Sample Structure Report

Crystal Data and Experimental

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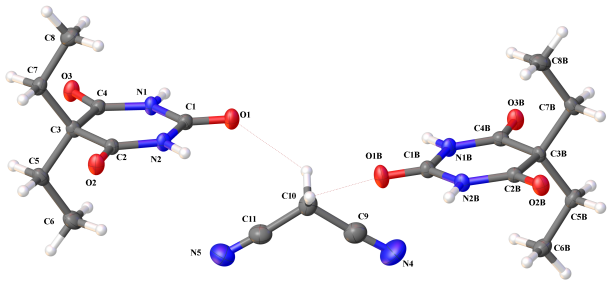

Crystal Data. $C_{19}H_{26}N_6O_6$, M = 434.46, orthorhombic, $Pna2_1$ (No. 33, a = 11.3799(9) Å, b = 12.1439(9) Å, c = 15.7504(12) Å, $\alpha = \beta = \gamma = 90°$, V = 2176.6(3)$Å^3$, T = 110(2) K, Z = 4, μ(Mo Kα) = 0.101, 21802 reflections measured, 5375 unique (R_{int} = 0.0367) which were used in all calculations. The final wR_2 was 0.1383 (all data) and R_1 was 0.0490 ($I > 2\sigma(I)$).

Experimental. Single crystals of $C_{19}H_{26}N_6O_6$ (**barbitalnitrile**) were recrystallised from a mixture of ethyl acetate and hexane by slow evaporation. A suitable crystal ($0.582 \times 0.166 \times 0.140 mm^3$) was selected and mounted on a loop on a Bruker APEX-II CCD diffractometer. The crystal was kept at 110(2) K during data collection. Using Olex2 [1], the structure was solved with the Superflip [2] structure solution program, using the Charge Flipping solution method. The model was refined with the ShelXL [3] refinement package using Least Squares minimisation.

Compound	barbitalnitrile
CCDC	
Formula	$C_{19}H_{26}N_6O_6$
$D_{calc.}$/ g cm^{-3}	1.326
μ/ mm^{-1}	0.101
Formula Weight	434.46
Colour	colourless
Shape	prism
Size/mm^3	$0.582 \times 0.166 \times 0.140$
T/K	110(2)
Crystal System	orthorhombic
Space Group	$Pna2_1$
a/Å	11.3799(9)
b/Å	12.1439(9)
c/Å	15.7504(12)
α/°	90
β/°	90
γ/°	90
V/ Å^3	2176.6(3)
Z	4
Θ_{min}/°	2.118
Θ_{max}/°	28.274
Measured Refl.	21802
Independent Refl.	5375
Reflections Used	3813
R_{int}	0.0367
Parameters	284
Restraints	1
Largest Peak Largest Peak	0.391
Deepest Hole	-0.232
GooF	1.064
wR_2(all data)	0.1383
wR_2	0.1182
R_1(all data)	0.0719
R_1	0.0490

References

[1] O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Cryst. (2009). 42, 339-341.

[2] SUPERFLIP, J. Appl. Cryst. (2007) 40, 786-790

[3] SHELX, G.M. Sheldrick, Acta Cryst. (2008). A64, 112-122

Table 1: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **barbitalnitrile**. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	**x**	**y**	**z**	**U(eq)**
O1	5049(2)	9194(2)	5816.9(17)	24.7(7)
O2	5227(2)	7214(2)	8240.8(17)	22.7(5)
O3	5292(2)	11107(2)	8268.2(16)	23.4(5)
N1	5156(2)	10138(3)	7051(2)	18.7(6)
N2	5125(2)	8215(3)	7036(2)	19.1(7)
C1	5108(3)	9185(3)	6579(2)	18.6(8)
C2	5202(2)	8115(3)	7897(2)	16.8(7)
C3	5256(3)	9158(3)	8427(2)	17.4(7)
C4	5232(2)	10215(3)	7916(2)	17.3(7)
C5	4174(3)	9164(3)	9021(2)	22.5(7)
C6	3009(3)	9212(3)	8542(2)	30.0(8)
C7	6414(3)	9131(3)	8937(2)	20.9(7)
C8	7504(3)	9186(3)	8374(2)	28.1(8)
O1B	4808(2)	5781(2)	3682.1(17)	26.3(7)
O2B	5025.3(18)	3897(2)	1220.0(18)	23.9(6)
O3B	4905.5(19)	7782(2)	1264.9(17)	23.8(6)
N1B	4873(2)	6778(3)	2464(2)	19.9(7)
N2B	4867(2)	4846(3)	2437(2)	19.2(6)
C1B	4846(3)	5802(3)	2916(2)	19.1(8)
C2B	4939(2)	4786(3)	1574(2)	17.6(7)
C3B	4889(2)	5842(3)	1066(2)	16.4(7)
C4B	4896(3)	6878(3)	1601(2)	18.1(7)
C5B	3716(3)	5831(3)	562(2)	22.1(7)
C6B	2625(3)	5776(3)	1132(2)	26.8(8)
C7B	5958(3)	5886(3)	462(2)	22.0(7)
C8B	7126(3)	5852(3)	939(2)	30.1(8)
N4	2172(3)	7401(3)	3461(3)	55.8(11)
N5	2457(3)	7670(3)	6246(3)	46.7(9)
C9	2734(3)	7443(3)	4055(3)	35.5(8)
C10	3520(3)	7503(4)	4794(2)	29.3(7)
C11	2889(3)	7598(3)	5603(3)	32.5(8)

