

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N-(3-Diacetylamino-2-methoxy-5-methylphenyl)acetamide

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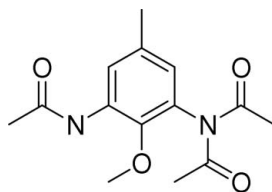
Received 1 November 2007; accepted 12 November 2007

Key indicators: single-crystal X-ray study; $T = 93$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.024; wR factor = 0.063; data-to-parameter ratio = 7.8.

The title compound, $\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}_4$, has a solid-state structure in which the imide unit is planar and perpendicular to the plane of the benzene ring. The structure is stabilized by intermolecular and intramolecular interactions involving the amide unit.

Related literature

For related literature, see: Manske & Kulka (1953); Polson & Steel (2006).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}_4$

$M_r = 278.30$

Orthorhombic, $P2_12_12_1$

$a = 7.2781$ (2) Å

$b = 9.5338$ (2) Å

$c = 20.4017$ (5) Å

$V = 1415.63$ (6) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.10$ mm⁻¹

$T = 93$ (2) K

$0.54 \times 0.24 \times 0.18$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (APEX2; Bruker, 2004)

$T_{\min} = 0.830$, $T_{\max} = 0.983$

18272 measured reflections

1481 independent reflections

1397 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.063$

$S = 1.03$

1481 reflections

190 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.14$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O4}$	0.83 (2)	2.42 (2)	2.7354 (18)	103.5 (16)
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.83 (2)	2.19 (2)	2.9731 (18)	158.6 (18)

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

Data collection: APEX2 (Bruker 2004); cell refinement: APEX2; data reduction: APEX2; program(s) used to solve structure: SHELXS97 (Sheldrick 1990); program(s) used to refine structure: SHELXL97 (Sheldrick 1997); molecular graphics: ORTEP-3 for Windows (Version 1.08; Farrugia, 1997) and Mercury (Version 1.4; Macrae *et al.*, 2006); software used to prepare material for publication: publCIF (Version 1.9.0_c; Westrip, 2007).

We thank the Foundation of Research Science and Technology for funding. PJS also thanks the Royal Society of New Zealand for the award of a James Cook Research Fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2043).

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supplementary materials

Acta Cryst. (2007). E63, o4796 [doi:10.1107/S1600536807058308]

N-(3-Diacetylamino-2-methoxy-5-methylphenyl)acetamide

M. I. J. Polson and P. J. Steel

Comment

Diazaanthracenols are of interest as binucleating ligands which may give interesting metal-metal interactions. They may be synthesized from the corresponding diaminophenols *via* the Skraup reaction (Manske & Kulka, 1953). The Skraup reaction is notoriously enthusiastic, and this can be controlled by acetylating the amine groups. During the course of such an acetylation we fortuitously synthesized the title compound (I) which crystallizes with one molecule in the asymmetric unit (Fig. 1). The imide unit of the molecule is, in essence, planar and perpendicular to the plane of the central benzene ring. In contrast the amide unit is close to coplanarity with the benzene ring with the NH hydrogen involved in an intramolecular N1—H1···O4 hydrogen bond to the methoxy oxygen atom. Molecules within the cell are linked together by a long intermolecular N1—H1···O1 hydrogen bond between the monoacetyl units (Fig. 2).

The structure is remarkably similar to the previously described *tert*-butyl analogue (Polson & Steel, 2006).

Experimental

The title compound was prepared by refluxing the free base (2,6-diamino-4-methylanisole, 1 g) in excess acetic anhydride (50 ml). After 1 h, water (50 ml) was added to the reaction mixture and the mixture was left to cool. Large blocks of (I) were found the next morning. Yield = 0.65 g (36%).

Refinement

Due to the structure only containing atoms lighter than Si, Friedel pairs were averaged. The hydrogen on N1 was found in the difference map and its position was refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N1})$. The rest of the H atoms were introduced in calculated positions as riding atoms, with distances: C—H = 0.95 and 0.98 Å for the aromatic and methyl H-atoms, respectively, and with fixed individual displacement parameters [$U(\text{H}) = 1.2 U_{\text{eq}}(\text{C}_{\text{aromatic}})$ or $U(\text{H}) = 1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$] during the refinements.

Figures

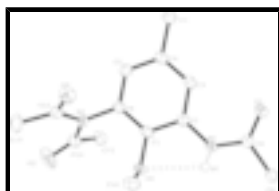


Fig. 1. The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. All H-atoms except amino-H have been omitted for clarity. The intramolecular hydrogen bond is shown as a double dashed line.

