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(3-Nitrophenyl)methanediyl diacetate

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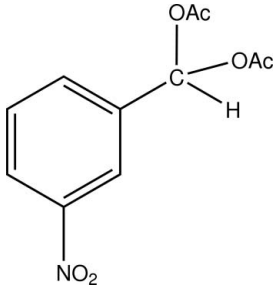
Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.098; data-to-parameter ratio = 12.9.

In the title compound, $C_{11}H_{11}NO_6$, only weak van der Waals interactions are found in the molecular packing. The compound is an efficient catalyst for the acetalization of the carbonyl group of aldehydes in nearly quantative yield.

Related literature

For background literature on silica-based sulfonic acid as catalyst in solvent-free acetalization, see: Karimi *et al.* (2000); Kumar *et al.* (2006); Smith & Reddy (2003).



Experimental

Crystal data

$C_{11}H_{11}NO_6$
M_r = 253.21
Triclinic, $P\overline{1}$
a = 7.8959 (9) Å
b = 8.4779 (10) Å
c = 9.7788 (13) Å
α = 109.094 (11)°
β = 98.031 (10)°
γ = 99.963 (10)°
V = 595.55 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 290 (2) K
0.3 × 0.2 × 0.2 mm

Data collection

Stoe IPDS diffractometer
Absorption correction: none
3869 measured reflections
2149 independent reflections
1224 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 0.91
2149 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *IPDS Software* (Stoe & Cie, 1997); cell refinement: *IPDS Software*; data reduction: *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2384).

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supporting information

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(3-Nitrophenyl)methanediyl diacetate

Ghodsi Mohammadi Ziarani, Alireza Abbasi, Alireza Badiei, Shima Ghorbi and Fatemeh Shahjafari

S1. Comment

Protection of aldehydes is important in organic chemistry. Many procedures have been done for this aim. In this work, silica based sulfonic acid was used as catalyst in solvent free condition for the acetalization of 3-nitro-benzaldehyde (Karimi, *et al.*, 2000; Kumar, *et al.*, 2006; Smith, & Reddy, 2003). The time of reaction was just 10 minutes at room temperature and the yield of reaction was more than 96%. Therefore, this catalyst was very efficient catalyst for acetalization of the carbonyl group of aldehydes.

The moleculare structure of (I) and the atom-numbering scheme are shown in Fig. 1. The nitro (NO_2) group is twisted regarding to phenyl ring by torsion angles of O3–N1–C3–C8, 4.8 (3)° and O6–N1–C3–C2, 7.1 (3)°. The methyne carbon are connected to two acetate ions and phenyl ring in a distorted tetrahedral configuration. The structure of the title compound was corroborated by IR and 1H NMR spectroscopies.

S2. Experimental

The catalyst (0.02 gr) was activated under vacuum at 100 °C followed by cooling to room temperature and then 3-nitro-benzaldehyde (3 mmol) was added to the catalyst. The mixture was stirred for two minutes and acetic anhydride (0.6 ml) was then added and stirred for 10 more minutes. The obtained solid was diluted with dichloromethane and filtered to remove the catalyst. The organic layer was washed with saturated $NaHCO_3$ solution and dried with Na_2SO_4. The solvent was evaporated under reduced pressure to obtain the title compound in yield of 96%. Crystals suitable for crystallography was obtained by crystallization from CH_2Cl_2.

S3. Refinement

All H atoms were geometrically positioned and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 and 1.5 for U_{eq}(CH and CH_3, respectively).

