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(4-Cyanophenyl)methylene diacetate

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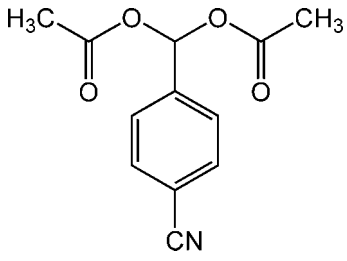
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.060; wR factor = 0.164; data-to-parameter ratio = 17.5.

In the title molecule, $C_{12}H_{11}NO_4$, the two acetyl groups are inclined by 71.3 (1) and 46.2 (1)° to the benzene ring. In the crystal structure, molecules are linked into a chain along the c axis by C—H···O hydrogen bonds.

Related literature

For general background on nitrile compounds, see: Jin *et al.* (1994); Radl *et al.* (2000). For a related structure, see: Fu & Zhao (2007).



Experimental

Crystal data

$C_{12}H_{11}NO_4$
M_r = 233.22
Monoclinic, $P2_1/c$
a = 8.1389 (15) Å
b = 20.919 (3) Å
c = 7.7748 (10) Å
β = 115.531 (7)°
V = 1194.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.35 × 0.30 × 0.30 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.951, T_{max} = 0.968
11568 measured reflections
2723 independent reflections
2096 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.163
S = 1.11
2723 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2^{i}	0.98	2.56	3.351 (3)	137

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This work was supported by a start-up grant from Southeast University to Professor Ren-Gen Xiong.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2587).

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supporting information

Acta Cryst. (2008). E**64**, o986 [doi:10.1107/S1600536808012543]

(4-Cyanophenyl)methylene diacetate

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S1. Comment

Nitrile compounds are used extensively in the chemical industry. They are discharged into the environment in industrial waste water, agricultural chemicals, *etc*. Nitrile derivatives are important materials in the synthesis of some heterocyclic molecules (Radl *et al.*, 2000), and they have been used as starting materials for phthalocyanines (Jin *et al.*, 1994). Recently, we have reported the crystal structure of a benzonitrile compound (Fu *et al.*, 2007). The title compound was unexpectedly obtained during our work on the nitrile compounds. Herein we report its crystal structure.

The bond lengths and angles have normal values. The O1/O2/C9/C10 and O3/O4/C11/C12 planes form dihedral angles of 71.3 (1)° and 46.2 (1)°, respectively, with the C2—C7 plane. The molecules are linked into a chain along the *c* axis by C—H···O hydrogen bonds (Table 1).

S2. Experimental

4-Formylbenzonitrile (0.262 mg, 2 mmol) was dissolved in acetic anhydride (5 ml) and heated under reflux for 3 h. The mixture was cooled to room temperature and the solution was filtered. The solvent was removed under vacuum from the filtrate to obtain a white precipitate of the title compound (yield 88%). Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol (15 ml) solution of the compound (100 mg) after 4 d.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2–1.5U_{eq}(C).

