Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 2-amino-4,5-dimethoxybenzoate

Tania N. Hill* and Naadiya Patel

Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, PO WITS 2050, Johannesburg, South Africa
Correspondence e-mail: tania.hill@gmail.com

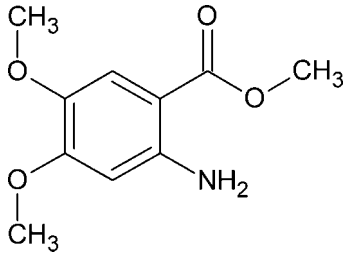
Received 7 November 2013; accepted 11 November 2013

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.109; data-to-parameter ratio = 17.3.

The title compound, $C_{10}H_{13}NO_4$, is essentially planar, with an r.m.s. deviation of 0.049 Å. An intramolecular C—H···O hydrogen bond occurs and the amino group forms an intramolecular N—H···O_{ester} hydrogen bond; the other H atom forms an intermolecular N—H···$O_{carbonyl}$ hydrogen bond, leading to the formation of a helical chain that runs along the *b*-axis direction.

Related literature

For similar crystal structures, see: Zhang *et al.* (2009); Smith & Elsegood (2002).



Experimental

Crystal data

$C_{10}H_{13}NO_4$
M_r = 211.21
Monoclinic, $P2_1/c$
a = 11.1933 (4) Å
b = 7.7564 (3) Å
c = 13.7728 (5) Å
β = 121.741 (2)°
V = 1016.91 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 173 K
0.5 × 0.29 × 0.25 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.948, T_{max} = 0.974
9994 measured reflections
2543 independent reflections
2080 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
S = 1.05
2543 reflections
147 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O2	0.95	2.37	2.7131 (14)	101
N1—H1A···O1	0.90 (2)	2.03 (2)	2.702 (2)	131 (2)
N1—H1B···O1^{i}	0.88 (2)	2.10 (2)	2.947 (1)	162 (1)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

The University of the Witwatersrand and the Molecular Sciences Institute are thanked for providing the infrastructure and financial support. Special thanks go to Dr Andreas Lemmerer of the University of the Witwatersrand for his contributions and insights toward this project. TNH wishes to thank the University of the Witwatersrand research committee for a postdoctoral fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5347).

References

Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Imapct GbR, Bonn, Germany.
Bruker (2004). *SAINT-Plus*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin. USA.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, M. B. & Elsegood, M. R. J. (2002). *Tetrahedron Lett.* **43**, 1299–1301.
Zhang, M., Lu, R., Han, L., Wei, W. & Wang, H. (2009). *Acta Cryst.* E**65**, o942.

supporting information

Acta Cryst. (2013). E**69**, o1779 [doi:10.1107/S1600536813030894]

Methyl 2-amino-4,5-dimethoxybenzoate

Tania N. Hill and Naadiya Patel

S1. Comment

The molecular structure of (I) is presented in Figure 1, and was obtained by recrystallization of the commercially available compound. The title compound consists of an amino (C2) and methoxy (C4 and C5) substituted benzoate which was found to be essentially planar with an *r.m.s.* deviation of 0.049 Å; the dihedral angles shown in Table 1 further reinforce the planarity of (I). The maximum deviation observed below the calculated mean plane was found for the carbonyl oxygen (O1) at -0.136 (1) Å. Two intramolecular hydrogen bonds found between the phenyl carbon (C6), the amino (N1) and the ester O atoms (O1 and O2) with distances of 2.713 (1) Å and 2.702 (2) Å, effectively lock the ester into the molecular plane. The last hydrogen bond interaction observed between the amino (N1) and an adjacent carbonyl oxygen (O1) (symmetry operator [-*x*, *y* + 1/2, -*z* - 1/2]) with a distance of 2.947 (1) Å results in a helical chain along [0 1 0] (Figure 2).

