

V. A. Tafeenko,^{a*} V. V. Chernyshev,^a A. V. Yatsenko,^a V. A. Makarov,^b E. J. Sonneveld,^c R. Peschar^c and H. Schenk^c

^aDepartment of Chemistry, Moscow State University, 119899 Moscow, Russia,

^bDepartment of Medicinal Chemistry, State Scientific Center 'NIOPIK', B. Sadovaya Str. 1-4, 103787 Moscow, Russia, and ^cLaboratory for Crystallography, Institute of Molecular Chemistry, University of Amsterdam, Nieuwe Achtergracht 166, Amsterdam 1018 WV, The Netherlands

Correspondence e-mail:

tafeenko@biocryst.phys.msu.ru

Intermolecular —CH₃ ··· O₂N— contacts in two polymorphic modifications of (1*E*)-*N*'-[(*E*)-2-cyano-1-(dimethylamino)-2-nitrovinyl]-*N,N*-dimethylethanamide

The title compound was synthesized and isolated in two crystal modifications. The structure of the orthorhombic modification was determined by the X-ray powder diffraction method and the structure of the monoclinic modification was determined using the X-ray single-crystal diffraction technique. The molecules in both polymorphs are *E,E* isomers. Intermolecular H₃C ··· NO₂ contacts and their role in the formation of the polymorphic modifications are analyzed.

Received 28 February 2003

Accepted 9 May 2003

1. Introduction

The title compound belongs to the class of highly polarized 'push-pull' enamines. These compounds are promising as starting reagents in the synthesis of various substituted heterocycles (Makarov, Sedov *et al.*, 1996). The chemical reactivity of these compounds depends to a large extent on the isomeric and conformational features of the molecules of interest. The determination of the configuration of a completely substituted ethylene fragment by spectral methods is a difficult task (Makarov *et al.*, 1996; Solov'eva *et al.*, 1997; Clark *et al.*, 1970).

Another problem that can hardly be solved using physical spectral methods is the determination of the type of possible *EE* ··· *ZZ* (Fig. 1) isomers of a compound.

Single-crystal X-ray diffraction allows the above problem to be solved most accurately. X-ray powder diffraction can also be applied, but the accuracy of the estimation of geometric parameters of a molecule by this method is rather low. Our attempts to obtain single crystals of the title compound were successful, but unit-cell parameters of the single crystals were different from those of the powder. The reason for this difference was not clear; therefore, an X-ray diffraction study was performed for both the powder and the single crystal. Results of both studies are reported in this paper.

2. Experimental and structure determination

2.1. Synthesis

A solution of 0.5 g (2.22 mmol) α -dimethylamino- β -cyano- β -nitroenamine in 1.5 ml (9.38 mmol) diethylacetal dimethylacetamide was refluxed for 2 h. The reaction mixture was cooled to room temperature and treated with 15 ml of boiled hexane. A white solid was filtered and crystallized from 10 ml of ethanol; m.p. 415–417 K. ¹H NMR (DMSO-*d*₆), δ : 3.11 (3H, s, NCH₃ enamine), 3.17 (3H, s, NCH₃ enamine), 3.13 (3H, s, NCH₃ amidine) and 2.07 (3H, s, CH₃). *M*⁺ (*m/z*) 225.

C₉H₁₅N₅O₂. Found (%): C 48.04, H 6.74, N 31.12; calculated (%): C 47.99, H 6.71, N 31.09.

Single crystals were obtained by slow evaporation from ethyl acetate solution; the substance used for the powder diffraction study was isolated from an ethanol solution.

2.2. Structure solution and refinement

Crystal data for the single-crystal (monoclinic, **M**) and powder diffraction (orthorhombic, **O**) studies are summarized in Table 1. The intensities of the reflections from a single crystal were collected at room temperature on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation with an ω -scan technique. Lattice constants were determined by diffractometer software and refined by least-squares fitting of the setting angles of 25 reflections. Intensities of two standard reflections were measured every 2 h and did not show significant variations. The structure was solved by direct methods and all other calculations were accomplished using the *SHELX97* (Sheldrick, 1990, 1997) programs. The structure was refined by a full-matrix least-squares method with anisotropic atomic displacement parameters for non-H atoms and isotropic parameters for the H atoms.

