Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
S1	0.4404 (1)	0.1225 (1)	0.4206 (1)	1.78 (3)
S2	0.5195 (1)	0.4646 (1)	0.68442 (9)	1.98 (3)
S3	0.8183 (1)	0.7628 (1)	0.68650 (9)	1.55 (2)
Si1	0.2207 (1)	−0.1051 (1)	0.3261 (1)	1.56 (3)
Si2	0.9075 (1)	1.0361 (1)	0.7938 (1)	1.42 (3)
C1	0.1244 (5)	−0.1965 (4)	0.4025 (4)	2.1 (1)
C2	0.0480 (6)	−0.3166 (5)	0.3340 (4)	3.1 (1)
C3	−0.0232 (5)	−0.3896 (4)	0.3993 (4)	2.7 (1)
C4	−0.0934 (6)	−0.5107 (5)	0.3280 (5)	4.7 (2)
C5	0.3443 (5)	−0.1905 (4)	0.2531 (4)	2.3 (1)
C6	0.4176 (5)	−0.1354 (4)	0.1758 (4)	2.5 (1)
C7	0.5215 (5)	−0.2054 (5)	0.1236 (5)	3.4 (1)
C8	0.4703 (6)	−0.3295 (5)	0.0515 (5)	4.9 (2)
C9	0.2979 (4)	0.0366 (4)	0.4341 (3)	1.6 (1)
C10	0.2476 (5)	0.0990 (4)	0.5243 (4)	2.0 (1)
C11	0.3182 (5)	0.2135 (4)	0.5820 (4)	2.0 (1)
C12	0.4273 (4)	0.2404 (4)	0.5342 (4)	1.8 (1)
C13	0.5212 (4)	0.3501 (4)	0.5648 (4)	1.7 (1)
C14	0.6193 (5)	0.3809 (4)	0.5082 (4)	2.2 (1)
C15	0.6912 (5)	0.4959 (4)	0.5603 (4)	2.1 (1)
C16	0.6493 (4)	0.5549 (4)	0.6563 (4)	1.45 (9)
C17	0.6950 (4)	0.6748 (4)	0.7254 (4)	1.6 (1)
C18	0.6547 (4)	0.7412 (4)	0.8196 (4)	1.7 (1)
C19	0.7194 (4)	0.8625 (4)	0.8578 (4)	1.5 (1)
C20	0.8120 (4)	0.8894 (4)	0.7941 (4)	1.5 (1)
C21	1.0617 (4)	0.9980 (4)	0.7338 (4)	1.6 (1)
C22	1.1445 (4)	0.9417 (4)	0.8013 (4)	2.1 (1)
C23	1.2556 (5)	0.8892 (5)	0.7421 (4)	2.7 (1)
C24	1.3380 (5)	0.8355 (5)	0.8116 (5)	3.7 (1)
C25	0.9441 (5)	1.1393 (4)	0.9366 (4)	1.9 (1)
C26	1.0342 (5)	1.2577 (4)	0.9465 (4)	2.5 (1)
C27	1.0609 (5)	1.3431 (5)	1.0606 (4)	2.6 (1)
C28	1.1611 (6)	1.4522 (5)	1.0674 (5)	3.4 (1)

Table 2. *Selected geometric parameters* (Å, °)

