$C_8H_{12}N^+.C_8H_7O_3^-.2C_8H_8O_3$

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

Mandelic acid moieties	Acid A	Acid B	Anion C
O(1)—C(1)	1.213 (3)	1.219 (2)	1.248 (2)
O(2)—C(1)	1.328 (2)	1.305 (2)	1.268 (3)
C(1)—C(2)	1.514 (3)	1.533 (3)	1.534 (3)
C(2)—O(3)	1.432 (2)	1.412 (2)	1.421 (2)
O(1)—C(1)—O(2)	124.2 (2)	123.8 (2)	125.2 (2)
O(1)—C(1)—C(2)	123.6 (2)	123.6 (2)	117.8 (2)
O(2)—C(1)—C(2)	112.1 (2)	112.6 (2)	117.0 (2)
O(3)—C(2)—C(1)	105.1 (2)	110.8 (2)	110.0 (2)
O(3)—C(2)—C(3)	113.2 (2)	109.8 (2)	111.4 (2)
C(1)—C(2)—C(3)	109.5 (2)	106.2 (2)	109.4 (2)
O(1)—C(1)—C(2)—O(3)	−12.6 (3)	−10.9 (3)	−12.2 (2)
O(3)—C(2)—C(3)—C(4)	−108.6 (2)	−137.4 (2)	−160.6 (2)

1-Phenylethylammonium ion	
N—C(10)	1.507 (3)
C(9)—C(10)	1.523 (3)
C(10)—C(11)	1.510 (3)
N—C(10)—C(11)	110.3 (2)
N—C(10)—C(9)	109.2 (2)
C(11)—C(10)—C(9)	113.2 (2)
N—C(10)—C(11)—C(12)	116.7 (2)
C(9)—C(10)—C(11)—C(12)	−120.6 (3)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···A	H···A	D···A	D—H···A
N—H(N1)···O(1C')	1.90 (3)	2.812 (2)	180 (3)
N—H(N2)···O(3C)	1.92 (3)	2.834 (2)	165 (3)
N—H(N3)···O(3A)	1.91 (3)	2.818 (2)	149 (2)
N—H(N3)···O(1A)	2.22 (3)	2.991 (2)	133 (2)
O(2A)—H(O2A)···O(1B)	1.71 (3)	2.645 (2)	169 (3)
O(3A)—H(O3A)···O(2C'')	1.85 (3)	2.616 (2)	173 (3)
O(2B)—H(O2B)···O(2C)	1.68 (4)	2.531 (2)	171 (3)
O(3B)—H(O3B)···O(3A''')	2.06 (3)	2.857 (2)	174 (3)
O(3C)—H(O3C)···O(1C)	2.03 (3)	2.626 (2)	123 (3)
O(3C)—H(O3C)···O(3B'')	2.17 (3)	2.875 (2)	135 (3)

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

The data reduction was performed using the *DREADD* programs (Blessing, 1987). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and refined by least-squares methods using *SHELXL*93 (Sheldrick, 1993). All H atoms were shown in a difference electron density map. H atoms bonded to tertiary and aromatic C atoms were fixed in ideal positions, the H atoms in the methyl group were refined with the restraint of a fixed angle and distance to the C atom and the remaining H atoms were located from a difference Fourier map. The absolute configuration was chosen to be in agreement with the known absolute configuration for (*S*)-mandelic acid and the choice was supported by a Flack (1983) χ parameter of −0.06 (16).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Three Polychloromononitrobenzenes: $C_6H_3Cl_2NO_2$, $C_6H_2Cl_3NO_2$ and $C_6HCl_4NO_2$

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Abstract

3,5-Dichloronitrobenzene (I), 3,4,5-trichloronitrobenzene (II) and 2,3,5,6-tetrachloronitrobenzene (III) are frequently used as intermediates in the manufacture of dyes as well as phyto-sanitary and drug products. In both compounds (I) and (II), the NO_2 plane and the benzene ring plane are coplanar, but in (III) these planes have a mean dihedral angle of 88°.

