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Packing polymorphism in the crystal structure of 4,5-dimethoxy-2-nitrobenzyl acetate

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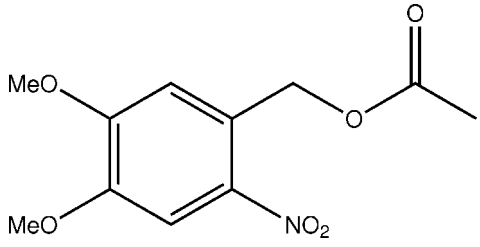
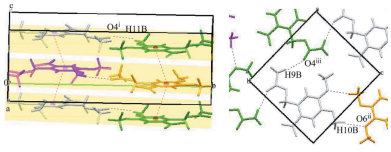
The title compound, $C_{11}H_{13}NO_6$, shows two polymorphs, orange and yellow forms, both of which crystallize in the space group $P2_1/c$. The molecular structures in the two polymorphs are essentially similar and adopt a planar structure, the maximum deviations for the non-H atoms being 0.1836 (13) and 0.1276 (13) Å, respectively, for the orange and yellow forms. In the orange crystal, molecules are linked by an intermolecular C—H···O interaction into a helical chain along the *b*-axis direction. The chains are stacked along the *c* axis through a π–π interaction [centroid–centroid distance = 3.6087 (11) Å], forming a layer parallel to the *bc* plane. In the yellow crystal, molecules are connected through C—H···O interactions into a sheet structure parallel to $(\bar{3}02)$. No significant π–π interaction is observed. The unit-cell volume of the orange crystal is larger than that of the yellow one, and this accounts for the predominant growth of the yellow crystal.

1. Chemical context

Polymorphism is of interest in crystallization, phase transition, material synthesis and the pharmaceutical industry because differences in the crystal packing and/or conformation of compounds with the same formula can change the chemical and physical properties, including solubility, bioavailability and so forth (Moulton & Zaworotko, 2001; Matsuo & Matsuoka, 2007; Yu, 2010). We have been investigating silane coupling agents and thiols with distal functional groups protected by photolabile 2-nitrobenzyl groups (Edagawa *et al.*, 2012). During the course of photoremoval studies of these materials, we found that the simple ester, 4,5-dimethoxy-2-nitrobenzyl acetate, which releases acetic acid on photo-irradiation, forms two different types of crystals, orange rods and yellow needles. Here, we report the crystal structures of these two polymorphs of the title compound.





2. Structural commentary

The molecular structures of the two crystals are approximately planar and almost identical, as shown in Fig. 1. The C2—C1—C7—O3, C9—C8—O3—C7, C5—C4—O5—C10 and C4—

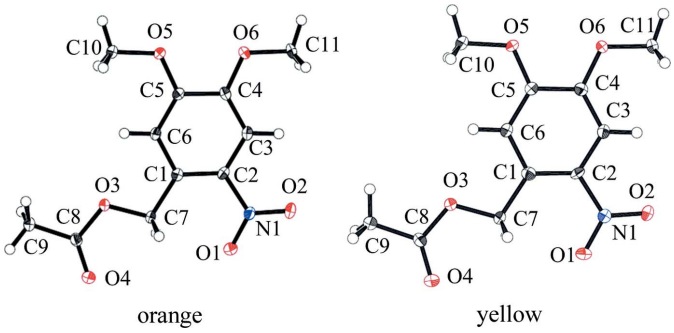


Figure 1
The molecular structures of the title compound polymorphs, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

C5—O6—C11 torsion angles in the two crystals are approximately 180°. The dihedral angles between the benzene ring (C1–C6) and the nitro group (O1/N1/O2) are 9.54 (11) and 4.15 (7)° for the orange and yellow polymorphs, respectively.