The unit-cell parameters for the **O** polymorph, which was available in the polycrystalline form only, were determined from the Guinier photograph with *TREOR90* (Werner *et al.*, 1985) and refined with *LSPAlD* (Visser, 1986) to $M_{20} = 23$ and $F_{30} = 47(0.010, 80)$. The space group *Pbca* was determined on the basis of the systematic extinctions. The unit-cell parameters and space group were tested further using Pawley's fit (Pawley, 1981) and confirmed by the crystal structure solution.

The X-ray powder diffraction pattern for structure solution and refinement was measured in the reflection mode on an X'Pert PRO X-ray powder diffraction system equipped with a PW 3050/60 standard resolution goniometer and PW 3011/20

Table 1

Experimental table.

Single crystal experiment	
Chemical formula	C ₉ H ₁₅ N ₅ O ₂
Crystal system, space group	Monoclinic, <i>P2₁/c</i>
Formula weight	225.26
Temperature (K)	303 (2)
Unit-cell dimensions:	
<i>a</i> (Å)	8.844 (1)
<i>b</i> (Å)	16.436 (2)
<i>c</i> (Å)	8.926 (1)
β (°)	114.8 (2)
<i>V</i> (Å ³)	1177.7 (4)
<i>Z</i> , calculated density (g m ⁻³)	4, 1.270
Wavelength (Å)	Mo $K\alpha$, 0.70926
θ range for data collection (°)	1.33–25.98
Limiting indices	–10 \Rightarrow <i>h</i> \Rightarrow 9 0 \Rightarrow <i>k</i> \Rightarrow 20 0 \Rightarrow <i>l</i> \Rightarrow 10
No. of measured reflections	
No. of reflections used in refinement ($I > 2\sigma$)	1343
No. of parameters used in refinement	206
<i>S</i> (goodness-of-fit on F^2)	0.980
Final <i>R</i> [$I > 2\sigma(I)$]	0.0540
Powder diffraction experiment	
Chemical formula	C ₉ H ₁₅ N ₅ O ₂
Crystal system, space group	Orthorhombic, <i>Pbca</i>
<i>M_r</i>	225.26
Temperature (K)	303 (2)
Unit-cell dimensions:	
<i>a</i> (Å)	11.627 (5)
<i>b</i> (Å)	30.77 (2)
<i>c</i> (Å)	6.642 (4)
<i>V</i> (Å ³)	2376 (2)
<i>Z</i> , calculated density (g m ⁻³)	8, 1.268
Wavelength (Å)	Cu $K\alpha_1$, 1.5406
2θ (°)	5–70
2θ step (°)	0.01
χ^2	1.7
<i>R</i> _{wp}	0.12
<i>R</i> _{exp}	0.09

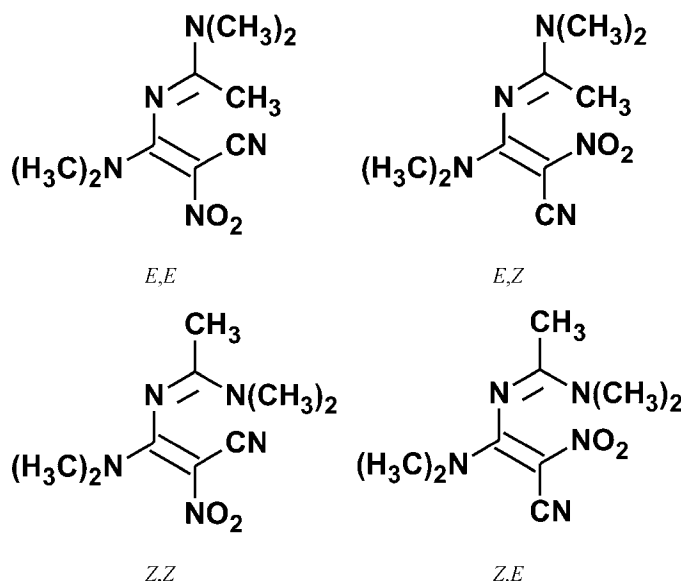


Figure 1

Four possible isomers of the title compound. The *E,E*-isomer was found in both polymorphic modifications.