Si1—Si2	2.363 (2)	C7—C8	1.488 (7)
S1—C9	1.734 (5)	C9—C10	1.359 (6)
S1—C12	1.723 (4)	C10—C11	1.410 (6)
S2—C13	1.726 (4)	C11—C12	1.375 (7)
S2—C16	1.734 (5)	C12—C13	1.449 (6)
S3—C17	1.737 (5)	C13—C14	1.367 (7)
S3—C20	1.723 (4)	C14—C15	1.407 (6)
Si1—C1	1.894 (5)	C15—C16	1.360 (6)
Si1—C5	1.876 (5)	C16—C17	1.428 (5)
Si1—C9	1.872 (4)	C17—C18	1.363 (6)
Si2—C20	1.876 (5)	C18—C19	1.422 (6)
Si2—C21	1.886 (5)	C19—C20	1.373 (7)
Si2—C25	1.864 (4)	C21—C22	1.528 (7)
C1—C2	1.512 (6)	C22—C23	1.531 (7)
C2—C3	1.525 (8)	C23—C24	1.523 (9)
C3—C4	1.510 (7)	C25—C26	1.540 (7)
C5—C6	1.529 (8)	C26—C27	1.501 (6)
C6—C7	1.524 (8)	C27—C28	1.522 (7)
C9—S1—C12	93.5 (2)	C13—C14—C15	113.3 (4)
C13—S2—C16	92.6 (2)	C14—C15—C16	114.1 (4)
C17—S3—C20	93.8 (2)	S2—C16—C15	109.8 (3)
S1—C9—Si1	122.8 (2)	S2—C16—C17	121.8 (3)
S1—C9—C10	108.4 (3)	S3—C17—C16	118.9 (3)
C9—C10—C11	115.9 (4)	S3—C17—C18	109.4 (3)
C10—C11—C12	111.9 (4)	C17—C18—C19	113.6 (4)
S1—C12—C11	110.3 (3)	C18—C19—C20	113.9 (4)
S1—C12—C13	120.6 (4)	S3—C20—Si2	118.2 (3)
S2—C13—C12	121.9 (4)	S3—C20—C19	109.3 (3)
S2—C13—C14	110.2 (3)		
C1—C2—C3—C4	−177.6 (5)	S2—C16—C17—S3	−175.7 (3)
C5—C6—C7—C8	63.2 (6)	S2—C16—C17—C18	0.3 (7)
S1—C12—C13—S2	−174.2 (3)	C15—C16—C17—C18	176.9 (5)
S1—C12—C13—C14	6.6 (7)	C21—C22—C23—C24	179.1 (4)
C11—C12—C13—C14	−170.8 (5)	C25—C26—C27—C28	174.0 (4)

Data collection, cell refinement, data reduction, structure solution and structure refinement: *MolEN* (Fair, 1990). Molecular graphics: *PLUTO* (Meetsma, 1991; Motherwell & Clegg, 1978).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1057). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Charge-Transfer Complexes. 3. 2,4,5,7-Tetranitro-9-fluorenone–2-Ethylnaphthalene (1/1)

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Abstract

The π–π* charge-transfer complex, $C_{13}H_4N_4O_9.C_{12}H_{12}$, stacks along the *c*-glide direction with the pair

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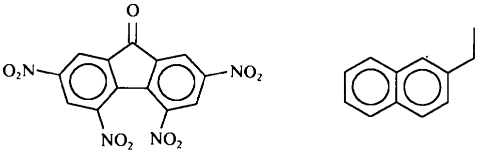
of molecules essentially parallel and at a van der Waals distance. The carbonyl vector in tetranitro-9-fluorenone (TNF) makes an angle of approximately 57° with the bridging C atoms in the 2-ethylnaphthalene, while the corresponding angle in the 1-ethylnaphthalene complex is 170°. No significant change in the TNF carbon skeleton appears to occur on adduct formation.

Comment

The crystal structure determination of the adduct of 2,4,5,7-tetranitro-9-fluorenone (TNF) with 2-ethylnaphthalene (2-ETNP) was performed as part of a study of π–π^* charge-transfer complexes (Baughman, 1982, 1987; Baldwin & Baughman, 1993). Views of the asymmetric unit and molecular stacking are given in Figs. 1 and 2, respectively (Johnson, 1971).



TNF-2-ETNP

TNF and 2-ETNP stack alternately along a *c*-glide direction (Fig. 1), while TNF and 1-ETNP (Baldwin & Baughman, 1993) stack by a simple translation along the *a* axis. In the adduct, 2-ETNP lies approximately 'across' the long axis of TNF (Fig. 1), which is quite unlike the stacking of the 1-ETNP isomer, which lies approximately along TNF in its adduct. The O9—C9···C22—C23 torsion angles in the two adducts are 57 and 170°, respectively.