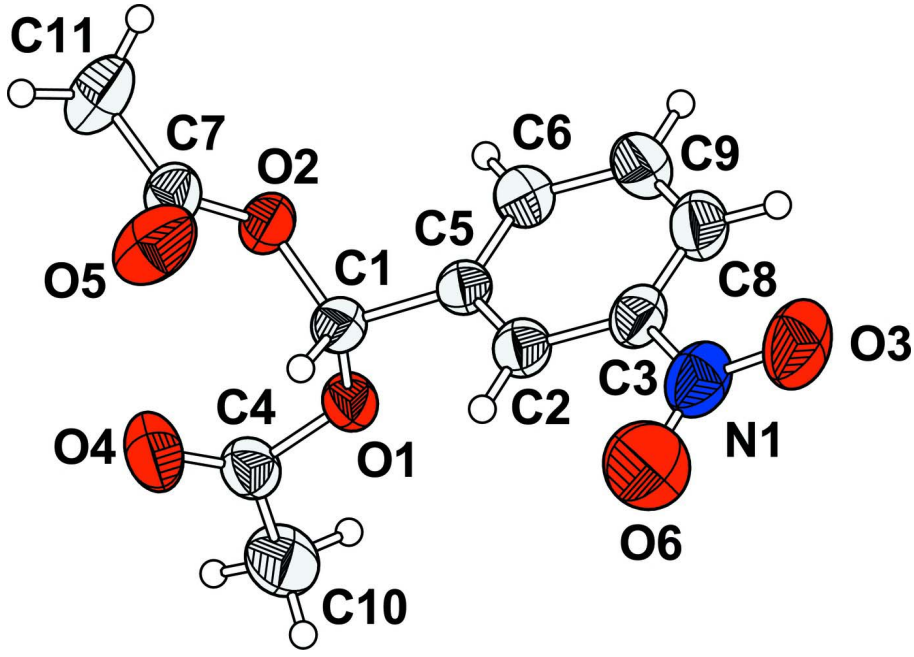

Figure 1

Molecular structure of (I), with 50% probability displacement ellipsoids. H atoms are shown as circles of arbitrary radii.

(3-Nitrophenyl)methanediyl diacetate

Crystal data

$C_{11}H_{11}NO_6$
$M_r = 253.21$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.8959\ (9)$ Å
$b = 8.4779\ (10)$ Å
$c = 9.7788\ (13)$ Å
$\alpha = 109.094\ (11)°$
$\beta = 98.031\ (10)°$
$\gamma = 99.963\ (10)°$
$V = 595.55\ (13)$ Å^3
$Z = 2$
$F(000) = 264$
$D_x = 1.412$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1543 reflections
$\theta = 3.5–25.5°$
$\mu = 0.12$ mm^{-1}
$T = 290$ K
Block shape, colorless
0.3 × 0.2 × 0.2 mm

Data collection

STOE IPDS diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Area detector – phi oscillation scans
3869 measured reflections
2149 independent reflections
1224 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 25.5°$, $\theta_{min} = 3.8°$
$h = -9\rightarrow8$
$k = -7\rightarrow10$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.098$
$S = 0.91$
2149 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 constrained
$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.100 (9)