3. Supramolecular features

Although the two crystals crystallize in the same space group ($P2_1/c$) with $Z' = 1$, their packing modes are different. In the orange crystal, the molecules are connected by an intermolecular C—H···O interaction [C11—H11*B*···O4^{i}; symmetry code: (i) $1-x, -\frac{1}{2}+y, \frac{3}{2}-z$; Table 1] between the methoxy group and the carbonyl group, forming a helical chain along the *b* axis as shown in Fig. 2, left. In addition, a π–π interaction between the benzene rings with a centroid–centroid distance of 3.6087 (11) Å links the chains to be stacked along the *c* axis. In the yellow crystal, the molecules located in the plane perpendicular to the *ac* plane are connected by C—H···O interactions (Table 2) between methoxy groups [C10—H10*B*···O6^{ii}; symmetry code: (ii) $1-x, 1-y, 2-z$] and between acetyl groups [C9—H9*B*···O4^{iii}; symmetry code: (iii) $-x, -\frac{1}{2}+y, \frac{1}{2}-z$], forming a sheet structure parallel to $(\bar{3}02)$ (Fig. 2, right).

Table 1
Hydrogen-bond geometry (Å, °) for orange.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···O4^{i}	0.98	2.50	3.369 (2)	147

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

Table 2
Hydrogen-bond geometry (Å, °) for yellow.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*B*···O4^{iii}	0.98	2.40	3.375 (2)	174
C10—H10*B*···O6^{ii}	0.98	2.51	3.472 (2)	169

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

In the orange crystal, the molecules are stacked in columnar structures *via* π–π interactions along the *c* axis (Fig. 3, left). In contrast, no π–π interactions are observed in the yellow crystal. The molecules are therefore terraced along the diagonal line of the *a* and *c* axes as shown in Fig. 3, right. As a result of these packing differences, the volume of the unit cell of the orange crystal is larger than that of the yellow one, *i.e.*, the orange crystal contains slightly more void space than the yellow one. This would account for the predominant growth of the yellow crystals.

4. Synthesis and crystallization

4,5-Dimethoxy-2-nitrobenzyl alcohol (0.714 g, 3.35 mmol), acetic anhydride (0.63 ml, 6.66 mmol), Et_3N (1 ml) and CH_2Cl_2 (20 ml) were placed in a 100 mL flask, and the mixture was stirred at ambient temperature overnight. The mixture was extracted with CH_2Cl_2 (20 ml × 3), washed with brine, dried over $MgSO_4$, and evaporated to give a yellow solid (0.773 g, 90% yield). The solid was crystallized by slow evaporation from a mixed solution of ethyl acetate and hexane

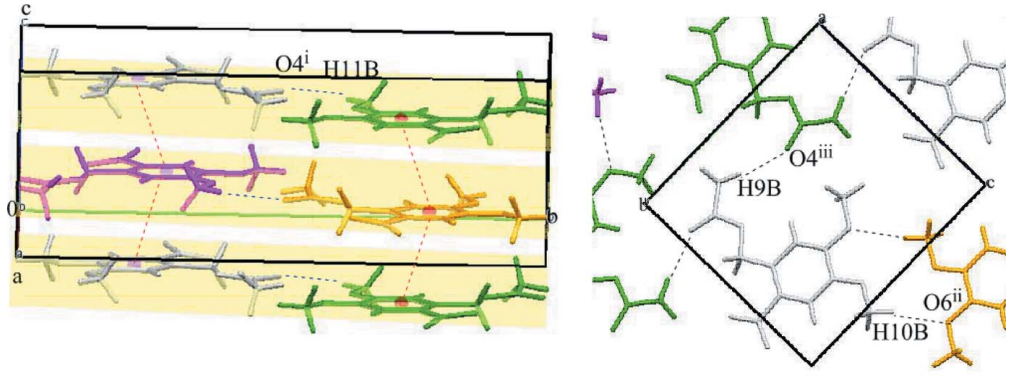


Figure 2
Intermolecular C—H···O (black dashed lines) and π–π (red dashed lines) interactions in the orange crystal (left), and intermolecular C—H···O interactions (black dashed lines) between methoxy groups and between acetyl groups in the yellow crystal (right). [Symmetry codes: (i) $1-x, -\frac{1}{2}+y, \frac{3}{2}-z$; (ii) $1-x, 1-y, 2-z$; (iii) $-x, -\frac{1}{2}+y, \frac{1}{2}-z$.]