Comment

In the field of substituted chloro derivatives of nitrobenzene, important synthetic problems often occur because some of these compounds can only be obtained using an indirect route. One method, of great potential interest, is the selective hydrodechlorination of 'overchlorinated' compounds (Tassara, Metzger & Aune, 1975). We are interested in this hydrodechlorination reaction in connection with polychloronitrobenzenes and we have undertaken the X-ray analysis of a

series of these compounds in order to investigate the relationship between their structures, and in particular, the Cl–NO$_2$ interactions.

(I)

(II)

(III)
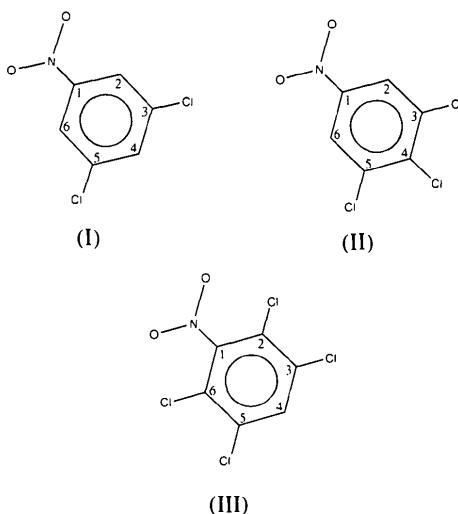
Compound (I) crystallizes in the space group $P2_1/m$, compounds (II) and (III) in $P\bar{1}$. The structure of (I) has three atoms [C(1), C(4) and N] that are located on a special position, the mirror m, while compounds (II) and (III) crystallize with two independent molecules in the asymmetric unit.

The C—Cl distances in all three compounds vary between 1.703 (4) and 1.734 (7) Å, which is in agreement with the C—Cl distances found in other benzene derivatives (Sakurai, 1962; Holden & Dickinson, 1967; Silverman, Soltberg, Yannoni & Krukonis, 1971; Tanaka, Iwasaki & Aihara, 1974; Sharma, Paulus, Weiden & Weiss, 1985).
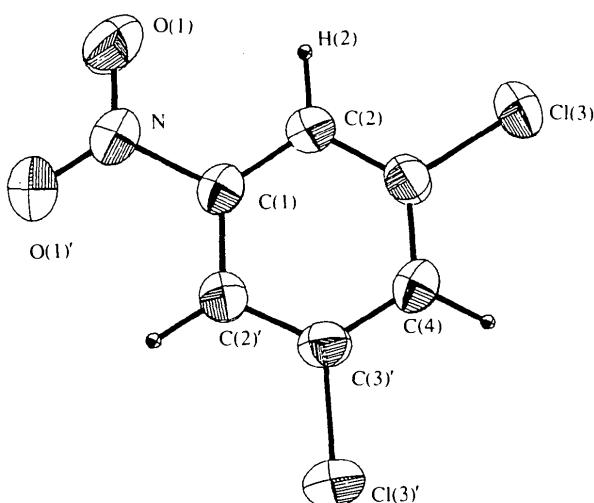
Fig. 1. *ORTEP*II (Johnson, 1976) view of compound (I). Displacement ellipsoids are shown at the 50% probability level.

