

X-ray structural and spectroscopic investigation of 1-piperidine-2,4-dinitrobenzene

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The crystal structure of the title compound (C₁₁H₁₃N₃O₄) has been determined by single-crystal X-ray diffraction. The compound is monoclinic, space group P2₁/n, with $a = 9.968(2)$, $b = 9.156(2)$, $c = 13.249(2)$ Å, $\beta = 102.05(2)^\circ$, and $D_x = 1.563$ g cm⁻³ for $Z = 4$. The aromatic ring shows a slight boat deformation. The *o*- and *p*-NO₂ groups are twisted out of the plane of the phenyl ring by 39.0(2)° and 4.4(1)°, respectively. The piperidine ring exhibits a slightly deformed chair conformation. Short C-H ··· O intermolecular contacts stabilize the three dimensional structure. UV and NMR data indicate that the molecule in solution presents a conformation similar to that of the the solid state.

KEY WORDS: Dinitrobenzene derivatives; 1-piperidine-2,4-dinitrobenzene; 2,4-dinitrobenzene; molecular conformation; spectroscopic study; structural determination.

Introduction

The investigation of the parameters that govern the molecular aggregation and the crystal packing of conjugated organic compounds presents interest due to the possible nonlinear optical behavior of these systems.¹ As part of a general study of the effect of substitution on the molecular geometry and organization of substituted 1-amine-2,4-nitrobenzenes²⁻⁶ a single crystal X-ray study of the molecular structure of 1-piperidine-2,4-dinitrobenzene (**I**) has been carried out and the UV, ¹H, and ¹³C NMR spectra have been obtained.

Experimental

The title compound was synthesized as reported⁷ and crystallized from slow evaporation of an ethanol

solution. Crystal data and data collection and refinement are shown in Table 1. Atomic parameters are given in Table 2. The UV spectra were recorded on a Shimadzu UV 260 spectrophotometer. The ¹H and ¹³C NMR spectra were obtained on a Bruker ACE 200 spectrometer and chemical shifts are referred to TMS.

Discussion

Molecular geometry in the solid state

A view of the molecule with atom labeling is shown in Fig. 1. The aromatic ring presents a slight boat deformation, C(1) and C(4) atoms are out of the mean ring plane, 0.085(1) Å and 0.039(1) Å, respectively. The Total Puckering Amplitude¹² is $QT = 0.074(2)$.

The *o*-nitro group is rotated out of the ring plane, the dihedral angle between the group and the mean plane of the aromatic ring is 39.0(2)°. The *p*-nitro group is also rotated out of the aromatic plane an angle of 4.4(1)°.

The C(1)-N(1), 1.354(2) Å, and C(4)-N(3), 1.453(2) Å, bond lengths indicate some degree of intra-

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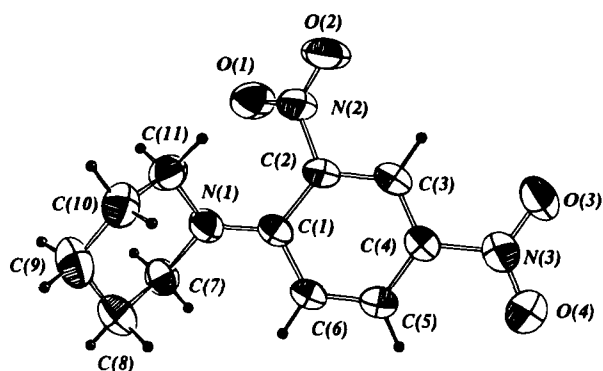


Fig. 1. An ORTEP-II¹⁰ view of the molecule showing the atom labelling and 50% probability ellipsoids.

molecular charge transfer. The C(2)–N(2) bond length, 1.465(2) Å, is larger than the previously mentioned bonds, but smaller than the mean, 1.471(2) Å, found by Domenicano¹³ for the C–N bond length in nitrobenzene derivatives for which the substituents were chosen to avoid conjugation. The C(2)–N(2) and C(4)–N(3) bond lengths are in agreement with the values found in other heavily amino substituted 2,4-dinitroanilines like N,N-diisopropyl-2,4-dinitroaniline (**II**)³ where the C(2)–N(2) bond length is 1.466 Å and the C(4)–N(3) one is 1.450 Å.

