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#### Key indicators

Single-crystal X-ray study  
 T = 120 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$   
 Disorder in main residue  
 R factor = 0.030  
 wR factor = 0.073  
 Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## Cocrystallized 1,2-dibromo-4,5-dimethyl-3-nitrobenzene and 1,2-dibromo-4,5,6-trimethyl-3-nitrobenzene

In the crystal structure of the title compound,  $\text{C}_8\text{H}_7\text{Br}_2\text{NO}_2 \cdot \text{C}_9\text{H}_9\text{Br}_2\text{NO}_2$ , the 1,2-dibromo-4,5-dimethyl-3-nitrobenzene and 1,2-dibromo-4,5,6-trimethyl-3-nitrobenzene molecules occupy the same crystallographic position, such that the aromatic H atom of the former compound is superimposed on the methyl group of the latter. The structure is thus best modelled by a 50:50 disorder of the two compounds. All non-H atoms are located on a mirror plane except the O atoms of the nitro group.

#### Comment

1,2-Dibromo-4,5-dimethyl-3-nitrobenzene was required as a reagent for the synthesis of 1,2-bis(mercapto)-4,5-dimethyl-3-nitrobenzene, which can be used as a 1,2-dithiolate ligand. However, melting point measurements revealed that this compound melts over a wide temperature range and NMR spectra were more complex than expected. Therefore, a single-crystal structure determination was performed, which shows that the sample is a cocrystallized mixture of the expected material and 1,2-dibromo-4,5,6-trimethyl-3-nitrobenzene.

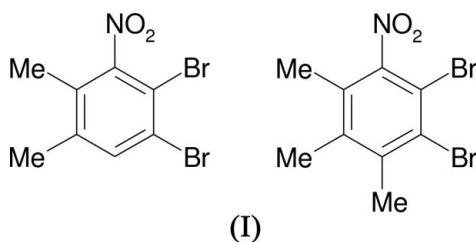


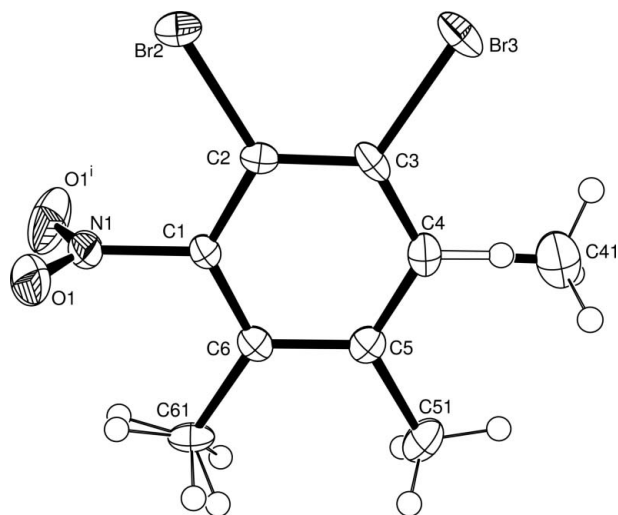
Fig. 1 shows the superimposed molecules within the crystal structure, the only difference lying in the replacement of the H atom at C4 by a methyl group. All non-H atoms are located on a crystallographic mirror plane, except the O atoms of the nitro group, which occupy general positions. The H atoms of one of the three crystallographically independent methyl groups are disordered over two orientations.

In the crystal structure, the molecules are stacked in the direction of the crystallographic *b* axis, but shifted in such a way that one C atom of the six-membered ring is located above and below the centroids of the six-membered rings of the neighbouring molecules (Fig. 2).

#### Experimental

A donated sample of 1,2-dibromo-4,5-dimethyl-3-nitrobenzene was recrystallized from ethanol (m.p. 381–390 K). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>,  $\delta$ , p.p.m.): 1,2-dibromo-4,5-dimethyl-3-nitrobenzene: 2.26 (s, 3H), 2.29 (s, 3H) (both Me), 7.54 (s, 1H, aryl-H); 1,2-dibromo-4,5,6-

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**Figure 1**

The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as circles of arbitrary radii. [Symmetry code: (i)  $x, \frac{1}{2} - y, z$ .] Disorder of the C6 methyl group is indicated.

trimethyl-3-nitrobenzene: 2.25 (s, 3H), 2.29 (s, 3H), 2.53 (s, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  14.7, 16.7, 19.8, 22.7, 112.5, 114.1, 122.9, 128.7, 131.5, 135.1, 139.5, 151.1 and 153.8. IR ( $\text{cm}^{-1}$ , KBr): 3094, 3026–2701, 1765, 1537, 1544, 1370, 1340, 1265, 1065, 895, 841, 738, 651, 532, 466.

