Table 2 (*cont.*)

C5*A*—C12*A*—C13*A*	128·1 (4)	O2*A*—C13*A*—C12*A*	60·3 (3)
O3*A*—C15*A*—C6*A*	108·9 (3)	O3*A*—C17*A*—O4*A*	124·6 (4)
O3*A*—C17*A*—C18*A*	110·3 (5)	O4*A*—C17*A*—C18*A*	125·1 (5)
O5*A*—C19*A*—O6*A*	122·2 (5)	O5*A*—C19*A*—C20*A*	111·1 (5)
O6*A*—C19*A*—C20*A*	126·7 (6)	O8*A*—C21*A*—O9*A*	120·2 (6)
O8*A*—C21*A*—C22*A*	111·8 (6)	O9*A*—C21*A*—C22*A*	128·0 (6)
C21*A*—C22*A*—C23*A*	115·3 (6)	C22*A*—C23*A*—C24*A*	113·4 (7)
C22*A*—C23*A*—C25*A*	113·5 (8)	C24*A*—C23*A*—C25*A*	104·1 (7)

in other mycotoxins: isororidin E (Flippen-Anderson & Gilardi, 1986), mycotoxin A and B, (Jarvis, Cömezoğlu, Lee, Flippen-Anderson, Gilardi & George, 1986) and verrucarin A (McPhail & Sim, 1966). The major feature of the packing is the presence of three-centered intermolecular hydrogen bonding in which O7 acts as a donor to both O1*A* ($x-1, y, z$) and O7*A* ($x-1, y, z$). Hydrogen bonding also occurs when O7*A* acts as a donor to O2 ($x+0{\cdot}5,\ 1{\cdot}5-y,\ 2-z$). The respective hydrogen bond parameters for H···O, O···O, and ∠O—H···O are 2·30 (4), 2·20 (4), 1·88 (4) Å; 2·97 (1), 2·89 (1), 2·74 (1) Å; and 134 (3), 136 (3) and 159 (3)° respectively. No other contacts less than van der Waals distances occur.

This work was supported in part by USAMRIID contract No. 85MM5511 and the Office of Naval Research.

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Acta Cryst. (1990). C**46**, 648–650

Structure of Ethyl (2,4,6-Trinitrophenyl)carbamate

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(*Received* 17 *August* 1988; *accepted* 31 *July* 1989)

Abstract. $C_9H_8N_4O_8$, $M_r = 300{\cdot}21$, monoclinic, $P2_1$, $a = 4{\cdot}978\ (2)$, $b = 6{\cdot}974\ (2)$, $c = 17{\cdot}991\ (6)$ Å, $\beta = 94{\cdot}69\ (3)°$, $V = 622{\cdot}7\ (3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}60$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}134$ mm^{-1}, $F(000) = 308$, $T = 225$ K, final $R = 0{\cdot}058$, $wR = 0{\cdot}063$ for 769 independent reflections. This trinitroaromatic compound forms an *intermolecular* hydrogen bond rather than a possible *intramolecular* hydrogen bond which would constrain one nitro group to be coplanar with the phenyl ring. The molecules form a hydrogen-bonded chain along the *a* axis.

Introduction. The crystal structure of the title compound was determined as part of an ongoing study of structure–reactivity relationships in polynitroaromatic compounds. Such compounds find application as propellants and explosives. A combination of quantum-mechanical (INDO) calculations and structural parameters (C—NO_2 distances) has been used (Delpuech & Cherville, 1979) to correlate sensitivity of explosives. Hence, structural data are needed to test the validity of this and other structure–reactivity correlations.