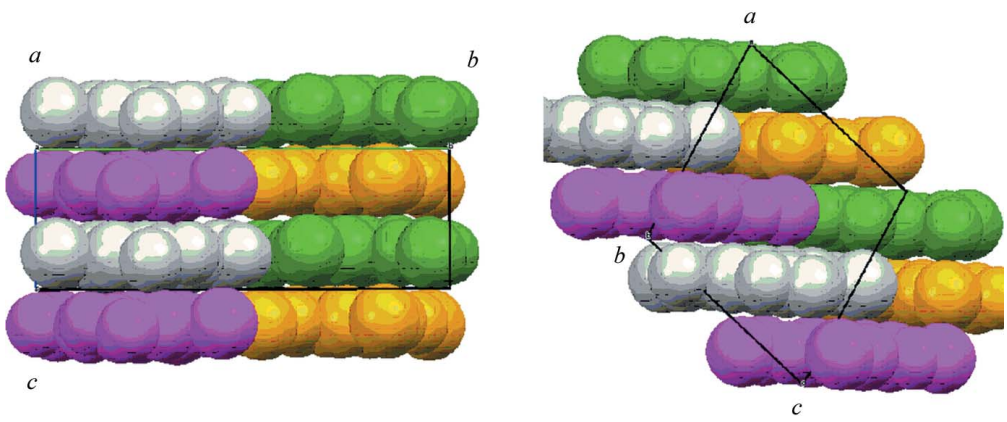


Figure 3
Side views of space-filling models of molecular packing of the orange (left) and yellow (right) crystals.

(1:1). Orange crystals were occasionally obtained in small amounts, but the yellow crystals grew predominantly.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were located geometrically and refined using a riding model, with C—H = 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene H atoms, C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms, and C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms.

Acknowledgements

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Table 3
Experimental details.

	orange	yellow
Crystal data		
Chemical formula	$C_{11}H_{13}NO_6$	$C_{11}H_{13}NO_6$
M_r	255.22	255.22
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	93	93
a, b, c (Å)	8.8751 (13), 19.555 (2), 6.8688 (9)	10.476 (3), 10.714 (3), 10.266 (3)
β (°)	106.298 (6)	105.077 (10)
V (Å^3)	1144.2 (3)	1112.6 (6)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.12	0.13
Crystal size (mm)	0.45 × 0.42 × 0.39	0.56 × 0.54 × 0.25
Data collection		
Diffractometer	Rigaku Mercury375R	Rigaku Mercury375R
Absorption correction	Multi-scan (*REQAB*; Rigaku, 1998)	Multi-scan (*REQAB*; Rigaku, 1998)
T_{min}, T_{max}	0.960, 0.970	0.797, 0.970
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11495, 2612, 2098	9498, 2058, 1769
R_{int}	0.047	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649	0.606
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.132, 1.11	0.049, 0.130, 1.13
No. of reflections	2612	2058
No. of parameters	166	166
H-atom treatment	H-atom parameters not refined	H-atom parameters not refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.30	0.38, −0.35

Computer programs: *CrystalClear-SM Expert* (Rigaku, 2011), *SIR2004* (Burla *et al.*, 2005), *SHELXL97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008), *Yadokari-XG* (Wakita, 2001).

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supporting information

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Computing details

For both compounds, data collection: *CrystalClear-SM Expert* (Rigaku, 2011). Cell refinement: *CrystalClear-SM Expert* (Rigaku, 2011) for orange; *CrystalClear-SM Expert* for yellow. Data reduction: *CrystalClear-SM Expert* (Rigaku, 2011) for orange; *CrystalClear-SM Expert* for yellow. For both compounds, program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *Yadokari-XG* (Wakita, 2001).

(orange) 4,5-Dimethoxy-2-nitrobenzyl acetate

Crystal data

$C_{11}H_{13}NO_6$
M_r = 255.22
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.8751 (13) Å
b = 19.555 (2) Å
c = 6.8688 (9) Å
β = 106.298 (6)°
V = 1144.2 (3) Å^3
Z = 4
$F(000)$ = 536
D_x = 1.48 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71069 Å
Cell parameters from 2655 reflections
θ = 3.1–27.5°
μ = 0.12 mm^{-1}
T = 93 K
Platelet, orange
0.45 × 0.42 × 0.39 mm

