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2,8-Dimethoxy-4,10-dimethyl-1,3,7,9-tetranitro-6*H*,12*H*-5,11-methanodibenzo[*b*,*f*][1,5]diazocine

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2,8-Dimethoxy-4,10-dimethyl-1,3,7,9-tetranitro-6*H*,12*H*-5,11-methanodibenzo[*b*,*f*][1,5]diazocine

M. Delower H. Bhuiyan,[a] Paul Jensen[b] and Andrew C. Try[a]*

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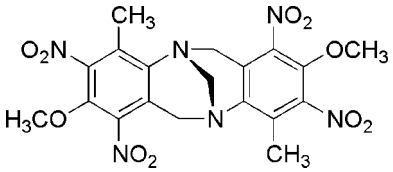
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.087; data-to-parameter ratio = 9.2.

In the molecule of the title compound, $C_{19}H_{18}N_6O_{10}$, the 2,8-dimethoxy-4,10-dimethyl-1,3,7,9-tetranitro analogue of Tröger's base, the diazocine bridge imparts a twist such that the two aryl rings are offset with respect to one another. The hinge angle of the molecule, measured as the dihedral angle between the two benzene rings, is 103.64 (5)°.

Related literature

For related literature on mononitro-substituted Tröger's base analogues, see: Webb & Wilcox (1990); Pardo *et al.* (1996). For dinitro-substituted Tröger's base analogues, see: Mederski *et al.* (2003); Li *et al.* (2005); Bhuiyan *et al.* (2007).

For related literature, see: Faroughi *et al.* (2006); Jensen & Wärnmark (2001); Kostyanovsky *et al.* (2003); Lenev *et al.* (2006); Mederski *et al.* (2003); Sergeyev *et al.* (2005); Solano *et al.* (2005); Sucholeiki *et al.* (1988).



Experimental

Crystal data

$C_{19}H_{18}N_6O_{10}$
M_r = 490.39
Orthorhombic, $P2_12_12_1$
a = 8.629 (2) Å
b = 9.155 (2) Å
c = 26.484 (5) Å
V = 2092.2 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 150 (2) K
0.50 × 0.47 × 0.20 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.913, T_{max} = 0.975
20912 measured reflections
2951 independent reflections
2796 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.087
S = 1.04
2951 reflections
320 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2003); software used to prepare material for publication: *modiCIFer* (Guzei, 2005).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2074).

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supplementary materials

Acta Cryst. (2007). E**63**, o4393 [**doi:10.1107/S1600536807050945**]

2,8-Dimethoxy-4,10-dimethyl-1,3,7,9-tetranitro-6*H*,12*H*-5,11-methanodibenzo[*b*,*f*][1,5]diazocine

M. D. H. Bhuiyan, P. Jensen and A. C. Try

Comment

For over 100 years since the first synthesis of Tröger's base it was believed that analogues bearing electron-withdrawing groups could not be prepared in good yields, if at all. This belief was dispelled with the first synthesis of dihalogenated analogues (Jensen & Wärnmark, 2001), tetrabromo (Faroughi *et al.*, 2006) and dinitro analogues (Mederski *et al.*, 2003; Li *et al.*, 2005; Bhuiyan *et al.*, 2007). Compound (I) is the first example of a tetranitro Tröger's base analogue and was prepared in racemic form by reacting 4-methoxy-2-methyl-3,5-dinitroaniline with diglycolic acid in polyphosphoric acid (PPA) as shown in Fig. 2. The molecular structure of (I) is shown in Fig. 1. It is interesting to note that in addition to (I), there are two other reports of simple dibenzo Tröger's base analogues with dihedral angles greater than 100° that bear substituents in the 2,4,8- and 10-positions (Sucholeiki *et al.*, 1988; Faroughi *et al.*, 2006), at the upper end of the the range of 82° (Solano *et al.*, 2005) to 108° (Faroughi *et al.*, 2006), that are the lower and upper limits, respectively, that have been measured for for over twenty simple dibenzo Tröger's base analogues. These results would tend to suggest that the placement of substituents in these positions may lead to an increase in the cavity size of the Tröger's base systems, at least in the crystalline state.