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}
O1	0.30507 (14)	0.16974 (14)	−0.04836 (12)	0.0445 (3)
O2	0.59909 (14)	0.22011 (14)	0.05498 (12)	0.0457 (4)
O3	0.16732 (19)	0.6263 (2)	0.60185 (17)	0.0836 (5)
O4	0.43191 (18)	0.23344 (17)	−0.22188 (14)	0.0668 (4)
O5	0.75926 (17)	0.47383 (18)	0.07041 (18)	0.0791 (5)
O6	0.1787 (2)	0.7027 (2)	0.4140 (2)	0.1013 (6)
N1	0.2009 (2)	0.6066 (2)	0.4801 (2)	0.0651 (5)
C1	0.4464 (2)	0.2888 (2)	0.06532 (17)	0.0404 (4)
H1	0.4676	0.4004	0.0533	0.048*
C2	0.3243 (2)	0.4433 (2)	0.27690 (19)	0.0440 (5)
H2	0.3153	0.5260	0.2347	0.053*
C3	0.2703 (2)	0.4571 (2)	0.40713 (19)	0.0479 (5)
C4	0.3077 (3)	0.1635 (2)	−0.1894 (2)	0.0485 (5)
C5	0.3922 (2)	0.3050 (2)	0.20946 (17)	0.0394 (4)
C6	0.4043 (2)	0.1838 (2)	0.2739 (2)	0.0501 (5)
H6	0.4494	0.0901	0.2287	0.060*
C7	0.7489 (2)	0.3270 (3)	0.0531 (2)	0.0500 (5)
C8	0.2821 (2)	0.3387 (3)	0.4739 (2)	0.0580 (5)
H8	0.2456	0.3512	0.5625	0.070*
C9	0.3497 (2)	0.2009 (3)	0.4059 (2)	0.0595 (5)
H9	0.3587	0.1188	0.4487	0.071*
C10	0.1372 (3)	0.0604 (3)	−0.2909 (2)	0.0687 (6)
H10A	0.1573	0.0030	−0.3875	0.103*
H10B	0.0600	0.1348	−0.2973	0.103*
H10C	0.0842	−0.0233	−0.2535	0.103*
C11	0.8926 (2)	0.2351 (3)	0.0284 (3)	0.0726 (7)
H11A	0.9436	0.2246	0.1192	0.109*
H11B	0.9812	0.2985	−0.0039	0.109*
H11C	0.8457	0.1228	−0.0459	0.109*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0425 (7)	0.0528 (8)	0.0365 (7)	0.0072 (6)	0.0073 (6)	0.0162 (6)
O2	0.0396 (7)	0.0477 (7)	0.0545 (8)	0.0125 (6)	0.0163 (6)	0.0208 (6)
O3	0.0737 (11)	0.1055 (12)	0.0530 (9)	0.0200 (9)	0.0252 (8)	0.0001 (9)
O4	0.0720 (10)	0.0778 (10)	0.0515 (8)	0.0062 (8)	0.0219 (8)	0.0267 (8)
O5	0.0537 (9)	0.0547 (9)	0.1318 (14)	0.0070 (7)	0.0210 (9)	0.0398 (9)
O6	0.1370 (17)	0.0995 (14)	0.0925 (13)	0.0722 (13)	0.0492 (12)	0.0343 (12)
N1	0.0562 (11)	0.0744 (13)	0.0545 (12)	0.0175 (9)	0.0168 (9)	0.0068 (10)
C1	0.0376 (10)	0.0417 (10)	0.0424 (10)	0.0089 (8)	0.0088 (8)	0.0159 (8)
C2	0.0395 (10)	0.0474 (11)	0.0420 (11)	0.0066 (8)	0.0068 (8)	0.0147 (9)
C3	0.0360 (10)	0.0582 (12)	0.0415 (11)	0.0068 (9)	0.0086 (8)	0.0097 (9)
C4	0.0571 (13)	0.0512 (11)	0.0413 (12)	0.0190 (10)	0.0143 (10)	0.0172 (9)
C5	0.0327 (10)	0.0457 (10)	0.0362 (10)	0.0047 (8)	0.0040 (8)	0.0136 (8)
C6	0.0487 (11)	0.0535 (11)	0.0526 (12)	0.0128 (9)	0.0126 (9)	0.0238 (9)
C7	0.0397 (11)	0.0588 (13)	0.0508 (12)	0.0049 (10)	0.0111 (9)	0.0214 (10)
C8	0.0499 (12)	0.0771 (14)	0.0444 (11)	0.0082 (11)	0.0127 (10)	0.0206 (11)
C9	0.0589 (13)	0.0731 (14)	0.0570 (12)	0.0124 (11)	0.0149 (11)	0.0375 (11)
C10	0.0693 (15)	0.0789 (15)	0.0459 (12)	0.0100 (12)	0.0025 (11)	0.0145 (11)
C11	0.0469 (13)	0.0767 (15)	0.0971 (18)	0.0168 (11)	0.0275 (12)	0.0290 (13)

Geometric parameters (Å, º)