The N–O bonds, N(2)–O(1), 1.224(2) Å, N(2)–O(2), 1.222(2) Å, N(3)–O(3), 1.227(2) Å, and N(3)–O(4), 1.216(2) Å are in agreement with the mean value obtained from 334 nitrobenzene fragments retrieved from the 1987 edition of the Cambridge Structural Database, 1.218(11) Å.¹⁴ The values obtained for the O–N–O bond angles, O(1)–N(2)–O(2), 123.7(2)°, and O(3)–N(3)–O(4), 122.9(2)°, are consistent with those expected.

The piperidine ring exhibits a slightly distorted chair conformation. The magnitude of the distortion can be described by the puckering parameters,¹² $QT = 0.544(2)$, $\phi = 48(1)^\circ$ and $\theta = 1.3(2)^\circ$. The piperidine ring bond lengths are in agreement with the mean values found by Herbstein and Schwotzer¹⁵ for 13 compounds containing the piperidine fragment. The piperidine ring bond angles present differences from the mean value obtained by the same authors. These discrepancies might be due to the partial double-bond character of the C(1)–N(1) bond. This double-bond character can be inferred from the short C(1)–N(1) bond length, 1.354(2) Å, and the small displacement of the N(1) atom, 0.066(1) Å, out of the plane defined by: C(1), C(7), and C(11). The C(1)–N(1) bond length and the C(11)–N(1)–C(7) angle, 113.1(1)°, agree well

with the values found by Herbstein and Schwotzer¹⁵ in five compounds with partial C–N double-bond character.

Influence of the substituents

The additivity of the effect of the substituents on the aromatic ring geometry was analyzed using the angular parameters of Domenicano and Murray-Rust,¹⁶ Table 4. It can be seen from the value of the nonadditivity parameters, NAP,¹⁷ that additivity is not accomplished in this compound. As neither the piperidine nor the cyclohexane parameters were available we approximate the effect of the piperidine ring by the effect of the NMe₂ group. For **I** the NAP parameter is larger than 2σ . This would indicate interactions between substituents that destroy the independence of the effect of each one. The interactions might be due not only to the conjugative effect described above but also to steric hindrance effects. Therefore, the deviation of the aromatic ring from the ideal geometry due to steric interaction between the *o*-nitro group and the piperidine ring was also analyzed. The repulsive deformation parameter, RDP [RDP = $\sum_{i=1-2} \{\theta'_i(\text{exp}) - \theta'_i(\text{cal})\}$; where: $\theta'_i(\text{cal}) = 1/2\{360 - \theta_i(\text{cal})\}$, $\theta'_1(\text{exp}) = \angle C(2) - C(1) - N(1)$ and $\theta'_2(\text{exp}) = \angle C(1) - C(2) - N(2)$]¹⁷ was used and the value obtained, 4.9(5)°, showed that steric interactions are not negligible.

Crystal packing

The analysis of the intermolecular contacts shows that some C–H···O distances are shorter than the sum of the van der Waals radii of the involved atoms (see Table 3). These contacts, that have been interpreted as attractive interactions by Taylor and Kennard¹⁸ and Desiraju,¹⁹ induce the formation of infinite polar chains along the [101] direction. The influence of weak interactions in the chain organization of nitroanilines and related compounds has been discussed previously by other authors.²⁰ They suggested that the chain organization might precede the crystallization process but they could not account for the relative arrangement of the chains. In the title compound the above described chains are disposed in an antiparallel fashion. Short intermolecular contacts are observed between *o*-nitro oxygens and piperidine hydrogens related by the inversion center in (1/2, 0, 1/2). The antiparallel array of the chains prevents the material from presenting a

Table 1. Crystal data and structure refinement, esd's in parentheses.^a

Crystal data	
Empirical formula	C ₁₁ H ₁₃ N ₃ O ₄
Formula weight	251.24
Crystal dimension	0.125 × 0.15 × 0.25 mm
Crystal system	Monoclinic
Space group	P2 ₁ /n
Unit cell dimensions	<i>a</i> = 9.968(2) Å <i>b</i> = 9.156(2) Å <i>β</i> = 102.05(2)° <i>c</i> = 13.249(2) Å
Volume	1182.6(6) Å ³
Z	4
Density (calculated)	1.563 g cm ⁻³
Absorption coefficient	0.109 mm ⁻¹
Data collection	
Temperature	293(2)°K
Wavelength	0.71073 Å (Mo Kα)
Scan type	θ/2θ
2-Theta range for data collection	4° to 59.90°
Index ranges	-1 ≤ <i>h</i> ≤ 14, 0 ≤ <i>k</i> ≤ 12, -18 ≤ <i>l</i> ≤ 18
Standard	$\bar{1}, \bar{2}, 6; 0, 0, 12$
Absorption correction	None
Refinement	
F(000)	528
Reflections collected	3976
Independent reflections	3434 [R(int) = 0.0271]
Refinement method	Full-matrix least-squares on F ²
Data/restraints/parameters	3434/0/216
Goodness-of-fit on F ²	1.091
Final R indices [<i>I</i> > 2σ(<i>I</i>)]	R1 = 0.0482, wR2 = 0.1107
R indices (all data)	R1 = 0.0806, wR2 = 0.1369
Calculated weights	w = [σ ² (Fo ²) + (0.0504P)] ² + 0.2611P] ⁻¹ where P = (Fo ² + 2Fc ²)/3.
Largest diff. peak and hole	0.172 and -0.213 e.Å ⁻³

