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N-(2,3-Dimethylphenyl)succinamic acid

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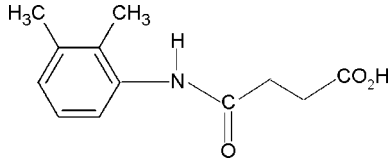
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.003 Å; R factor = 0.064; wR factor = 0.183; data-to-parameter ratio = 13.1.

In the title compound, $C_{12}H_{15}NO_3$, the conformations of N—H and C=O bonds in the amide segment are *anti* to each other and that of the amide H atom is *syn* to the *ortho*- and *meta*-methyl groups in the benzene ring. In the crystal, the molecules are linked into infinite chains through intermolecular O—H···O and N—H···O hydrogen bonds.

Related literature

For background to our study of the effect of ring and side-chain substitutions on the crystal structures of anilides, see: Gowda *et al.* (2010*a*,*b*,*c*). For the modes of interlinking carboxylic acids by hydrogen bonds, see: Leiserowitz (1976). The packing of molecules involving dimeric hydrogen-bonded association of each carboxyl group with a centrosymmetrically related neighbor has also been observed, see: Jagannathan *et al.* (1994).



Experimental

Crystal data

$C_{12}H_{15}NO_3$
M_r = 221.25
Triclinic, $P\overline{1}$
a = 4.8379 (4) Å
b = 10.0424 (6) Å
c = 11.9876 (8) Å
α = 90.222 (6)°
β = 99.614 (7)°
γ = 98.506 (6)°
V = 567.67 (7) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 299 K
0.40 × 0.25 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
3962 measured reflections
2017 independent reflections
1751 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
3 standard reflections every 120 min
intensity decay: 0.5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.183
S = 1.11
2017 reflections
154 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.87 (3)	2.04 (3)	2.909 (2)	174 (2)
O2—H2*O*···O3^{ii}	0.85 (3)	1.90 (4)	2.679 (2)	152 (3)

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+3$.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2264).

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supplementary materials

Acta Cryst. (2011). E**67**, o236 [**doi:10.1107/S160053681005292X**]

N-(2,3-Dimethylphenyl)succinamic acid

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Comment

In the present work, as a part of studying the effect of ring and side chain substitutions on the crystal structures of anilides (Gowda *et al.*, 2010*a*,*b*,*c*), the crystal structure of *N*-(2,3-dimethylphenyl)-succinamic acid (I) has been determined. The conformations of N—H and C═O bonds in the amide segment are *anti* to each other (Fig. 1). The conformation of the amide oxygen and the carbonyl oxygen of the acid segment are almost midway between the *syn* and *anti* conformations, in contrast to the *anti* conformation observed in *N*-(2-methylphenyl)succinamic acid (II) (Gowda *et al.*, 2010*c*) and the *syn* conformation observed in *N*-(3-methylphenyl)succinamic acid (III) (Gowda *et al.*, 2010*a*). Further, the conformation of the amide C═O bond is *anti* to the H atoms of its adjacent $-CH_2$ groups (Fig. 1) and that of the carbonyl oxygen of the acid segment is almost midway between the *syn* and *anti* conformations. The C═O and O—H bonds of the acid group are in *syn* position to each other, similar to that observed in (II) and (III).

The conformation of the amide hydrogen is *syn* to the *ortho*- and *meta*-methyl groups in the benzene ring, similar to that observed between the amide hydrogen and the *ortho*-methyl group in (II), but contrary to the *anti* conformation observed between the amide hydrogen and the *meta*-methyl group in the benzene ring of (III).

The intermolecular O—H···O and N—H···O hydrogen bonds pack the molecules into infinite chains in the structure (Table 1, Fig.2).

The modes of interlinking carboxylic acids by hydrogen bonds is described elsewhere (Leiserowitz, 1976). The packing of molecules involving dimeric hydrogen bonded association of each carboxyl group with a centrosymmetrically related neighbor has also been observed (Jagannathan *et al.*, 1994).

Experimental

The solution of succinic anhydride (0.01 mole) in toluene (25 ml) was treated dropwise with the solution of 2,3-dimethylaniline (0.01 mole) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for about one h and set aside for an additional hour at room temperature for completion of the reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted 2,3-dimethylaniline. The resultant solid *N*-(2,3-dimethylphenyl)-succinamic acid was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. It was recrystallized to constant melting point from ethanol. The purity of the compound was checked by elemental analysis and characterized by its infrared and NMR spectra.