proportional point detector; Cu $K\alpha_1$ radiation was used. To avoid the preferred orientation the powder was sprinkled onto the sample holder using a small sieve. The thickness of the sample was less than 0.1 mm. During the exposure, the sample was spun in its plane to improve particle statistics. The structure of the orthorhombic polymorph was solved by the grid-search procedure (Chernyshev & Schenk, 1998) on the basis of the powder diffraction data. Four possible molecular structures – *EE*, *EZ*, *ZZ* and *ZE* (Fig. 1) – were tested with the grid search and a solution was unambiguously found for the *EE* configuration. The conformation of the initial model changed slightly during the subsequent bond-restrained Rietveld refinement with the *MRIA* program (Zlokazov & Chernyshev, 1992). The strength of the restraints was a function of interatomic separation and for intramolecular bond lengths corresponded to an r.m.s. deviation of 0.03 Å. H atoms were placed in geometrically calculated positions and allowed to refine using bond restraints, with a common isotropic displacement parameter U_{iso} fixed at 0.05 Å². The diffraction profiles and the differences between the observed and calculated profiles are shown in Fig. 2. An *ORTEPII* (Johnson, 1976) diagram of the **M** (monoclinic) molecule showing the numbering scheme used for **M** and **O** molecules is presented in

Table 2

Torsion angles ($^{\circ}$) for the molecules in the **M** and **O** phases.

	Angle, M	Angle, O
C6–N4–C3–N1	10.9 (4)	2 (4)
C6–N4–C3–C4	–173.7 (3)	–178 (2)
C3–N1–C1–C2	40.9 (4)	49 (5)
C3–N1–C1–N3	–146.4 (3)	–137 (4)
C8–N3–C1–N1	18.5 (4)	12 (5)
C8–N3–C1–C2	–169.0 (3)	–175 (3)
C9–N3–C1–C2	17.0 (4)	11 (5)
C9–N3–C1–N1	–155.5 (3)	–163 (3)
O1–N2–C2–C1	–169.0 (3)	–175 (3)
O1–N2–C2–C5	–163.8 (2)	–171 (2)

Fig. 3. The crystal packing of **M** (Fig. 4) and **O** (Fig. 5) structures were analysed with the program *PLUTON92* (Spek, 1992).

3. Results and discussion

The studies have revealed that the molecule has an *E,E* configuration in both polymorphic modifications (Fig. 1). The differences in the molecular geometry parameters in the **M** and **O** phases are insignificant and apparently arise from the packing effects (see Fig. 3, Tables 2 and 3).

The values of the torsion angles and bond lengths (Tables 2 and 3) in the molecules indicate that π -electron density of the nitro group is strongly conjugated through the double C1=C2 bond with the lone electron pair of the nitrogen atom N3 (π - π - n conjugation). On the other hand, the planarity of the dimethylamino groups (CH₃)₂N4 (the sums of the bond angles at the N4 atoms are equal to 359.8 and 360 $^{\circ}$ for the **M** and **O** crystals, respectively) and the value of the torsion angles C6–N4–C3–N1 10.9 (4), 2 (4) $^{\circ}$ (for **M** and **O**) as well as the short bond N4–C3 1.317 (3), 1.32 (3) Å indicate the transfer of a lone pair from N4 to N1 (n - π - n conjugation). When there are two electron pairs located on the N1 atom the direction of one of them is orthogonal to the C1=C2 π -system and conse-