Compounds (I) and (II) are nearly planar, but compound (III) has a large dihedral angle between the NO$_2$ group and the benzene ring in both molecules of the asymmetric unit [84.8 (1) and 90.8 (1)°]. This is due, in particular, to the steric repulsion between the electronegative O atoms and the Cl atoms in the *ortho* positions.
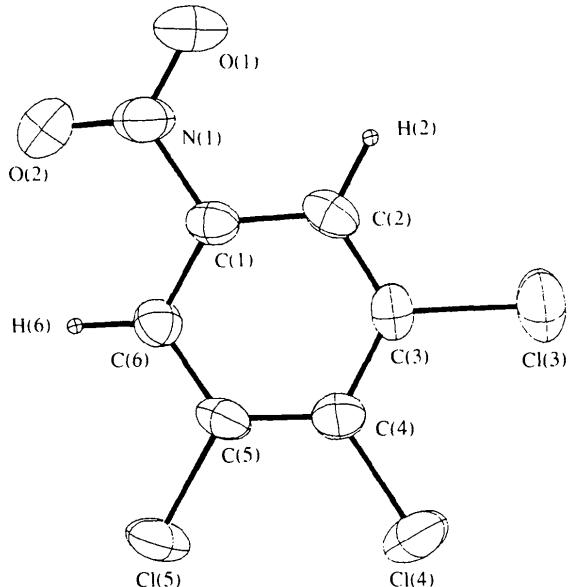
Fig. 2. *ORTEP*II view of one independent molecule of compound (II). Displacement ellipsoids are shown at the 50% probability level.
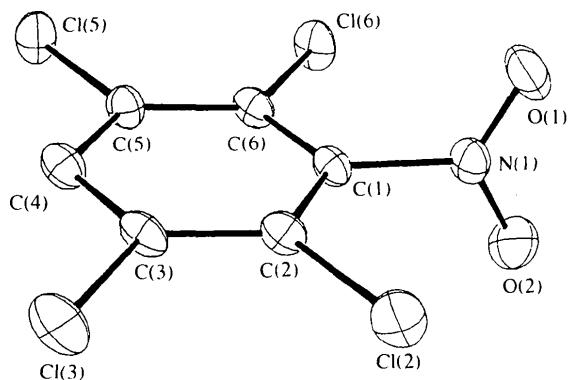
Fig. 3. *ORTEP*II view of one independent molecule of compound (III). Displacement ellipsoids are shown at the 50% probability level.

Experimental

Crystals suitable for X-ray work were obtained by slow evaporation of a saturated acetonitrile solution for compounds (I) and (II), and of a saturated hexane solution for (III), all at room temperature.

Compound (I)

Crystal data

C$_6$H$_3$Cl$_2$NO$_2$ Mo $K\alpha$ radiation
$M_r = 192$ $\lambda = 0.71069$ Å

Monoclinic

$P2_1/m$

$a = 3.873$ (2) Å

$b = 13.687$ (2) Å

$c = 7.013$ (3) Å

$\beta = 92.94$ (6)°

$V = 371.3$ (1) Å^3

$Z = 2$

$D_x = 1.716$ Mg m^{-3}

Cell parameters from 25 reflections

$\theta = 10-16°$

$\mu = 0.826$ mm^{-1}

$T = 293$ K

Rectangular

$0.3 \times 0.2 \times 0.2$ mm

Yellow

Triclinic

$P\bar{1}$

$a = 7.842$ (2) Å

$b = 14.450$ (5) Å

$c = 7.548$ (2) Å

$\alpha = 101.08$ (2)°

$\beta = 98.10$ (2)°

$\gamma = 84.38$ (3)°

$V = 828.9$ (4) Å^3

$Z = 4$

$D_x = 1.814$ Mg m^{-3}

Cell parameters from 25 reflections

$\theta = 11-17°$

$\mu = 1.062$ mm^{-1}

$T = 293$ K

Rectangular

$0.5 \times 0.3 \times 0.3$ mm

Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer

θ–2θ scans

Absorption correction: none

665 measured reflections

559 independent reflections

495 observed reflections
[$I > 3\sigma(I)$]

$R_{int} = 0.102$

$\theta_{max} = 24°$

$h = -4 \to 4$

$k = 0 \to 15$

$l = 0 \to 8$

2 standard reflections frequency: 60 min intensity decay: 39.3%

Data collection

Enraf–Nonius CAD-4 diffractometer

θ–2θ scans

Absorption correction: none

1587 measured reflections

1571 independent reflections

1384 observed reflections
[$I > 3\sigma(I)$]