Experimental. A clear, colorless 0·03 × 0·22 × 0·48 mm data crystal from material supplied by Dr Paul Bolduc, Eglin Air Force Base. Automated Nicolet *R*3*m* diffractometer with incident beam monochromator. 25 centered reflections within $21 \le 2\theta \le 30°$ used for determining lattice parameters. $[\sin(\theta)/\lambda]_{\max} = 0{\cdot}54$ Å^{-1}, range of *hkl*: $0 \le h \le 5$, $-7 \le k \le 0$, $-19 \le l \le 19$. Standards $\bar{1}\bar{1}0$, $\bar{1}\bar{3}0$, 006 monitored every 100 reflections showed no decrease in intensity. θ–2θ mode, θ scan width 0·9°, θ scan rate a function of count rate, 3° min^{-1} minimum, 15°

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Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* (Å^2 × 10^3)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	4752 (18)	6210 (15)	2355 (4)	29 (2)
C(2)	2814 (18)	6551 (16)	1757 (5)	25 (2)
N(2)	1263 (14)	8359 (15)	1701 (4)	29 (2)
O(2*a*)	− 1154 (13)	8224 (13)	1493 (3)	46 (2)
O(2*b*)	2422 (14)	9839 (13)	1831 (4)	48 (2)
C(3)	2389 (18)	5291 (15)	1163 (4)	29 (2)
C(4)	3991 (18)	3707 (15)	1150 (5)	28 (2)
N(4)	3551 (18)	2347 (16)	520 (4)	42 (2)
O(4*a*)	5338 (17)	1187 (16)	429 (4)	66 (2)
O(4*b*)	1400 (15)	2483 (14)	155 (4)	61 (2)
C(5)	6017 (16)	3328 (15)	1707 (4)	24 (2)
N(6)	8193 (15)	3953 (14)	2933 (4)	30 (2)
C(6)	6259 (16)	4544 (15)	2310 (4)	22 (2)
O(6*a*)	10275 (14)	3175 (13)	2776 (3)	45 (2)
O(6*b*)	7568 (13)	4243 (14)	3572 (3)	45 (2)
N(7)	5150 (16)	7522 (14)	2950 (3)	27 (2)
C(8)	3136 (18)	8243 (17)	3310 (4)	27 (2)
O(8)	736 (13)	7812	3183 (3)	37 (2)
O(9)	4079 (11)	9491 (13)	3828 (3)	30 (1)
C(10)	2084 (19)	10269 (17)	4295 (5)	38 (2)
C(11)	3479 (23)	11809 (19)	4765 (6)	54 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·405 (12)	C(1)—C(6)	1·390 (14)
C(1)—N(7)	1·409 (12)	C(2)—N(2)	1·477 (14)
C(2)—C(3)	1·387 (13)	N(2)—O(2*a*)	1·235 (9)
N(2)—O(2*b*)	1·196 (13)	C(3)—C(4)	1·364 (14)
C(4)—N(4)	1·480 (13)	C(4)—C(5)	1·388 (11)
N(4)—O(4*a*)	1·224 (14)	N(4)—O(4*b*)	1·213 (11)
C(5)—C(6)	1·375 (12)	N(6)—C(6)	1·475 (10)
N(6)—O(6*a*)	1·223 (11)	N(6)—O(6*b*)	1·231 (10)
N(7)—C(8)	1·336 (12)	C(8)—O(8)	1·235 (11)
C(8)—O(9)	1·333 (12)	O(9)—C(10)	1·458 (12)
C(10)—C(11)	1·501 (16)		
C(2)—C(1)—C(6)	115·9 (8)	C(2)—C(1)—N(7)	121·1 (9)
C(6)—C(1)—N(7)	123·0 (7)	C(1)—C(2)—N(2)	121·2 (8)
C(1)—C(2)—C(3)	122·3 (9)	N(2)—C(2)—C(3)	116·3 (7)
C(2)—N(2)—O(2*a*)	116·5 (9)	C(2)—N(2)—O(2*b*)	118·8 (7)
O(2*a*)—N(2)—O(2*b*)	124·6 (10)	C(2)—C(3)—C(4)	118·3 (8)
C(3)—C(4)—N(4)	118·8 (8)	C(3)—C(4)—C(5)	122·2 (8)
N(4)—C(4)—C(5)	118·9 (9)	C(4)—N(4)—O(4*a*)	117·5 (8)
C(4)—N(4)—O(4*b*)	115·7 (9)	O(4*a*)—N(4)—O(4*b*)	126·8 (9)
C(4)—C(5)—C(6)	117·7 (9)	C(6)—N(6)—O(6*a*)	117·5 (7)
C(6)—N(6)—O(6*b*)	117·7 (7)	O(6*a*)—N(6)—O(6*b*)	124·8 (7)
C(1)—C(6)—C(5)	123·3 (7)	C(1)—C(6)—N(6)	120·8 (7)
C(5)—C(6)—N(6)	115·9 (8)	C(1)—N(7)—C(8)	123·2 (8)
N(7)—C(8)—O(8)	124·9 (9)	N(7)—C(8)—O(9)	110·5 (8)
O(8)—C(8)—O(9)	124·6 (8)	C(8)—O(9)—C(10)	115·4 (7)
O(9)—C(10)—C(11)	106·6 (8)		