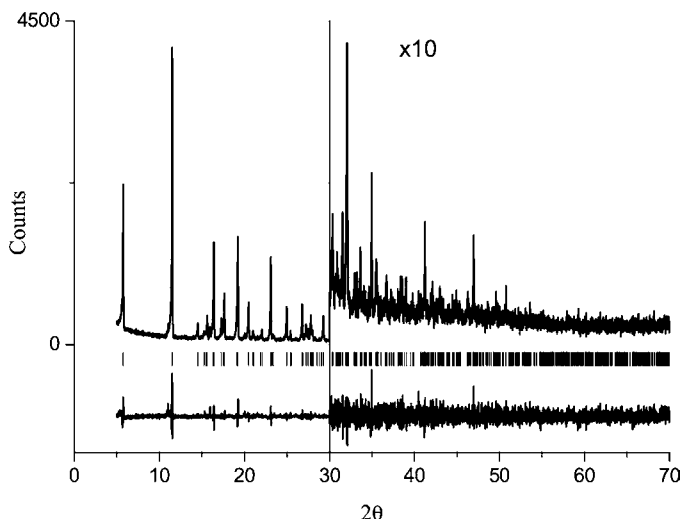


Figure 2

The Rietveld plot showing the observed and difference profiles for **O**. The reflection positions are shown above the difference profiles.

Table 3

Selected angles ($^{\circ}$) and bonds (Å) for molecules in **M** and **O** phases.

	Angle, M	Angle, O	Bond, M	Bond O
C3–N1–C1	122.1 (2)	122 (3)	O1–N2	1.250 (3)
O2–N2–O1	121.3 (2)	122 (2)	O2–N2	1.245 (3)
O2–N2–C2	119.1 (2)	118 (3)	N1–C3	1.324 (3)
O1–N2–C2	119.6 (2)	120 (2)	N1–C1	1.342 (3)
C1–N3–C9	123.2 (2)	124 (2)	N2–C2	1.377 (3)
C1–N3–C8	120.2 (2)	119 (3)	N3–C1	1.335 (3)
C9–N3–C8	116.3 (3)	117 (2)	N3–C9	1.458 (4)
C3–N4–C6	120.5 (3)	120 (2)	N3–C8	1.462 (4)
C3–N4–C7	123.1 (3)	124 (2)	N4–C3	1.317 (3)
C6–N4–C7	116.4 (3)	116 (2)	N4–C6	1.458 (4)
N3–C1–N1	117.8 (2)	119 (3)	N4–C7	1.466 (4)
N3–C1–C2	122.0 (2)	122 (4)	N5–C5	1.146 (3)
N1–C1–C2	119.7 (2)	119 (4)	C1–C2	1.441 (3)
N2–C2–C5	115.9 (2)	118 (2)	C2–C5	1.405 (3)
N2–C2–C1	123.7 (2)	122 (3)	C3–C4	1.496 (4)

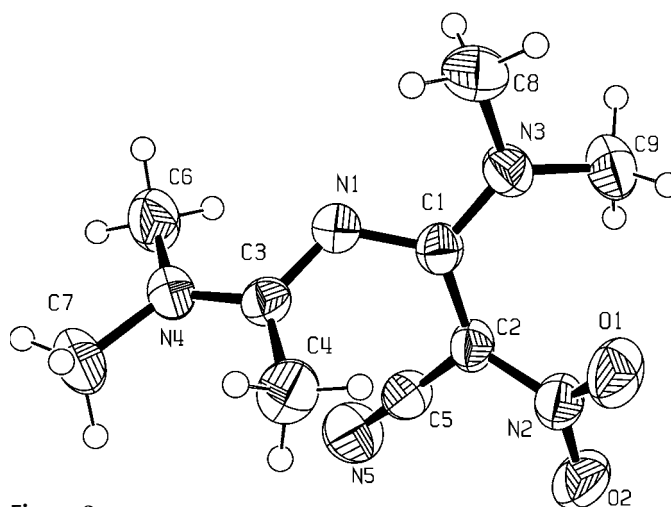


Figure 3

ORTEPII (Johnson, 1976) diagram of the **M** molecule, showing the numbering scheme used in this work for **M** and **O** molecules. Thermal ellipsoids are drawn at the 50% probability level.