The distance from the TNF carbon skeleton least-squares plane to the naphthalene portion of 2-ETNP is 3.4 (2) Å; the corresponding distance from TNF to 2-ETNP *via* a *c* glide is 3.42 (4) Å, and that from 2-ETNP to TNF is 3.4 (1) Å (Fig. 2). All these distances agree with the van der Waals thickness of an aromatic ring (3.40 Å; Pauling, 1960) and are also in agreement with the same distances in TNF–1-ETNP, as well as in TNF–3,6-dimethylphenanthrene (3,6-DMP) (Baldwin & Baughman, 1993).

Both TNF–2-ETNP and TNF itself (Baughman, 1987) have centrosymmetric space groups; therefore, both positive and negative values of the torsion angles will be present in the unit cell. Values of the C5—C12—C11—C4 and C10—C11—C12—C13 torsion angles in the TNF portion of the 2-ETNP adduct are ± 9 (2) and ± 8 (1)°, respectively. These values compare favorably with those of molecule *B* (±7.5 and ±5.1°) in the asymmetric unit of the parent TNF and also with the corresponding angles in TNF–1-ETNP and TNF–3,6-DMP (Baldwin & Baughman, 1993). Based on a comparison of these torsion angles, it would seem that TNF does not undergo a significant change in its carbon-skeleton geometry during the formation of this charge-transfer adduct.

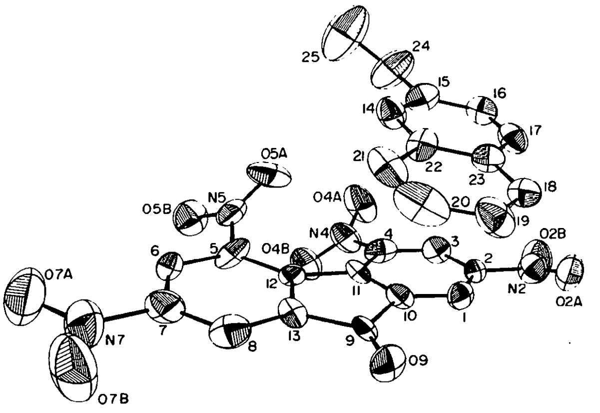


Fig. 1. Atom-labeled TNF-2-ETNP molecules showing 50% probability ellipsoids. The C atoms are represented only by numbers.

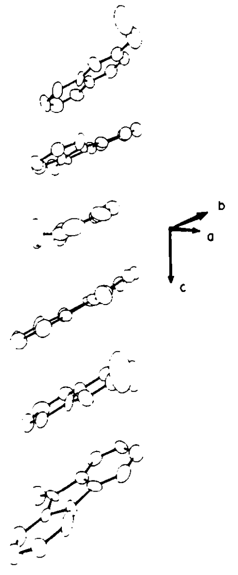


Fig. 2. Stacking (three asymmetric units), viewed perpendicular to c. The nitro groups are not shown for clarity.

Experimental

Crystal data

$C_{13}H_4N_4O_9.C_{12}H_{12}$
$M_r = 516.42$
Monoclinic
$P2_1/c$
$a = 9.462\ (4)$ Å
$b = 15.04\ (1)$ Å
$c = 16.149\ (5)$ Å
$\beta = 99.55\ (3)°$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 23 reflections
$\theta = 4.6–11.9°$
$\mu = 0.127\ \text{mm}^{-1}$
$T = 288$ K
Prism

$V = 2266$ (2) Å^3
$Z = 4$
$D_x = 1.514$ Mg m^{-3}

0.44 × 0.16 × 0.088 mm
Red
Crystal source: 1:1 mol ratio of each reactant

Data collection

Siemens *P*3 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
$T_{\min} = 0.946$, $T_{\max} = 0.989$
8562 measured reflections
4830 independent reflections
1852 observed reflections [$F \geq 3\sigma(F)$]
1046 unique observed reflections

$R_{\rm int} = 0.028$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 11$
$k = -17 \rightarrow 17$
$l = -19 \rightarrow 18$
3 standard reflections monitored every 50 reflections
intensity variation: ±3.8%

Refinement

Refinement on *F*
$R = 0.065$
$wR = 0.034$
$S = 1.58$
1046 reflections
343 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$

