etry in the present study is more precise, with bond-length e.s.d.'s 0·002–0·003 Å (*cf.* 0·004–0·007 Å) and valence-angle e.s.d.'s 0·1–0·2° (*cf.* 0·2 – 0·4°). Aromatic C—C distances here are all in the range 1·375 (2)–1·395 (2) Å compared with the slightly wider range of 1·365 (7)–1·402 (5) Å obtained in the earlier study.

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Structure of *N*-(*p*-Nitrophenyl)ethylenediamine

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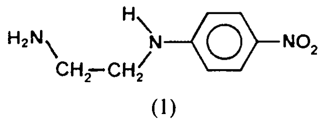
Abstract. $C_8H_{11}N_3O_2$, $M_r = 181{\cdot}19$, monoclinic, $P2_1/c$, $a = 10{\cdot}668\ (2)$, $b = 8{\cdot}958\ (2)$, $c = 10{\cdot}308\ (2)$ Å, $\beta = 115{\cdot}75\ (2)^\circ$, $U = 887{\cdot}2\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}356\ \mathrm{g\ cm^{-3}}$, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}085\ \mathrm{cm^{-1}}$, $F(000) = 384$, $T = 295$ K, $R = 0{\cdot}054$ for 1136 observed reflections. All non-H atoms except C(8) and N(2) are coplanar, the maximum atomic deviation from the best plane being 0·08 Å. A comparison of C—C and N—C bond lengths in the aromatic ring indicates significant contributions from the quinonoid canonical forms owing to through-conjugation effects as also found in *p*-nitroaniline derivatives. An intermolecular hydrogen bond is formed between N(1)(x, y, z) and N(2)($-x, -\frac{1}{2}+y, \frac{3}{2}-z$) [N(1)···N(2) = 2·994 (4) Å and N(1)—H(9)··· N(2) = 170 (3)°].

Experimental. The title compound (1) was prepared by a previously reported method (Linsker & Evans, 1945). Crystals were grown as brown plates from boiling water. A crystal with approximate dimensions 0·15 × 0·50 × 0·55 mm was mounted on a Rigaku automated four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å). Accurate unit-cell parameters were determined by a least-squares fit of 2θ values of 25 centered reflections in the 2θ range 20·1–28·6°. Intensities were measured by the θ–2θ scan technique with a scan rate of 4° min^{-1} in 2θ and a scan width of $\Delta(2\theta) = (2{\cdot}5 + 0{\cdot}70\tan\theta)^\circ$. Background intensities were measured for 5 s at both ends of a scan. Four standard reflections (00$\bar{2}$, 04$\bar{4}$, 040, 500) were remeasured after every 60 reflections; no significant loss of intensity was observed throughout data collection. 1610 independent reflections ($R_{\rm int} = 0{\cdot}015$) were collected with 2θ up to 50·5° [$(\sin\theta)/\lambda = 0{\cdot}600$ Å^{-1}] and index range of $h = -11$ to 12, $k = 0$ to 10, $l = -12$ to 0. Corrections for Lorentz and polarization effects were applied to the intensity data; no absorption or extinction corrections were carried out.



(1)

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters of non*-H *atoms with e.s.d.'s in parentheses*

	x	*y*	*z*	B_{eq}*(Å^2)
C(1)	0·3298 (3)	0·1467 (3)	0·8791 (3)	3·77
C(2)	0·4249 (3)	0·2577 (3)	0·9609 (3)	4·23
C(3)	0·5545 (3)	0·2670 (3)	0·9650 (3)	4·36
C(4)	0·5939 (3)	0·1676 (3)	0·8868 (3)	4·16
C(5)	0·5023 (3)	0·0573 (3)	0·8039 (3)	4·33
C(6)	0·3729 (3)	0·0483 (3)	0·7999 (3)	4·19
C(7)	0·1507 (3)	0·2155 (4)	0·9615 (3)	4·96
C(8)	0·0908 (3)	0·3663 (3)	0·8981 (4)	5·60
N(1)	0·2025 (2)	0·1301 (3)	0·8755 (3)	4·34
N(2)	−0·0290 (3)	0·3603 (3)	0·7593 (3)	6·34
N(3)	0·7289 (3)	0·1779 (3)	0·8885 (3)	5·21
O(1)	0·7614 (3)	0·0892 (3)	0·8172 (3)	7·31
O(2)	0·8086 (2)	0·2783 (3)	0·9617 (3)	7·03

* As defined by Hamilton (1959).