Although the compound was prepared in racemic form, the crystal chosen for analysis crystallized in enantiopure form, however the absolute configuration of the structure has not been established by X-ray methods. This appears to be the fourth example of conglomerate crystallization among Tröger's base systems (Kostyanovsky *et al.*, 2003; Sergeyev *et al.*, 2005; Lenev *et al.*, 2006).

We were interested in preparing a range of nitro-substituted Tröger's base compounds as precursors for supramolecular recognition elements.

Experimental

Synthetic details will be reported elsewhere. Crystals of (I) were obtained by slow evaporation of a dichloromethane solution.

Figures

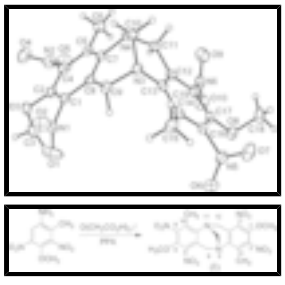


Fig. 1. The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

2,8-Dimethoxy-4,10-dimethyl-1,3,7,9-tetranitro-6*H*,12*H*- 5,11-methanodibenzo[*b*,*f*][1,5]diazocine

Crystal data

$C_{19}H_{18}N_6O_{10}$	D_x = 1.557 Mg m^{-3}
M_r = 490.39	Melting point: 509 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 6736 reflections
a = 8.629 (2) Å	θ = 2.4–28.3º
b = 9.155 (2) Å	μ = 0.13 mm^{-1}
c = 26.484 (5) Å	T = 150 (2) K
V = 2092.2 (8) Å^3	Plate, pale yellow
Z = 4	0.50 × 0.47 × 0.20 mm
F_{000} = 1016	

Data collection

Bruker CCD-1000 area-detector diffractometer	2951 independent reflections
Radiation source: fine-focus sealed tube	2796 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 150(2) K	θ_{max} = 28.4º
ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −11→11
T_{min} = 0.913, T_{max} = 0.975	k = −12→11
20912 measured reflections	l = −35→35

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.4264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.087	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
2951 reflections	Extinction correction: none
320 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.24794 (17)	0.33842 (19)	0.07709 (8)	0.0502 (5)
O2	−0.08837 (18)	0.48777 (15)	0.04288 (6)	0.0389 (4)
O3	−0.16534 (14)	0.11061 (15)	0.01060 (4)	0.0271 (3)
O4	0.0242 (2)	−0.11653 (17)	−0.02943 (5)	0.0412 (4)
O5	0.0452 (3)	−0.23330 (16)	0.04133 (7)	0.0519 (4)
O6	−0.03451 (19)	0.5055 (2)	0.31937 (6)	0.0516 (5)
O7	0.1945 (2)	0.5605 (2)	0.34557 (6)	0.0503 (4)
O8	0.16799 (16)	0.21859 (15)	0.34025 (4)	0.0293 (3)
O9	0.42213 (18)	−0.03735 (17)	0.25715 (6)	0.0405 (4)
O10	0.1932 (2)	−0.04848 (15)	0.29035 (6)	0.0390 (3)
N1	−0.12096 (16)	0.36974 (16)	0.06071 (5)	0.0229 (3)
N2	0.04626 (19)	−0.12154 (16)	0.01626 (6)	0.0293 (3)
N3	0.30705 (16)	0.42473 (15)	0.14649 (5)	0.0204 (3)
N4	0.38756 (16)	0.18709 (16)	0.11780 (5)	0.0205 (3)
N5	0.10563 (19)	0.49762 (18)	0.31727 (5)	0.0289 (3)
N6	0.29756 (19)	0.01726 (17)	0.26895 (5)	0.0267 (3)
C1	−0.00005 (17)	0.25689 (17)	0.06230 (6)	0.0192 (3)
C2	−0.03352 (19)	0.12601 (18)	0.03837 (6)	0.0210 (3)
C3	−0.2842 (2)	0.0221 (3)	0.03419 (7)	0.0362 (4)
H3A	−0.2384	−0.0695	0.0462	0.054*
H3B	−0.3657	0.0005	0.0095	0.054*
H3C	−0.3288	0.0754	0.0628	0.054*
C4	0.0768 (2)	0.01678 (18)	0.04267 (6)	0.0217 (3)
C5	0.21390 (19)	0.03065 (17)	0.06985 (6)	0.0202 (3)
C6	0.3289 (2)	−0.09185 (19)	0.07389 (7)	0.0274 (3)
H6A	0.2939	−0.1617	0.0995	0.041*
H6B	0.4302	−0.0525	0.0836	0.041*
H6C	0.3376	−0.1414	0.0412	0.041*
C7	0.24331 (18)	0.16794 (17)	0.09196 (5)	0.0180 (3)
C8	0.13757 (18)	0.28300 (17)	0.08822 (5)	0.0181 (3)
C9	0.17197 (19)	0.43057 (17)	0.11245 (6)	0.0205 (3)
H9A	0.1917	0.5034	0.0856	0.025*
H9B	0.0800	0.4631	0.1318	0.025*