$\theta_{max} = 24°$

$h = -8 \to 8$

$k = -16 \to 16$

$l = 0 \to 8$

2 standard reflections frequency: 60 min intensity decay: 34.7%

Refinement

Refinement on F

$R = 0.046$

$wR = 0.059$

$S = 2.024$

460 reflections

55 parameters

H-atom parameters not refined

$w = 1/[\sigma^2(I_o) + (0.04I_o)^2]$

$(\Delta/\sigma)_{max} = 0.03$

$\Delta\rho_{max} = 0.37$ (4) e Å^{-3}

$\Delta\rho_{min} = -0.36$ (4) e Å^{-3}

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Refinement

Refinement on F

$R = 0.06$

$wR = 0.08$

$S = 3.086$

1293 reflections

217 parameters

H-atom parameters not refined

$w = 1/[\sigma^2(I_o) + (0.04I_o)^2]$

$(\Delta/\sigma)_{max} = 0.08$

$\Delta\rho_{max} = 0.80$ (2) e Å^{-3}

$\Delta\rho_{min} = -0.69$ (2) e Å^{-3}

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	B_{eq}
Cl(3)	0.8704 (3)	0.44558 (7)	0.2327 (1)	5.40 (2)
O(1)	0.3460 (8)	0.3268 (2)	0.8587 (4)	6.99 (8)
N	0.4100 (1)	1/4	0.7874 (6)	4.4 (1)
C(1)	0.5670 (1)	1/4	0.5998 (6)	3.4 (1)
C(2)	0.6365 (9)	0.3387 (2)	0.5188 (5)	3.49 (7)
C(3)	0.7812 (8)	0.3366 (2)	0.3426 (5)	3.57 (7)
C(4)	0.8540 (1)	1/4	0.2539 (6)	3.6 (1)

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

Cl(3)—C(3)	1.722 (3)	C(1)—C(2)	1.372 (4)
N—O(1)	1.196 (3)	C(2)—C(3)	1.382 (5)
N—C(1)	1.478 (6)	C(3)—C(4)	1.375 (4)
C(1)—N—O(1)	118.4 (2)	Cl(3)—C(3)—C(2)	118.9 (3)
N—C(1)—C(2)	117.8 (2)	Cl(3)—C(3)—C(4)	119.6 (3)
C(1)—C(2)—C(3)	116.7 (3)	C(2)—C(3)—C(4)	121.6 (3)

Compound (II)