S2. Experimental

Methyl 2-amino-4,5-dimethoxybenzoate was obtained commercially. (I) was redissolved in warm MeOH and allowed to cool to room terperature. Yellow crystals suitable for single-crystal diffraction were obtained by slow evaporation over a few days.

S3. Refinement

All hydrogen atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å U_{iso}(H)= 1.2 U_{eq}(C) for the aromatic H and with C—H = 0.98 Å U_{iso}(H)= 1.5 U_{eq}(C)for methyl H atoms. The methyl groups were allowed to rotate with a fixed angle arround the C—C bond to best fit the experimental electron density. The amino hydrogens were freely refined.

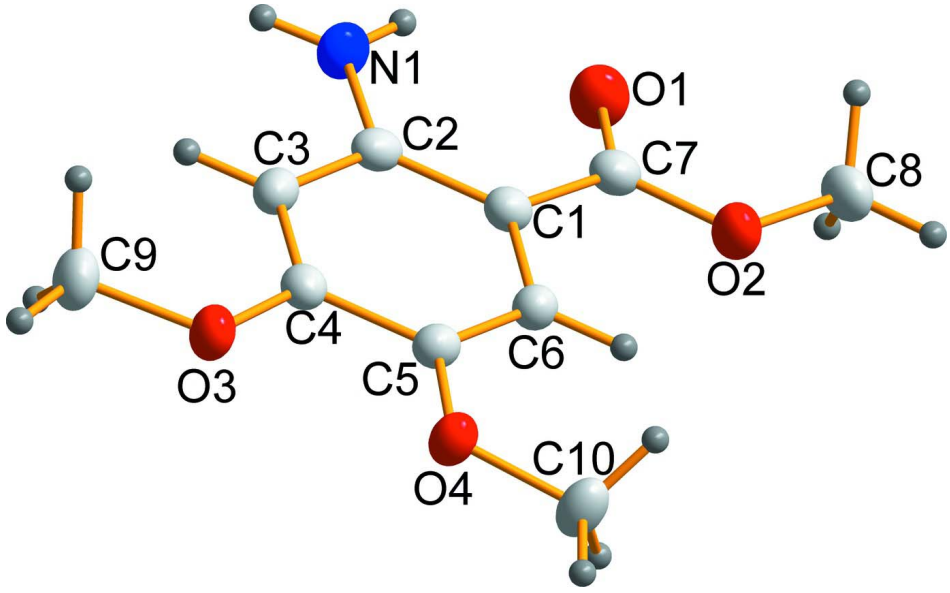


Figure 1
View of (I) (50% probability displacement ellipsoids)