Data collection

Rigaku Mercury375R (2x2 bin mode) diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
profile data from ω–scans
Absorption correction: multi-scan (*REQAB*; Rigaku, 1998)
T_{min} = 0.960, T_{max} = 0.970
11495 measured reflections
2612 independent reflections
2098 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.5°, θ_{min} = 3.2°
h = −11→11
k = −25→25
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.132
S = 1.11
2612 reflections
166 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.5862P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.19328 (19)	0.29434 (8)	0.7664 (2)	0.0124 (3)
C2	0.04911 (19)	0.26435 (9)	0.7614 (3)	0.0139 (3)
C3	0.02606 (19)	0.19342 (8)	0.7577 (3)	0.0140 (3)
H3	−0.0739	0.1752	0.7541	0.017*
C4	0.1487 (2)	0.15013 (8)	0.7593 (3)	0.0139 (3)
C5	0.29755 (19)	0.17865 (8)	0.7678 (2)	0.0125 (3)
C6	0.31701 (19)	0.24911 (8)	0.7700 (2)	0.0128 (3)
H6	0.4171	0.2673	0.7740	0.015*
C7	0.22141 (19)	0.37064 (8)	0.7674 (3)	0.0138 (3)
H7A	0.1502	0.3919	0.6450	0.017*
H7B	0.2010	0.3916	0.8887	0.017*
C8	0.4225 (2)	0.44786 (9)	0.7575 (3)	0.0155 (4)
C9	0.5882 (2)	0.45412 (9)	0.7490 (3)	0.0218 (4)
H9A	0.5938	0.4425	0.6124	0.033*
H9B	0.6550	0.4227	0.8475	0.033*
H9C	0.6247	0.5012	0.7816	0.033*
C10	−0.0084 (2)	0.05009 (9)	0.7387 (3)	0.0200 (4)
H10A	−0.0436	0.0632	0.8565	0.030*
H10B	0.0007	0.0002	0.7344	0.030*
H10C	−0.0848	0.0660	0.6144	0.030*
C11	0.5657 (2)	0.15867 (9)	0.7809 (3)	0.0187 (4)
H11A	0.5598	0.1883	0.6639	0.028*
H11B	0.6370	0.1205	0.7803	0.028*
H11C	0.6051	0.1850	0.9063	0.028*
N1	−0.08677 (17)	0.30611 (7)	0.7608 (2)	0.0154 (3)
O1	−0.08009 (15)	0.36827 (7)	0.7379 (2)	0.0252 (3)
O2	−0.20491 (15)	0.27751 (7)	0.7846 (2)	0.0236 (3)
O3	0.38271 (14)	0.38139 (6)	0.7701 (2)	0.0154 (3)
O4	0.33181 (16)	0.49408 (7)	0.7520 (2)	0.0257 (3)
O5	0.14161 (14)	0.08062 (6)	0.7543 (2)	0.0176 (3)
O6	0.41110 (14)	0.13243 (6)	0.7695 (2)	0.0164 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0128 (8)	0.0155 (8)	0.0088 (8)	0.0001 (6)	0.0027 (6)	0.0011 (6)
C2	0.0112 (8)	0.0174 (8)	0.0137 (8)	0.0022 (6)	0.0046 (6)	0.0009 (6)
C3	0.0121 (8)	0.0174 (8)	0.0127 (8)	−0.0021 (6)	0.0037 (6)	0.0004 (6)
C4	0.0147 (8)	0.0137 (8)	0.0133 (8)	−0.0031 (6)	0.0040 (6)	0.0003 (6)
C5	0.0134 (8)	0.0152 (8)	0.0095 (8)	0.0014 (6)	0.0041 (6)	−0.0001 (6)
C6	0.0113 (8)	0.0160 (8)	0.0117 (8)	−0.0008 (6)	0.0043 (6)	−0.0002 (6)
C7	0.0105 (8)	0.0147 (8)	0.0177 (9)	−0.0003 (6)	0.0065 (6)	0.0002 (6)
C8	0.0161 (8)	0.0149 (8)	0.0164 (9)	−0.0025 (6)	0.0063 (7)	−0.0006 (6)
C9	0.0140 (8)	0.0182 (9)	0.0347 (11)	−0.0021 (7)	0.0094 (8)	−0.0009 (8)
C10	0.0173 (9)	0.0163 (8)	0.0271 (10)	−0.0074 (7)	0.0075 (7)	−0.0016 (7)
C11	0.0120 (8)	0.0170 (8)	0.0277 (10)	−0.0006 (6)	0.0065 (7)	−0.0011 (7)
N1	0.0113 (7)	0.0171 (7)	0.0177 (8)	−0.0005 (5)	0.0041 (6)	−0.0007 (5)
O1	0.0179 (7)	0.0159 (6)	0.0437 (9)	0.0031 (5)	0.0119 (6)	0.0037 (6)
O2	0.0133 (6)	0.0236 (7)	0.0364 (8)	−0.0016 (5)	0.0111 (6)	0.0010 (6)
O3	0.0114 (6)	0.0131 (6)	0.0227 (7)	−0.0011 (4)	0.0063 (5)	0.0000 (5)
O4	0.0203 (7)	0.0141 (6)	0.0461 (9)	0.0008 (5)	0.0147 (6)	0.0018 (6)
O5	0.0160 (6)	0.0120 (6)	0.0264 (7)	−0.0022 (5)	0.0085 (5)	−0.0001 (5)
O6	0.0124 (6)	0.0137 (6)	0.0244 (7)	0.0013 (5)	0.0071 (5)	0.0005 (5)