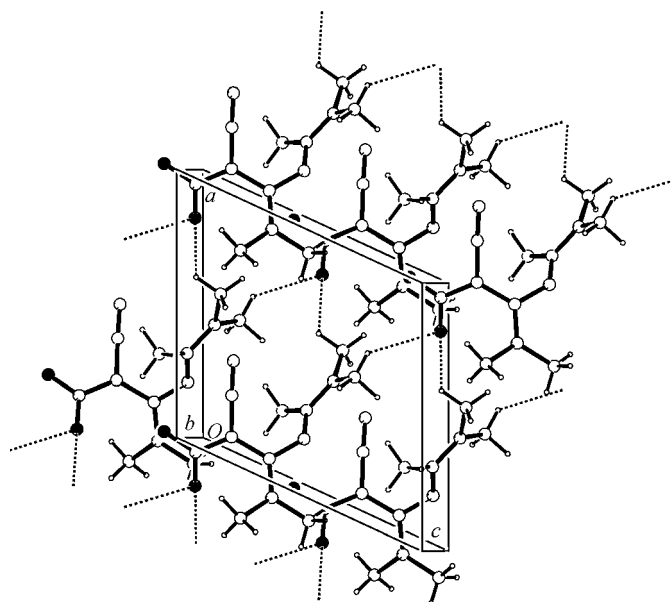


Figure 4

Molecular packing of **M** along the *b* axis (O atoms in black).

Table 4
Crystal field effects on charge distribution in the **M** and **O** molecules.

Fragment	Monoclinic M		Orthorhombic O	
	Isolate	In the crystal	Isolate	In the crystal
$q(\text{Me}_2\text{N3})$	+0.236	+0.297	+0.277	+0.336
$q(\text{Me}_2\text{N4})$	+0.214	+0.260	+0.197	+0.240
$q(\text{C2NO}_2\text{CN})$	-0.808	-0.901	-0.828	-0.934
$q(\text{O1})$	-0.461	-0.477	-0.469	-0.479
$q(\text{O2})$	-0.406	-0.473	-0.419	-0.490
$\mu(\text{D})$	10.87	13.06	11.26	14.03

quently the electron density from the N1 atom may effectively be transferring through $\text{C1}=\text{C2}$ to the nitro group ($n-\pi-\pi$ conjugation). Note that the $\text{N2}-\text{C2}$ bond length in **M** is 1.377 (3) Å, among the shortest ones observed for the nitro groups connected to a π -system (Allen & Kennard, 1993).

It is evident from our findings that the O atoms of the nitro group bear the excess negative charge and the N3 and N4 atoms bear the positive charge, which should enhance the acidity of the methyl protons in the dimethylamino fragments. The charges of the fragments considered were calculated by the quantum-chemical method using the semi-empirical AM1 Hamiltonian for the molecules in the isolated form and in the crystal field (Yatsenko & Paseshnichenko, 2000; Table 4). The calculated charges completely support the above conclusions. The calculations revealed that the molecule studied has a considerable dipole moment ($\mu \simeq 11 \text{ D}$). On going from the isolated molecules to crystals, the dipole moments of the molecules in both modifications increase and the charge transfer to the acceptor is enhanced. In the crystal form, characteristic of the isolated molecules, the charges at the O1 and O2 (see Fig. 3) atoms actually become identical. This shows the role of the intermolecular contacts in the stabilization of the intramolecular charge transfer. It should also be noted that in both crystal polymorphs the excess positive charge at $\text{Me}_2\text{N3}-$ increases more and is larger than at $\text{Me}_2\text{N4}-$ (by 0.061 and 0.046 in **M**, and 0.059 and 0.043 in **O**, Table 4) when compared with the isolated molecules.

A large dipole moment of the molecule and the atomic charge distribution can provide insight into the intermolecular interactions in the crystals which are responsible for the formation of different polymorphic modifications. Evidently, the negatively charged O atoms of the nitro group are able to interact with the H atoms of the methyl groups at the N3 or/and N4 atoms of neighboring molecules. The crystal structure of the polymorphic modifications reported here support this conclusion.

