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3-Methyl-4-nitrophenol–4-dimethylaminopyridine (1/1)

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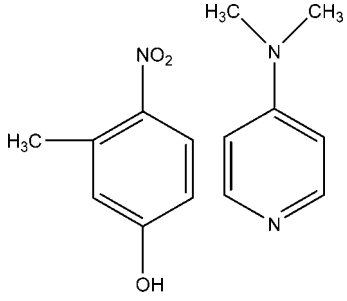
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.056; wR factor = 0.185; data-to-parameter ratio = 19.0.

In the title adduct, $C_7H_7NO_3{\cdot}C_7H_{10}N_2$, the dihedral angle betwen the benzene ring and pyridine rings is 9.60 (8)° while the nitro group attached to the benzene ring makes a dihedral angle of 21.76 (13)°. The hydroxyl O atom deviates by 0.0247 (15) Å from the plane of the benzene ring. The crystal packing features O—H···N hydrogen bonds.

Related literature

For a related structure, see: Dong & Cheng (2012).



Experimental

Crystal data

$C_7H_7NO_3{\cdot}C_7H_{10}N_2$ M_r = 275.31
Monoclinic, $P2_1/c$
a = 11.4923 (9) Å
b = 9.8362 (8) Å
c = 12.7781 (10) Å
β = 103.870 (5)°
V = 1402.3 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.35 × 0.30 × 0.30 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.968, T_{max} = 0.973
13307 measured reflections
3498 independent reflections
2469 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.185
S = 1.03
3498 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···N3^{i}	0.82	1.79	2.594 (2)	168

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection. TS thanks the DST for an Inspire fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2590).

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supporting information

Acta Cryst. (2012). E**68**, o3106 [doi:10.1107/S1600536812041670]

3-Methyl-4-nitrophenol–4-dimethylaminopyridine (1/1)

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S1. Comment

In the molecule (fig.1),the pyridine ring (N3/C10/C11/C12/C13/C14) makes a dihedral angle of 9.60 (8)° with the phenyl ring (C1/C2/C3/C4/C5/C6) system. The oxygen atom O3 deviates by -0.0247 (15)Å from the plane of the phenyl ring. The carbon atom C7 deviates by deviates by 0.0677 (21)Å from the plane of the phenyl ring.

The nitrogen atom N1 deviates by -0.0285 (18)Å from the plane of the phenyl ring. The nitrogen atom N2 devaites by -0.0292 (18)Å from the plane of the pyridine ring. The crystal packing is stabilized by intermolecular O—H···N hydrogen bonds

S2. Experimental

4-Dimethylaminopyridine and 3-methyl-4-nitrophenol were taken in equimolar (1:1) ratio using acetone as solvent. The solution was filtered in a clean beaker and optimally closed. The prepared solution was kept at room temperature for two days after which crystals suitable for X-ray diffraction were obtained.

S3. Refinement

The hydrogen atoms were placed in calculated positions with C—H = 0.93 Å to 1.08 Å refined in the riding model with fixed isotropic displacement parameters:U_{iso}(H) = 1.5U_{eq}(C) for methyl group and U_{iso}(H) = 1.2U_{eq}(C) for other groups.