Geometric parameters (Å, º)

C1—C2	1.399 (2)	C8—O3	1.356 (2)
C1—C6	1.405 (2)	C8—C9	1.494 (2)
C1—C7	1.512 (2)	C9—H9A	0.9800
C2—C3	1.401 (2)	C9—H9B	0.9800
C2—N1	1.456 (2)	C9—H9C	0.9800
C3—C4	1.377 (2)	C10—O5	1.436 (2)
C3—H3	0.9500	C10—H10A	0.9800
C4—O5	1.361 (2)	C10—H10B	0.9800
C4—C5	1.420 (2)	C10—H10C	0.9800
C5—O6	1.351 (2)	C11—O6	1.446 (2)
C5—C6	1.388 (2)	C11—H11A	0.9800
C6—H6	0.9500	C11—H11B	0.9800
C7—O3	1.4419 (19)	C11—H11C	0.9800
C7—H7A	0.9900	N1—O1	1.229 (2)
C7—H7B	0.9900	N1—O2	1.2390 (19)
C8—O4	1.204 (2)		
C2—C1—C6	116.20 (15)	O3—C8—C9	110.96 (15)
C2—C1—C7	124.21 (15)	C8—C9—H9A	109.5
C6—C1—C7	119.59 (15)	C8—C9—H9B	109.5
C1—C2—C3	122.90 (15)	H9A—C9—H9B	109.5
C1—C2—N1	121.08 (15)	C8—C9—H9C	109.5
C3—C2—N1	116.02 (15)	H9A—C9—H9C	109.5
C4—C3—C2	119.83 (15)	H9B—C9—H9C	109.5

C4—C3—H3	120.1	O5—C10—H10A	109.5
C2—C3—H3	120.1	O5—C10—H10B	109.5
O5—C4—C3	125.67 (15)	H10A—C10—H10B	109.5
O5—C4—C5	115.43 (15)	O5—C10—H10C	109.5
C3—C4—C5	118.90 (15)	H10A—C10—H10C	109.5
O6—C5—C6	125.00 (15)	H10B—C10—H10C	109.5
O6—C5—C4	114.87 (15)	O6—C11—H11A	109.5
C6—C5—C4	120.12 (15)	O6—C11—H11B	109.5
C5—C6—C1	122.03 (15)	H11A—C11—H11B	109.5
C5—C6—H6	119.0	O6—C11—H11C	109.5
C1—C6—H6	119.0	H11A—C11—H11C	109.5
O3—C7—C1	107.82 (13)	H11B—C11—H11C	109.5
O3—C7—H7A	110.1	O1—N1—O2	122.43 (15)
C1—C7—H7A	110.1	O1—N1—C2	119.04 (14)
O3—C7—H7B	110.1	O2—N1—C2	118.53 (14)
C1—C7—H7B	110.1	C8—O3—C7	114.47 (13)
H7A—C7—H7B	108.5	C4—O5—C10	116.93 (13)
O4—C8—O3	122.58 (16)	C5—O6—C11	117.20 (13)
O4—C8—C9	126.45 (16)		

