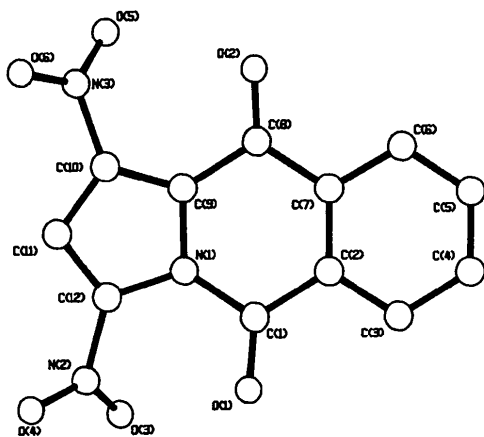


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) drawing of the title compound showing atomic labeling scheme.

Table 1. Fractional atomic coordinates ($\times 10^4$) and isotropic or equivalent isotropic thermal parameters ($\times 10^3$)

O atoms and N(1) and C(9) were refined anisotropically; U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor. Other C and N atoms have isotropic U_{iso} values. Atom sites N(1) and C(9) have occupancy 0.5N/0.5C.

	x	y	z	U_{iso}/U_{eq}
O(1)	5726 (5)	2723 (5)	6243 (3)	60 (4)
O(2)	2310 (6)	-329 (6)	5343 (3)	81 (4)
O(3)	6986 (5)	855 (6)	6969 (3)	67 (4)
O(4)	5988 (6)	1082 (7)	7870 (3)	75 (4)
O(5)	2419 (5)	-2419 (5)	6195 (3)	69 (4)
O(6)	1946 (5)	-1763 (6)	7172 (3)	74 (4)
N(1)	4752 (5)	758 (6)	6315 (3)	27 (3)
N(2)	6067 (5)	840 (6)	7283 (3)	44 (2)
N(3)	2516 (7)	-1695 (7)	6669 (3)	55 (2)
C(1)	5116 (7)	1920 (8)	5959 (4)	44 (2)
C(2)	4680 (7)	2029 (8)	5279 (4)	43 (2)
C(3)	5161 (8)	3034 (9)	4894 (4)	63 (3)
C(4)	4703 (9)	3169 (10)	4249 (5)	75 (3)
C(5)	3850 (8)	2370 (10)	4023 (5)	74 (3)
C(6)	3354 (8)	1386 (9)	4409 (5)	65 (3)
C(7)	3780 (7)	1222 (7)	5056 (4)	43 (2)
C(8)	3212 (8)	250 (8)	5478 (4)	48 (2)
C(9)	3783 (6)	19 (6)	6126 (3)	39 (4)
C(10)	3480 (6)	-745 (7)	6653 (3)	33 (2)
C(11)	4219 (7)	-496 (7)	7181 (4)	38 (2)
C(12)	5006 (7)	413 (7)	6954 (3)	35 (2)

Table 2. Intramolecular distances (\AA) and angles ($^\circ$)

O(1)—C(1)	1.213 (9)	O(2)—C(8)	1.210 (11)
O(3)—N(2)	1.221 (8)	O(4)—N(2)	1.219 (8)
O(5)—N(3)	1.213 (9)	O(6)—N(3)	1.210 (9)
N(1)—C(1)	1.441 (10)	N(1)—C(9)	1.384 (9)
N(1)—C(12)	1.374 (9)	N(2)—C(12)	1.442 (10)
N(3)—C(10)	1.457 (10)	C(1)—C(2)	1.470 (11)
C(2)—C(3)	1.393 (12)	C(2)—C(7)	1.383 (11)
C(3)—C(4)	1.413 (13)	C(4)—C(5)	1.342 (14)
C(5)—C(6)	1.386 (14)	C(6)—C(7)	1.409 (12)
C(7)—C(8)	1.455 (11)	C(8)—C(9)	1.483 (11)
C(9)—C(10)	1.364 (10)	C(10)—C(11)	1.381 (10)
C(11)—C(12)	1.362 (10)		
C(1)—N(1)—C(9)	122.1 (6)	C(1)—N(1)—C(12)	128.3 (6)
C(9)—N(1)—C(12)	106.8 (6)	O(3)—N(2)—O(4)	124.8 (7)
O(3)—N(2)—C(12)	118.3 (6)	O(4)—N(2)—C(12)	116.8 (6)
O(5)—N(3)—O(6)	125.8 (7)	O(5)—N(3)—C(10)	116.8 (6)
O(6)—N(3)—C(10)	117.1 (6)	O(1)—C(1)—N(1)	118.3 (7)
O(1)—C(1)—C(2)	125.9 (7)	N(1)—C(1)—C(2)	115.8 (6)
C(1)—C(2)—C(3)	116.7 (7)	C(1)—C(2)—C(7)	120.7 (7)
C(3)—C(2)—C(7)	122.5 (7)	C(2)—C(3)—C(4)	116.4 (8)
C(3)—C(4)—C(5)	121.5 (9)	C(4)—C(5)—C(6)	122.3 (9)
C(5)—C(6)—C(7)	118.1 (8)	C(2)—C(7)—C(6)	119.2 (7)
C(2)—C(7)—C(8)	122.3 (7)	C(6)—C(7)—C(8)	118.4 (7)
O(2)—C(8)—C(7)	124.7 (8)	O(2)—C(8)—C(9)	119.5 (7)
C(7)—C(8)—C(9)	115.7 (7)	N(1)—C(9)—C(8)	120.4 (6)
N(1)—C(9)—C(10)	106.8 (6)	C(8)—C(9)—C(10)	132.4 (7)
N(3)—C(10)—C(9)	125.5 (6)	N(3)—C(10)—C(11)	124.0 (6)
C(9)—C(10)—C(11)	110.5 (6)	C(10)—C(11)—C(12)	105.1 (7)
N(1)—C(12)—N(2)	122.4 (6)	N(1)—C(12)—C(11)	110.7 (7)
N(2)—C(12)—C(11)	126.4 (6)		