Crystal data

$C_6H_2Cl_3NO_2$

$M_r = 226.45$

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

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

	x	y	z	B_{eq}
Cl(3)	0.8492 (2)	0.3602 (1)	0.4305 (3)	4.04 (4)
Cl(4)	0.4752 (3)	0.2984 (1)	0.3036 (3)	4.42 (4)
Cl(5)	0.1909 (2)	0.4417 (2)	0.1544 (3)	4.46 (5)
Cl(9)	0.7766 (3)	0.0878 (1)	1.3222 (3)	4.91 (5)
Cl(10)	0.6209 (2)	0.0835 (1)	0.9153 (3)	4.23 (4)
Cl(11)	0.7077 (3)	-0.0945 (1)	0.6217 (3)	4.93 (5)
O(1)	0.5460 (7)	0.7279 (3)	0.1707 (9)	6.6 (1)
O(2)	0.7993 (7)	0.7007 (3)	0.3050 (8)	5.6 (1)
O(3)	1.1517 (7)	-0.2271 (3)	1.2822 (7)	4.2 (1)
O(4)	1.1042 (8)	-0.3073 (4)	1.0117 (9)	5.1 (1)
N(1)	0.6551 (8)	0.6783 (3)	0.2451 (8)	3.6 (1)
N(2)	1.0824 (7)	-0.2372 (3)	1.1279 (8)	3.4 (1)
C(1)	0.6082 (8)	0.5840 (4)	0.2571 (9)	2.4 (1)
C(2)	0.7370 (8)	0.5239 (4)	0.3271 (9)	2.5 (1)
C(3)	0.6911 (8)	0.4348 (4)	0.3388 (9)	2.7 (1)
C(4)	0.5262 (8)	0.4084 (4)	0.2868 (9)	2.7 (1)
C(5)	0.4000 (8)	0.4733 (5)	0.2211 (9)	2.7 (1)
C(6)	0.4393 (9)	0.5622 (4)	0.2075 (9)	2.8 (1)
C(7)	0.9633 (8)	-0.1597 (4)	1.0742 (9)	2.3 (1)
C(8)	0.9297 (8)	-0.0853 (4)	1.2041 (9)	3.2 (1)
C(9)	0.8189 (8)	-0.0076 (4)	1.157 (1)	2.7 (1)
C(10)	0.7502 (8)	-0.0101 (4)	0.9754 (9)	2.6 (1)
C(11)	0.7897 (8)	-0.0876 (4)	0.8491 (9)	3.3 (1)
C(12)	0.8969 (8)	-0.1657 (4)	0.8944 (9)	2.6 (1)

Table 4. Selected geometric parameters (Å, °) for (II)

Cl(3)—C(3)	1.721 (6)	C(1)—C(2)	1.381 (8)
Cl(4)—C(4)	1.709 (7)	C(1)—C(6)	1.375 (9)
Cl(5)—C(5)	1.725 (6)	C(2)—C(3)	1.392 (9)
Cl(9)—C(9)	1.711 (6)	C(3)—C(4)	1.366 (9)
Cl(10)—C(10)	1.704 (6)	C(4)—C(5)	1.401 (9)
Cl(11)—C(11)	1.734 (7)	C(5)—C(6)	1.38 (1)
O(1)—N(1)	1.210 (8)	C(7)—C(8)	1.342 (8)
O(2)—N(1)	1.212 (8)	C(7)—C(12)	1.373 (9)
O(3)—N(2)	1.202 (7)	C(8)—C(9)	1.420 (9)
O(4)—N(2)	1.221 (7)	C(9)—C(10)	1.40 (1)
N(1)—C(1)	1.468 (8)	C(10)—C(11)	1.366 (8)
N(2)—C(7)	1.468 (8)	C(11)—C(12)	1.406 (9)
O(1)—N(1)—O(2)	124.6 (6)	Cl(5)—C(5)—C(6)	118.9 (5)
O(1)—N(1)—C(1)	116.5 (5)	C(4)—C(5)—C(6)	121.3 (6)
O(2)—N(1)—C(1)	118.8 (5)	C(1)—C(6)—C(5)	117.3 (6)
O(3)—N(2)—O(4)	124.8 (6)	N(2)—C(7)—C(8)	117.6 (6)
O(3)—N(2)—C(7)	117.9 (5)	N(2)—C(7)—C(12)	118.7 (5)
O(4)—N(2)—C(7)	117.3 (5)	C(8)—C(7)—C(12)	123.7 (6)
N(1)—C(1)—C(2)	117.7 (5)	C(7)—C(8)—C(9)	119.4 (6)
N(1)—C(1)—C(6)	118.9 (5)	Cl(9)—C(9)—C(8)	119.6 (5)
C(2)—C(1)—C(6)	123.9 (6)	Cl(9)—C(9)—C(10)	121.6 (5)
C(1)—C(2)—C(3)	116.7 (6)	C(8)—C(9)—C(10)	118.8 (5)
Cl(3)—C(3)—C(2)	117.1 (5)	Cl(10)—C(10)—C(9)	119.6 (4)
Cl(3)—C(3)—C(4)	121.0 (5)	Cl(10)—C(10)—C(11)	121.4 (5)
C(2)—C(3)—C(4)	121.8 (6)	C(9)—C(10)—C(11)	119.0 (5)
Cl(4)—C(4)—C(3)	120.4 (5)	Cl(11)—C(11)—C(10)	120.9 (5)
Cl(4)—C(4)—C(5)	120.7 (5)	Cl(11)—C(11)—C(12)	116.4 (5)
C(3)—C(4)—C(5)	118.9 (6)	C(10)—C(11)—C(12)	122.7 (6)
Cl(5)—C(5)—C(4)	119.8 (5)	C(7)—C(12)—C(11)	116.3 (5)