$\Delta\rho_{\max} = 0.3$ e Å^{-3}
$\Delta\rho_{\min} = -0.2$ e Å^{-3}
Atomic scattering factors from Hanson, Herman, Lea & Skillman (1964) (non-H atoms) and Stewart, Davidson & Simpson (1965) (H atoms)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$B_{\rm eq}$
O2*A*	0.6345 (9)	0.8939 (5)	0.5767 (6)	6.1
O2*B*	0.7584 (9)	0.7760 (6)	0.6161 (6)	7.3
O4*A*	0.4892 (9)	0.5187 (5)	0.6916 (5)	6.5
O4*B*	0.4134 (8)	0.5560 (5)	0.8070 (4)	4.8
O5*A*	0.1689 (8)	0.5285 (5)	0.6816 (5)	5.6
O5*B*	0.1239 (8)	0.4784 (4)	0.8017 (5)	5.1
O7*A*	−0.2773 (9)	0.6623 (6)	0.8666 (7)	7.4
O7*B*	−0.252 (1)	0.8007 (6)	0.8707 (7)	9.6
O9	0.1881 (7)	0.9390 (4)	0.7434 (5)	3.9
N2	0.653 (1)	0.8234 (7)	0.6120 (7)	5.5
N4	0.441 (1)	0.5728 (8)	0.7372 (6)	5.2
N5	0.136 (1)	0.5391 (7)	0.7523 (6)	4.4
N7	−0.210 (1)	0.7286 (8)	0.8565 (8)	7.6
C1	0.433 (1)	0.8456 (7)	0.6737 (7)	2.8
C2	0.537 (1)	0.7881 (8)	0.6535 (7)	2.4
C3	0.534 (1)	0.6984 (8)	0.6699 (7)	4.0
C4	0.422 (1)	0.6662 (8)	0.7069 (7)	3.0
C5	0.096 (1)	0.6308 (7)	0.7759 (7)	2.9
C6	−0.027 (1)	0.6372 (8)	0.8087 (7)	3.0
C7	−0.077 (1)	0.7224 (8)	0.8237 (7)	3.2
C8	−0.007 (1)	0.7990 (8)	0.8041 (7)	4.0
C9	0.208 (1)	0.8592 (7)	0.7421 (7)	3.0
C10	0.325 (1)	0.8098 (7)	0.7098 (7)	2.1
C11	0.314 (1)	0.7203 (8)	0.7254 (7)	2.7
C12	0.177 (1)	0.7028 (7)	0.7589 (6)	2.1
C13	0.119 (1)	0.7880 (7)	0.7726 (6)	1.9
C14	0.186 (1)	0.6471 (8)	0.4985 (7)	3.7
C15	0.293 (1)	0.6053 (8)	0.4665 (8)	4.2
C16	0.399 (1)	0.6586 (9)	0.4390 (7)	3.4
C17	0.395 (1)	0.7488 (9)	0.4433 (7)	3.4
C18	0.284 (1)	0.8877 (8)	0.4818 (7)	4.0
C19	0.176 (2)	0.9279 (8)	0.5152 (8)	4.1
C20	0.067 (1)	0.878 (1)	0.5422 (8)	4.2
C21	0.071 (1)	0.7859 (9)	0.5374 (7)	3.2
C22	0.180 (1)	0.7413 (8)	0.5041 (8)	2.5
C23	0.287 (1)	0.7945 (8)	0.4761 (7)	3.3
C24	0.300 (1)	0.5043 (9)	0.4620 (8)	5.9
C25	0.177 (2)	0.4546 (8)	0.449 (1)	8.7

Table 2. *Selected geometric parameters* (Å, °)

