Crystal structure of $C_{12}H_{10}N_2O_5$ — ban0835*b*

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Abstract

The crystal structure of $C_{12}H_{10}N_2O_5$ is reported.

Comment

The crystallographic asymmetric unit consists of one $C_{12}H_{10}N_2O_5$ molecule.

Experimental

The compound was prepared by DMP and recrystallized from ethylacetate/n-hexane. The sample ID is 7DP31.

Refinement

H atoms bonded to C were included at geometrically determined positions with the methyl group on N9 oriented to best-match peaks in the difference electron density map. H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom). A difference map showed peaks between the H atoms on C18 so the original sites were assigned occupancies of 0.7 and additional sites of occupancy 0.3 were placed between them. In the final cycles of refinement the H atom positions were refined with riding constraints.

The largest feature in the final difference electron density map is a peak just beyond H51. This perhaps suggests the presence of a very small amount of an impurity in the sample. Data sets were collected on three different crystals, and all three showed this feature. The next largest peaks in the difference map are located along C—C bonds.

Computing details

Data collection: *COLLECT* (Nonius, 1997-2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP*II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

(ban0835b)

Crystal data

$C_{12}H_{10}N_2O_5$	$V = 1140.26\ (4)$ Å^3
$M_r = 262.22$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$
$a = 5.8333\ (1)$ Å	$\mu = 0.12$ mm^{-1}
$b = 11.3778\ (3)$ Å	$T = 200$ K
$c = 17.2688\ (4)$ Å	0.46 × 0.08 × 0.07 mm
$\beta = 95.8027\ (17)^o$	

Data collection

Area diffractometer	2611 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	1942 reflections with $I > 2.0\sigma(I)$
$T_{min} = 0.966$, $T_{max} = 0.994$	$R_{int} = 0.062$
21901 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	172 parameters
$wR(F^2) = 0.106$	H-atom parameters constrained
$S = 0.92$	$\Delta\rho_{max} = 0.51$ e Å^{-3}
2611 reflections	$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °)

C1—C2	1.4055 (18)	C7—C11	1.3304 (18)
C1—C6	1.3791 (18)	C8—N9	1.3758 (17)
C1—N12	1.4611 (16)	C8—O17	1.2149 (15)
C2—C3	1.3865 (18)	N9—C10	1.3901 (16)
C2—C7	1.4724 (17)	N9—C18	1.4499 (17)
C3—C4	1.3933 (19)	C10—C11	1.4883 (18)
C4—C5	1.390 (2)	C10—O19	1.2104 (17)
C4—O15	1.3569 (16)	N12—O13	1.2285 (16)
C5—C6	1.383 (2)	N12—O14	1.2355 (15)
C7—C8	1.5132 (17)	O15—C16	1.4305 (16)
C2—C1—C6	121.23 (12)	C7—C8—N9	106.21 (10)
C2—C1—N12	121.04 (12)	C7—C8—O17	127.68 (12)
C6—C1—N12	117.67 (12)	N9—C8—O17	126.04 (12)
C1—C2—C3	117.18 (12)	C8—N9—C10	110.74 (10)
C1—C2—C7	125.79 (12)	C8—N9—C18	124.66 (11)
C3—C2—C7	116.97 (11)	C10—N9—C18	124.25 (12)
C2—C3—C4	121.50 (12)	N9—C10—C11	106.29 (11)

C3—C4—C5	120.56 (13)	N9—C10—O19	125.05 (13)
C3—C4—O15	114.90 (12)	C11—C10—O19	128.65 (12)
C5—C4—O15	124.54 (13)	C10—C11—C7	108.96 (11)
C4—C5—C6	118.30 (13)	C1—N12—O13	118.96 (11)
C5—C6—C1	121.20 (13)	C1—N12—O14	118.11 (12)
C2—C7—C8	122.59 (11)	O13—N12—O14	122.93 (11)
C2—C7—C11	129.08 (11)	C4—O15—C16	116.85 (11)
C8—C7—C11	107.75 (11)		

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supplementary materials

Crystal structure of $C_{12}H_{10}N_2O_5$ — ban0835*b*

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(ban0835b)