Compound (III)

Crystal data

C$_6$HCl$_4$NO$_2$
M_r = 260.89
Triclinic
$P\bar{1}$
a = 10.920 (3) Å
b = 10.944 (2) Å
c = 8.740 (1) Å
α = 103.17 (1)°
β = 112.68 (2)°
γ = 78.25 (2)°
V = 930.7 (4) Å^3
Z = 4
D_x = 1.862 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 10–16°
μ = 1.24 mm^{-1}
T = 293 K
Cubic
0.4 × 0.4 × 0.4 mm
Transparent

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
3130 measured reflections
2910 independent reflections
2910 observed reflections

θ_{max} = 24°
h = -12 → 12
k = -12 → 12
l = 0 → 10
2 standard reflections frequency: 60 min intensity decay: 2.3%

Refinement

Refinement on F
R = 0.039
wR = 0.056
S = 2.194
2408 reflections
235 parameters
H-atom parameters not refined

w = 1/[$\sigma^2(I_o)$ + (0.04I_o)^2]
(Δ/σ)$_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.25 (1) e Å^{-3}
$\Delta\rho_{min}$ = -0.40 (1) e Å^{-3}
Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV)

Table 5. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (III)

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

	x	y	z	B_{eq}
Cl(2)	0.45863 (8)	0.15662 (9)	0.8604 (1)	6.15 (2)
Cl(3)	0.31551 (8)	0.24535 (8)	0.5065 (1)	5.96 (2)
Cl(5)	0.76650 (9)	0.0950 (1)	0.3764 (1)	7.23 (2)
Cl(6)	0.91023 (7)	0.00425 (8)	0.7299 (1)	6.00 (2)
Cl(8)	0.79164 (8)	0.42742 (8)	-0.41950 (9)	5.79 (2)
Cl(9)	1.07961 (7)	0.28088 (8)	-0.24892 (9)	5.92 (2)
Cl(11)	0.97747 (8)	0.33376 (8)	0.31483 (9)	5.60 (2)
Cl(12)	0.69019 (7)	0.47222 (8)	0.14247 (9)	5.31 (2)
O(1)	0.7227 (3)	-0.0660 (2)	0.9310 (3)	7.18 (7)
O(2)	0.8035 (2)	0.1098 (3)	1.0472 (3)	6.98 (7)
O(3)	0.6296 (2)	0.6281 (2)	-0.1799 (3)	6.67 (7)
O(4)	0.5378 (2)	0.4575 (2)	-0.2796 (3)	6.70 (7)
N(1)	0.7388 (2)	0.0391 (2)	0.9293 (3)	4.87 (6)
N(2)	0.6328 (2)	0.5146 (2)	-0.2023 (3)	4.53 (6)
C(1)	0.6732 (2)	0.0857 (2)	0.7675 (3)	3.72 (6)
C(2)	0.5411 (2)	0.1388 (2)	0.7244 (3)	3.76 (6)
C(3)	0.4798 (3)	0.1783 (2)	0.5690 (3)	4.07 (7)
C(4)	0.5504 (3)	0.1647 (3)	0.4641 (4)	4.74 (7)
C(5)	0.6823 (3)	0.1119 (3)	0.5105 (3)	4.46 (7)
C(6)	0.7467 (3)	0.0709 (2)	0.6652 (3)	4.10 (7)
C(7)	0.7635 (2)	0.4419 (2)	-0.1260 (3)	3.69 (6)
C(8)	0.8462 (2)	0.3997 (2)	-0.2181 (3)	4.01 (6)
C(9)	0.9716 (2)	0.3356 (3)	-0.1401 (3)	4.17 (6)
C(10)	1.0111 (3)	0.3171 (3)	0.0226 (3)	4.31 (7)
C(11)	0.9267 (3)	0.3587 (3)	0.1115 (3)	4.04 (6)
C(12)	0.8000 (2)	0.4218 (3)	0.0370 (3)	3.71 (6)

