the *SHELXTL-Plus* program package (Sheldrick, 1988) on a MicroVAX II computer. H atoms were placed in idealized positions and constrained to have C—H = 0·96 Å and isotropic thermal parameters, $U = 0{\cdot}08$ Å^2. All non-H atoms treated as anisotropic, except the disordered atoms in (III). No absorption correction necessary for any of the structures. Details of the data collection are given in Table 1. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates for (I), (II) and (III) are contained in Tables 2, 3 and 4.* Figs. 1, 2 and 3 illustrate the molecules with the numbering scheme employed. Figs. 4, 5 and 6 illustrate the projected packing of the molecules viewed down the *a* axis.

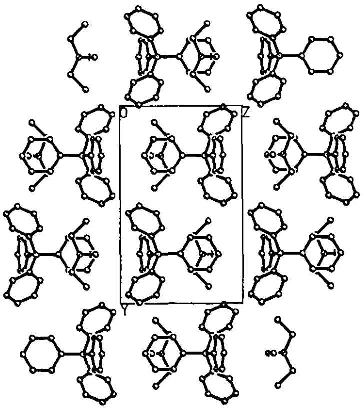

Fig. 6. Projected packing plot of (III) viewed down the *a* axis.

Related literature. For additional information on related *N*-acyl ammonium salt reactions and chemistry see Gresser & Jencks (1977), Jencks & Gilchrist (1968), Kim, Lee & Kim (1985), Bayliss, Homer & Shepherd (1990) and Brunelle, Boden & Shannon (1990).

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54207 (54 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,3-Dinitronaphthalene

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Abstract. $C_{10}H_6N_2O_4$, $M_r = 218{\cdot}2$, triclinic, $P\bar{1}$, $a = 7{\cdot}5552\,(8)$, $b = 7{\cdot}763\,(1)$, $c = 9{\cdot}073\,(1)$ Å, $\alpha = 110{\cdot}742\,(9)$, $\beta = 99{\cdot}570\,(9)$, $\gamma = 100{\cdot}309\,(9)$°, $V = 474{\cdot}1\,(2)$ Å^3, $D_x = 1{\cdot}528$ g cm^{-3}, $Z = 2$, Cu $K\alpha$ ($\lambda = 1{\cdot}54178$ Å, graphite monochromator) $\mu = 10{\cdot}5$ cm^{-1}, $F(000) = 224$, $T = 293$ K, final $R = 0{\cdot}064$, $wR = 0{\cdot}092$ for 1213 reflections with $I > 3\sigma(I)$. The naphthalene ring is planar with an average deviation of 0·004 Å from the ten-atom least-squares plane. The C—C distances show the typical naphthalene alternation: C1—C2, C3—C4, C5—C6 and C7—C8 average 1·359 Å; C2—C3 and C6—C7 average 1·391 Å. The C1 nitro group is twisted out of the ring plane by approximately 37° to relieve non-bonded interactions between an oxygen and the C8—H8 region.

Experimental. Commercial sample (Aldrich Chemical Co.), orange-brown needle-like crystals from cyclohexane/1,2-dichloroethane by vapor diffusion, 0·2 × 0·23 × 0·46 mm specimen; Enraf–Nonius CAD-4 diffractometer; cell parameters and crystal orientation from 25 automatically centered reflections in the range $7{\cdot}1 < \theta < 43{\cdot}2$°; 2θ–θ scans over $\Delta\theta$ range of 1·5 $(1{\cdot}1 + 0{\cdot}14\tan\theta)$°; variable θ scan speed of 1·18–5·49° min^{-1}; each scan recorded in 96 steps with two outermost 16-step intensity blocks used for background determination; diffractometer controlled with Digital Equipment Corporation MicroVaxII computer and standard Enraf–Nonius programs (version 5·0); nine standard reflections monitored at 1 h intervals of X-ray exposure, −2·4 to 0·4% intensity variation, −0·9% average, correc-