Crystal data

$C_{12}H_{10}N_2O_5$	F_{000} = 544
M_r = 262.22	D_x = 1.527 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 5.8333 (1) Å	Cell parameters from 21484 reflections
b = 11.3778 (3) Å	θ = 2.7–27.5º
c = 17.2688 (4) Å	μ = 0.12 mm^{-1}
β = 95.8027 (17)º	T = 200 K
V = 1140.26 (4) Å^3	Needle, colourless
Z = 4	0.46 × 0.08 × 0.07 mm

Data collection

Area diffractometer	1942 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	R_{int} = 0.062
T = 200 K	θ_{max} = 27.5º
φ and ω scans with CCD	θ_{min} = 3.0º
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	h = −7→7
T_{min} = 0.966, T_{max} = 0.994	k = −13→14
21901 measured reflections	l = −22→22
2611 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.038	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.06P)^2 + 0.0P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$wR(F^2)$ = 0.106	$(\Delta/\sigma)_{max}$ = 0.0004
S = 0.92	$\Delta\rho_{max}$ = 0.51 e Å^{-3}
2611 reflections	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
172 parameters	Extinction correction: None
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.2692 (2)	0.79114 (12)	0.56450 (8)	0.0303	
C2	0.4415 (2)	0.71943 (11)	0.60258 (7)	0.0279	
C3	0.6338 (2)	0.77518 (12)	0.63949 (8)	0.0302	
C4	0.6533 (2)	0.89724 (12)	0.64045 (8)	0.0312	
C5	0.4790 (2)	0.96694 (13)	0.60382 (8)	0.0353	
C6	0.2888 (2)	0.91193 (12)	0.56546 (8)	0.0352	
C7	0.4314 (2)	0.59064 (11)	0.60988 (7)	0.0277	
C8	0.2519 (2)	0.52902 (12)	0.65265 (7)	0.0286	
N9	0.30935 (18)	0.41170 (9)	0.65408 (7)	0.0309	
C10	0.5104 (2)	0.39313 (12)	0.61892 (8)	0.0319	
C11	0.5831 (2)	0.51030 (12)	0.59190 (8)	0.0311	
N12	0.06814 (18)	0.73999 (11)	0.51944 (6)	0.0331	
O13	0.05347 (16)	0.63256 (9)	0.51382 (6)	0.0389	
O14	−0.07898 (18)	0.80745 (10)	0.48798 (7)	0.0484	
O15	0.85022 (17)	0.93928 (9)	0.67919 (6)	0.0409	
C16	0.8796 (3)	1.06411 (13)	0.68048 (10)	0.0464	
O17	0.09401 (16)	0.57328 (8)	0.68306 (6)	0.0377	
C18	0.1919 (3)	0.32159 (13)	0.69443 (10)	0.0431	
O19	0.60327 (18)	0.29883 (9)	0.61326 (7)	0.0445	
H31	0.7528	0.7298	0.6658	0.0358*	
H51	0.4969	1.0619	0.6060	0.0390*	
H61	0.1699	0.9582	0.5393	0.0411*	
H111	0.7165	0.5238	0.5661	0.0380*	
H161	1.0349	1.0809	0.7070	0.0667*	
H162	0.7571	1.1033	0.7086	0.0665*	
H163	0.8706	1.0943	0.6256	0.0658*	
H181	0.2539	0.2470	0.6855	0.0640*	0.7000
H182	0.2121	0.3339	0.7488	0.0640*	0.7000
H183	0.0363	0.3201	0.6779	0.0640*	0.7000
H184	0.2988	0.2824	0.7298	0.0640*	0.3000
H185	0.0780	0.3567	0.7216	0.0640*	0.3000
H186	0.1229	0.2674	0.6582	0.0640*	0.3000

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0281 (6)	0.0320 (7)	0.0308 (7)	−0.0006 (5)	0.0034 (5)	0.0019 (6)
C2	0.0280 (6)	0.0263 (7)	0.0304 (7)	0.0000 (5)	0.0072 (5)	0.0014 (5)
C3	0.0282 (6)	0.0270 (7)	0.0356 (7)	0.0003 (5)	0.0040 (5)	0.0042 (5)
C4	0.0322 (6)	0.0280 (7)	0.0336 (7)	−0.0047 (5)	0.0038 (5)	0.0017 (5)
C5	0.0397 (7)	0.0265 (7)	0.0399 (8)	0.0006 (6)	0.0055 (6)	0.0027 (6)
C6	0.0356 (7)	0.0310 (8)	0.0386 (8)	0.0065 (6)	0.0020 (6)	0.0052 (6)
C7	0.0267 (6)	0.0261 (7)	0.0303 (7)	−0.0036 (5)	0.0025 (5)	0.0013 (5)
C8	0.0256 (6)	0.0299 (7)	0.0302 (6)	−0.0025 (5)	0.0017 (5)	0.0001 (5)