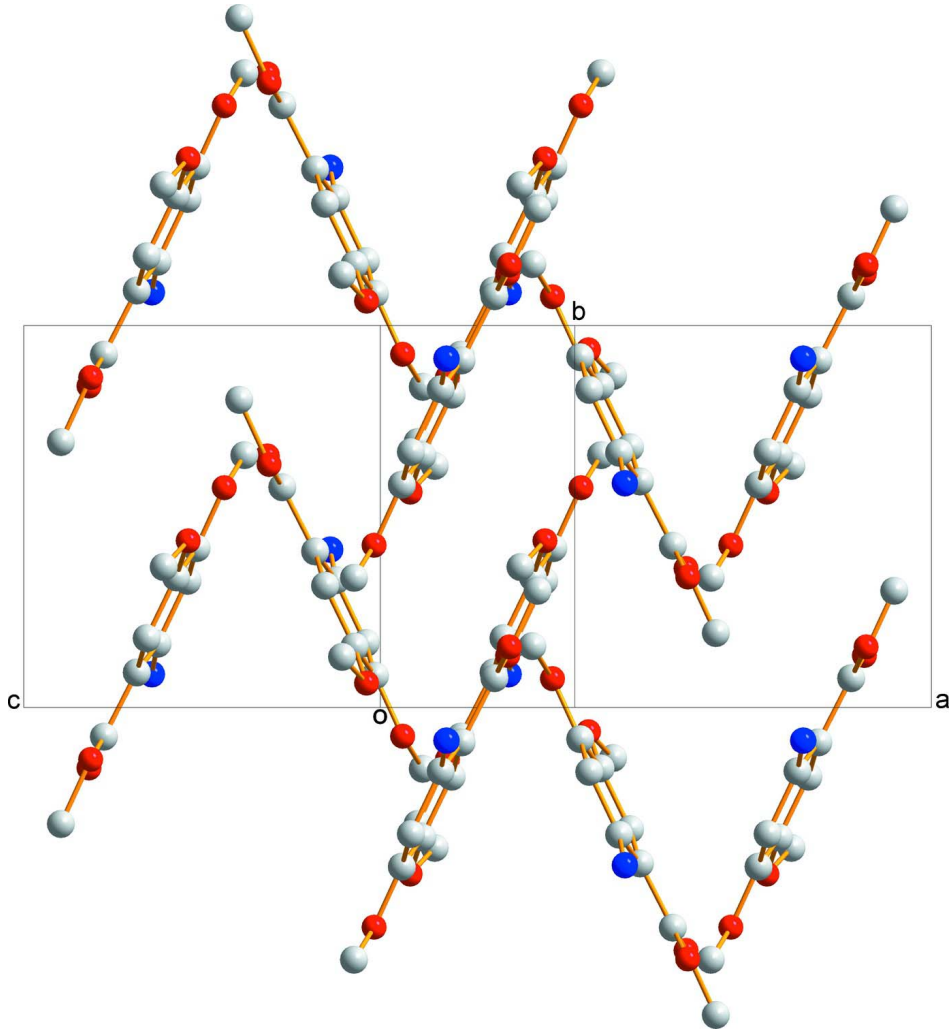


Figure 2

Packing of (I) viewed along [0 1 0]. H atoms omitted.

Methyl 2-amino-4,5-dimethoxybenzoate

Crystal data

$C_{10}H_{13}NO_4$
$M_r = 211.21$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.1933\ (4)$ Å
$b = 7.7564\ (3)$ Å
$c = 13.7728\ (5)$ Å
$\beta = 121.741\ (2)°$
$V = 1016.91\ (7)$ Å^3
$Z = 4$
$F(000) = 448$
$D_x = 1.38$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3834 reflections
$\theta = 3.0–28.3°$
$\mu = 0.11$ mm^{-1}
$T = 173$ K
Cuboid, yellow
0.5 × 0.29 × 0.25 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 512 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.948, T_{max} = 0.974

9994 measured reflections
2543 independent reflections
2080 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 28.3°, θ_{min} = 2.1°
h = −14→14
k = −10→10
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
S = 1.05
2543 reflections
147 parameters
0 restraints

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.2646P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.19769 (12)	0.09141 (14)	−0.01177 (10)	0.0237 (2)
C2	0.16865 (12)	0.16680 (14)	−0.11496 (10)	0.0243 (2)
C3	0.22538 (12)	0.33134 (15)	−0.11104 (10)	0.0250 (2)
H3	0.2062	0.3839	−0.1801	0.03*
C4	0.30761 (12)	0.41704 (14)	−0.00949 (10)	0.0239 (2)
C5	0.33814 (12)	0.34050 (14)	0.09484 (9)	0.0234 (2)
C6	0.28295 (12)	0.18137 (14)	0.09202 (9)	0.0235 (2)
H6	0.3024	0.1301	0.1615	0.028*
C7	0.13820 (12)	−0.07648 (14)	−0.01151 (10)	0.0256 (2)
C8	0.13103 (15)	−0.30520 (15)	0.09811 (12)	0.0340 (3)
H8A	0.0291	−0.3006	0.064	0.051*
H8B	0.1763	−0.3421	0.1779	0.051*
H8C	0.1531	−0.3875	0.0558	0.051*
C9	0.33489 (15)	0.66056 (17)	−0.10146 (11)	0.0345 (3)
H9A	0.3706	0.591	−0.1402	0.052*
H9B	0.3805	0.7738	−0.0826	0.052*
H9C	0.2331	0.6751	−0.1518	0.052*
C10	0.44564 (14)	0.36738 (17)	0.29532 (10)	0.0328 (3)
H10A	0.3554	0.3526	0.29	0.049*
H10B	0.5049	0.4467	0.3579	0.049*
H10C	0.4927	0.2554	0.31	0.049*