O2*A*—N2	1.20 (1)	C7—C8	1.39 (1)
O2*B*—N2	1.22 (1)	C8—C13	1.39 (1)
O4*A*—N4	1.24 (1)	C9—C10	1.50 (1)
O4*B*—N4	1.22 (1)	C9—C13	1.49 (1)
O5*A*—N5	1.24 (1)	C10—C11	1.38 (1)
O5*B*—N5	1.23 (1)	C11—C12	1.51 (1)
O7*A*—N7	1.20 (1)	C12—C13	1.42 (1)
O7*B* —N7	1.19 (1)	C14—C15	1.37 (1)
O9—C9	1.22 (1)	C14—C22	1.42 (1)
N2—C2	1.48 (1)	C15—C16	1.41 (1)
N4—C4	1.49 (1)	C15—C24	1.52 (1)
N5—C5	1.50 (1)	C16—C17	1.37 (1)
N7—C7	1.45 (1)	C17—C23	1.40 (1)
C1—C2	1.38 (1)	C18—C19	1.37 (1)
C1—C10	1.37 (1)	C18—C23	1.41 (1)
C2—C3	1.38 (1)	C19—C20	1.41 (2)
C3—C4	1.39 (1)	C20—C21	1.38 (1)
C4—C11	1.38 (1)	C21—C22	1.41 (1)
C5—C6	1.36 (1)	C22—C23	1.43 (1)
C5—C12	1.38 (1)	C24—C25	1.37 (1)
C6—C7	1.40 (1)		
O2*A*—N2—O2*B*	126 (1)	C7—C8—C13	117 (1)
O2*A*—N2—C2	118 (1)	C1—C10—C9	127 (1)
O2*B*—N2—C2	115 (1)	C1—C10—C11	123 (1)
O4*A*—N4—O4*B*	125 (1)	C9—C10—C11	109 (1)
O4*A*—N4—C4	118 (1)	C4—C11—C10	117 (1)
O4*B*—N4—C4	118 (1)	C4—C11—C12	133 (1)
O5*A*—N5—O5*B*	124 (1)	C10—C11—C12	110 (1)
O5*A*—N5—C5	118 (1)	C5—C12—C11	138 (1)
O5*B*—N5—C5	118 (1)	C5—C12—C13	116 (1)
O7*A*—N7—O7*B*	122 (1)	C11—C12—C13	106 (1)
O7*A*—N7—C7	120 (1)	C8—C13—C9	127 (1)
O7*B*—N7—C7	118 (1)	C8—C13—C12	123 (1)
O9—C9—C10	128 (1)	C9—C13—C12	110 (1)
O9—C9—C13	127 (1)	C15—C14—C22	122 (1)
C10—C9—C13	104 (1)	C14—C15—C16	118 (1)
C2—C1—C10	117 (1)	C14—C15—C24	121 (1)
N2—C2—C1	119 (1)	C16—C15—C24	121 (1)
N2—C2—C3	119 (1)	C15—C16—C17	121 (1)
C1—C2—C3	123 (1)	C16—C17—C23	123 (1)
C2—C3—C4	118 (1)	C19—C18—C23	119 (1)
N4—C4—C3	114 (1)	C18—C19—C20	121 (1)
N4—C4—C11	123 (1)	C19—C20—C21	120 (1)
C3—C4—C11	122 (1)	C20—C21—C22	121 (1)
N5—C5—C6	116 (1)	C14—C22—C21	123 (1)
N5—C5—C12	120 (1)	C14—C22—C23	120 (1)
C6—C5—C12	124 (1)	C21—C22—C23	117 (1)
C5—C6—C7	118 (1)	C17—C23—C18	123 (1)
N7—C7—C6	117 (1)	C17—C23—C22	116 (1)
N7—C7—C8	120 (1)	C18—C23—C22	121 (1)
C6—C7—C8	122 (1)	C15—C24—C25	121 (1)
O2*A*—N2—C2—C1	−20 (2)	O5*B*—N5—C5—C12	141 (1)
O2*B*—N2—C2—C1	161 (1)	O7*A*—N7—C7—C6	−3 (1)
O2*A*—N2—C2—C3	159 (1)	O7*B*—N7—C7—C6	179 (1)
O2*B*—N2—C2—C3	−19 (1)	O7*A*—N7—C7—C8	173 (1)
O4*A*—N4—C4—C3	−42 (2)	O7*B*—N7—C7—C8	−5 (2)
O4*B*—N4—C4—C3	135 (1)	C4—C11—C12—C5	−9 (2)
O4*A*—N4—C4—C11	147 (1)	C4—C11—C12—C13	174 (1)
O4*B*—N4—C4—C11	−35 (2)	C10—C11—C12—C5	170 (1)
O5*A*—N5—C5—C6	131 (1)	C10—C11—C12—C13	−8 (1)
O5*B*—N5—C5—C6	−43 (1)	C14—C15—C24—C25	33 (2)
O5*A*—N5—C5—C12	−45 (1)	C16—C15—C24—C25	−148 (1)