N9	0.0300 (5)	0.0261 (6)	0.0369 (6)	−0.0040 (4)	0.0052 (5)	0.0035 (5)
C10	0.0295 (6)	0.0294 (7)	0.0362 (7)	0.0001 (5)	0.0006 (5)	−0.0004 (6)
C11	0.0276 (6)	0.0291 (7)	0.0367 (7)	−0.0022 (5)	0.0045 (5)	0.0007 (6)
N12	0.0303 (5)	0.0370 (7)	0.0322 (6)	−0.0003 (5)	0.0035 (5)	0.0034 (5)
O13	0.0372 (5)	0.0353 (6)	0.0432 (6)	−0.0069 (4)	−0.0006 (4)	0.0011 (4)
O14	0.0379 (6)	0.0497 (7)	0.0546 (7)	0.0074 (5)	−0.0096 (5)	0.0064 (5)
O15	0.0407 (5)	0.0274 (5)	0.0524 (6)	−0.0083 (4)	−0.0065 (5)	0.0035 (4)
C16	0.0593 (10)	0.0282 (8)	0.0486 (9)	−0.0138 (7)	−0.0092 (8)	0.0030 (7)
O17	0.0325 (5)	0.0383 (6)	0.0440 (6)	0.0019 (4)	0.0118 (4)	0.0009 (4)
C18	0.0438 (8)	0.0326 (8)	0.0540 (9)	−0.0089 (6)	0.0103 (7)	0.0092 (7)
O19	0.0428 (6)	0.0284 (6)	0.0629 (7)	0.0057 (4)	0.0082 (5)	0.0013 (5)

Geometric parameters (Å, °)

C1—C2	1.4055 (18)	N9—C18	1.4499 (17)
C1—C6	1.3791 (18)	C10—C11	1.4883 (18)
C1—N12	1.4611 (16)	C10—O19	1.2104 (17)
C2—C3	1.3865 (18)	C11—H111	0.948
C2—C7	1.4724 (17)	N12—O13	1.2285 (16)
C3—C4	1.3933 (19)	N12—O14	1.2355 (15)
C3—H31	0.944	O15—C16	1.4305 (16)
C4—C5	1.390 (2)	C16—H161	0.992
C4—O15	1.3569 (16)	C16—H162	1.007
C5—C6	1.383 (2)	C16—H163	1.005
C5—H51	1.086	C18—H181	0.941
C6—H61	0.948	C18—H182	0.945
C7—C8	1.5132 (17)	C18—H183	0.924
C7—C11	1.3304 (18)	C18—H184	0.940
C8—N9	1.3758 (17)	C18—H185	0.940
C8—O17	1.2149 (15)	C18—H186	0.940
N9—C10	1.3901 (16)		
O13···O13^{i}	3.107 (2)	O15···C18^{vi}	3.537 (2)
O13···O19^{ii}	3.213 (2)	O15···C10^{vi}	3.538 (2)
O13···C11^{ii}	3.354 (2)	O17···C11^{iii}	3.305 (2)
O13···N9^{i}	3.450 (2)	O17···C3^{iii}	3.556 (2)
O13···C11^{iii}	3.468 (2)	O19···N12^{ii}	3.164 (2)
O14···C3^{iii}	3.267 (2)	O19···C16^{viii}	3.271 (2)
O14···C4^{iii}	3.354 (2)	O19···C1^{ii}	3.389 (2)
O14···C18^{i}	3.477 (2)	O19···C18^{vii}	3.585 (2)
O14···C6^{iv}	3.509 (2)	N12···C3^{iii}	3.455 (2)
O14···C16^{v}	3.555 (2)	C8···C16^{ix}	3.440 (2)
O14···O19^{i}	3.576 (2)	C11···C11^{ii}	3.232 (3)
O15···N9^{vi}	3.343 (2)	C16···C18^{x}	3.445 (2)
O15···C6^{vii}	3.393 (2)		
C2—C1—C6	121.23 (12)	N9—C10—O19	125.05 (13)
C2—C1—N12	121.04 (12)	C11—C10—O19	128.65 (12)