C6—C1—C2—C3	−0.7 (2)	C7—C1—C6—C5	−179.55 (15)
C7—C1—C2—C3	179.08 (16)	C2—C1—C7—O3	−179.18 (15)
C6—C1—C2—N1	178.95 (15)	C6—C1—C7—O3	0.6 (2)
C7—C1—C2—N1	−1.2 (3)	C1—C2—N1—O1	9.5 (2)
C1—C2—C3—C4	0.1 (3)	C3—C2—N1—O1	−170.83 (16)
N1—C2—C3—C4	−179.59 (15)	C1—C2—N1—O2	−170.13 (16)
C2—C3—C4—O5	−179.39 (16)	C3—C2—N1—O2	9.6 (2)
C2—C3—C4—C5	1.0 (2)	O4—C8—O3—C7	2.5 (2)
O5—C4—C5—O6	0.1 (2)	C9—C8—O3—C7	−176.69 (15)
C3—C4—C5—O6	179.78 (15)	C1—C7—O3—C8	175.79 (14)
O5—C4—C5—C6	178.91 (14)	C3—C4—O5—C10	2.4 (3)
C3—C4—C5—C6	−1.4 (2)	C5—C4—O5—C10	−177.96 (15)
O6—C5—C6—C1	179.47 (15)	C6—C5—O6—C11	2.1 (2)
C4—C5—C6—C1	0.8 (2)	C4—C5—O6—C11	−179.17 (15)
C2—C1—C6—C5	0.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···O4^{i}	0.98	2.50	3.369 (2)	147

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+3/2.

(yellow) 4,5-Dimethoxy-2-nitrobenzyl Acetate

Crystal data

$C_{11}H_{13}NO_6$
M_r = 255.22
Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc
a = 10.476 (3) Å
b = 10.714 (3) Å

c = 10.266 (3) Å
β = 105.077 (10)°
V = 1112.6 (6) Å^3
Z = 4
F(000) = 536
D_x = 1.52 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71069 Å

Cell parameters from 2424 reflections
θ = 3.1–27.5°
μ = 0.13 mm^{-1}
T = 93 K
Neecle, yellow
0.56 × 0.54 × 0.25 mm

Data collection

Rigaku Mercury375R (2x2 bin mode) diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
profile data from ω–scan
Absorption correction: multi-scan (*REQAB*; Rigaku, 1998)
T_{min} = 0.797, T_{max} = 0.970

9498 measured reflections
2058 independent reflections
1769 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 25.5°, θ_{min} = 3.1°
h = −12→12
k = −12→12
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.130
S = 1.13
2058 reflections
166 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2 + 0.4454P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Special details

Experimental. Rigaku (1998). REQAB. Rigaku Corporation, Tokyo, Japan.

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.22297 (15)	0.82305 (16)	0.58154 (16)	0.0158 (4)
C2	0.29963 (16)	0.88543 (15)	0.69423 (17)	0.0154 (4)
C3	0.37895 (16)	0.82230 (16)	0.80564 (16)	0.0168 (4)
H3	0.4307	0.8681	0.8801	0.020*
C4	0.38181 (16)	0.69396 (16)	0.80717 (16)	0.0167 (4)
C5	0.30317 (15)	0.62775 (16)	0.69555 (17)	0.0154 (4)
C6	0.22676 (16)	0.69258 (16)	0.58567 (16)	0.0158 (4)
H6	0.1754	0.6469	0.5109	0.019*