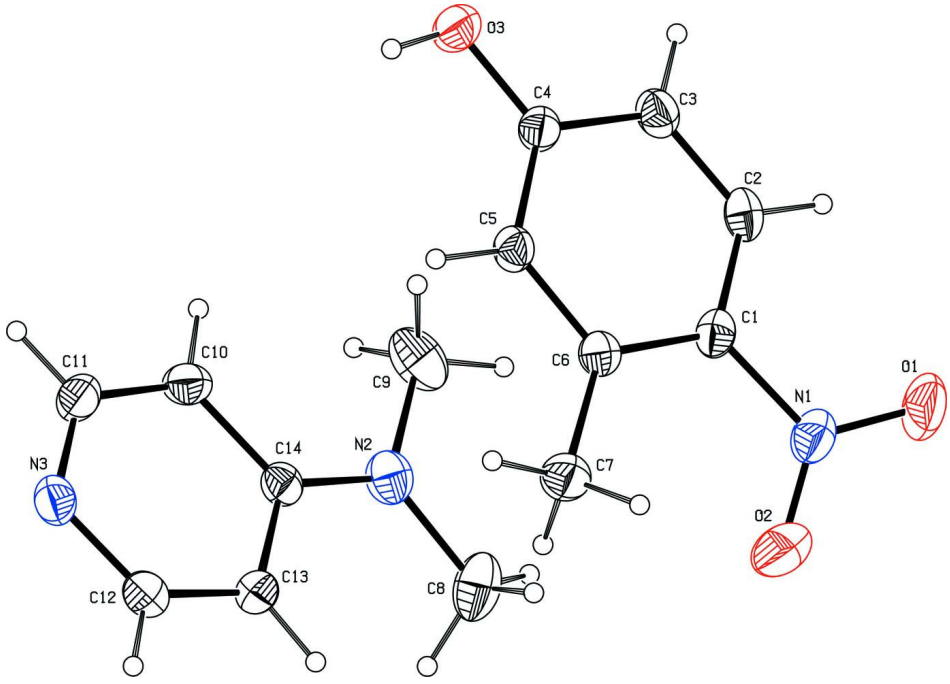

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radius.

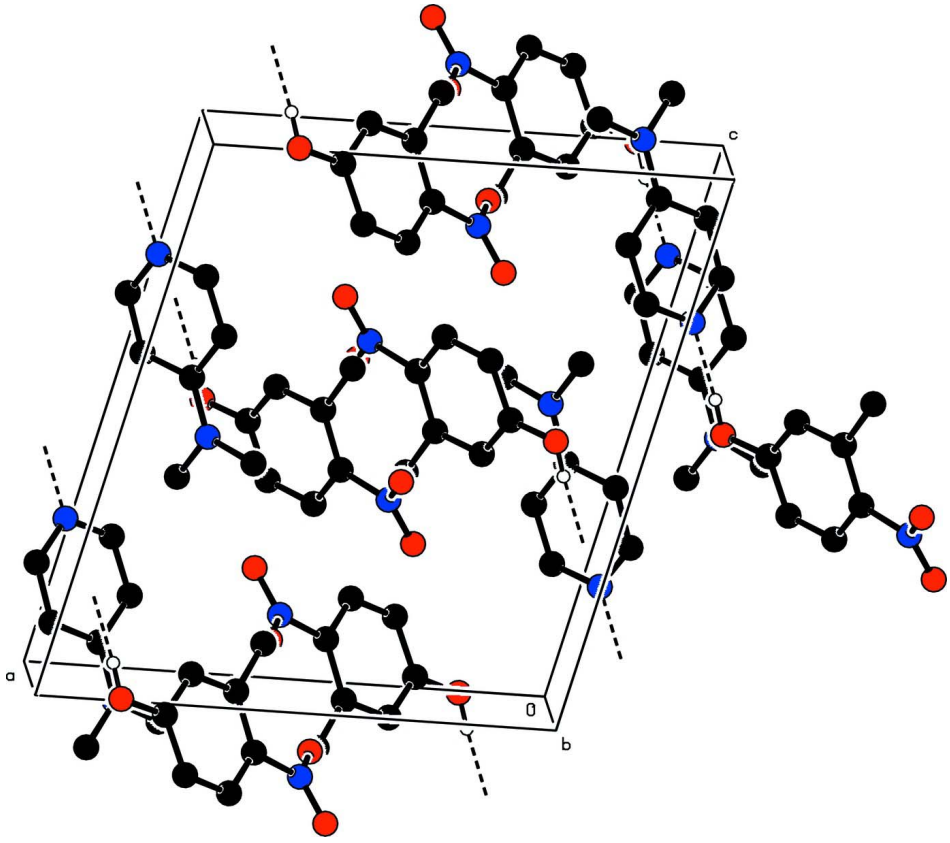

Figure 2

The crystal packing of the title compound viewed down *c* axis. H-atoms not involved in H-bonds have been excluded for clarity.