C10	0.42902 (19)	0.34144 (19)	0.12147 (6)	0.0232 (3)
H10A	0.5268	0.3514	0.1407	0.028*
H10B	0.4463	0.3813	0.0872	0.028*
C11	0.38882 (19)	0.12370 (18)	0.16884 (6)	0.0217 (3)
H11A	0.4975	0.1073	0.1794	0.026*
H11B	0.3361	0.0277	0.1680	0.026*
C12	0.30895 (18)	0.22120 (17)	0.20748 (6)	0.0199 (3)
C13	0.27142 (18)	0.36599 (17)	0.19513 (6)	0.0189 (3)
C14	0.20377 (19)	0.46122 (18)	0.23053 (6)	0.0204 (3)
C15	0.1778 (2)	0.61959 (19)	0.21860 (6)	0.0273 (3)
H15A	0.0893	0.6292	0.1957	0.041*
H15B	0.2707	0.6599	0.2025	0.041*
H15C	0.1566	0.6732	0.2499	0.041*
C16	0.17340 (19)	0.40371 (19)	0.27790 (6)	0.0221 (3)
C17	0.20533 (19)	0.26139 (18)	0.29235 (6)	0.0223 (3)
C18	0.2990 (3)	0.1949 (2)	0.37300 (6)	0.0335 (4)
H18A	0.3633	0.1159	0.3594	0.050*
H18B	0.2623	0.1679	0.4068	0.050*
H18C	0.3605	0.2847	0.3751	0.050*
C19	0.27199 (19)	0.17181 (18)	0.25601 (6)	0.0216 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0255 (7)	0.0472 (9)	0.0781 (12)	0.0093 (7)	0.0215 (7)	0.0217 (9)
O2	0.0358 (7)	0.0257 (6)	0.0553 (9)	0.0065 (6)	0.0150 (7)	0.0123 (6)
O3	0.0214 (5)	0.0388 (7)	0.0213 (5)	−0.0075 (5)	−0.0039 (4)	0.0033 (5)
O4	0.0496 (8)	0.0450 (8)	0.0290 (6)	−0.0131 (8)	0.0026 (6)	−0.0152 (6)
O5	0.0773 (12)	0.0237 (6)	0.0548 (9)	−0.0119 (8)	−0.0092 (10)	−0.0007 (6)
O6	0.0351 (8)	0.0801 (13)	0.0394 (8)	0.0226 (9)	0.0029 (7)	−0.0153 (9)
O7	0.0524 (9)	0.0621 (10)	0.0363 (7)	−0.0002 (9)	−0.0028 (7)	−0.0274 (7)
O8	0.0311 (7)	0.0394 (7)	0.0174 (5)	0.0006 (6)	−0.0004 (5)	0.0032 (5)
O9	0.0401 (8)	0.0389 (7)	0.0426 (8)	0.0175 (7)	−0.0053 (6)	0.0050 (7)
O10	0.0501 (9)	0.0298 (7)	0.0371 (7)	−0.0035 (7)	0.0030 (7)	0.0049 (6)
N1	0.0191 (6)	0.0267 (7)	0.0229 (6)	0.0022 (6)	0.0006 (5)	0.0020 (5)
N2	0.0309 (7)	0.0242 (7)	0.0329 (7)	−0.0076 (6)	0.0027 (6)	−0.0084 (6)
N3	0.0205 (6)	0.0222 (6)	0.0187 (6)	−0.0035 (5)	0.0011 (5)	−0.0020 (5)
N4	0.0160 (6)	0.0244 (6)	0.0211 (6)	0.0001 (5)	−0.0001 (5)	−0.0031 (5)
N5	0.0331 (8)	0.0341 (8)	0.0196 (6)	0.0090 (7)	0.0013 (6)	−0.0031 (6)
N6	0.0333 (8)	0.0261 (7)	0.0209 (6)	0.0035 (6)	−0.0078 (6)	0.0011 (5)
C1	0.0176 (7)	0.0212 (7)	0.0187 (6)	0.0011 (6)	0.0023 (5)	0.0020 (5)
C2	0.0201 (7)	0.0260 (7)	0.0167 (6)	−0.0049 (6)	0.0004 (6)	0.0008 (6)
C3	0.0268 (9)	0.0514 (12)	0.0302 (9)	−0.0165 (9)	−0.0057 (7)	0.0059 (9)
C4	0.0245 (8)	0.0199 (7)	0.0207 (7)	−0.0043 (6)	0.0026 (6)	−0.0032 (6)
C5	0.0213 (7)	0.0200 (7)	0.0192 (6)	0.0005 (6)	0.0038 (6)	−0.0011 (6)
C6	0.0284 (8)	0.0227 (7)	0.0310 (8)	0.0053 (7)	0.0030 (7)	−0.0027 (6)
C7	0.0173 (7)	0.0206 (7)	0.0163 (6)	−0.0006 (6)	0.0025 (5)	−0.0017 (5)
C8	0.0184 (7)	0.0202 (7)	0.0157 (6)	−0.0026 (6)	0.0036 (5)	−0.0007 (5)