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O1	0·2694 (6)	−0·3127 (5)	0·2059 (4)	9·6 (2)
O2	0·2778 (6)	−0·0770 (5)	0·4188 (4)	9·0 (2)
O3	0·6779 (4)	0·4995 (4)	0·4419 (3)	6·2 (1)
O4	0·6418 (5)	0·5701 (4)	0·2343 (4)	7·6 (1)
N1	0·2788 (5)	−0·1446 (5)	0·2785 (4)	5·2 (1)
N2	0·6059 (4)	0·4647 (4)	0·3022 (3)	4·5 (1)
C1	0·3106 (4)	−0·0181 (4)	0·1931 (4)	3·7 (1)
C2	0·4304 (5)	0·1555 (5)	0·2828 (4)	3·8 (1)
C3	0·4699 (4)	0·2791 (4)	0·2062 (3)	3·6 (1)
C4*A*	0·2640 (4)	0·0543 (4)	−0·0463 (3)	3·31 (9)
C4	0·3912 (4)	0·2343 (4)	0·0475 (4)	3·7 (1)
C5	0·1809 (5)	0·0048 (5)	−0·2130 (4)	4·2 (1)
C6	0·0585 (5)	−0·1681 (5)	−0·3032 (4)	4·6 (1)
C7	0·0144 (5)	−0·2977 (5)	−0·2326 (4)	4·5 (1)
C8*A*	0·2212 (4)	−0·0790 (4)	0·0250 (3)	3·46 (9)
C8	0·0917 (5)	−0·2568 (5)	−0·0731 (4)	4·2 (1)

Table 2. *Bond lengths* (Å), *angles* (°) *and nitro-group torsion angles* (°) *with e.s.d.'s in parentheses*

O1—N1	1·219 (5)	C3—C4	1·354 (4)
O2—N1	1·195 (4)	C4*A*—C4	1·416 (4)
O3—N2	1·205 (3)	C4*A*—C8*A*	1·420 (4)
O4—N2	1·207 (4)	C4*A*—C5	1·420 (4)
N1—C1	1·462 (4)	C5—C6	1·361 (5)
N2—C3	1·474 (4)	C6—C7	1·393 (5)
C1—C2	1·356 (4)	C7—C8	1·365 (5)
C1—C8*A*	1·428 (4)	C8*A*—C8	1·419 (4)
C2—C3	1·390 (4)		

O2—N1—O1	123·0 (3)	C2—C3—N2	118·1 (3)
O2—N1—C1	118·9 (3)	C4—C4*A*—C8*A*	120·3 (3)
O1—N1—C1	117·8 (3)	C4—C4*A*—C5	120·1 (3)
O3—N2—O4	124·1 (3)	C8*A*—C4*A*—C5	119·6 (3)
O3—N2—C3	118·1 (3)	C3—C4—C4*A*	119·4 (3)
O4—N2—C3	117·7 (3)	C6—C5—C4*A*	120·2 (3)
C2—C1—C8*A*	123·3 (3)	C5—C6—C7	120·2 (3)
C2—C1—N1	115·8 (3)	C8—C7—C6	121·7 (3)
C8*A*—C1—N1	120·8 (3)	C8—C8*A*—C4*A*	118·3 (3)
C1—C2—C3	118·2 (3)	C8—C8*A*—C1	125·6 (3)
C4—C3—C2	122·7 (3)	C4*A*—C8*A*—C1	116·1 (3)
C4—C3—N2	119·2 (3)	C7—C8—C8*A*	120·0 (3)