N1	0.09141 (12)	0.08643 (15)	−0.21889 (9)	0.0326 (3)
O1	0.05611 (10)	−0.15847 (12)	−0.09659 (8)	0.0376 (2)
O2	0.18202 (10)	−0.13617 (11)	0.09315 (7)	0.0324 (2)
O3	0.36486 (9)	0.57535 (10)	0.00121 (7)	0.0293 (2)
O4	0.42222 (9)	0.43624 (10)	0.19078 (7)	0.0279 (2)
H1B	0.0560 (16)	0.1496 (19)	−0.2812 (14)	0.037 (4)*
H1A	0.0410 (18)	−0.007 (3)	−0.2225 (15)	0.056 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0235 (5)	0.0231 (5)	0.0239 (5)	0.0032 (4)	0.0121 (4)	−0.0001 (4)
C2	0.0221 (5)	0.0261 (5)	0.0232 (5)	0.0050 (4)	0.0110 (4)	−0.0010 (4)
C3	0.0261 (6)	0.0278 (6)	0.0223 (5)	0.0056 (4)	0.0137 (5)	0.0038 (4)
C4	0.0248 (6)	0.0229 (5)	0.0268 (5)	0.0033 (4)	0.0155 (5)	0.0024 (4)
C5	0.0236 (5)	0.0249 (5)	0.0219 (5)	0.0017 (4)	0.0121 (4)	−0.0004 (4)
C6	0.0255 (6)	0.0237 (5)	0.0216 (5)	0.0021 (4)	0.0127 (4)	0.0011 (4)
C7	0.0250 (6)	0.0246 (5)	0.0272 (6)	0.0032 (4)	0.0137 (5)	−0.0011 (4)
C8	0.0438 (8)	0.0221 (5)	0.0417 (7)	−0.0015 (5)	0.0263 (6)	0.0015 (5)
C9	0.0402 (7)	0.0328 (6)	0.0336 (6)	0.0003 (5)	0.0214 (6)	0.0092 (5)
C10	0.0398 (7)	0.0348 (6)	0.0218 (6)	−0.0095 (5)	0.0148 (5)	−0.0019 (5)
N1	0.0371 (6)	0.0324 (6)	0.0211 (5)	−0.0008 (5)	0.0102 (5)	−0.0008 (4)
O1	0.0411 (5)	0.0323 (5)	0.0305 (5)	−0.0088 (4)	0.0127 (4)	−0.0055 (4)
O2	0.0431 (5)	0.0241 (4)	0.0295 (4)	−0.0050 (4)	0.0188 (4)	−0.0004 (3)
O3	0.0363 (5)	0.0258 (4)	0.0271 (4)	−0.0031 (3)	0.0176 (4)	0.0029 (3)
O4	0.0341 (5)	0.0271 (4)	0.0215 (4)	−0.0063 (3)	0.0140 (4)	−0.0018 (3)

Geometric parameters (Å, º)