36 out of 38 non-H atoms were located by *MULTAN*76 (Main, Woolfson & Germain, 1976) using coordinates for TNF and 2-ETNP, and a *K* curve to compute the *E* values. Aromatic H atoms were set at 1.00 Å from the respective C atoms while bisect-

ing the C—C—C angle; methyl and ethyl H atoms were placed in tetrahedral positions at 1.10 Å. Anisotropic displacement parameters were refined for all non-H atoms. H atoms were assigned the B_{eq} value of the C atom to which they were bonded. Programs used: *MULTAN76*; *ALLS* (Lapp & Jacobson, 1979); *FOUR* (Powell & Jacobson, 1980); *ORFFE* (Busing, Martin & Levy, 1964); *ORTEP*II (Johnson, 1971).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71606 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1051]

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A 2,5-Diketomorpholine

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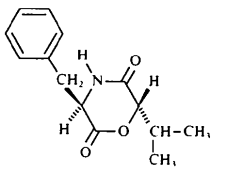
Abstract

We report the crystal structure of (3*R*,6*S*)-3-benzyl-6-isopropylmorpholine-2,5-dione, $C_{14}H_{17}NO_3$. The morpholine heterocycle adopts a boat conformation with the isopropyl group in an equatorial position and the benzyl substituent in an axial position. An intermolecular hydrogen bond connects symmetry-related amide groups to form an infinite chain.

Comment

The title compound (I) is a synthetic intermediate in the diastereoselective synthesis of amino acids (Maywald, 1987). The boat conformation of the morpholine heterocycle is clearly defined: O(1), C(2), N(4) and C(5) form the base [σ = 0.04 (1) Å] while C(3) and C(6) deviate from the base by 0.39 (1) and 0.46 (1) Å, respectively. The phenyl ring of the axial benzyl substituent is oriented towards the N atom.



(I)

The intermolecular packing is stabilized by a hydrogen bond from N(4) to O(51) [N(4)···O(51) 2.864 (2), H(4)···O(51) 1.93 (1) Å, N(4)—H(4)···O(51) 165 (2)°] with the two molecules related by a 2_1 axis ($1 - x$, $-\frac{1}{2} + y$, $1 - z$) thus forming an infinite chain parallel to **b**.

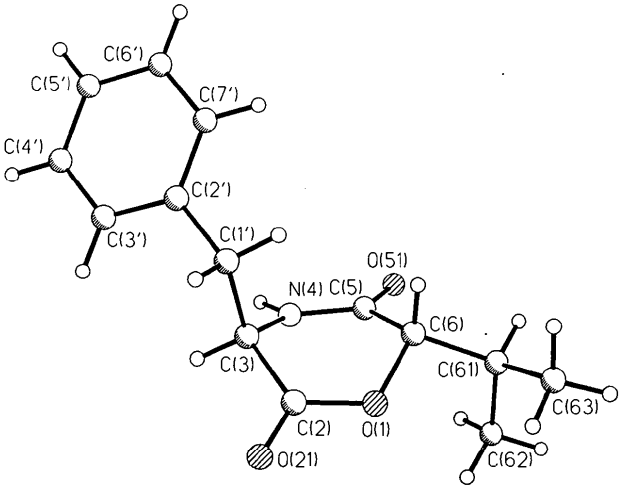


Fig. 1. A perspective view of the title compound showing the atom-numbering scheme.

Experimental

Crystal data

$C_{14}H_{17}NO_3$ Mo $K\alpha$ radiation
M_r = 247.3 λ = 0.71069 Å

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