C7	0.13844 (16)	0.88840 (15)	0.45857 (17)	0.0166 (4)
H7A	0.0754	0.9456	0.4852	0.020*
H7B	0.1948	0.9379	0.4140	0.020*
C8	−0.01205 (16)	0.83679 (16)	0.25084 (16)	0.0175 (4)
C9	−0.08377 (18)	0.73342 (16)	0.16403 (18)	0.0216 (4)
H9A	−0.1781	0.7378	0.1603	0.032*
H9B	−0.0481	0.6529	0.2022	0.032*
H9C	−0.0721	0.7418	0.0728	0.032*
C10	0.53711 (16)	0.68572 (16)	1.02246 (16)	0.0186 (4)
H10A	0.6026	0.7354	0.9923	0.028*
H10B	0.5826	0.6245	1.0895	0.028*
H10C	0.4834	0.7409	1.0630	0.028*
C11	0.22990 (18)	0.43083 (16)	0.59702 (17)	0.0211 (4)
H11A	0.1368	0.4538	0.5832	0.032*
H11B	0.2410	0.3417	0.6185	0.032*
H11C	0.2574	0.4480	0.5146	0.032*
O1	0.23683 (12)	1.08224 (11)	0.60592 (12)	0.0219 (3)
O2	0.36433 (12)	1.07109 (11)	0.80903 (12)	0.0228 (3)
O3	0.06713 (11)	0.79354 (11)	0.36698 (12)	0.0186 (3)
O4	−0.02320 (12)	0.94638 (11)	0.22241 (12)	0.0229 (3)
O5	0.45330 (11)	0.62196 (11)	0.90924 (12)	0.0183 (3)
O6	0.30975 (12)	0.50251 (11)	0.70663 (12)	0.0187 (3)
N1	0.30069 (14)	1.02142 (14)	0.70366 (14)	0.0175 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0163 (8)	0.0161 (8)	0.0155 (8)	0.0025 (6)	0.0052 (7)	0.0004 (6)
C2	0.0196 (8)	0.0084 (8)	0.0189 (9)	0.0000 (6)	0.0064 (7)	−0.0011 (6)
C3	0.0179 (8)	0.0159 (8)	0.0154 (8)	−0.0026 (7)	0.0023 (7)	−0.0015 (6)
C4	0.0189 (8)	0.0155 (9)	0.0149 (8)	0.0008 (7)	0.0031 (7)	0.0008 (6)
C5	0.0163 (8)	0.0136 (9)	0.0158 (8)	−0.0006 (6)	0.0033 (7)	−0.0002 (6)
C6	0.0177 (8)	0.0137 (9)	0.0153 (8)	−0.0009 (6)	0.0033 (7)	−0.0023 (6)
C7	0.0193 (8)	0.0115 (8)	0.0163 (8)	−0.0006 (6)	0.0000 (7)	−0.0022 (6)
C8	0.0169 (8)	0.0188 (9)	0.0148 (8)	0.0001 (7)	0.0006 (7)	0.0014 (7)
C9	0.0233 (9)	0.0158 (9)	0.0212 (9)	0.0007 (7)	−0.0025 (7)	0.0003 (7)
C10	0.0199 (8)	0.0181 (9)	0.0144 (8)	−0.0017 (7)	−0.0018 (7)	−0.0009 (7)
C11	0.0278 (9)	0.0134 (9)	0.0188 (9)	−0.0016 (7)	0.0000 (7)	−0.0029 (6)
O1	0.0277 (7)	0.0145 (6)	0.0204 (7)	0.0032 (5)	0.0009 (5)	0.0035 (5)
O2	0.0307 (7)	0.0153 (7)	0.0189 (7)	−0.0016 (5)	−0.0001 (5)	−0.0052 (5)
O3	0.0215 (6)	0.0127 (6)	0.0173 (6)	0.0004 (5)	−0.0027 (5)	−0.0002 (5)
O4	0.0277 (7)	0.0140 (6)	0.0229 (7)	0.0001 (5)	−0.0008 (5)	0.0031 (5)
O5	0.0222 (6)	0.0131 (6)	0.0146 (6)	−0.0007 (5)	−0.0040 (5)	0.0008 (5)
O6	0.0245 (6)	0.0095 (6)	0.0183 (6)	−0.0001 (5)	−0.0013 (5)	−0.0001 (4)
N1	0.0195 (7)	0.0153 (8)	0.0168 (7)	−0.0007 (6)	0.0029 (6)	−0.0007 (6)

Geometric parameters (Å, º)