C1—C2	1.4077 (15)	C8—O2	1.4458 (14)
C1—C6	1.4162 (15)	C8—H8A	0.98
C1—C7	1.4634 (16)	C8—H8B	0.98
C2—N1	1.3722 (15)	C8—H8C	0.98
C2—C3	1.4138 (16)	C9—O3	1.4317 (14)
C3—C4	1.3751 (16)	C9—H9A	0.98
C3—H3	0.95	C9—H9B	0.98
C4—O3	1.3568 (13)	C9—H9C	0.98
C4—C5	1.4203 (15)	C10—O4	1.4247 (14)
C5—O4	1.3692 (13)	C10—H10A	0.98
C5—C6	1.3716 (15)	C10—H10B	0.98
C6—H6	0.95	C10—H10C	0.98
C7—O1	1.2202 (14)	N1—H1B	0.881 (16)
C7—O2	1.3374 (14)	N1—H1A	0.90 (2)
C2—C1—C6	119.31 (10)	H8A—C8—H8B	109.5
C2—C1—C7	120.56 (10)	O2—C8—H8C	109.5
C6—C1—C7	120.12 (10)	H8A—C8—H8C	109.5
N1—C2—C1	123.27 (11)	H8B—C8—H8C	109.5

N1—C2—C3	118.28 (10)	O3—C9—H9A	109.5
C1—C2—C3	118.42 (10)	O3—C9—H9B	109.5
C4—C3—C2	121.47 (10)	H9A—C9—H9B	109.5
C4—C3—H3	119.3	O3—C9—H9C	109.5
C2—C3—H3	119.3	H9A—C9—H9C	109.5
O3—C4—C3	124.97 (10)	H9B—C9—H9C	109.5
O3—C4—C5	114.83 (10)	O4—C10—H10A	109.5
C3—C4—C5	120.20 (10)	O4—C10—H10B	109.5
O4—C5—C6	125.87 (10)	H10A—C10—H10B	109.5
O4—C5—C4	115.28 (10)	O4—C10—H10C	109.5
C6—C5—C4	118.85 (10)	H10A—C10—H10C	109.5
C5—C6—C1	121.75 (10)	H10B—C10—H10C	109.5
C5—C6—H6	119.1	C2—N1—H1B	118.5 (10)
C1—C6—H6	119.1	C2—N1—H1A	117.0 (12)
O1—C7—O2	121.23 (11)	H1B—N1—H1A	116.4 (15)
O1—C7—C1	125.11 (11)	C7—O2—C8	115.77 (9)
O2—C7—C1	113.66 (10)	C4—O3—C9	117.30 (9)
O2—C8—H8A	109.5	C5—O4—C10	116.08 (9)
O2—C8—H8B	109.5		

C6—C1—C2—N1	177.57 (11)	C4—C5—C6—C1	0.54 (17)
C7—C1—C2—N1	−3.72 (17)	C2—C1—C6—C5	0.06 (17)
C6—C1—C2—C3	−0.46 (16)	C7—C1—C6—C5	−178.65 (10)
C7—C1—C2—C3	178.25 (10)	C2—C1—C7—O1	−3.96 (18)
N1—C2—C3—C4	−177.87 (11)	C6—C1—C7—O1	174.73 (11)
C1—C2—C3—C4	0.26 (16)	C2—C1—C7—O2	176.09 (10)
C2—C3—C4—O3	−179.49 (10)	C6—C1—C7—O2	−5.22 (15)
C2—C3—C4—C5	0.33 (17)	O1—C7—O2—C8	2.67 (16)
O3—C4—C5—O4	−0.88 (14)	C1—C7—O2—C8	−177.38 (10)
C3—C4—C5—O4	179.28 (10)	C3—C4—O3—C9	1.26 (16)
O3—C4—C5—C6	179.10 (10)	C5—C4—O3—C9	−178.57 (10)
C3—C4—C5—C6	−0.73 (17)	C6—C5—O4—C10	−4.40 (16)
O4—C5—C6—C1	−179.48 (10)	C4—C5—O4—C10	175.59 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2	0.95	2.37	2.7131 (14)	101
N1—H1*A*···O1	0.90 (2)	2.03 (2)	2.702 (2)	131 (2)
N1—H1*B*···O1^{i}	0.88 (2)	2.10 (2)	2.947 (1)	162 (1)

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