Table 6. Selected geometric parameters (Å, °) for (III)

Cl(2)—C(2)	1.707 (3)	N(2)—C(7)	1.470 (3)
Cl(3)—C(3)	1.717 (3)	C(1)—C(2)	1.378 (3)
Cl(5)—C(5)	1.703 (4)	C(1)—C(6)	1.381 (5)
Cl(6)—C(6)	1.709 (3)	C(2)—C(3)	1.388 (4)
Cl(8)—C(8)	1.705 (3)	C(3)—C(4)	1.376 (5)
Cl(9)—C(9)	1.725 (3)	C(4)—C(5)	1.380 (4)
Cl(11)—C(11)	1.714 (3)	C(5)—C(6)	1.392 (4)
Cl(12)—C(12)	1.716 (3)	C(7)—C(8)	1.378 (4)
O(1)—N(1)	1.202 (3)	C(7)—C(12)	1.380 (4)
O(2)—N(1)	1.207 (3)	C(8)—C(9)	1.392 (3)
O(3)—N(2)	1.208 (3)	C(9)—C(10)	1.368 (4)
O(4)—N(2)	1.206 (3)	C(10)—C(11)	1.372 (4)
N(1)—C(1)	1.474 (4)	C(11)—C(12)	1.392 (3)
O(1)—N(1)—O(2)	126.3 (3)	N(2)—C(7)—C(8)	118.7 (2)
O(1)—N(1)—C(1)	116.7 (2)	N(2)—C(7)—C(12)	118.6 (3)
O(2)—N(1)—C(1)	117.0 (3)	C(8)—C(7)—C(12)	122.6 (2)
O(3)—N(2)—O(4)	125.3 (3)	Cl(8)—C(8)—C(7)	119.3 (2)
O(3)—N(2)—C(7)	116.5 (2)	Cl(8)—C(8)—C(9)	123.3 (2)
O(4)—N(2)—C(7)	118.2 (3)	C(7)—C(8)—C(9)	117.4 (3)
N(1)—C(1)—C(2)	118.5 (3)	Cl(9)—C(9)—C(8)	119.5 (2)
N(1)—C(1)—C(6)	118.0 (2)	Cl(9)—C(9)—C(10)	119.5 (2)
C(2)—C(1)—C(6)	123.4 (2)	C(8)—C(9)—C(10)	120.9 (3)
Cl(2)—C(2)—C(1)	120.4 (2)	C(9)—C(10)—C(11)	120.7 (2)
Cl(2)—C(2)—C(3)	122.0 (2)	Cl(11)—C(11)—C(10)	120.1 (2)
C(1)—C(2)—C(3)	117.6 (3)	Cl(11)—C(11)—C(12)	120.0 (2)
Cl(3)—C(3)—C(2)	120.3 (3)	C(10)—C(11)—C(12)	119.9 (3)
Cl(3)—C(3)—C(4)	119.4 (2)	Cl(12)—C(12)—C(7)	120.3 (2)
C(2)—C(3)—C(4)	120.3 (2)	Cl(12)—C(12)—C(11)	121.4 (2)
C(3)—C(4)—C(5)	121.0 (3)	C(7)—C(12)—C(11)	118.3 (3)
Cl(5)—C(5)—C(4)	119.7 (2)		
Cl(5)—C(5)—C(6)	120.4 (2)		
C(4)—C(5)—C(6)	119.9 (3)		

During X-ray exposure measurements compounds (I) and (II) showed a significant loss in intensity (39 and 34°, respectively). However, a linear plot of standard intensity *versus* time allowed us to apply a decay correction on all reflections. The crystals are very stable outside the X-ray beam.