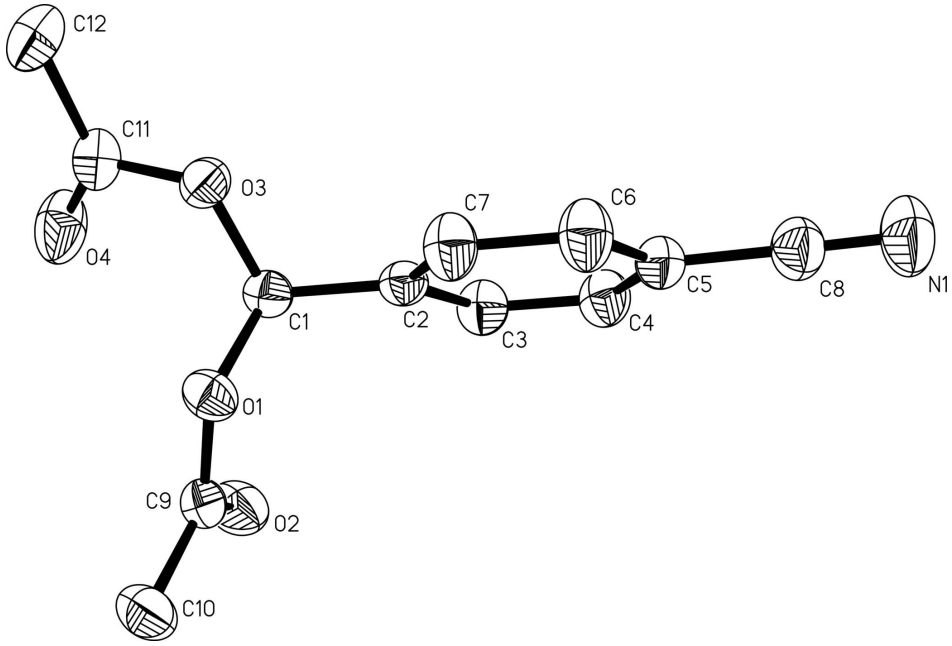

Figure 1

The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

(4-Cyanophenyl)methylene diacetate

Crystal data

$C_{12}H_{11}NO_4$
$M_r = 233.22$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.1389\ (15)$ Å
$b = 20.919\ (3)$ Å
$c = 7.7748\ (10)$ Å
$\beta = 115.531\ (7)°$
$V = 1194.5\ (3)$ Å^3
$Z = 4$
$F(000) = 488$
$D_x = 1.297$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2461 reflections
$\theta = 3.1–27.5°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, colourless
0.35 × 0.30 × 0.30 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.951$, $T_{max} = 0.968$
11568 measured reflections
2723 independent reflections
2096 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -10\rightarrow10$
$k = -27\rightarrow27$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.163$
$S = 1.11$
2723 reflections
156 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 constrained
$w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.2026P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4962 (2)	0.34433 (9)	0.7629 (3)	0.0477 (4)
H1	0.5242	0.2985	0.7757	0.057*
C2	0.6526 (2)	0.38246 (9)	0.7624 (2)	0.0462 (4)
C3	0.8165 (3)	0.35208 (10)	0.8026 (3)	0.0586 (5)
H3	0.8287	0.3086	0.8300	0.070*
C4	0.9621 (3)	0.38610 (11)	0.8021 (3)	0.0626 (5)
H4	1.0722	0.3656	0.8301	0.075*
C5	0.9433 (3)	0.45058 (10)	0.7598 (3)	0.0565 (5)
C6	0.7805 (3)	0.48137 (11)	0.7208 (4)	0.0693 (6)
H6	0.7685	0.5249	0.6936	0.083*
C7	0.6354 (3)	0.44710 (10)	0.7223 (3)	0.0627 (5)
H7	0.5260	0.4677	0.6962	0.075*
C8	1.0936 (3)	0.48634 (12)	0.7541 (4)	0.0701 (6)
C9	0.5275 (3)	0.33307 (10)	1.0797 (3)	0.0535 (5)
C10	0.4826 (4)	0.36266 (13)	1.2275 (4)	0.0779 (7)
H10A	0.5070	0.3327	1.3292	0.117*
H10B	0.3561	0.3742	1.1723	0.117*
H10C	0.5557	0.4002	1.2768	0.117*
C11	0.2023 (3)	0.31319 (10)	0.5370 (4)	0.0608 (5)
C12	0.0457 (3)	0.33230 (13)	0.3577 (4)	0.0799 (7)
H12A	−0.0238	0.2951	0.2961	0.120*
H12B	0.0895	0.3526	0.2747	0.120*
H12C	−0.0300	0.3616	0.3860	0.120*
N1	1.2104 (3)	0.51476 (12)	0.7475 (4)	0.0967 (8)
O1	0.45266 (18)	0.36634 (6)	0.91409 (19)	0.0536 (4)