#### Crystal data

$\text{C}_8\text{H}_7\text{Br}_2\text{NO}_2 \cdot \text{C}_9\text{H}_9\text{Br}_2\text{NO}_2$   
 $M_r = 631.96$   
 Orthorhombic,  $Pnma$   
 $a = 8.9730$  (3) Å  
 $b = 7.1165$  (2) Å  
 $c = 15.2972$  (5) Å  
 $V = 976.82$  (5) Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 2.149$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 1315 reflections  
 $\theta = 2.9$ – $27.5^\circ$   
 $\mu = 8.27$  mm<sup>-1</sup>  
 $T = 120$  (2) K  
 Rod, colourless  
 $0.60 \times 0.15 \times 0.15$  mm

#### Data collection

Nonius KappaCCD diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.101$ ,  $T_{\max} = 0.289$   
 10287 measured reflections  
 1199 independent reflections

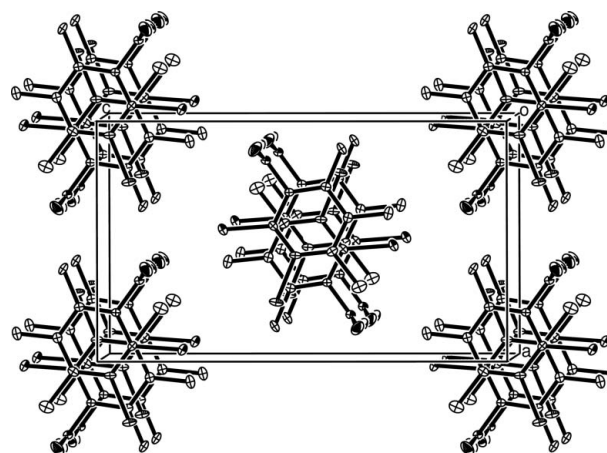
1047 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.039$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -9 \rightarrow 8$   
 $l = -17 \rightarrow 19$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.073$   
 $S = 1.11$   
 1199 reflections  
 84 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 1.2866P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.72$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.82$  e Å<sup>-3</sup>

The space groups  $Pnma$  and  $Pna2_1$  were permitted by the systematic absences;  $Pnma$  was selected and confirmed by the structure analysis. To check that the disorder was not an artefact of the selected space group, the structure was also solved in  $Pna2_1$  and in the triclinic spacegroup  $\bar{P}1$ . In both space groups the disorder was also evident. In addition, no superstructure reflections were found.


**Figure 2**

Part of the crystal structure of the title compound, showing the packing of molecules along [010]. Displacement ellipsoids are shown at the 30% level and H atoms have been omitted for clarity. Only one component is shown for each disordered group.

All H atoms were located in difference maps and then treated as riding atoms, with C–H distances of 0.95 Å (aromatic) or 0.98 Å (methyl), and  $U_{\text{iso}}(\text{H})$  values of  $1.2U_{\text{eq}}(\text{C})$  for aromatic and  $1.5U_{\text{eq}}(\text{C})$  for methyl H atoms. The occupancy of the disordered methyl (C41) group was initially refined freely, and converged to a low value (0.27) but with non-positive displacement parameters for this atom, so the occupancy was gradually increased to give displacement parameters similar to those of the other methyl groups. Finally, they were fixed at  $\frac{1}{2}$ , representing a 50:50 mixture of the cocrystallized molecules. The H atoms of one of the three methyl groups are disordered over two orientations with equal occupancies.