3-Methyl-4-nitrophenol–4-dimethylaminopyridine (1/1)

Crystal data

$C_7H_7NO_3 \cdot C_7H_{10}N_2$
$M_r = 275.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.4923\ (9)$ Å
$b = 9.8362\ (8)$ Å
$c = 12.7781\ (10)$ Å
$\beta = 103.870\ (5)°$
$V = 1402.3\ (2)$ Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.304$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3498 reflections
$\theta = 1.8–28.4°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colourless
0.35 × 0.30 × 0.30 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.968$, $T_{max} = 0.973$
13307 measured reflections
3498 independent reflections
2469 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 28.4°$, $\theta_{min} = 1.8°$
$h = -15 \rightarrow 12$
$k = -12 \rightarrow 13$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.185$
$S = 1.03$
3498 reflections
184 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
$w = 1/[\sigma^2(F_o^2) + (0.0939P)^2 + 0.361P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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}
O3	0.15952 (12)	−0.10464 (16)	0.99019 (11)	0.0722 (4)
H3	0.1231	−0.0878	0.9281	0.108*
N3	−0.03318 (13)	0.41197 (16)	0.70694 (12)	0.0580 (4)
N1	0.55651 (14)	0.22673 (19)	1.12638 (14)	0.0661 (4)
O1	0.63106 (15)	0.1883 (2)	1.20437 (15)	0.0964 (6)
N2	0.17044 (15)	0.41211 (18)	1.02568 (13)	0.0637 (4)
C14	0.10524 (14)	0.41079 (16)	0.92229 (13)	0.0474 (4)
C6	0.38178 (14)	0.15782 (17)	0.97841 (13)	0.0475 (4)
C1	0.45173 (14)	0.14230 (17)	1.08398 (13)	0.0498 (4)
C13	0.13746 (15)	0.48787 (18)	0.84064 (15)	0.0524 (4)
H13	0.2061	0.5415	0.8567	0.063*
C3	0.32858 (16)	−0.0365 (2)	1.12182 (14)	0.0568 (4)
H3A	0.3113	−0.1011	1.1691	0.068*
C10	0.00038 (16)	0.33308 (18)	0.88898 (16)	0.0569 (4)
H10	−0.0253	0.2778	0.9381	0.068*
C5	0.28341 (14)	0.07288 (18)	0.94833 (13)	0.0499 (4)
H5	0.2347	0.0806	0.8791	0.060*
C4	0.25448 (14)	−0.02386 (17)	1.01789 (13)	0.0507 (4)
C11	−0.06391 (16)	0.33887 (19)	0.78414 (17)	0.0615 (5)
H11	−0.1342	0.2882	0.7652	0.074*
C2	0.42568 (15)	0.0457 (2)	1.15360 (13)	0.0558 (4)
H2	0.4749	0.0369	1.2226	0.067*
O2	0.56459 (19)	0.3367 (2)	1.08688 (19)	0.1206 (8)
C12	0.06747 (16)	0.48361 (18)	0.73744 (15)	0.0560 (4)
H12	0.0920	0.5343	0.6852	0.067*