C1—C2	1.395 (2)	C8—O3	1.345 (2)
C1—C6	1.399 (2)	C8—C9	1.496 (2)
C1—C7	1.512 (2)	C9—H9A	0.9800
C2—C3	1.401 (2)	C9—H9B	0.9800
C2—N1	1.460 (2)	C9—H9C	0.9800
C3—C4	1.375 (3)	C10—O5	1.4344 (19)
C3—H3	0.9500	C10—H10A	0.9800
C4—O5	1.359 (2)	C10—H10B	0.9800
C4—C5	1.416 (2)	C10—H10C	0.9800
C5—O6	1.347 (2)	C11—O6	1.437 (2)
C5—C6	1.388 (2)	C11—H11A	0.9800
C6—H6	0.9500	C11—H11B	0.9800
C7—O3	1.4524 (19)	C11—H11C	0.9800
C7—H7A	0.9900	O1—N1	1.2368 (19)
C7—H7B	0.9900	O2—N1	1.2339 (19)
C8—O4	1.208 (2)		
C2—C1—C6	116.61 (15)	O3—C8—C9	111.81 (14)
C2—C1—C7	123.79 (16)	C8—C9—H9A	109.5
C6—C1—C7	119.60 (14)	C8—C9—H9B	109.5
C1—C2—C3	122.48 (16)	H9A—C9—H9B	109.5
C1—C2—N1	121.72 (15)	C8—C9—H9C	109.5
C3—C2—N1	115.79 (15)	H9A—C9—H9C	109.5
C4—C3—C2	119.91 (15)	H9B—C9—H9C	109.5
C4—C3—H3	120.0	O5—C10—H10A	109.5
C2—C3—H3	120.0	O5—C10—H10B	109.5
O5—C4—C3	125.62 (15)	H10A—C10—H10B	109.5
O5—C4—C5	115.33 (15)	O5—C10—H10C	109.5
C3—C4—C5	119.04 (15)	H10A—C10—H10C	109.5
O6—C5—C6	124.98 (15)	H10B—C10—H10C	109.5
O6—C5—C4	115.13 (14)	O6—C11—H11A	109.5
C6—C5—C4	119.89 (16)	O6—C11—H11B	109.5
C5—C6—C1	122.05 (15)	H11A—C11—H11B	109.5
C5—C6—H6	119.0	O6—C11—H11C	109.5
C1—C6—H6	119.0	H11A—C11—H11C	109.5
O3—C7—C1	107.90 (13)	H11B—C11—H11C	109.5
O3—C7—H7A	110.1	C8—O3—C7	115.31 (13)
C1—C7—H7A	110.1	C4—O5—C10	116.94 (13)
O3—C7—H7B	110.1	C5—O6—C11	117.37 (13)
C1—C7—H7B	110.1	O2—N1—O1	122.61 (15)
H7A—C7—H7B	108.4	O2—N1—C2	118.78 (14)
O4—C8—O3	123.19 (15)	O1—N1—C2	118.60 (13)
O4—C8—C9	125.01 (15)		
C6—C1—C2—C3	1.2 (2)	C7—C1—C6—C5	179.61 (14)
C7—C1—C2—C3	−178.75 (15)	C2—C1—C7—O3	−176.32 (14)

C6—C1—C2—N1	−178.07 (14)	C6—C1—C7—O3	3.7 (2)
C7—C1—C2—N1	2.0 (2)	O4—C8—O3—C7	0.8 (2)
C1—C2—C3—C4	−0.8 (2)	C9—C8—O3—C7	−178.75 (13)
N1—C2—C3—C4	178.48 (15)	C1—C7—O3—C8	−179.24 (13)
C2—C3—C4—O5	−179.33 (14)	C3—C4—O5—C10	−2.8 (2)
C2—C3—C4—C5	−0.4 (2)	C5—C4—O5—C10	178.28 (13)
O5—C4—C5—O6	0.4 (2)	C6—C5—O6—C11	−1.0 (2)
C3—C4—C5—O6	−178.65 (15)	C4—C5—O6—C11	178.91 (13)
O5—C4—C5—C6	−179.73 (14)	C1—C2—N1—O2	175.79 (14)
C3—C4—C5—C6	1.3 (2)	C3—C2—N1—O2	−3.5 (2)
O6—C5—C6—C1	179.03 (15)	C1—C2—N1—O1	−3.7 (2)
C4—C5—C6—C1	−0.9 (2)	C3—C2—N1—O1	177.02 (14)
C2—C1—C6—C5	−0.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*B*···O4^{i}	0.98	2.40	3.375 (2)	174
C10—H10*B*···O6^{ii}	0.98	2.51	3.472 (2)	169

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2; (ii) −*x*+1, −*y*+1, −*z*+2.