Program used for data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Programs used for data reduction and structure resolution: *SDP* software (Frenz, 1978) *via* direct methods *MULTAN* (Main *et al.*, 1980), completed by difference Fourier synthesis.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: PA1066). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,5-Di-*tert*-butyl-2,4-dinitrobenzene, $C_{14}H_{20}N_2O_4$

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Abstract

The present crystal study was undertaken to gain a better insight into the geometry of the nitro group. The nitro groups in 1,5-di-*tert*-butyl-2,4-dinitrobenzene are

rotated by approximately 65° out of the plane of the aromatic ring.

Comment

Crystals suitable for X-ray diffraction were grown *in vacuo* at 473 K by sublimation of a crude sample of 3,5-di-*tert*-butyl-2-nitrobenzoic acid (2) prepared according to Franck & Leser (1970); this crude sample probably also contained compound (3). The m.p. of the title compound (5) was 432–433 K (*cf.* 436.5–437 K; Knoester, De Konig, Verkade & Wepster, 1967). The crystals were of poor quality.
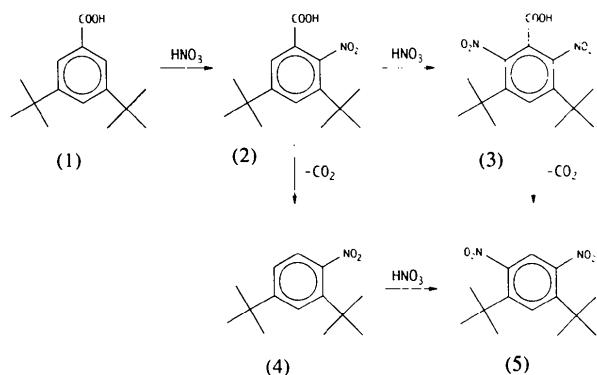
The maximum distance of a ring atom from the best plane through the benzene ring is 0.019 (5) Å. The nitro groups are tilted by 63.0 (2) and 68.0 (2)° out of the plane of the aromatic ring, whereas an average of 35 (2)° is reported for a set of nitrobenzenes with only one *ortho* substituent (De Ridder, 1992). In the title compound, the endocyclic angles are increased at the substituent sites carrying the nitro groups and decreased at the sites carrying the alkyl groups, in accordance with the observations of Domenicano & Murray-Rust (1979).
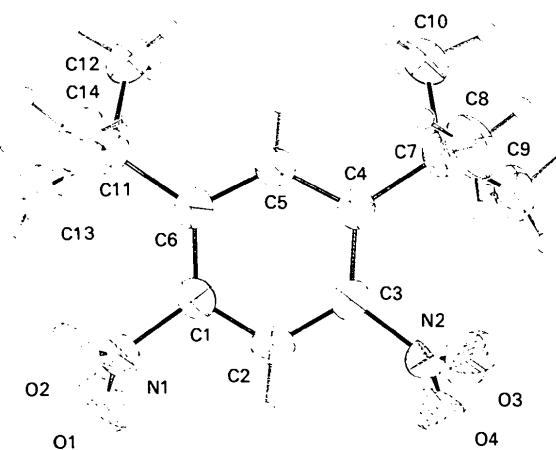

Fig. 1. *PLATON* (Spek, 1990) drawing showing the numbering system for the title compound. Displacement ellipsoids are drawn at the 50% probability level. The H atoms are shown but not labelled.