C9	0.0232 (7)	0.0193 (7)	0.0189 (6)	−0.0003 (6)	0.0005 (6)	−0.0004 (5)
C10	0.0182 (7)	0.0290 (8)	0.0226 (7)	−0.0057 (6)	0.0027 (6)	−0.0046 (6)
C11	0.0192 (7)	0.0251 (7)	0.0209 (7)	0.0048 (6)	−0.0011 (6)	−0.0027 (6)
C12	0.0170 (6)	0.0230 (7)	0.0198 (6)	0.0008 (6)	−0.0017 (6)	−0.0023 (6)
C13	0.0165 (6)	0.0217 (7)	0.0185 (6)	−0.0026 (6)	−0.0011 (5)	−0.0021 (5)
C14	0.0185 (7)	0.0222 (7)	0.0204 (7)	−0.0006 (6)	−0.0016 (6)	−0.0032 (6)
C15	0.0355 (9)	0.0218 (7)	0.0245 (7)	0.0031 (7)	0.0004 (7)	−0.0027 (6)
C16	0.0201 (7)	0.0282 (8)	0.0180 (7)	0.0024 (6)	−0.0014 (6)	−0.0053 (6)
C17	0.0212 (7)	0.0288 (8)	0.0168 (6)	0.0001 (7)	−0.0021 (6)	0.0000 (6)
C18	0.0414 (10)	0.0366 (10)	0.0227 (8)	0.0015 (9)	−0.0101 (7)	0.0019 (7)
C19	0.0207 (7)	0.0240 (7)	0.0201 (7)	0.0011 (6)	−0.0039 (6)	0.0004 (6)

Geometric parameters (Å, °)