O2	0.6196 (2)	0.28659 (9)	1.0993 (2)	0.0757 (5)
O3	0.33842 (17)	0.35734 (6)	0.59147 (18)	0.0545 (4)
O4	0.2134 (2)	0.26588 (8)	0.6276 (3)	0.0905 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0498 (10)	0.0476 (9)	0.0466 (9)	0.0048 (8)	0.0217 (8)	0.0008 (7)
C2	0.0465 (9)	0.0503 (10)	0.0406 (9)	0.0022 (8)	0.0178 (7)	−0.0018 (7)
C3	0.0548 (11)	0.0518 (11)	0.0689 (13)	0.0068 (9)	0.0266 (10)	0.0037 (9)
C4	0.0480 (11)	0.0687 (13)	0.0728 (14)	0.0092 (9)	0.0277 (10)	0.0020 (10)
C5	0.0496 (10)	0.0654 (12)	0.0553 (11)	−0.0037 (9)	0.0234 (9)	−0.0012 (9)
C6	0.0612 (13)	0.0515 (11)	0.0986 (18)	−0.0007 (10)	0.0377 (13)	0.0069 (11)
C7	0.0524 (11)	0.0526 (11)	0.0866 (15)	0.0078 (9)	0.0333 (11)	0.0069 (10)
C8	0.0574 (13)	0.0760 (15)	0.0783 (16)	−0.0037 (11)	0.0304 (11)	0.0028 (12)
C9	0.0505 (10)	0.0609 (12)	0.0505 (11)	−0.0104 (9)	0.0230 (9)	0.0027 (9)
C10	0.0912 (17)	0.0936 (18)	0.0642 (14)	−0.0232 (14)	0.0478 (13)	−0.0149 (12)
C11	0.0513 (11)	0.0548 (11)	0.0786 (14)	−0.0018 (9)	0.0303 (10)	−0.0020 (10)
C12	0.0525 (12)	0.0849 (16)	0.0844 (17)	−0.0072 (11)	0.0126 (11)	−0.0059 (13)
N1	0.0668 (14)	0.0997 (17)	0.129 (2)	−0.0114 (12)	0.0470 (14)	0.0127 (15)
O1	0.0614 (8)	0.0528 (7)	0.0533 (8)	0.0088 (6)	0.0310 (6)	0.0040 (6)
O2	0.0770 (11)	0.0842 (11)	0.0665 (10)	0.0217 (9)	0.0315 (8)	0.0249 (8)
O3	0.0500 (7)	0.0556 (8)	0.0513 (8)	−0.0048 (6)	0.0158 (6)	0.0033 (6)
O4	0.0642 (10)	0.0731 (11)	0.1270 (16)	−0.0078 (8)	0.0344 (10)	0.0250 (11)

Geometric parameters (Å, º)

C1—O3	1.423 (2)	C7—H7	0.93
C1—O1	1.441 (2)	C8—N1	1.141 (3)
C1—C2	1.504 (3)	C9—O2	1.197 (3)
C1—H1	0.98	C9—O1	1.355 (2)
C2—C7	1.381 (3)	C9—C10	1.483 (3)
C2—C3	1.386 (3)	C10—H10A	0.96
C3—C4	1.384 (3)	C10—H10B	0.96
C3—H3	0.93	C10—H10C	0.96
C4—C5	1.381 (3)	C11—O4	1.196 (3)
C4—H4	0.93	C11—O3	1.362 (2)
C5—C6	1.384 (3)	C11—C12	1.482 (3)
C5—C8	1.451 (3)	C12—H12A	0.96
C6—C7	1.386 (3)	C12—H12B	0.96
C6—H6	0.93	C12—H12C	0.96
O3—C1—O1	105.26 (14)	C6—C7—H7	119.8
O3—C1—C2	108.62 (14)	N1—C8—C5	179.2 (3)
O1—C1—C2	109.80 (14)	O2—C9—O1	122.60 (18)
O3—C1—H1	111.0	O2—C9—C10	126.2 (2)
O1—C1—H1	111.0	O1—C9—C10	111.2 (2)
C2—C1—H1	111.0	C9—C10—H10A	109.5

C7—C2—C3	119.60 (18)	C9—C10—H10B	109.5
C7—C2—C1	121.08 (17)	H10A—C10—H10B	109.5
C3—C2—C1	119.32 (16)	C9—C10—H10C	109.5
C4—C3—C2	120.36 (19)	H10A—C10—H10C	109.5
C4—C3—H3	119.8	H10B—C10—H10C	109.5
C2—C3—H3	119.8	O4—C11—O3	122.2 (2)
C5—C4—C3	119.73 (19)	O4—C11—C12	126.4 (2)
C5—C4—H4	120.1	O3—C11—C12	111.37 (19)
C3—C4—H4	120.1	C11—C12—H12A	109.5
C4—C5—C6	120.28 (19)	C11—C12—H12B	109.5
C4—C5—C8	120.14 (19)	H12A—C12—H12B	109.5
C6—C5—C8	119.6 (2)	C11—C12—H12C	109.5
C5—C6—C7	119.7 (2)	H12A—C12—H12C	109.5
C5—C6—H6	120.1	H12B—C12—H12C	109.5
C7—C6—H6	120.1	C9—O1—C1	116.28 (15)
C2—C7—C6	120.30 (19)	C11—O3—C1	116.52 (15)
C2—C7—H7	119.8		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2^{i}	0.98	2.56	3.351 (3)	137

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