min^{-1} maximum, 1014 reflections measured, 856 unique, $R_{int} = 0{\cdot}016$, 769 observed with $F_o > 3\sigma(|F_o|)$. Data corrected for Lorentz and polarization but not absorption effects. Structure solved by direct methods. Least-squares refinement using *SHELXTL*80 (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}000225$. No correction for secondary extinction was made. Twin lattices, unequal in intensity and related by a twofold rotation about **a**, were observed. Data were collected from the stronger twin lattice. Because of definite twin overlaps on the $h = 0$ layer, six interlayer (on h) scale factors were refined. Their relative values, for $h = 0,1...,5$ were, respectively, 1·000, 0·769, 0·788, 0·761, 0·777 and 1·061. 197 parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms; position refined for H(7*n*), while its U_{iso} was set to $1{\cdot}1U_{eq}$(N7); other H atoms included using riding model (coordinate shifts of C applied to attached H atoms, methyl group initially staggered, but allowed to rotate), C—H = 0·96 Å, H—C—H = 109·5°, U(H) = $1{\cdot}1U_{eq}$(C). $(\Delta/\sigma)_{max} = 0{\cdot}01$, $R = 0{\cdot}058$, $wR = 0{\cdot}063$, $S = 2{\cdot}36$. Final difference Fourier excursions 0·27 and $-0{\cdot}33$ e Å^{-3}. No attempt was made to determine the enantiomorph. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Atom numbering for Table 1,* atom coordinates, and Table 2, bond distances and angles, follows that shown in Fig. 1.

Discussion. The molecules hydrogen bond (N—H···O) to one another parallel to the *a* axis. Thus,

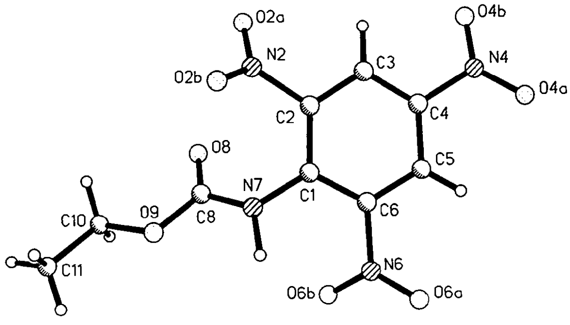

Fig. 1. Perspective drawing of the title compound.

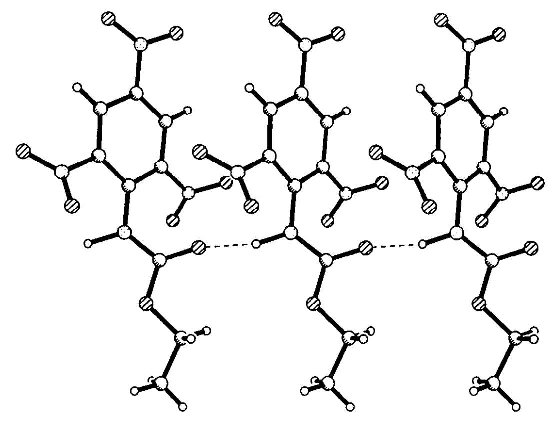

Fig. 2. Intermolecular N—H···O hydrogen bonding (dashed) parallel to the *a* axis.