The prism like colorless single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation at room temperature.

Refinement

The H atoms of the OH group and of the NH group were located in a difference map and their positions refined [O—H = 0.85 (3) Å, N—H = 0.87 (3) Å]. The other H atoms were positioned with idealized geometry using a riding model [C—H = 0.93–0.97 Å]. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Figures

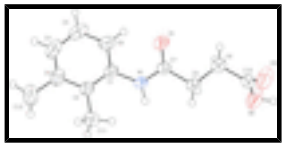

Fig. 1. Molecular structure of the title compound, showing the atom labeling scheme. The displacement ellipsoids are drawn at the 50% probability level.

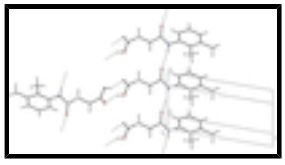

Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

N-(2,3-Dimethylphenyl)succinamic acid

Crystal data

$C_{12}H_{15}NO_3$	$Z = 2$
$M_r = 221.25$	$F(000) = 236$
Triclinic, $P\overline{1}$	$D_x = 1.294$ Mg m^{-3}
Hall symbol: -P 1	Cu $K\alpha$ radiation, $\lambda = 1.54180$ Å
$a = 4.8379$ (4) Å	Cell parameters from 25 reflections
$b = 10.0424$ (6) Å	$\theta = 5.9$–$22.4°$
$c = 11.9876$ (8) Å	$\mu = 0.77$ mm^{-1}
$\alpha = 90.222$ (6)°	$T = 299$ K
$\beta = 99.614$ (7)°	Prism, colorless
$\gamma = 98.506$ (6)°	0.40 × 0.25 × 0.10 mm
$V = 567.67$ (7) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.035$
Radiation source: fine-focus sealed tube	$\theta_{max} = 66.9°$, $\theta_{min} = 3.7°$
graphite	$h = -5 \rightarrow 5$
$\omega/2\theta$ scans	$k = -11 \rightarrow 11$
3962 measured reflections	$l = -14 \rightarrow 14$
2017 independent reflections	3 standard reflections every 120 min
1751 reflections with $I > 2\sigma(I)$	intensity decay: 0.5%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.064$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.183$	$w = 1/[\sigma^2(F_o^2) + (0.1035P)^2 + 0.1611P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} = 0.003$
2017 reflections	$\Delta\rho_{max} = 0.42$ e Å^{-3}
154 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.025 (4)

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}
C1	−0.0305 (4)	0.17546 (19)	0.88221 (15)	0.0389 (5)
C2	0.0537 (4)	0.21089 (18)	0.77926 (15)	0.0396 (5)
C3	−0.0852 (4)	0.1370 (2)	0.68124 (17)	0.0481 (5)
C4	−0.3018 (5)	0.0324 (2)	0.6889 (2)	0.0597 (6)
H4	−0.3937	−0.0163	0.6236	0.072*
C5	−0.3839 (5)	−0.0011 (2)	0.7909 (2)	0.0655 (7)
H5	−0.5314	−0.0710	0.7942	0.079*
C6	−0.2461 (4)	0.0697 (2)	0.88872 (19)	0.0534 (6)
H6	−0.2976	0.0464	0.9582	0.064*
C7	−0.0169 (4)	0.3000 (2)	1.05958 (16)	0.0502 (6)
C8	0.1749 (4)	0.3738 (3)	1.15994 (17)	0.0560 (6)
H8A	0.3676	0.3570	1.1608	0.067*
H8B	0.1734	0.4699	1.1529	0.067*
C9	0.0814 (5)	0.3293 (2)	1.26929 (17)	0.0545 (6)
H9A	−0.1128	0.3442	1.2677	0.065*