C6—C1—N12	117.67 (12)	C10—C11—C7	108.96 (11)
C1—C2—C3	117.18 (12)	C10—C11—H111	124.5
C1—C2—C7	125.79 (12)	C7—C11—H111	126.6
C3—C2—C7	116.97 (11)	C1—N12—O13	118.96 (11)
C2—C3—C4	121.50 (12)	C1—N12—O14	118.11 (12)
C2—C3—H31	119.5	O13—N12—O14	122.93 (11)
C4—C3—H31	119.0	C4—O15—C16	116.85 (11)
C3—C4—C5	120.56 (13)	O15—C16—H161	107.5
C3—C4—O15	114.90 (12)	O15—C16—H162	110.9
C5—C4—O15	124.54 (13)	H161—C16—H162	110.6
C4—C5—C6	118.30 (13)	O15—C16—H163	109.2
C4—C5—H51	119.3	H161—C16—H163	109.2
C6—C5—H51	122.4	H162—C16—H163	109.4
C5—C6—C1	121.20 (13)	N9—C18—H181	110.6
C5—C6—H61	119.3	N9—C18—H182	110.8
C1—C6—H61	119.5	H181—C18—H182	106.6
C2—C7—C8	122.59 (11)	N9—C18—H183	111.2
C2—C7—C11	129.08 (11)	H181—C18—H183	108.3
C8—C7—C11	107.75 (11)	H182—C18—H183	109.3
C7—C8—N9	106.21 (10)	N9—C18—H184	109.5
C7—C8—O17	127.68 (12)	N9—C18—H185	109.4
N9—C8—O17	126.04 (12)	H184—C18—H185	109.5
C8—N9—C10	110.74 (10)	N9—C18—H186	109.6
C8—N9—C18	124.66 (11)	H184—C18—H186	109.5
C10—N9—C18	124.25 (12)	H185—C18—H186	109.5
N9—C10—C11	106.29 (11)		
O13—N12—C1—C2	−1.5 (2)	C1—C2—C7—C8	−62.5 (2)
O13—N12—C1—C6	175.7 (1)	C1—C2—C7—C11	127.4 (2)
O14—N12—C1—C2	179.2 (1)	C1—C6—C5—C4	1.2 (2)
O14—N12—C1—C6	−3.6 (2)	C2—C1—C6—C5	0.1 (2)
O15—C4—C3—C2	179.6 (1)	C2—C3—C4—C5	−0.2 (2)
O15—C4—C5—C6	179.1 (1)	C2—C7—C11—C10	173.0 (1)
O17—C8—N9—C10	−175.1 (1)	C3—C2—C1—C6	−1.4 (2)
O17—C8—N9—C18	−1.6 (2)	C3—C2—C7—C8	114.5 (1)
O17—C8—C7—C2	2.7 (2)	C3—C2—C7—C11	−55.6 (2)
O17—C8—C7—C11	174.7 (1)	C3—C4—O15—C16	178.9 (1)
O19—C10—N9—C8	178.5 (1)	C3—C4—C5—C6	−1.1 (2)
O19—C10—N9—C18	5.0 (2)	C4—C3—C2—C7	−175.8 (1)
O19—C10—C11—C7	−180.0 (1)	C5—C4—O15—C16	−1.3 (2)
N9—C8—C7—C2	−174.2 (1)	C6—C1—C2—C7	175.6 (1)
N9—C8—C7—C11	−2.3 (1)	C7—C8—N9—C10	1.9 (1)
N9—C10—C11—C7	−0.6 (1)	C7—C8—N9—C18	175.4 (1)
N12—C1—C2—C3	175.6 (1)	C8—N9—C10—C11	−0.9 (1)
N12—C1—C2—C7	−7.3 (2)	C8—C7—C11—C10	1.7 (1)
N12—C1—C6—C5	−177.1 (1)	C11—C10—N9—C18	−174.4 (1)
C1—C2—C3—C4	1.5 (2)		

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) −*x*, −*y*+2, −*z*+1; (v) −*x*+1, −*y*+2, −*z*+1; (vi) −*x*+3/2, *y*+1/2, −*z*+3/2; (vii) *x*+1, *y*, *z*; (viii) *x*, *y*−1, *z*; (ix) −*x*+3/2, *y*−1/2, −*z*+3/2; (x) *x*+1, *y*+1, *z*.