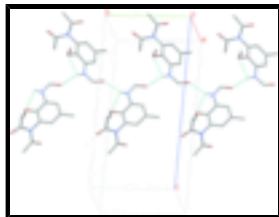


Fig. 2. A packing diagram showing the two-dimensional hydrogen-bonded sheets. Hydrogen atoms not involved in H-bonding have been omitted for clarity. Hydrogen bonds are drawn as dashed lines.

N-(3-Diacetylamino-2-methoxy-5-methylphenyl)acetamide

Crystal data

$C_{14}H_{18}N_2O_4$	$F_{000} = 592$
$M_r = 278.30$	$D_x = 1.306 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
Hall symbol: P 2ac 2ab	$\lambda = 0.71073 \text{ \AA}$
$a = 7.2781 (2) \text{ \AA}$	Cell parameters from 9389 reflections
$b = 9.5338 (2) \text{ \AA}$	$\theta = 2.4\text{--}26.4^\circ$
$c = 20.4017 (5) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$V = 1415.63 (6) \text{ \AA}^3$	$T = 93 (2) \text{ K}$
$Z = 4$	Block, colourless
	$0.54 \times 0.24 \times 0.18 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector diffractometer	1481 independent reflections
Radiation source: sealed tube	1397 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.030$
$T = 93(2) \text{ K}$	$\theta_{\text{max}} = 25.1^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.9^\circ$
Absorption correction: multi-scan (APEX2; Bruker, 2004)	$h = -8 \rightarrow 8$
$T_{\text{min}} = 0.830$, $T_{\text{max}} = 0.983$	$k = -11 \rightarrow 11$
18272 measured reflections	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.5007P]$
$R[F^2 > 2\sigma(F^2)] = 0.024$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.063$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
1481 reflections	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
190 parameters	Extinction correction: none

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

Special details

Experimental. Spectroscopic data: ¹H NMR (CDCl₃): δ 2.22 (3H, s, ArCH₃), 2.30 (6H, s, N(COCH₃)₂), 2.33 (3H, s, NHCOCH₃), 3.69 (3H, s, OCH₃), 6.63 (1H, s, ArH), 7.62 (1H, s, NH), 8.25 (1H, s, ArH);

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 F² against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F², conventional R-factors R are based on F, with F set to zero for negative F². The threshold expression of F² > 2σ(F²) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F² 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	0.6869 (2)	0.39316 (16)	0.12672 (8)	0.0187 (4)
C2	0.7441 (2)	0.29237 (18)	0.17230 (7)	0.0184 (4)
C3	0.6392 (2)	0.17077 (17)	0.17974 (8)	0.0199 (4)
H3	0.6772	0.1016	0.2104	0.024*
C4	0.4808 (3)	0.14888 (16)	0.14330 (8)	0.0212 (4)
C5	0.4215 (2)	0.25222 (17)	0.10010 (8)	0.0217 (4)
H5	0.3115	0.2394	0.0758	0.026*
C6	0.5234 (2)	0.37381 (17)	0.09270 (8)	0.0200 (4)
C7	0.3752 (3)	0.01341 (18)	0.15072 (9)	0.0275 (4)
H7A	0.4128	-0.0524	0.1163	0.041*
H7B	0.2432	0.0323	0.1468	0.041*
H7C	0.4010	-0.0276	0.1938	0.041*
N1	0.9042 (2)	0.32107 (15)	0.20881 (7)	0.0202 (3)
O1	0.98350 (19)	0.09950 (11)	0.23920 (6)	0.0258 (3)
C8	1.0144 (2)	0.22637 (16)	0.23946 (7)	0.0191 (3)
C9	1.1803 (2)	0.28573 (17)	0.27277 (8)	0.0227 (4)
H9A	1.1997	0.2373	0.3146	0.034*
H9B	1.1615	0.3860	0.2809	0.034*
H9C	1.2882	0.2728	0.2447	0.034*
N2	0.4618 (2)	0.48325 (15)	0.04880 (7)	0.0205 (3)
O2	0.3874 (3)	0.71670 (13)	0.04256 (6)	0.0468 (4)
C10	0.4050 (3)	0.61289 (18)	0.07548 (9)	0.0257 (4)
C11	0.3640 (3)	0.61353 (19)	0.14793 (8)	0.0277 (4)
H11A	0.4794	0.6100	0.1726	0.042*
H11B	0.2888	0.5316	0.1590	0.042*