O1—N1—C1—C2	−139·1 (4)	O3—N2—C3—C4	−178·1 (3)
O1—N1—C1—C8*A*	39·9 (5)	O3—N2—C3—C2	0·7 (5)
O2—N1—C1—C2	35·1 (5)	O4—N2—C3—C4	0 (4)
O2—N1—C1—C8*A*	−145·9 (4)	O4—N2—C3—C2	178·9 (4)

tion applied; absorption correction applied with method of Walker & Stuart (1983), transmission factor range 0·700–1·503, average 0·963; $4 < \theta < 67°$, *hkl*'s selected to maintain diffractometer χ angle within 0–90° range; 1888 total data measured, 1690 unique data, 1213 data with $I > 3\sigma(I)$, $R_{int} = 0{\cdot}002$ for 63 twice-measured data. All crystallographic calculations performed with the *TEXSAN* (Molecular Structure Corporation, 1989) system on DEC MicroVaxII or VaxStationII computers; structure solved with the *SIR* direct-methods program (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989) incorporated in *TEXSAN*. Full-matrix least-squares refinement, $\sum[w(F_o - F_c)^2]$ minimized with $w = 1/\sigma^2(F_o)$, reflections with $I < 3\sigma(I)$ excluded from refinement, correction for secondary isotropic extinction (Zachariasen, 1968) applied; C, O and N refined with anisotropic temperature factors; H atoms initially positioned from the C-atom framework and refined with individual isotropic temperature factors; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 155–175); minimum and maximum $\Delta\rho$ −0·28 and 0·44 e Å^{-3}; maximum Δ/σ in final least-squares cycle 0·45; R, wR and S 0·064, 0·092 and 2·52. Atomic coordinates are listed in Table 1;* bond lengths, angles and nitro-group torsion angles are given in Table 2. The *PLOTMD* program (Luo, Ammon & Gilliland, 1989) was used to display the *ORTEP* drawing (Johnson, 1965) (Fig. 1) on a VaxStationII monitor, label the drawing, and prepare a print file for a Hewlett-Packard Laser-Jet II printer.

Related literature. The determination was performed to increase our database of nitro-aromatic structures. These data are being used in the development of force-field parameters for crystal lattice computations.

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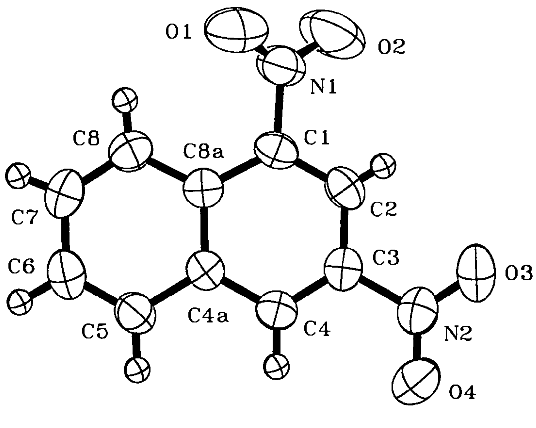

Fig. 1. *ORTEP* drawing. The C, O and N atoms are shown as 50% ellipsoids, and the H atoms are depicted as spheres with $B = 1{\cdot}5$ Å^2.

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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 54096 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Possible Precursor to Harringtonolide