Table 2. *Bond distances* (Å) *and bond angles* (°) *for non*-H *atoms with e.s.d.'s in parentheses*

C(1)—C(2)	1·409 (4)	C(1)—C(6)	1·407 (4)
C(1)—N(1)	1·351 (4)	C(2)—C(3)	1·367 (4)
C(3)—C(4)	1·383 (4)	C(4)—C(5)	1·391 (4)
C(4)—N(3)	1·436 (4)	C(5)—C(6)	1·366 (4)
C(7)—C(8)	1·516 (5)	C(7)—N(1)	1·451 (4)
C(8)—N(2)	1·447 (5)	N(3)—O(1)	1·230 (4)
N(3)—O(2)	1·242 (4)		
C(2)—C(1)—C(6)	117·3 (3)	C(2)—C(1)—N(1)	123·2 (3)
C(6)—C(1)—N(1)	119·5 (3)	C(1)—C(2)—C(3)	121·1 (3)
C(2)—C(3)—C(4)	120·1 (3)	C(3)—C(4)—C(5)	120·3 (3)
C(3)—C(4)—N(3)	120·6 (3)	C(5)—C(4)—N(3)	119·1 (3)
C(4)—C(5)—C(6)	119·4 (3)	C(1)—C(6)—C(5)	121·7 (3)
C(8)—C(7)—N(1)	114·0 (3)	C(7)—C(8)—N(2)	114·7 (3)
C(1)—N(1)—C(7)	124·8 (3)	C(4)—N(3)—O(1)	119·5 (3)
C(4)—N(3)—O(2)	118·2 (3)	O(1)—N(3)—O(2)	122·3 (3)

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986) and refined by the block-diagonal least-squares procedure using *HBLSV* (Ashida, 1979). 1136 observed reflections $[|F_o| \geq 3\sigma(|F_o|)]$ were included in the refinement; the function minimized was $\sum w(|F_o| - |F_c|)^2$. On the difference Fourier map calculated after several cycles of anisotropic refinement, positions of all the H atoms were reasonably found at essentially the same positions as those estimated on the basis of stereochemical considerations. These H-atom parameters were refined isotropically in the further refinement cycles. The weighting scheme used was $w = [\sigma(F_o)^2 + 0{\cdot}0005|F_o|^2]^{-1}$, although unit weights were employed in the early stages of the refinements. The number of observations per refined parameter is 1136/163 = 6·97 and $S = 1{\cdot}72$. The final *R* and *wR* values are 0·054 and 0·061, respectively. $(\Delta/\sigma)_{\max}$ of non-H atoms in the final refinement cycle was 0·05 and 0·04 for positional and thermal parameters, respectively. The peaks in the final $\Delta\rho$ map were between 0·15 and −0·19 e Å^{-3}. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are listed in Table 1.* The molecular structure with the atomic numbering system is shown in Fig. 1 and the crystal-packing diagram is depicted in Fig. 2. Bond distances and angles are presented in Table 2.

All the computations were performed on an ACOS 930 computer at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University.

Related literature. The structure of *p*-nitroaniline has been determined by Trueblood, Goldish & Donohue (1961) and later redetermined by Colapietro, Domenicano, Marciante & Portalone (1982). That of *N*,*N*-dimethyl-*p*-nitroaniline has also been reported (Mak & Trotter, 1965). The average values of the related bond lengths in these two compounds are respectively: R_2N—CH [corresponding to N(1)—C(1) in the present compound] = 1·355 and 1·358 Å, R_2NCH—CH [C(1)—C(2) and C(1)—C(6)]

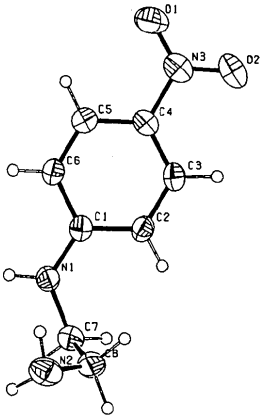

Fig. 1. *ORTEP* drawing (Johnson, 1976) of the molecular structure together with the atomic numbering system. Non-H atoms are represented by thermal ellipsoids with 30% probability levels, whereas H atoms are drawn by a sphere with $B = 1{\cdot}0$ Å^2.