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H11C	0.2970	0.6993	0.1593	0.042*
O3	0.53026 (19)	0.34119 (13)	-0.03624 (6)	0.0292 (3)
C12	0.4667 (3)	0.45270 (18)	-0.01891 (8)	0.0229 (4)
C13	0.3934 (3)	0.5565 (2)	-0.06700 (9)	0.0369 (5)
H13A	0.4767	0.6371	-0.0695	0.055*
H13B	0.2716	0.5880	-0.0529	0.055*
H13C	0.3840	0.5124	-0.1103	0.055*
O4	0.78823 (16)	0.51451 (12)	0.11885 (5)	0.0215 (3)
C14	0.9138 (3)	0.5064 (2)	0.06425 (9)	0.0299 (4)
H14A	1.0012	0.4297	0.0716	0.045*
H14B	0.9808	0.5951	0.0602	0.045*
H14C	0.8447	0.4887	0.0239	0.045*
H1	0.932 (3)	0.405 (2)	0.2128 (9)	0.025 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0246 (8)	0.0136 (7)	0.0179 (8)	-0.0006 (7)	0.0021 (7)	-0.0019 (6)
C2	0.0215 (8)	0.0171 (8)	0.0167 (8)	0.0024 (7)	-0.0002 (7)	-0.0031 (7)
C3	0.0246 (9)	0.0160 (8)	0.0189 (8)	0.0019 (7)	0.0022 (7)	0.0010 (6)
C4	0.0250 (9)	0.0172 (8)	0.0214 (8)	0.0002 (8)	0.0043 (7)	-0.0021 (6)
C5	0.0224 (8)	0.0209 (8)	0.0218 (8)	0.0005 (7)	-0.0018 (7)	-0.0033 (7)
C6	0.0240 (8)	0.0175 (8)	0.0184 (7)	0.0025 (7)	-0.0019 (7)	-0.0007 (6)
C7	0.0280 (9)	0.0218 (8)	0.0326 (9)	-0.0060 (8)	0.0002 (9)	0.0005 (8)
N1	0.0259 (8)	0.0119 (7)	0.0226 (7)	-0.0010 (6)	-0.0032 (6)	-0.0001 (6)
O1	0.0297 (7)	0.0146 (5)	0.0332 (6)	0.0012 (5)	-0.0056 (6)	0.0014 (5)
C8	0.0238 (9)	0.0173 (8)	0.0163 (7)	0.0017 (7)	0.0007 (7)	-0.0004 (6)
C9	0.0263 (9)	0.0186 (8)	0.0231 (8)	0.0018 (8)	-0.0020 (7)	0.0016 (7)
N2	0.0246 (7)	0.0184 (7)	0.0186 (7)	0.0009 (6)	-0.0036 (6)	-0.0003 (5)
O2	0.0910 (13)	0.0220 (7)	0.0273 (7)	0.0128 (8)	0.0027 (8)	0.0026 (6)
C10	0.0329 (10)	0.0205 (8)	0.0237 (9)	0.0023 (8)	-0.0029 (8)	-0.0016 (7)
C11	0.0353 (10)	0.0233 (9)	0.0244 (9)	0.0032 (8)	0.0029 (8)	-0.0017 (7)
O3	0.0363 (7)	0.0259 (7)	0.0255 (6)	0.0043 (6)	0.0028 (6)	-0.0047 (5)
C12	0.0225 (8)	0.0243 (9)	0.0220 (8)	-0.0027 (8)	-0.0013 (8)	-0.0018 (7)
C13	0.0569 (14)	0.0328 (10)	0.0209 (9)	0.0044 (11)	-0.0112 (10)	-0.0012 (8)
O4	0.0265 (6)	0.0150 (6)	0.0229 (6)	-0.0024 (5)	-0.0032 (5)	0.0006 (5)
C14	0.0313 (10)	0.0266 (9)	0.0318 (10)	-0.0072 (9)	0.0042 (8)	0.0010 (8)

Geometric parameters (\AA , $^\circ$)

C1—O4	1.381 (2)	C9—H9A	0.9800
C1—C6	1.390 (2)	C9—H9B	0.9800
C1—C2	1.400 (2)	C9—H9C	0.9800
C2—C3	1.396 (2)	N2—C10	1.412 (2)
C2—N1	1.410 (2)	N2—C12	1.412 (2)
C3—C4	1.388 (2)	O2—C10	1.203 (2)
C3—H3	0.9500	C10—C11	1.508 (2)
C4—C5	1.391 (2)	C11—H11A	0.9800
C4—C7	1.510 (2)	C11—H11B	0.9800