C7	0.40796 (19)	0.2548 (2)	0.89618 (16)	0.0650 (5)
H7A	0.3587	0.2327	0.8264	0.098*
H7B	0.4909	0.2478	0.8948	0.098*
H7C	0.3910	0.3460	0.9148	0.098*
C9	0.1410 (3)	0.3213 (3)	1.10553 (18)	0.0842 (7)
H9A	0.0657	0.3478	1.1194	0.126*
H9B	0.2026	0.3264	1.1711	0.126*
H9C	0.1353	0.2297	1.0787	0.126*
C8	0.26892 (19)	0.5044 (3)	1.06203 (19)	0.0842 (7)
H8A	0.3380	0.4714	1.0400	0.126*
H8B	0.2870	0.5109	1.1392	0.126*
H8C	0.2475	0.5926	1.0311	0.126*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0643 (8)	0.0827 (10)	0.0624 (8)	−0.0234 (7)	0.0014 (6)	0.0170 (7)
N3	0.0535 (8)	0.0548 (8)	0.0598 (9)	0.0094 (7)	0.0020 (6)	−0.0021 (7)
N1	0.0526 (9)	0.0713 (11)	0.0682 (10)	−0.0036 (8)	0.0023 (7)	−0.0149 (8)
O1	0.0690 (10)	0.1098 (13)	0.0881 (11)	−0.0055 (9)	−0.0249 (8)	−0.0143 (10)
N2	0.0660 (10)	0.0714 (10)	0.0511 (8)	0.0051 (8)	0.0087 (7)	−0.0005 (7)
C14	0.0456 (8)	0.0435 (8)	0.0533 (9)	0.0073 (6)	0.0119 (6)	−0.0005 (7)
C6	0.0458 (8)	0.0496 (9)	0.0482 (8)	0.0043 (6)	0.0132 (6)	−0.0018 (6)
C1	0.0406 (8)	0.0544 (9)	0.0515 (9)	0.0044 (7)	0.0056 (6)	−0.0099 (7)
C13	0.0444 (8)	0.0520 (9)	0.0602 (10)	−0.0030 (7)	0.0112 (7)	0.0025 (7)
C3	0.0553 (9)	0.0649 (11)	0.0485 (9)	0.0058 (8)	0.0093 (7)	0.0111 (8)
C10	0.0567 (10)	0.0480 (9)	0.0698 (11)	−0.0029 (7)	0.0224 (8)	0.0054 (8)
C5	0.0484 (8)	0.0584 (9)	0.0399 (8)	−0.0007 (7)	0.0047 (6)	0.0034 (7)
C4	0.0459 (8)	0.0555 (9)	0.0488 (9)	−0.0016 (7)	0.0078 (6)	0.0023 (7)
C11	0.0453 (9)	0.0537 (10)	0.0813 (13)	−0.0029 (7)	0.0069 (8)	−0.0095 (9)
C2	0.0508 (9)	0.0687 (11)	0.0422 (8)	0.0103 (8)	0.0001 (7)	−0.0002 (7)
O2	0.1045 (15)	0.1048 (15)	0.1303 (17)	−0.0483 (12)	−0.0155 (12)	0.0154 (13)
C12	0.0585 (10)	0.0538 (9)	0.0563 (10)	0.0064 (8)	0.0150 (8)	0.0068 (7)
C7	0.0666 (11)	0.0673 (12)	0.0613 (11)	−0.0061 (9)	0.0157 (9)	0.0084 (9)
C9	0.1090 (18)	0.0927 (16)	0.0549 (11)	0.0277 (14)	0.0273 (11)	0.0153 (11)
C8	0.0637 (12)	0.115 (2)	0.0667 (13)	0.0000 (12)	0.0009 (10)	−0.0245 (13)

Geometric parameters (Å, º)

O3—C4	1.328 (2)	C3—C4	1.401 (2)
O3—H3	0.8200	C3—H3A	0.9300
N3—C12	1.331 (2)	C10—C11	1.367 (3)
N3—C11	1.335 (3)	C10—H10	0.9300
N1—O2	1.207 (3)	C5—C4	1.396 (2)
N1—O1	1.209 (2)	C5—H5	0.9300
N1—C1	1.456 (2)	C11—H11	0.9300
N2—C14	1.354 (2)	C2—H2	0.9300
N2—C8	1.439 (3)	C12—H12	0.9300

N2—C9	1.456 (3)	C7—H7A	0.9600
C14—C10	1.404 (2)	C7—H7B	0.9600
C14—C13	1.409 (2)	C7—H7C	0.9600
C6—C5	1.384 (2)	C9—H9A	0.9600
C6—C1	1.403 (2)	C9—H9B	0.9600
C6—C7	1.502 (3)	C9—H9C	0.9600
C1—C2	1.383 (3)	C8—H8A	0.9600
C13—C12	1.371 (3)	C8—H8B	0.9600
C13—H13	0.9300	C8—H8C	0.9600
C3—C2	1.359 (2)		