Considering the intermolecular contacts, we assume the van der Waals radii to be: H 1.2, O 1.52, N 1.55 Å (Bondi, 1964). For **M** (Fig. 4) molecules the shortest contacts $\text{C7}-\text{H7}\cdots\text{O1}^{\text{i}}$ and $\text{C6}-\text{H61}\cdots\text{O1}^{\text{ii}}$ are smaller than the sum of the radii contact for O and H atoms assumed above: 2.42 (4) Å, the angle 123° and 2.70 (4) Å, 128 (3)°, respectively (Table 5). That is, the molecules are arranged in a 'head-to-tail' manner and the O1 atom of the nitro group is attached to the atoms of the methyl groups at the N4 atom of the neighboring mole-

cules. These interactions result in the formation of layers. In Fig. 4 only one layer is shown; the other layer is related by a center of symmetry.

A more intricate system of the shortest intermolecular contacts was revealed in the orthorhombic crystal. As in the case of the monoclinic crystal, only the $\text{O}\cdots\text{H}$ contacts that are less than 2.72 Å were considered. In the orthorhombic modification, compared with the monoclinic form, the O atoms of the nitro group form short contacts with the methyl groups at the N3 atom, namely, $\text{C8}-\text{H8}\cdots\text{O1}^{\text{iv}}$ 2.59 Å, angle 164°, $\text{C8}-\text{H81}\cdots\text{O2}^{\text{v}}$ 2.60 Å, angle 169°, and $\text{C9}-\text{H92}\cdots\text{O2}^{\text{vi}}$ 2.58 Å, angle 128° (see Table 5).

The data obtained support the conclusion that the Coulombic interactions are essential, possibly even decisive, for the packing of the molecules in question. This conclusion is supported by the data available in the literature. We analyzed the data on the molecules that contain the dimethylamino- and nitro- groups linked by a π -system.

A search of the Cambridge Structural Database (Allen & Kennard, 1993) has been performed for the fragments shown in Fig. 6. The packings of all the structures found, for which the coordinates are available in the Database, were analyzed to establish the shortest (<2.72 Å) intermolecular contacts

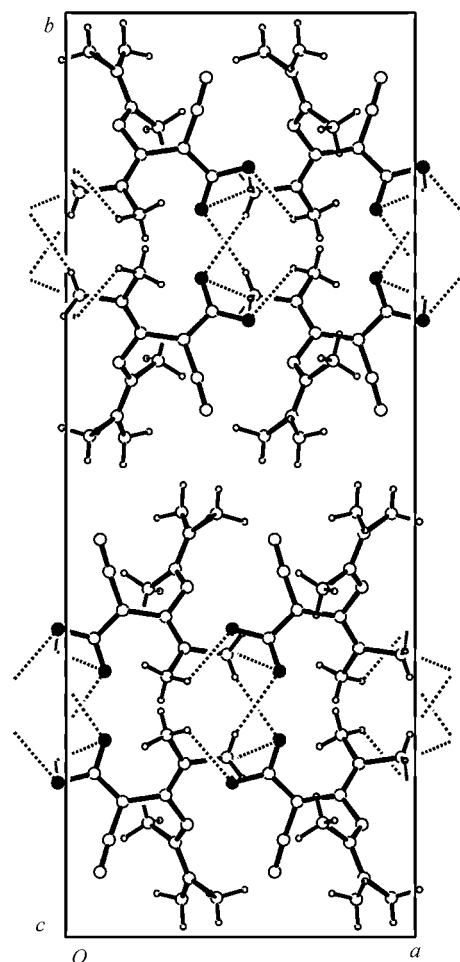


Figure 5
Molecular packing of **O** along the *c* axis (O atoms in black).

Table 5
Selected (<2.72 Å) intra- and intermolecular C—H···N,O contacts (Å, °) for **M** and **O**.