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* 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 52469 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

H(7*n*) of a molecule centered at x,y,z hydrogen bonds to O(8) of a molecule centered at $1 + x,y,z$. This interaction [H···O, 1·71 (8) Å, N···O, 2·785 (8) Å; N—H···O, 160·4°] is shown by a dashed line in Fig. 2. Though the molecule could form a six-membered ring [C(1), C(6), N(6), O(6*b*), H(7*n*), N(7)] by hydrogen bonding intramolecularly, a close intermolecular association is adopted in this crystal form. In fact, H(7*n*) points 'above' the average molecular plane whereas O(6*b*) points 'below' the molecular plane, giving an H(7*n*)···O(6*b*) intramolecular distance of 2·47 Å.

The N(nitro)—C distances are all 1·48 (1) Å and are significantly longer than the N(7)—C(1) (amide—phenyl ring) linkage [1·41 (1) Å], consistent with previous observations that C—NO_2 distances tend to be longer than C—N(amine) bonds to aromatic rings (Ammon & Prasad, 1985; Cady & Larson, 1965).

The nitro groups display a range of torsion angles relative to the aromatic ring. For the N(2), N(4) and N(6) nitro groups, the signed twist angles [averaged torsion angles defined by Winkler & Dunitz (1971)] are +44·2 (5), −16·5 (5) and +37·2 (5)°, respectively. Though the torsion angle of N(4) is small enough to consider a cross-ring resonance with the amide nitrogen, the twist angle of the amide moiety relative to the phenyl ring is +47·8°, making its resonance with the ring unlikely. The similarity of the ring—nitro group distances also argues against any appreciable resonance effects. Indeed, resonance of the nitro group of nitrobenzene, which is coplanar with the benzene ring, has recently been challenged (Politzer, Lane, Jayasuriya & Domelsmith, 1987).

The authors thank the Air Force Armament Laboratory, Energetic Materials Branch, for support of this work.

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Acta Cryst. (1990). **C46**, 650–652

Structure of Ethyl (3-Methyl-2,4,6-trinitrophenyl)carbamate Dimethyl Sulfoxide Solvate

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(*Received* 17 *August* 1988; *accepted* 31 *July* 1989)

Abstract. $C_{10}H_{10}N_4O_8.C_2H_6OS$, $M_r = 392{\cdot}38$, triclinic, $P\bar{1}$, $a = 8{\cdot}516\ (2)$, $b = 9{\cdot}688\ (2)$, $c = 11{\cdot}895\ (3)$ Å, $\alpha = 73{\cdot}43\ (2)$, $\beta = 80{\cdot}78\ (2)$, $\gamma = 72{\cdot}07\ (2)°$, $V = 892{\cdot}1\ (4)$ Å^3, $Z = 2$, $D_x = 1{\cdot}461$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}224$ mm^{-1}, $F(000) = 408$, $T = 275$ K, final $R = 0{\cdot}051$, $wR = 0{\cdot}050$ for 1821 independent reflections. The title compound is hydrogen bonded through the amide hydrogen to the dimethyl sulfoxide (DMSO) molecule. The molecule is virtually identical to ethyl (2,4,6-trinitrophenyl)carbamate except for (*a*) the intermolecular hydrogen bonding and (*b*) the methyl group of the title compound which significantly distorts the intra-annular angle of the aromatic carbon to which it is bonded.

Introduction. Polynitroaromatic compounds are important as high-energy materials (propellants and explosives). They have also received attention since the nitro groups can cause significant distortions of aromatic rings from idealized structures. Though chemically very similar to the previously reported (Dudis & Gilardi, 1990) ethyl (2,4,6-trinitrophenyl)carbamate (II), the title compound (I) has a

0108-2701/90/040650-03$03.00