O1—N1	1.213 (2)	C4—C5	1.391 (2)
O2—N1	1.212 (2)	C5—C7	1.410 (2)
O3—C2	1.3618 (19)	C5—C6	1.501 (2)
O3—C3	1.449 (2)	C6—H6A	0.9800
O4—N2	1.226 (2)	C6—H6B	0.9800
O5—N2	1.220 (2)	C6—H6C	0.9800
O6—N5	1.213 (2)	C7—C8	1.397 (2)
O7—N5	1.217 (2)	C8—C9	1.525 (2)
O8—C17	1.366 (2)	C9—H9A	0.9900
O8—C18	1.441 (2)	C9—H9B	0.9900
O9—N6	1.226 (2)	C10—H10A	0.9900
O10—N6	1.223 (2)	C10—H10B	0.9900
N1—C1	1.469 (2)	C11—C12	1.523 (2)
N2—C4	1.470 (2)	C11—H11A	0.9900
N3—C13	1.4295 (19)	C11—H11B	0.9900
N3—C10	1.459 (2)	C12—C19	1.399 (2)
N3—C9	1.475 (2)	C12—C13	1.403 (2)
N4—C7	1.431 (2)	C13—C14	1.407 (2)
N4—C10	1.461 (2)	C14—C16	1.386 (2)
N4—C11	1.471 (2)	C14—C15	1.501 (2)
N5—C16	1.472 (2)	C15—H15A	0.9800
N6—C19	1.473 (2)	C15—H15B	0.9800
C1—C2	1.386 (2)	C15—H15C	0.9800
C1—C8	1.392 (2)	C16—C17	1.386 (2)
C2—C4	1.385 (2)	C17—C19	1.389 (2)
C3—H3A	0.9800	C18—H18A	0.9800
C3—H3B	0.9800	C18—H18B	0.9800
C3—H3C	0.9800	C18—H18C	0.9800
C2—O3—C3	114.59 (13)	C7—C8—C9	120.73 (14)
C17—O8—C18	114.64 (14)	N3—C9—C8	112.28 (13)
O2—N1—O1	124.03 (16)	N3—C9—H9A	109.1
O2—N1—C1	118.24 (14)	C8—C9—H9A	109.1
O1—N1—C1	117.72 (14)	N3—C9—H9B	109.1
O5—N2—O4	124.57 (16)	C8—C9—H9B	109.1
O5—N2—C4	117.72 (15)	H9A—C9—H9B	107.9

O4—N2—C4	117.71 (15)	N3—C10—N4	111.05 (13)
C13—N3—C10	111.58 (13)	N3—C10—H10A	109.4
C13—N3—C9	113.24 (12)	N4—C10—H10A	109.4
C10—N3—C9	108.16 (12)	N3—C10—H10B	109.4
C7—N4—C10	111.31 (13)	N4—C10—H10B	109.4
C7—N4—C11	113.42 (13)	H10A—C10—H10B	108.0
C10—N4—C11	108.57 (13)	N4—C11—C12	112.52 (13)
O6—N5—O7	124.88 (18)	N4—C11—H11A	109.1
O6—N5—C16	117.60 (17)	C12—C11—H11A	109.1
O7—N5—C16	117.52 (16)	N4—C11—H11B	109.1
O10—N6—O9	124.29 (16)	C12—C11—H11B	109.1
O10—N6—C19	118.07 (16)	H11A—C11—H11B	107.8
O9—N6—C19	117.63 (16)	C19—C12—C13	117.83 (15)
C2—C1—C8	123.48 (15)	C19—C12—C11	122.07 (15)
C2—C1—N1	116.55 (14)	C13—C12—C11	120.10 (14)
C8—C1—N1	119.94 (14)	C12—C13—C14	121.72 (14)
O3—C2—C4	122.92 (15)	C12—C13—N3	121.06 (14)
O3—C2—C1	120.70 (15)	C14—C13—N3	117.17 (14)
C4—C2—C1	116.32 (15)	C16—C14—C13	116.51 (15)
O3—C3—H3A	109.5	C16—C14—C15	121.97 (15)
O3—C3—H3B	109.5	C13—C14—C15	121.35 (15)
H3A—C3—H3B	109.5	C14—C15—H15A	109.5
O3—C3—H3C	109.5	C14—C15—H15B	109.5
H3A—C3—H3C	109.5	H15A—C15—H15B	109.5
H3B—C3—H3C	109.5	C14—C15—H15C	109.5
C2—C4—C5	124.14 (15)	H15A—C15—H15C	109.5
C2—C4—N2	117.37 (15)	H15B—C15—H15C	109.5
C5—C4—N2	118.49 (15)	C14—C16—C17	124.73 (15)
C4—C5—C7	116.71 (14)	C14—C16—N5	119.63 (15)
C4—C5—C6	122.07 (15)	C17—C16—N5	115.62 (14)
C7—C5—C6	121.17 (15)	O8—C17—C16	118.64 (15)
C5—C6—H6A	109.5	O8—C17—C19	124.86 (16)
C5—C6—H6B	109.5	C16—C17—C19	116.49 (15)
H6A—C6—H6B	109.5	O8—C18—H18A	109.5
C5—C6—H6C	109.5	O8—C18—H18B	109.5
H6A—C6—H6C	109.5	H18A—C18—H18B	109.5
H6B—C6—H6C	109.5	O8—C18—H18C	109.5
C8—C7—C5	121.68 (14)	H18A—C18—H18C	109.5
C8—C7—N4	120.63 (14)	H18B—C18—H18C	109.5
C5—C7—N4	117.67 (14)	C17—C19—C12	122.68 (15)
C1—C8—C7	117.55 (14)	C17—C19—N6	117.90 (15)
C1—C8—C9	121.71 (14)	C12—C19—N6	119.36 (15)
O2—N1—C1—C2	−123.01 (17)	C7—N4—C10—N3	56.74 (17)
O1—N1—C1—C2	56.5 (2)	C11—N4—C10—N3	−68.78 (16)
O2—N1—C1—C8	58.8 (2)	C7—N4—C11—C12	−79.62 (16)
O1—N1—C1—C8	−121.7 (2)	C10—N4—C11—C12	44.66 (17)
C3—O3—C2—C4	76.4 (2)	N4—C11—C12—C19	168.27 (14)
C3—O3—C2—C1	−106.45 (19)	N4—C11—C12—C13	−12.4 (2)
C8—C1—C2—O3	−175.31 (14)	C19—C12—C13—C14	2.2 (2)