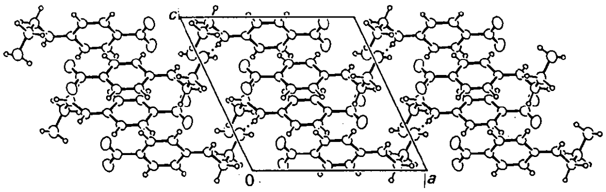

Fig. 2. *ORTEP* (Johnson, 1976) crystal-packing diagram as viewed along the *b* axis. Atoms are represented as in Fig. 1. The intermolecular hydrogen bonding is shown by dotted lines. The shortest intermolecular atomic contact apart from the hydrogen bonds is N(2)$(x, y, z)\cdots$O(2)$(-1 + x, \frac{1}{2} - y, -\frac{1}{2} + z)$ [3·056 (4) Å].

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* Lists of anisotropic temperature factors for non-H atoms, atomic parameters for H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52140 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

= 1·407 and 1·435 Å, CH—CH [C(2)—C(3) and C(6)—C(5)] = 1·367 and 1·375 Å, CH—CNO_2 [C(3)—C(4) and C(5)—C(4)] = 1·392 and 1·400 Å, and C—NO_2 [C(4)—N(3)] = 1·434 and 1·405 Å.

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Structure of 1-(2,4-Dinitrophenyl)pyrazole

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Abstract. 1-(2,4-Dinitrophenyl)pyrazole, $C_9H_6N_4O_4$, M_r = 234·2, orthorhombic, *Pbca*, *a* = 8·958 (2), *b* = 19·103 (3), *c* = 11·806 (3) Å, *V* = 2020·2 (8) Å^3, *Z* = 8, D_x = 1·540 g cm^{-3}, Cu *Kα*, λ = 1·54184 Å, μ = 10·3 cm^{-1}, *F*(000) = 960, *T* = 299 K, *R* = 0·037 for 1798 observations having $I > 3\sigma(I)$ (of 2077 unique data). The phenyl ring is nearly planar with maximum deviation 0·014 (1) Å. The pyrazole ring is planar and forms a 26·24 (7)° dihedral angle with the phenyl ring. The nitro groups are twisted out of the best plane of the phenyl ring by 69·0 (1) and 9·1 (2)°.

Experimental. 1-(2,4-Dinitrophenyl)pyrazole was prepared by the modification of the procedure described by Shriner, Fuson & Curtin (1970). Crystals were obtained by slow evaporation of a $CDCl_3$ solution, as yellow needles, dimensions 0·30 × 0·40 × 0·48 mm. Space group from absences 0*kl* with *k* odd, *h*0*l* with *l* odd, *hk*0 with *h* odd. Enraf–Nonius CAD-4 diffractometer with graphite monochromator, cell dimensions from setting angles of 25 reflections having $30 > \theta > 25°$. Data collection by ω-2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s; scan rates varied 0·53–3·30° min^{-1}. Two octants of data having $2 < \theta < 75°$, $0 \leq h \leq 11$, $-23 \leq k \leq 23$, $0 \leq l \leq 14$ were measured, corrected for background, Lorentz–polarization, and absorption by ψ scans, minimum relative transmission 94·1%. Two equivalent octants merged to give 2077 unique data, $R_{\rm int}$ = 0·012. Standard reflections 400, 0,10,0, 008 displayed only random variation, no decay correction. Structure solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares based on *F* with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ with 1798 data for which $I > 3\sigma(I)$ (279 unobserved reflections), using Enraf–Nonius *SDP* (Frenz & Okaya, 1980). Non-H atoms anisotropic; H atoms located by ΔF synthesis and refined isotropically. Atomic scattering factors from Cromer & Waber (1974) and anomalous coefficients from Cromer (1974). Final *R* = 0·037, *wR* = 0·051, *S* = 2·814 for 179 variables, extinction coefficient *g* = 8·1 (2) × 10^{-6}, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c, max. shift in final cycle 0·05σ, max. residual density 0·21, min. −0·18 e Å^{-3}. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond distances and angles in Table 2. Fig. 1 shows the atom-numbering scheme.

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* Tables of H-atom parameters, distances and angles involving H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52085 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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