<i>D</i> —H··· <i>A</i>	H··· <i>A</i> (Å)	<i>D</i> —H··· <i>A</i> (°)	<i>D</i> —H··· <i>A</i>	H··· <i>A</i> (Å)	<i>D</i> —H··· <i>A</i> (°)
C9—H92···O1	2.49 (4)	101 (3)	C9—H9···O1	2.65	95
C8—H8···N1	2.44 (4)	94 (4)	C8—H8···N1	2.58	85
C6—H62···N1	2.38 (5)	99 (4)	C8—H82···N1	2.64	82
C7—H7···O1 ⁱ	2.42 (4)	123 (3)	C6—H62···N1	2.44	93
C6—H61···O1 ⁱⁱ	2.70 (4)	128 (3)	C7—H7···N5 ⁱⁱⁱ	2.67	166
C8—H8···O1 ^{iv}	2.59	164			
C8—H81···O2 ^v	2.60	169			
C9—H92···O2 ^{vi}	2.58	128			

Symmetry codes: (i) $x+1, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x+1, y, z+1$; (iii) $-x+\frac{1}{2}, -y, z-\frac{1}{2}$; (iv) $\frac{1}{2}+x, y, \frac{3}{2}-z$; (v) $\frac{1}{2}+x, y, -z-\frac{1}{2}$; (vi) $\frac{1}{2}+x, y, -z+\frac{1}{2}$.

Table 6
Bond lengths (Å) and the number of —N(CH₃)₂···O₂N— intermolecular contacts in the molecules deposited at Cambridge Structural Database.

Refcodes (1a)	Numbers and lengths (Å) of the contacts	Refcode (1b)	Numbers and lengths (Å) of the contacts
FAWJIJ	(3), 2.54–2.69	CAVJOY	(2), 2.41, 2.49
FNDMAN	(2), 2.27, 2.66	DUPHIF	(3), 2.60–2.64
GAHKAB02	(1), 2.69	KOVGIL	(7), 2.49–2.70
GIMRUP10	(1), 2.47	MNETAM	(2), 2.53–2.62
JUPRIV	(4), 2.54–2.60	MNETAM01	(2), 2.60, 2.69
TACWID*	(0)	—	—
TEWHOS	(6), 2.39–2.71		
TEWHUY	(1), 2.66	Refcode (1c)	
ZOSKEX	(1), 2.67	MANCPP	(3), 2.56–2.60
TEWJEK	(2), 2.60, 2.66		
ZOSKAT**	(1), 2.75		
ZUHMUK	(1), 2.63		

between the H atoms of the dimethylamino group and the O atom of the nitro group. The results of the study are summarized in Table 6.

The required —CH₃···O₂N— contact was found in 17 compounds (95% of the total number). The crystal structures with an asterisk in Table 6 (refcodes ZOSKAT and TAKWID) require a more detailed consideration. Under the ZOSKAT refcode, the data on the crystal structure of 4-nitro-1,8-bis(dimethylamino)naphthalene (C₁₄H₁₇N₃O₂) have been deposited.¹ Two dimethylamino groups occupy the *peri* positions relative to each other (positions 1 and 8 of naphthalene) and the steric interactions between them result in a decrease in conjugation of the dimethylamino group with the nitro group through the π-system of the naphthalene ring. Actually, the torsion angles that characterize the rotation of the methyl groups relative to the naphthalene bicycle are 17 and 38°, the coordination of the N atom becomes pyramidal and, as a result, the transfer of the electron density to the nitro group decreases. Nevertheless, a contact of 2.75 Å and the head-to-tail (nitro group-to-dimethylamino group) arrangement of molecules allow this compound to be included in the list whose molecules form the required contacts. Note that some authors considering the —O···H—C— hydrogen bonds tolerate the

¹ Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV0064). Services for accessing these data are described at the back of the journal.

contacts which are even larger than 2.72 Å. For example, the criteria used by Steiner (1996) are H···O < 2.8 Å and the angle at the H atom > 90°. The increase in the H···O distance to 2.75 Å and the NO₂ and dimethylamino group conjugation decrease provides more support for the importance of charge transfer in the molecules of this type to the formation of the C—H···O interactions. In this connection the absence of the required contacts in the structure with the TAKWID refcode is understood. Under this refcode, the data on the crystal structure of 1-amino-*N*-(2-dimethylamino)-3,5-dinitrophenylcyclopentanocarboxamide (C₁₄H₁₉N₅O₅) are deposited.