N1—C1—C2—O3	6.6 (2)	C11—C12—C13—C14	−177.18 (14)
C8—C1—C2—C4	2.0 (2)	C19—C12—C13—N3	179.33 (14)
N1—C1—C2—C4	−176.08 (13)	C11—C12—C13—N3	0.0 (2)
O3—C2—C4—C5	178.54 (15)	C10—N3—C13—C12	−20.9 (2)
C1—C2—C4—C5	1.3 (2)	C9—N3—C13—C12	101.40 (17)
O3—C2—C4—N2	−0.9 (2)	C10—N3—C13—C14	156.36 (14)
C1—C2—C4—N2	−178.10 (14)	C9—N3—C13—C14	−81.33 (17)
O5—N2—C4—C2	−123.3 (2)	C12—C13—C14—C16	−1.2 (2)
O4—N2—C4—C2	57.0 (2)	N3—C13—C14—C16	−178.47 (14)
O5—N2—C4—C5	57.3 (2)	C12—C13—C14—C15	174.14 (15)
O4—N2—C4—C5	−122.48 (18)	N3—C13—C14—C15	−3.1 (2)
C2—C4—C5—C7	−3.3 (2)	C13—C14—C16—C17	0.2 (2)
N2—C4—C5—C7	176.13 (14)	C15—C14—C16—C17	−175.08 (16)
C2—C4—C5—C6	179.20 (15)	C13—C14—C16—N5	178.75 (15)
N2—C4—C5—C6	−1.4 (2)	C15—C14—C16—N5	3.4 (3)
C4—C5—C7—C8	2.1 (2)	O6—N5—C16—C14	88.4 (2)
C6—C5—C7—C8	179.65 (14)	O7—N5—C16—C14	−92.3 (2)
C4—C5—C7—N4	−176.71 (14)	O6—N5—C16—C17	−93.0 (2)
C6—C5—C7—N4	0.9 (2)	O7—N5—C16—C17	86.4 (2)
C10—N4—C7—C8	−20.73 (19)	C18—O8—C17—C16	−109.32 (18)
C11—N4—C7—C8	102.05 (17)	C18—O8—C17—C19	71.6 (2)
C10—N4—C7—C5	158.08 (14)	C14—C16—C17—O8	−179.42 (15)
C11—N4—C7—C5	−79.14 (16)	N5—C16—C17—O8	2.0 (2)
C2—C1—C8—C7	−3.0 (2)	C14—C16—C17—C19	−0.3 (2)
N1—C1—C8—C7	174.98 (13)	N5—C16—C17—C19	−178.84 (15)
C2—C1—C8—C9	177.82 (14)	O8—C17—C19—C12	−179.61 (15)
N1—C1—C8—C9	−4.1 (2)	C16—C17—C19—C12	1.3 (2)
C5—C7—C8—C1	0.9 (2)	O8—C17—C19—N6	3.2 (2)
N4—C7—C8—C1	179.63 (13)	C16—C17—C19—N6	−175.87 (15)
C5—C7—C8—C9	−179.99 (13)	C13—C12—C19—C17	−2.3 (2)
N4—C7—C8—C9	−1.2 (2)	C11—C12—C19—C17	177.10 (15)
C13—N3—C9—C8	−80.15 (16)	C13—C12—C19—N6	174.90 (14)
C10—N3—C9—C8	44.05 (17)	C11—C12—C19—N6	−5.7 (2)
C1—C8—C9—N3	168.06 (13)	O10—N6—C19—C17	44.5 (2)
C7—C8—C9—N3	−11.0 (2)	O9—N6—C19—C17	−136.02 (17)
C13—N3—C10—N4	55.74 (17)	O10—N6—C19—C12	−132.75 (17)
C9—N3—C10—N4	−69.45 (17)	O9—N6—C19—C12	46.7 (2)