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Abstract. Methyl (1α,3α,$3a\alpha$,$10a\alpha$,$10b\alpha$)-1,2,3,3a,6,9,10,10bα-octahydro-3-hydroxy-7-methoxy-1,5-dimethyl-4-oxocyclohept[*bc*]acenaphthalene-10a(4*H*)-carboxylate, $C_{21}H_{26}O_5$, $M_r = 358{\cdot}43$, triclinic, $P\bar{1}$, $a = 16{\cdot}215\,(2)$, $b = 11{\cdot}985\,(1)$, $c = 10{\cdot}480\,(2)$ Å, $\alpha = 69{\cdot}40\,(1)$, $\beta = 75{\cdot}28\,(1)$, $\gamma = 76{\cdot}11\,(2)°$, $V = 1818$ Å^3, $Z = 4$, $D_x = 1{\cdot}31$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}667$ mm^{-1}, $F(000) = 768$, $T = 298$ K, $R = 0{\cdot}040$ for 4553 observed reflections. The structure contains two independent molecules in the asymmetric unit which have essentially identical conformations. The cycloheptatriene ring adopts the expected boat conformation with C(6) at the 'prow'. The methoxy substituent on the cycloheptatriene ring is eclipsed with the C(7)—C(8) bond [C(11)—O(1)—C(7)—C(8) −5·8 (3)°]. This eclipsing results in close intramolecular contacts between the hydrogens attached to C(11) and the hydrogen on C(8). The structure is held together by weak intermolecular hydrogen bonds: O(2)···O(4) 2·890 (2), H(16)···O(4) 2·03 (4) Å, O(2)—H(16)···O(7) 153 (2)°; O(7)···O(6) 3·005 (2), H(26)···O(6) 2·15 (4), O(7)—H(26)···O(6) 151 (2)°. There also exist weak intramolecular hydrogen bonds within the β-hydroxy ketone: O(2)···O(4) 3·091 (2), H(16)···O(4) 2·52 (3) Å, O(2)—H(16)···O(4) 120 (3)°; O(7)···O(6) 3·089 (2), H(26)···O(6) 2·45 (3) Å, O(7)—H(26)···O(6) 125 (2)°.

Experimental. Colourless crystal 0·28 × 0·20 × 0·24 mm. Philips PW 1100/20 diffractometer, graphite monochromator. Lattice parameters from least-squares analysis of setting angles of 25 reflections $50 < 2\theta < 60°$, λ(Cu $K\alpha$) = 1·5418 Å. θ–2θ scans of width $(1{\cdot}0 + 0{\cdot}142\tan\theta)°$ in θ and rate 8° min^{-1} in θ with 5 s backgrounds on each side of every scan. $2\theta_{\max} = 120°$ with $-18 \le h \le 18$, $-13 \le k \le 13$, $0 \le l \le 12$, 5170 unique reflections, 4553 with $I > 3\sigma(I)$ regarded as observed. Three check reflections measured every 120 min showed no significant decrease in intensity during data collection. Data corrected for absorption (maximum/minimum transmission 0·91/0·83). Structure solution by direct methods (*SHELXS*86, Sheldrick, 1985), ΔF synthesis and full-matrix least-squares refinement. Non-H atoms refined with anisotropic displacement factors, H atoms refined with isotropic thermal parameters. Refinement on F, 678 parameters, to $R = 0{\cdot}040$, $wR = 0{\cdot}058$, $S = 1{\cdot}94$, weighting scheme $w = [\sigma^2(F) + (0{\cdot}0005)F^2]^{-1}$, max. $\Delta/\sigma = 0{\cdot}03$, max. and min. heights in final $\Delta\rho$ map 0·2 and $-0{\cdot}2$ e Å^{-3}. Atomic

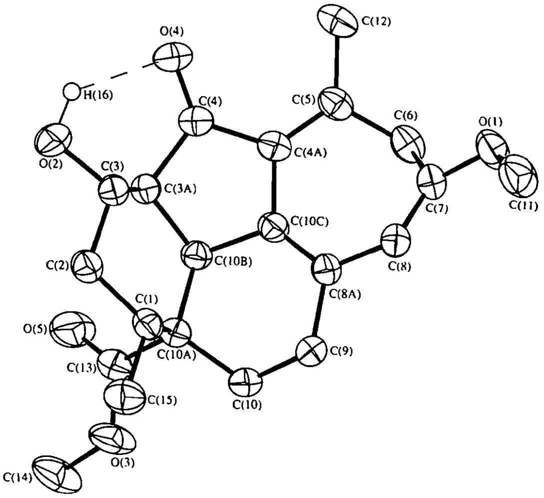

Fig. 1. Thermal-ellipsoid diagram of one of the independent molecules of (1) showing labelling of non-H atoms. Ellipsoids show 50% probability levels. The hydroxyl hydrogen is drawn as a small circle.

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