Under the influence of two *ortho* substituents, the dimethylamino groups are rotated relative to the benzene ring (the rotation angles of the methyl groups are 38 and 77°) even to a larger degree than in the ZOSCAT molecule, which completely eliminates the possibility of conjugation of the nitro group and the lone electron pair of the N atom [the absence of conjugation is evident in particular from the O₂N—C and (CH₃)₂N—C bond lengths, which are 1.47 and 1.41 Å]. Thus, the TAKWID molecule does not belong to highly polarized ('push-pull') organic molecules, although it contains the required functional groups. On the whole, the data obtained agree with one of the conclusions of Taylor & Kennard (1982): 'The tendency of the H(C) to form a short contact with the oxygen atom increases if C is bonded to an N atom; this tendency is most pronounced for a C atom directly adjacent to N⁺ atom'.

Knowledge of the donating-withdrawing properties of the substituents allows the most probable intermolecular contacts in the crystal to be evaluated. The —NO₂···H₃C— contacts discussed in this paper provide an example of such interactions. Strictly speaking, the nature of these interactions is ambiguous: is it a purely Coulomb interaction or do other components of the hydrogen bond make their contributions? According to Singh & Kollman (1985), a hydrogen bond includes generally different types of interactions, but in the range of medium and weak hydrogen bonds the Coulomb interaction is the most important and essential component.

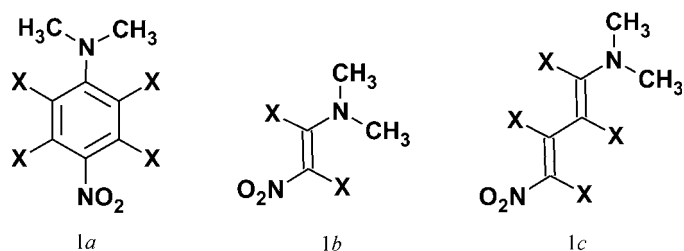


Figure 6
The molecular fragments used in the search of the Cambridge Structural Database.

This work has been supported by The Netherlands Organization for Scientific Research, NWO and Russian Fund for Basic Research (RFBR); licence payment for using the Cambridge Structural Database: project 02-07-90322.

References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441.
- Chernyshev, V. V. & Schenk, H. (1998). *Z. Kristallogr.* **213**, 1–3.
- Clark, J., Gelling, I., Southon, I. W. & Morton, M. S. (1970). *J. Chem. Soc.* **3**, 498–502.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Makarov, V. A., Sedov, A. L., Anisimova, O. S. & Granik, V. G. (1996). *Chem. Heterocycl. Compds*, **6**, 811–820 (in Russian).
- Pawley, G. S. (1981). *J. Appl. Cryst.* **14**, 357–361.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Gottingen, Germany.
- Singh, U. C. & Kollman, P. A. (1985). *J. Chem. Phys.* **83**, 4033–4040.
- Solov'eva, N. P., Makarov, V. A. & Granik, V. G. (1997). *Chem. Heterocycl. Compds*, **1**, 89–98. (In Russian.)
- Spek, A. L. (1992). *PLUTON92*. University of Utrecht, The Netherlands.
- Steiner, T. (1996). *Cryst. Rev.* **6**, 1–57.
- Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.
- Visser, J. W. (1986). *Powder Diffr.* **1**, 66–76.
- Werner, P. E., Eriksson, L. & Westdahl, M. (1985). *J. Appl. Cryst.* **18**, 367.
- Yatsenko, A. V. & Paseshnikchenko, K. A. (2000). *Chem. Phys.* **262**, 292–301.
- Zlokazov, V. B. & Chernyshev, V. V. (1992). *J. Appl. Cryst.* **25**, 447–451.