^a Data collection: CAD-4 (Enraf Nonius) software 1990. Cell refinement: CAD-4 (Enraf-Nonius) software 1990. Data reduction: MoLEN²⁶ software 1990. Structure solved: SHELXS-86.⁸ Structure refinement: SHELXL93.⁹ Molecular graphics: ORTEP-II.¹⁰ Software used to prepare material for publication: PARST.¹¹

nonvanishing macroscopic quadratic electrical susceptibility. A similar organization of the molecules in the crystalline state has been observed in other substituted 1-amino-2,4-dinitrobenzenes as **II**³ and N,N-isopropylcyclohexyl-2,4-dinitroaniline.⁴

Molecular geometry in solution

NMR spectra. In order to get information about the geometry of **I** in solution the ¹³C chemical shifts

Table 2. Atomic coordinates (×10⁴) and equivalent isotropic displacement parameters (Å² × 10³), esd's in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} ^a
C(1)	8474(1)	314(2)	5763(1)	42(1)
C(2)	7499(1)	211(2)	4810(1)	43(1)
C(3)	7385(2)	1243(2)	4043(1)	45(1)
C(4)	8292(2)	2393(2)	4176(1)	44(1)
C(5)	9353(2)	2477(2)	5045(1)	46(1)
C(6)	9442(2)	1458(2)	5807(1)	46(1)
C(7)	9673(2)	-802(2)	7409(1)	50(1)
C(8)	9499(2)	-168(3)	8426(1)	61(1)
C(9)	8210(2)	-763(3)	8713(2)	76(1)
C(10)	6979(2)	-556(2)	7838(2)	61(1)
C(11)	7206(2)	-1163(2)	6821(2)	56(1)
O(1)	7198(2)	-2293(2)	4806(1)	73(1)
O(2)	5523(1)	-972(2)	3998(1)	74(1)
O(3)	7312(1)	3336(2)	2582(1)	78(1)
O(4)	8955(2)	4544(2)	3522(1)	87(1)
N(1)	8461(1)	-559(2)	6585(1)	51(1)
N(2)	6674(1)	-1110(2)	4532(1)	54(1)
N(3)	8181(2)	3503(2)	3379(1)	55(1)

^a *U*_{eq} is defined as one third of the trace of the orthogonalized *U*_{ij} tensor.

Table 3. Bond lengths (Å), angles (°) and relevant intermolecular contacts, esd's in parentheses.^a

C(1)–C(2)	1.426(2)	C(7)–N(1)	1.466(2)
C(1)–C(6)	1.417(2)	C(8)–C(9)	1.514(3)
C(1)–N(1)	1.354(2)	C(9)–C(10)	1.514(3)
C(2)–C(3)	1.376(2)	C(10)–C(11)	1.518(3)
C(2)–N(2)	1.465(2)	C(11)–N(1)	1.460(2)
C(3)–C(4)	1.375(2)	N(2)–O(1)	1.224(2)
C(4)–C(5)	1.394(2)	N(2)–O(2)	1.222(2)
C(4)–N(3)	1.453(2)	N(3)–O(3)	1.227(2)
C(5)–C(6)	1.365(2)	N(3)–O(4)	1.216(2)
C(7)–C(8)	1.510(3)		
C(1)–C(2)–C(3)	122.9(1)	C(7)–N(1)–C(11)	113.1(1)
C(1)–C(2)–N(2)	121.4(1)	C(8)–C(9)–C(10)	111.0(2)
C(1)–C(6)–C(5)	122.4(1)	C(8)–C(7)–N(1)	112.1(1)
C(1)–N(1)–C(7)	123.0(1)	C(9)–C(10)–C(11)	112.7(2)
C(1)–N(1)–C(11)	123.2(1)	C(10)–C(11)–N(1)	110.2(2)
C(2)–C(1)–C(6)	114.7(1)	N(1)–C(1)–C(2)	123.6(1)
C(3)–C(2)–N(2)	115.2(1)	N(1)–C(1)–C(6)	121.6(1)
C(3)–C(4)–C(5)	120.9(1)	O(1)–N(2)–C(2)	118.2(1)
C(3)–C(4)–N(3)	119.6(1)	O(2)–N(2)–C(2)	118.0(2)
C(4)–C(3)–C(2)	118.8(1)	O(2)–N(2)–O(1)	123.7(2)
C(4)–C(5)–C(6)	119.6(2)	O(3)–N(3)–C(4)	118.1(2)
C(5)–C(4)–N(3)	119.4(1)	O(4)–N(3)–C(4)	119.0(1)
C(7)–C(8)–C(9)	110.3(2)	O(4)–N(3)–O(3)	122.9(2)
C(6)–O(3) ⁽ⁱ⁾	3.308(2)		
O(3)–C(6) ⁽ⁱⁱ⁾	3.308(2)		
C(11)–O(2) ⁽ⁱⁱⁱ⁾	3.342(2)		