H9B	0.0872	0.2336	1.2772	0.065*
C10	0.2680 (4)	0.4050 (2)	1.36885 (16)	0.0481 (5)
C11	0.2885 (4)	0.3245 (2)	0.77306 (18)	0.0518 (5)
H11A	0.4581	0.2888	0.7655	0.062*
H11B	0.3218	0.3797	0.8409	0.062*
H11C	0.2361	0.3778	0.7088	0.062*
C12	−0.0018 (6)	0.1712 (3)	0.56799 (19)	0.0684 (7)
H12A	0.1952	0.1644	0.5709	0.082*
H12B	−0.0329	0.2615	0.5500	0.082*
H12C	−0.1144	0.1096	0.5109	0.082*
N1	0.1111 (3)	0.24747 (17)	0.98335 (13)	0.0432 (5)
H1N	0.295 (6)	0.262 (2)	0.998 (2)	0.052*
O1	−0.2754 (3)	0.2922 (2)	1.05008 (14)	0.0823 (7)
O2	0.4948 (4)	0.3603 (2)	1.40631 (16)	0.0769 (6)
H2O	0.538 (7)	0.397 (3)	1.472 (3)	0.092*
O3	0.2004 (4)	0.50537 (19)	1.40937 (15)	0.0762 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0239 (9)	0.0495 (10)	0.0406 (9)	0.0034 (7)	−0.0001 (7)	−0.0042 (7)
C2	0.0310 (10)	0.0455 (10)	0.0418 (10)	0.0092 (7)	0.0022 (7)	−0.0021 (7)
C3	0.0462 (12)	0.0536 (11)	0.0440 (11)	0.0177 (9)	−0.0027 (8)	−0.0079 (8)
C4	0.0548 (14)	0.0571 (12)	0.0587 (13)	0.0069 (10)	−0.0133 (10)	−0.0185 (10)
C5	0.0453 (13)	0.0560 (12)	0.0837 (16)	−0.0122 (10)	−0.0043 (11)	−0.0092 (11)
C6	0.0394 (11)	0.0598 (12)	0.0556 (12)	−0.0062 (9)	0.0045 (9)	0.0024 (9)
C7	0.0230 (9)	0.0834 (14)	0.0413 (10)	0.0000 (8)	0.0041 (7)	−0.0105 (9)
C8	0.0285 (10)	0.0906 (16)	0.0439 (11)	−0.0054 (9)	0.0051 (8)	−0.0160 (10)
C9	0.0400 (11)	0.0723 (14)	0.0469 (11)	−0.0019 (9)	0.0045 (8)	−0.0115 (9)
C10	0.0388 (11)	0.0676 (13)	0.0375 (10)	0.0039 (9)	0.0089 (8)	−0.0060 (9)
C11	0.0449 (12)	0.0574 (12)	0.0525 (11)	0.0008 (9)	0.0124 (9)	0.0030 (9)
C12	0.0806 (18)	0.0824 (16)	0.0441 (12)	0.0265 (13)	0.0034 (11)	−0.0066 (10)
N1	0.0196 (8)	0.0677 (11)	0.0391 (8)	−0.0010 (7)	0.0026 (6)	−0.0072 (7)
O1	0.0220 (8)	0.1555 (19)	0.0645 (10)	0.0054 (9)	0.0015 (7)	−0.0449 (11)
O2	0.0544 (11)	0.1007 (14)	0.0710 (11)	0.0252 (9)	−0.0140 (8)	−0.0343 (10)
O3	0.0715 (12)	0.0869 (12)	0.0660 (11)	0.0300 (10)	−0.0155 (9)	−0.0277 (9)

Geometric parameters (Å, °)

C1—C6	1.387 (3)	C8—H8A	0.9700
C1—C2	1.394 (3)	C8—H8B	0.9700
C1—N1	1.425 (2)	C9—C10	1.499 (3)
C2—C3	1.402 (3)	C9—H9A	0.9700
C2—C11	1.498 (3)	C9—H9B	0.9700
C3—C4	1.385 (3)	C10—O3	1.227 (3)
C3—C12	1.507 (3)	C10—O2	1.261 (3)
C4—C5	1.376 (4)	C11—H11A	0.9600
C4—H4	0.9300	C11—H11B	0.9600
C5—C6	1.384 (3)	C11—H11C	0.9600