Fig. 1

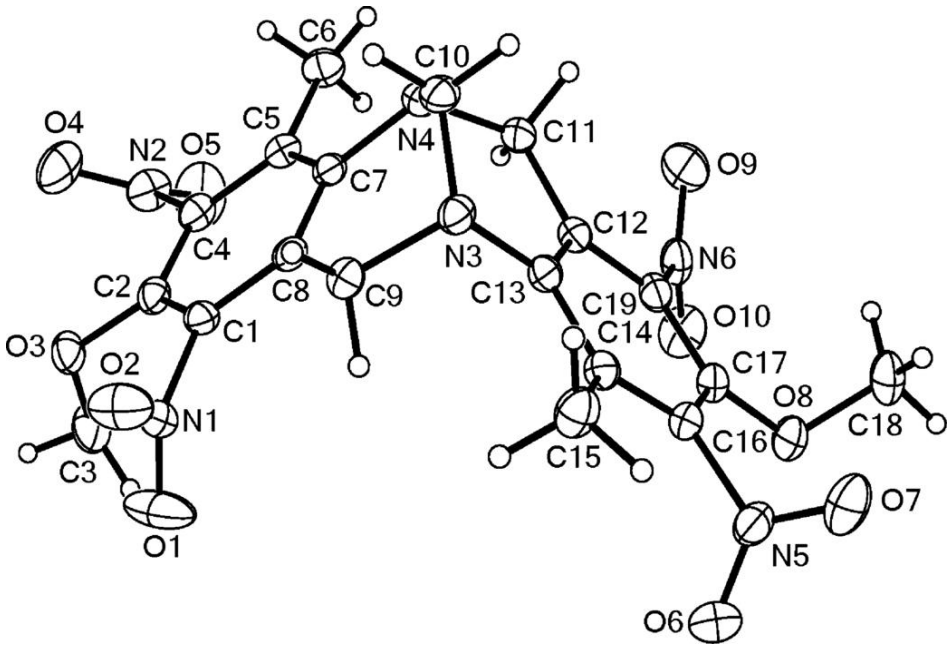
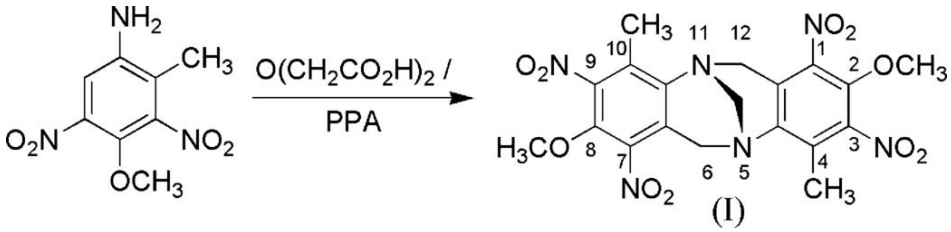


Fig. 2



(I)