^a (i): *x* + 1/2, -*y* + 1/2, *z* + 1/2, (ii): *x* - 1/2, -*y* + 1/2, *z* + 1/2, (iii): -*x* + 1, -*y*, -*z* + 1.

Table 4. Experimental and calculated endocyclic angles, and nonadditivity parameter value, esd's in parentheses.

θ_i	Angles(°)	D.M.R. ^a	Exper.
θ_1	C(6)–C(1)–C(2)	116.1(6)	114.7(1)
θ_2	C(1)–C(2)–C(3)	123.8(5)	122.9(1)
θ_3	C(2)–C(3)–C(4)	117.6(4)	118.8(1)
θ_4	C(3)–C(4)–C(5)	121.5(6)	120.9(1)
θ_5	C(4)–C(5)–C(6)	119.9(5)	119.6(2)
θ_6	C(5)–C(6)–C(1)	121.2(4)	122.4(1)
	N.A.P. ^b	5.6 ($\sigma = 1.5$)	

^a Calculated from Domenicano and Murray-Rust¹⁶ angular parameters.

^b N.A.P. = $\sum_i |\theta_i(\text{exp}) - \theta_i(\text{calc})|$; $\sigma = [\sum(\sigma_{\text{calc.}} + \sigma_{\text{exp.}})^2]^{1/2}$.

were determined and the values obtained are shown in Table 5 together with the corresponding values for related compounds, namely N,N-diisopropyl-2,4-dinitroaniline (**II**) and N,N-diethyl-2,4-dinitroaniline (**III**).

The lack of coplanarity between the *o*-nitro substituent and the aromatic ring in a series of N-dialkylsubstituted dinitroanilines has been inferred from ¹³C NMR data.⁴ Chemical shifts of **III** were calculated using the additivity rule of substituent effects with data from the literature.²¹ The rotation of the 2-nitro group in **I** and **II** induces shielding in the *ipso* carbon (C(2)) and decreases the shielding in the *ortho* carbon (C(3)). Studies in the solid state have shown that when the size of the alkyl substituent of the amine increases, rotation of both groups (amino and *o*-nitro) contribute to reduce hindrance.⁴ The displacement of the ¹³C NMR signals of C(4) and C(6) in solution in **I** and **II** is in agreement with the twisting of both groups. It can be seen in Table 5 that the $\Delta\delta$ for **I** is somewhere in between those of

Table 5. Experimental and calculated ¹³C NMR chemical shift of compounds (**I**) 1-piperidine-2,4-dinitrobenzene, (**II**) N,N-diisopropyl-2,4-dinitroaniline, and (**III**) N,N-Diethyl-2,4-dinitroaniline.

C	I ^a	$\Delta\delta(\text{I})$	II ^b	$\Delta\delta(\text{II})$	III ^c	III ^b	$\Delta\delta(\text{III})$
1	149.7	+1.2	147.9	-0.6	148.5	147.9	-0.6
2	137.2	+4.8	138.5	+6.1	132.4	129.7	-2.7
3	123.9	+5.4	123.7	+5.2	118.5	123.7	+5.2
4	137.2	+1.3	142.6	+6.7	135.9	135.3	-0.6
5	127.9	-1.4	126.1	-3.2	129.3	129.9	+0.6
6	119.1	+5.5	122.3	+8.7	113.6	113.8	+0.2

^a Experimental values, this work.