C5—H5	0.9300	C12—H12A	0.9600
C6—H6	0.9300	C12—H12B	0.9600
C7—O1	1.227 (2)	C12—H12C	0.9600
C7—N1	1.334 (3)	N1—H1N	0.87 (3)
C7—C8	1.508 (3)	O2—H2O	0.85 (3)
C8—C9	1.505 (3)		
C6—C1—C2	121.34 (18)	H8A—C8—H8B	108.0
C6—C1—N1	119.13 (17)	C10—C9—C8	111.26 (17)
C2—C1—N1	119.52 (16)	C10—C9—H9A	109.4
C1—C2—C3	118.49 (18)	C8—C9—H9A	109.4
C1—C2—C11	121.03 (16)	C10—C9—H9B	109.4
C3—C2—C11	120.48 (17)	C8—C9—H9B	109.4
C4—C3—C2	119.54 (19)	H9A—C9—H9B	108.0
C4—C3—C12	119.9 (2)	O3—C10—O2	123.04 (19)
C2—C3—C12	120.5 (2)	O3—C10—C9	120.7 (2)
C5—C4—C3	121.38 (19)	O2—C10—C9	116.29 (19)
C5—C4—H4	119.3	C2—C11—H11A	109.5
C3—C4—H4	119.3	C2—C11—H11B	109.5
C4—C5—C6	119.8 (2)	H11A—C11—H11B	109.5
C4—C5—H5	120.1	C2—C11—H11C	109.5
C6—C5—H5	120.1	H11A—C11—H11C	109.5
C5—C6—C1	119.5 (2)	H11B—C11—H11C	109.5
C5—C6—H6	120.3	C3—C12—H12A	109.5
C1—C6—H6	120.3	C3—C12—H12B	109.5
O1—C7—N1	123.30 (18)	H12A—C12—H12B	109.5
O1—C7—C8	120.46 (18)	C3—C12—H12C	109.5
N1—C7—C8	116.23 (16)	H12A—C12—H12C	109.5
C9—C8—C7	111.27 (17)	H12B—C12—H12C	109.5
C9—C8—H8A	109.4	C7—N1—C1	125.13 (16)
C7—C8—H8A	109.4	C7—N1—H1N	115.0 (16)
C9—C8—H8B	109.4	C1—N1—H1N	119.8 (16)
C7—C8—H8B	109.4	C10—O2—H2O	101 (2)
C6—C1—C2—C3	0.1 (3)	C2—C1—C6—C5	−1.0 (3)
N1—C1—C2—C3	178.85 (16)	N1—C1—C6—C5	−179.77 (19)
C6—C1—C2—C11	−179.43 (19)	O1—C7—C8—C9	49.2 (3)
N1—C1—C2—C11	−0.7 (3)	N1—C7—C8—C9	−131.8 (2)
C1—C2—C3—C4	0.4 (3)	C7—C8—C9—C10	−178.67 (19)
C11—C2—C3—C4	180.00 (19)	C8—C9—C10—O3	95.5 (3)
C1—C2—C3—C12	−179.90 (18)	C8—C9—C10—O2	−83.7 (3)
C11—C2—C3—C12	−0.4 (3)	O1—C7—N1—C1	−0.3 (4)
C2—C3—C4—C5	−0.1 (3)	C8—C7—N1—C1	−179.29 (19)
C12—C3—C4—C5	−179.7 (2)	C6—C1—N1—C7	−51.1 (3)
C3—C4—C5—C6	−0.8 (4)	C2—C1—N1—C7	130.1 (2)
C4—C5—C6—C1	1.4 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N1—H1N···O1^{i}	0.87 (3)	2.04 (3)	2.909 (2)	174 (2)
O2—H2O···O3^{ii}	0.85 (3)	1.90 (4)	2.679 (2)	152 (3)

Symmetry codes: (i) $x+1$, y, z; (ii) $-x+1$, $-y+1$, $-z+3$.

Fig. 1

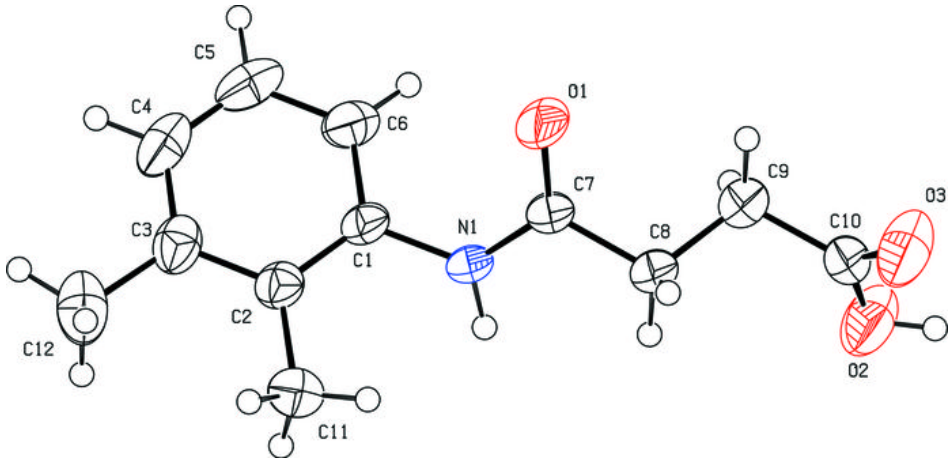

Fig. 2