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: OSCAIL (McArdle, 2003) and SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: OSCAIL and SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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## supporting information

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## Cocrystallized 1,2-dibromo-4,5-dimethyl-3-nitrobenzene and 1,2-dibromo-4,5,6-trimethyl-3-nitrobenzene

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1,2-dibromo-4,5-dimethyl-3-nitrobenzene– 1,2-dibromo-4,5,6-trimethyl-3-nitrobenzene (1/1)

### Crystal data

$C_8H_7Br_2NO_2 \cdot C_9H_9Br_2NO_2$

$M_r = 631.96$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 8.9730$  (3) Å

$b = 7.1165$  (2) Å

$c = 15.2972$  (5) Å

$V = 976.82$  (5) Å<sup>3</sup>

$Z = 2$

$F(000) = 608$

$D_x = 2.149$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1315 reflections

$\theta = 2.9$ – $27.5^\circ$

$\mu = 8.27$  mm<sup>-1</sup>

$T = 120$  K

Rod, colourless

$0.60 \times 0.15 \times 0.15$  mm

### Data collection

Nonius KappaCCD  
diffractometer

Radiation source: Bruker-Nonius FR591  
rotating anode

Graphite monochromator

Detector resolution: 9.091 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Sheldrick, 2003)

$T_{\min} = 0.101$ ,  $T_{\max} = 0.289$

10287 measured reflections

1199 independent reflections

1047 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.5^\circ$

$h = -11 \rightarrow 11$

$k = -9 \rightarrow 8$

$l = -17 \rightarrow 19$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.073$

$S = 1.11$

1199 reflections

84 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 1.2866P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.72$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.82$  e Å<sup>-3</sup>

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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.6963 (5)	0.2500	0.4618 (3)	0.0192 (8)	
N1	0.8300 (4)	0.2500	0.4048 (2)	0.0323 (9)	
O1	0.8807 (3)	0.0978 (4)	0.38353 (17)	0.0532 (8)	
C2	0.5567 (5)	0.2500	0.4221 (2)	0.0189 (8)	
Br2	0.54155 (5)	0.2500	0.29954 (3)	0.03157 (15)	
C3	0.4341 (4)	0.2500	0.4764 (3)	0.0213 (8)	
Br3	0.23875 (5)	0.2500	0.42946 (3)	0.03008 (15)	
C4	0.4506 (5)	0.2500	0.5661 (3)	0.0239 (9)	
H4	0.3644	0.2500	0.6021	0.029*	0.50
C41	0.3033 (12)	0.2500	0.6320 (8)	0.043 (2)	0.50
H41A	0.2119	0.2500	0.5969	0.065*	0.50
H41B	0.3063	0.3616	0.6695	0.065*	0.50
C5	0.5912 (5)	0.2500	0.6050 (3)	0.0226 (8)	
C51	0.6091 (6)	0.2500	0.7037 (3)	0.0331 (11)	
H51A	0.6604	0.1350	0.7220	0.050*	
H51B	0.5106	0.2500	0.7313	0.050*	
C6	0.7192 (5)	0.2500	0.5512 (3)	0.0207 (8)	
C61	0.8780 (5)	0.2500	0.5900 (3)	0.0255 (9)	
H61A	0.8847	0.1545	0.6360	0.038*	0.50
H61B	0.8998	0.3739	0.6148	0.038*	0.50
H61C	0.9502	0.2216	0.5438	0.038*	0.50

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.014 (2)	0.0232 (19)	0.0200 (18)	0.000	0.0002 (15)	0.000
N1	0.0165 (19)	0.059 (3)	0.0216 (17)	0.000	-0.0007 (15)	0.000
O1	0.0462 (16)	0.0744 (19)	0.0391 (13)	0.0353 (15)	0.0170 (12)	0.0122 (14)
C2	0.019 (2)	0.0188 (18)	0.0186 (18)	0.000	-0.0043 (16)	0.000
Br2	0.0290 (3)	0.0456 (3)	0.0201 (2)	0.000	-0.00710 (17)	0.000
C3	0.0109 (19)	0.0193 (18)	0.034 (2)	0.000	-0.0014 (17)	0.000
Br3	0.0140 (2)	0.0289 (2)	0.0473 (3)	0.000	-0.00640 (19)	0.000
C4	0.022 (2)	0.022 (2)	0.028 (2)	0.000	0.0064 (18)	0.000
C41	0.034 (6)	0.044 (6)	0.051 (6)	0.000	0.008 (5)	0.000
C5	0.025 (2)	0.0222 (19)	0.0210 (19)	0.000	0.0010 (17)	0.000