^b Experimental values taken from Punte *et al.*⁴

^c Calculated values.²¹

II and **III** indicating that the steric hindrance of the cyclic amine is smaller than that of diisopropyl group and bigger than that of the diethyl group.

There is a reduction of the shielding of C(1) in **I** ($\Delta\delta = +1.2$), that can be compared with the value obtained for N,N-dicyclohexyl-2,4-dinitroaniline (**IV**) ($\Delta\delta = 0.7$),⁵ which may indicate that the interaction of the lone pair of the nitrogen atom with the π system of the aromatic ring in **I** is small due to rotation of the group combined with the strain of the cyclic amine. On the other hand in **IV** the reduced resonance is due only to the extensive rotation of the amino and *ortho* nitro groups.

We can use the chemical shift of the methylene protons α to N (see Table 6) to confirm the rotation of the amino group. The chemical shift of the hydrogens of the methylene groups in cyclohexane is 1.44 ppm.²² This signal appears 0.24 ppm at lower fields than that of methylene groups in alkanes (i.e., 1.2 ppm)²² and the difference is attributed to ring strain. Assuming that the difference in chemical shifts between the methylene protons in **III** and the methyl protons in N,N-dimethyl-2,4-dinitroaniline (**V**) is the same as that of any methylene and methyl protons, we can calculate the chemical shift of the methylene protons in **III** as 3.36 ppm. Considering the ring strain, the value calculated for α methylene protons in **I** is 3.60 ppm. The difference between this value and the observed one (i.e., 3.21–3.60 = -0.39) shows a shielding of the protons in piperidine that could be attributed to the ring current produced by the electrons of benzene.²³ Therefore, the piperidine moiety must be rotated with respect to the main plane of the aromatic ring.

Table 6. Experimental ¹H NMR chemical shift of compounds (**I**) 1-piperidine-2,4-dinitrobenzene and (**V**) N,N-dimethyl-2,4-dinitrobenzene.

Atoms	I	V
H(3)	7.04	6.95
H(5)	8.19	8.25
H(6)	8.60	8.60
H α N ^a	3.21	3.06
H β N ^b	1.41	—
H γ N ^c	1.41	—

^a H(111), H(112), H(71), H(72).

^b H(101), H(102), H(81), H(82).

^c H(91), H(92).

UV spectra. The rotation of the *o*-NO₂ group out of the aromatic ring in solution can also be inferred from UV data. The UV spectrum of **I** shows only an absorption band at 374 nm ($\epsilon = 11500$). It is known that 2,4-dinitroaniline (**VI**)²⁴ in methanol shows two charge transfer (CT) UV bands: one at 336nm ($\epsilon = 14450$) and the other at 390nm ($\epsilon = 6460$), these CT bands correspond to the electronic transitions from the amino group to the *p*- (band 1) and *o*-nitro (band 2) groups, respectively. As some of us have shown in a previous paper, N-monoalkylsubstitution of 2,4-dinitroaniline essentially does not modify the UV spectra.³ This was in accordance with previous ¹H NMR predictions.²⁵ On the other hand N,N-dialkylation induces a bathochromic displacement and steric enhancement of the resonance of band 1, while steric inhibition of band 2 is observed.³ These results indicate that the electron withdrawing *ortho* substituent is twisted out of the phenyl ring in solution. Besides, if the amino group was rotated out of the ring plane the intensity of band 1 would be reduced. We found that in **I** band 1 shows a bathochromic displacement compared with **VI** and band 2 disappears completely indicating a rotation of the *o*-nitro group out of the plane of the aromatic ring. The rotation of piperidine out of this plane in **I** is inferred from the comparison of the value of ϵ of band 1 with that value in compound **VI**, as can be seen above. The examination of the values of λ_{\max} and ϵ of the CT band between the amino and *p*-nitro groups in **II** and **III** helps to analyze the **I** data. In fact, the bathochromic shift in the wavelength of maximum absorption (λ_{\max} (**I**) = 374, λ_{\max} (**II**) = 375 and λ_{\max} (**III**) = 380 nm) is in accordance with the steric enhancement of the resonance. The decrease in the intensity of the band 1 in **I** and **II** relative to **VI** is consistent with a decrease in the probability of the transition due to the noncoplanarity of the amino group with the phenyl ring.

Conclusions

The present X-ray single crystal analysis, UV, and ¹H and ¹³C NMR results allow a comparison of the conformation of **I** in solid state and in solution. The comparison led us to conclude that the main features of the molecular conformation do not change due to the packing forces that induce the chain formation.

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