C4—O3—H3	109.5	O3—C4—C3	118.28 (15)
C12—N3—C11	115.74 (15)	C5—C4—C3	118.74 (15)
O2—N1—O1	121.00 (19)	N3—C11—C10	124.75 (16)
O2—N1—C1	119.70 (17)	N3—C11—H11	117.6
O1—N1—C1	119.19 (19)	C10—C11—H11	117.6
C14—N2—C8	121.99 (18)	C3—C2—C1	120.37 (15)
C14—N2—C9	120.72 (18)	C3—C2—H2	119.8
C8—N2—C9	117.27 (18)	C1—C2—H2	119.8
N2—C14—C10	122.35 (17)	N3—C12—C13	124.50 (17)
N2—C14—C13	122.36 (16)	N3—C12—H12	117.8
C10—C14—C13	115.29 (15)	C13—C12—H12	117.8
C5—C6—C1	116.28 (15)	C6—C7—H7A	109.5
C5—C6—C7	118.45 (15)	C6—C7—H7B	109.5
C1—C6—C7	125.23 (16)	H7A—C7—H7B	109.5
C2—C1—C6	122.06 (15)	C6—C7—H7C	109.5
C2—C1—N1	116.10 (15)	H7A—C7—H7C	109.5
C6—C1—N1	121.85 (17)	H7B—C7—H7C	109.5
C12—C13—C14	119.88 (16)	N2—C9—H9A	109.5
C12—C13—H13	120.1	N2—C9—H9B	109.5
C14—C13—H13	120.1	H9A—C9—H9B	109.5
C2—C3—C4	119.98 (16)	N2—C9—H9C	109.5
C2—C3—H3A	120.0	H9A—C9—H9C	109.5
C4—C3—H3A	120.0	H9B—C9—H9C	109.5
C11—C10—C14	119.82 (17)	N2—C8—H8A	109.5
C11—C10—H10	120.1	N2—C8—H8B	109.5
C14—C10—H10	120.1	H8A—C8—H8B	109.5
C6—C5—C4	122.56 (14)	N2—C8—H8C	109.5
C6—C5—H5	118.7	H8A—C8—H8C	109.5
C4—C5—H5	118.7	H8B—C8—H8C	109.5
O3—C4—C5	122.98 (15)		

C8—N2—C14—C10	−172.07 (18)	C13—C14—C10—C11	−1.7 (2)
C9—N2—C14—C10	6.4 (3)	C1—C6—C5—C4	−0.3 (2)
C8—N2—C14—C13	7.6 (3)	C7—C6—C5—C4	177.63 (17)
C9—N2—C14—C13	−174.00 (17)	C6—C5—C4—O3	179.17 (16)
C5—C6—C1—C2	1.3 (2)	C6—C5—C4—C3	−0.7 (3)
C7—C6—C1—C2	−176.47 (17)	C2—C3—C4—O3	−179.11 (17)

C5—C6—C1—N1	−178.98 (15)	C2—C3—C4—C5	0.7 (3)
C7—C6—C1—N1	3.2 (3)	C12—N3—C11—C10	−0.4 (3)
O2—N1—C1—C2	−156.9 (2)	C14—C10—C11—N3	1.8 (3)
O1—N1—C1—C2	19.3 (3)	C4—C3—C2—C1	0.2 (3)
O2—N1—C1—C6	23.4 (3)	C6—C1—C2—C3	−1.3 (3)
O1—N1—C1—C6	−160.43 (18)	N1—C1—C2—C3	178.99 (16)
N2—C14—C13—C12	−179.41 (16)	C11—N3—C12—C13	−1.2 (3)
C10—C14—C13—C12	0.3 (2)	C14—C13—C12—N3	1.2 (3)
N2—C14—C10—C11	178.01 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N3^{i}	0.82	1.79	2.594 (2)	168

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