O1—C4	1.366 (2)	C4—C10	1.488 (3)
O1—C1	1.4249 (19)	C5—C6	1.380 (2)
O2—C7	1.367 (2)	C6—C9	1.388 (2)
O2—C1	1.4287 (18)	C6—H6	0.9300
O3—N1	1.221 (2)	C7—C11	1.487 (2)
O4—C4	1.193 (2)	C8—C9	1.378 (2)
O5—C7	1.187 (2)	C8—H8	0.9300
O6—N1	1.215 (2)	C9—H9	0.9300
N1—C3	1.474 (2)	C10—H10A	0.9600
C1—C5	1.500 (2)	C10—H10B	0.9600
C1—H1	0.9800	C10—H10C	0.9600
C2—C3	1.374 (2)	C11—H11A	0.9600
C2—C5	1.381 (2)	C11—H11B	0.9600
C2—H2	0.9300	C11—H11C	0.9600
C3—C8	1.374 (3)		
C4—O1—C1	116.57 (13)	C5—C6—H6	119.8
C7—O2—C1	116.69 (13)	C9—C6—H6	119.8
O6—N1—O3	123.62 (18)	O5—C7—O2	122.99 (17)
O6—N1—C3	117.60 (17)	O5—C7—C11	125.89 (18)
O3—N1—C3	118.8 (2)	O2—C7—C11	111.12 (17)
O1—C1—O2	107.75 (12)	C3—C8—C9	118.09 (17)
O1—C1—C5	106.49 (12)	C3—C8—H8	121.0
O2—C1—C5	111.36 (13)	C9—C8—H8	121.0

O1—C1—H1	110.4	C8—C9—C6	120.49 (18)
O2—C1—H1	110.4	C8—C9—H9	119.8
C5—C1—H1	110.4	C6—C9—H9	119.8
C3—C2—C5	119.16 (16)	C4—C10—H10A	109.5
C3—C2—H2	120.4	C4—C10—H10B	109.5
C5—C2—H2	120.4	H10A—C10—H10B	109.5
C8—C3—C2	122.43 (17)	C4—C10—H10C	109.5
C8—C3—N1	118.60 (18)	H10A—C10—H10C	109.5
C2—C3—N1	118.95 (18)	H10B—C10—H10C	109.5
O4—C4—O1	122.89 (18)	C7—C11—H11A	109.5
O4—C4—C10	126.65 (18)	C7—C11—H11B	109.5
O1—C4—C10	110.46 (16)	H11A—C11—H11B	109.5
C6—C5—C2	119.45 (16)	C7—C11—H11C	109.5
C6—C5—C1	121.85 (15)	H11A—C11—H11C	109.5
C2—C5—C1	118.68 (15)	H11B—C11—H11C	109.5
C5—C6—C9	120.38 (18)		

C4—O1—C1—O2	−74.57 (15)	C3—C2—C5—C1	178.43 (15)
C4—O1—C1—C5	165.86 (13)	O1—C1—C5—C6	79.55 (18)
C7—O2—C1—O1	129.88 (14)	O2—C1—C5—C6	−37.7 (2)
C7—O2—C1—C5	−113.68 (15)	O1—C1—C5—C2	−98.83 (16)
C5—C2—C3—C8	0.4 (3)	O2—C1—C5—C2	143.97 (14)
C5—C2—C3—N1	178.90 (15)	C2—C5—C6—C9	−0.3 (2)
O6—N1—C3—C8	−174.42 (18)	C1—C5—C6—C9	−178.70 (16)
O3—N1—C3—C8	4.8 (3)	C1—O2—C7—O5	5.7 (3)
O6—N1—C3—C2	7.1 (3)	C1—O2—C7—C11	−174.89 (14)
O3—N1—C3—C2	−173.74 (16)	C2—C3—C8—C9	−0.5 (3)
C1—O1—C4—O4	10.7 (2)	N1—C3—C8—C9	−179.01 (15)
C1—O1—C4—C10	−169.00 (14)	C3—C8—C9—C6	0.2 (3)
C3—C2—C5—C6	0.0 (2)	C5—C6—C9—C8	0.2 (3)