C5—C6	1.384 (2)	C11—H11C	0.9800
C5—H5	0.9500	O3—C12	1.212 (2)
C6—N2	1.446 (2)	C12—C13	1.492 (3)
C7—H7A	0.9800	C13—H13A	0.9800
C7—H7B	0.9800	C13—H13B	0.9800
C7—H7C	0.9800	C13—H13C	0.9800
N1—C8	1.360 (2)	O4—C14	1.443 (2)
N1—H1	0.83 (2)	C14—H14A	0.9800
O1—C8	1.230 (2)	C14—H14B	0.9800
C8—C9	1.496 (2)	C14—H14C	0.9800
O4—C1—C6	120.67 (14)	H9A—C9—H9B	109.5
O4—C1—C2	119.56 (15)	C8—C9—H9C	109.5
C6—C1—C2	119.67 (15)	H9A—C9—H9C	109.5
C3—C2—C1	118.65 (15)	H9B—C9—H9C	109.5
C3—C2—N1	123.74 (15)	C10—N2—C12	124.39 (14)
C1—C2—N1	117.60 (15)	C10—N2—C6	118.92 (13)
C4—C3—C2	121.39 (15)	C12—N2—C6	116.68 (14)
C4—C3—H3	119.3	O2—C10—N2	122.41 (15)
C2—C3—H3	119.3	O2—C10—C11	121.53 (16)
C3—C4—C5	119.39 (15)	N2—C10—C11	116.05 (15)
C3—C4—C7	119.85 (15)	C10—C11—H11A	109.5
C5—C4—C7	120.76 (16)	C10—C11—H11B	109.5
C6—C5—C4	119.75 (16)	H11A—C11—H11B	109.5
C6—C5—H5	120.1	C10—C11—H11C	109.5
C4—C5—H5	120.1	H11A—C11—H11C	109.5
C5—C6—C1	121.02 (15)	H11B—C11—H11C	109.5
C5—C6—N2	120.37 (15)	O3—C12—N2	118.42 (15)
C1—C6—N2	118.62 (15)	O3—C12—C13	121.74 (15)
C4—C7—H7A	109.5	N2—C12—C13	119.84 (16)
C4—C7—H7B	109.5	C12—C13—H13A	109.5
H7A—C7—H7B	109.5	C12—C13—H13B	109.5
C4—C7—H7C	109.5	H13A—C13—H13B	109.5
H7A—C7—H7C	109.5	C12—C13—H13C	109.5
H7B—C7—H7C	109.5	H13A—C13—H13C	109.5
C8—N1—C2	126.97 (15)	H13B—C13—H13C	109.5
C8—N1—H1	116.9 (14)	C1—O4—C14	112.50 (13)
C2—N1—H1	116.1 (14)	O4—C14—H14A	109.5
O1—C8—N1	122.89 (17)	O4—C14—H14B	109.5
O1—C8—C9	121.41 (15)	H14A—C14—H14B	109.5
N1—C8—C9	115.68 (14)	O4—C14—H14C	109.5
C8—C9—H9A	109.5	H14A—C14—H14C	109.5
C8—C9—H9B	109.5	H14B—C14—H14C	109.5

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O4	0.83 (2)	2.42 (2)	2.7354 (18)	103.5 (16)
N1—H1 \cdots O1 ⁱ	0.83 (2)	2.19 (2)	2.9731 (18)	158.6 (18)

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

Fig. 1

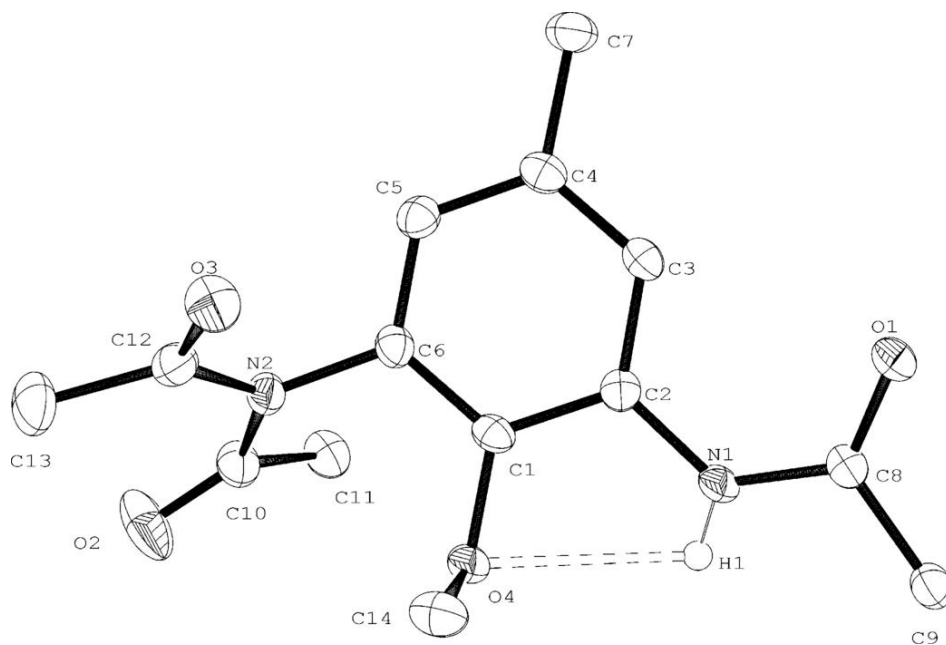


Fig. 2