N(1) and C(9) were refined anisotropically, but other C and N atoms were isotropic because of limited data. H atoms were fixed at calculated positions with $U_{iso} = 1.3U_{eq}$ of the parent atom. The function $\sum w(|F_o| - |F_c|)^2$ was minimized [$w = \sigma^{-2}(F)$]; $R = 0.063$, $wR = 0.071$, for 638 observed reflections and 125 variables; $S = 1.8$, $(\Delta/\sigma)_{max} = 0.01$, $(\Delta\rho)_{max,min} = 0.37, -0.25$ e \AA^{-3} . Atomic scattering factors were as imbedded in the *SDP* package. Final atomic parameters are given in Table 1, bond lengths and angles in

Table 2.* The molecular structure is shown in Fig. 1, drawn using *PLUTO* (Motherwell & Clegg, 1978).

Related literature. There appear to have been no previous crystal structure determinations of molecules having the pyrrolo[1,2-*b*]isoquinoline-5,10-dione ring nucleus. The bond lengths and angles

* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55268 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0528]

involving the disordered atoms are unreliable. Other molecular dimensions show no abnormal features.

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Structure of 1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]-1-propanone

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Abstract. $C_{14}H_{20}O_2$, $M_r = 220.3$, monoclinic, $C2/m$, $a = 16.200$ (2), $b = 7.182$ (2), $c = 13.758$ (2) Å, $\beta = 126.99$ (1)°, $V = 1278.6$ (5) Å³, $Z = 4$, $D_x = 1.14$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.70$ cm⁻¹, $F(000) = 480$, $T = 294$ K, $R = 0.056$ and $wR = 0.051$ for 701 significant reflections. An intramolecular hydrogen bond exists between the carbonyl and hydroxyl groups.

Experimental. A colourless crystal $0.70 \times 0.60 \times 0.20$ mm [obtained as described by Martin, Lafrance & Demerseman (1991) and recrystallized from ethanol], was used for data collection on an Enraf-Nonius diffractometer, with graphite-monochromated Mo $K\alpha$ radiation. Intensities were measured using θ - 2θ scans at room temperature. Unit-cell dimensions were determined by least-squares treatment of 25 reflections ($17.5 < \theta < 18^\circ$). No absorption correction was applied to the data, which were collected to $(\sin\theta/\lambda)_{\max} = 0.59$ Å⁻¹. Two standard reflections measured every 2 h showed no significant variation. 1229 unique reflections were measured, of

which 701 were used in calculations [$I \geq 3\sigma(I)$]; $h - 15 \rightarrow 15$, $k 0 \rightarrow 8$, $l 0 \rightarrow 16$. Calculations were performed using the program *CRYSTALS* (Carruthers & Watkin, 1985) on a VAX 11/725 computer; the structure was solved by direct methods (Sheldrick, 1985). The molecule lies in a crystallographic mirror plane, except C(12) and C(12') of the *tert*-butyl group and some H atoms which are in general position in the space group $C2/m$. Refinements in $C2$ did not significantly improve the R factor. H atoms were localized on a difference Fourier synthesis and refined with an overall isotropic thermal parameter. Refinement of 129 least-squares parameters [(number of reflections)/(number of varied parameters) = 5.4 (refined by full-matrix least squares); no extinction correction] converged at $R(F) = 0.056$, $wR = 0.051$ ($w = 1$); average shift/e.s.d. = 0.17, maximum shift/e.s.d. = 1.0 in the last cycle; maximum $\Delta\rho = 0.20$, minimum $\Delta\rho = -0.15$ e Å⁻³ in the final difference Fourier synthesis. Scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 shows atomic parameters