C51	0.040 (3)	0.040 (3)	0.019 (2)	0.000	0.0057 (19)	0.000
C6	0.018 (2)	0.0199 (18)	0.024 (2)	0.000	-0.0012 (16)	0.000
C61	0.030 (2)	0.027 (2)	0.0199 (18)	0.000	-0.0119 (17)	0.000

*Geometric parameters (Å, °)*

C1—C6	1.383 (5)	C4—H4	0.9500
C1—C2	1.392 (6)	C41—H41A	0.9799
C1—N1	1.483 (6)	C41—H41B	0.9800
N1—O1 <sup>i</sup>	1.219 (3)	C5—C6	1.413 (6)
N1—O1	1.219 (3)	C5—C51	1.519 (6)
C2—C3	1.378 (6)	C51—H51A	0.9800
C2—Br2	1.880 (4)	C51—H51B	0.9793
C3—C4	1.380 (6)	C6—C61	1.543 (6)
C3—Br3	1.894 (4)	C61—H61A	0.9800
C4—C5	1.394 (6)	C61—H61B	0.9800
C4—C41	1.662 (11)	C61—H61C	0.9800
C6—C1—C2	124.4 (4)	C4—C41—H41A	109.5
C6—C1—N1	117.5 (4)	C4—C41—H41B	109.5
C2—C1—N1	118.1 (3)	H41A—C41—H41B	110.0
O1 <sup>i</sup> —N1—O1	125.4 (4)	C4—C5—C6	119.1 (4)
O1 <sup>i</sup> —N1—C1	117.3 (2)	C4—C5—C51	121.3 (4)
O1—N1—C1	117.3 (2)	C6—C5—C51	119.5 (4)
C3—C2—C1	117.1 (4)	C5—C51—H51A	109.5
C3—C2—Br2	122.9 (3)	C5—C51—H51B	109.4
C1—C2—Br2	120.0 (3)	H51A—C51—H51B	107.5
C2—C3—C4	120.8 (4)	C1—C6—C5	117.1 (4)
C2—C3—Br3	120.7 (3)	C1—C6—C61	121.1 (4)
C4—C3—Br3	118.5 (3)	C5—C6—C61	121.8 (4)
C3—C4—C5	121.4 (4)	C6—C61—H61A	109.5
C3—C4—C41	121.1 (5)	C6—C61—H61B	109.5
C5—C4—C41	117.4 (5)	H61A—C61—H61B	109.5
C3—C4—H4	119.3	C6—C61—H61C	109.5
C5—C4—H4	119.3	H61A—C61—H61C	109.5
C41—C4—H4	1.9	H61B—C61—H61C	109.5
C6—C1—N1—O1 <sup>i</sup>	-89.7 (3)	C2—C3—C4—C41	180.000 (2)
C2—C1—N1—O1 <sup>i</sup>	90.3 (3)	Br3—C3—C4—C41	0.000 (2)
C6—C1—N1—O1	89.7 (3)	C3—C4—C5—C6	0.000 (1)
C2—C1—N1—O1	-90.3 (3)	C41—C4—C5—C6	180.0
C6—C1—C2—C3	0.000 (1)	C3—C4—C5—C51	180.000 (1)
N1—C1—C2—C3	180.0	C41—C4—C5—C51	0.000 (2)
C6—C1—C2—Br2	180.0	C2—C1—C6—C5	0.000 (1)
N1—C1—C2—Br2	0.0	N1—C1—C6—C5	180.0
C1—C2—C3—C4	0.000 (1)	C2—C1—C6—C61	180.0
Br2—C2—C3—C4	180.0	N1—C1—C6—C61	0.000 (1)
C1—C2—C3—Br3	180.0	C4—C5—C6—C1	0.000 (1)

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Br2—C2—C3—Br3	0.0	C51—C5—C6—C1	180.000 (1)
C2—C3—C4—C5	0.000 (1)	C4—C5—C6—C61	180.000 (1)
Br3—C3—C4—C5	180.0	C51—C5—C6—C61	0.000 (1)

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Symmetry code: (i)  $x, -y+1/2, z$ .