Table 2: Anisotropic Displacement Parameters ($\times 10^4$) **barbitalnitrile**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a*^2 U_{11} + ... + 2hka \times b \times U_{12}]$

Atom	$\mathbf{U}_{11}$	$\mathbf{U}_{22}$	$\mathbf{U}_{33}$	$\mathbf{U}_{23}$	$\mathbf{U}_{13}$	$\mathbf{U}_{12}$
O1	35.4(13)	21.2(18)	17.4(15)	−1.8(11)	−0.4(9)	−2.8(10)
O2	35.5(13)	13.2(13)	19.5(12)	1.7(12)	−1.5(10)	−0.2(9)
O3	36.7(12)	13.9(13)	19.5(12)	−1.8(11)	−2.6(10)	−0.6(9)
N1	27.9(14)	13.9(16)	14.3(13)	0.1(13)	−0.3(10)	−0.5(10)
N2	28.1(14)	11.9(17)	17.4(16)	−2.9(14)	0.8(11)	−1(1)
C1	20.7(15)	16(2)	18.5(19)	−1.3(15)	0.6(11)	−2.1(12)
C2	18.7(15)	14.9(19)	16.7(17)	−1.2(15)	0.3(11)	1.2(11)
C3	22.9(15)	12.3(19)	16.9(17)	−0.3(13)	2.2(12)	0.4(12)
C4	18.6(15)	15.3(19)	18.1(17)	−0.6(15)	−1.0(11)	−1.1(11)
C5	28.1(16)	18.9(18)	20.5(16)	−0.1(13)	6.3(13)	−3.3(12)
C6	26.9(17)	34(2)	28.9(19)	−3.5(16)	7.5(14)	−1.6(15)
C7	25.3(16)	19.2(18)	18.2(15)	0.2(13)	−4.8(12)	−0.2(12)
C8	24.4(16)	33(2)	27.4(19)	1.0(16)	−3.9(14)	−2.1(14)
O1B	41.8(15)	20.9(18)	16.2(14)	0.2(11)	2.8(10)	0.7(10)
O2B	33.3(12)	16.6(13)	21.8(13)	−2.4(11)	2.4(10)	1.5(9)
O3B	36.7(13)	14.1(14)	20.6(13)	−0.6(12)	0.9(10)	−1.2(8)
N1B	27.8(14)	14.1(18)	17.6(15)	−2.9(15)	0.4(11)	−1.3(10)
N2B	26.9(13)	12.8(16)	18.0(14)	3.0(13)	0.4(11)	−0.9(10)
C1B	22.7(16)	16(2)	18.3(18)	−1.0(15)	1.1(12)	0.7(12)
C2B	18.1(15)	16(2)	18.9(17)	1.1(15)	0.3(11)	−0.5(11)
C3B	20.8(14)	14(2)	14.0(16)	1.3(13)	1.0(11)	−0.8(11)
C4B	17.2(15)	16(2)	21.4(18)	−0.3(15)	1.3(11)	−0.2(11)
C5B	25.7(15)	19.7(18)	20.8(17)	−1.6(14)	−6.2(13)	0.4(12)
C6B	23.0(15)	29(2)	28.8(19)	−4.7(16)	−4.6(14)	−0.3(13)
C7B	28.5(16)	19.7(18)	17.7(16)	0.6(13)	6.5(13)	−1.5(12)
C8B	22.9(16)	33(2)	35(2)	−3.3(17)	7.6(14)	−3.5(14)
N4	42.4(19)	75(3)	50(2)	0(2)	−16.3(18)	−2.7(19)
N5	37.2(17)	60(3)	43(2)	1(2)	7.1(15)	−3.0(16)
C9	34.0(19)	33(2)	40(2)	1.3(18)	−1.1(16)	−2.6(15)
C10	32.6(17)	29.0(14)	26.5(15)	−1.7(13)	−2.6(14)	2.3(15)
C11	25.8(15)	32(2)	40(2)	1.9(18)	0.1(16)	2.2(14)

Table 3: Bond Lengths in Å for **barbitalnitrile**.

Atom	**Atom**	**Length/Å**	**Atom**	**Atom**	**Length/Å**
O1	C1	1.203(5)	O3B	C4B	1.219(5)
O2	C2	1.221(5)	N1B	C1B	1.384(5)
O3	C4	1.219(4)	N1B	C4B	1.364(5)
N1	C1	1.377(5)	N2B	C1B	1.384(5)
N1	C4	1.367(4)	N2B	C2B	1.364(5)
N2	C1	1.380(5)	C2B	C3B	1.512(5)
N2	C2	1.365(5)	C3B	C4B	1.515(5)
C2	C3	1.518(5)	C3B	C5B	1.553(4)
C3	C4	1.516(5)	C3B	C7B	1.545(4)
C3	C5	1.547(4)	C5B	C6B	1.533(5)
C3	C7	1.544(4)	C7B	C8B	1.527(5)
C5	C6	1.526(5)	N4	C9	1.134(5)
C7	C8	1.527(5)	N5	C11	1.129(5)
O1B	C1B	1.208(5)	C9	C10	1.469(5)
O2B	C2B	1.220(5)	C10	C11	1.467(5)

Table 4: Bond Angles in ° for **barbitalnitrile**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C4	N1	C1	126.7(3)	O1B	C1B	N1B	122.3(4)
C2	N2	C1	126.5(3)	O1B	C1B	N2B	121.8(4)
O1	C1	N1	122.3(4)	N1B	C1B	N2B	116.0(3)
O1	C1	N2	122.0(4)	O2B	C2B	N2B	120.5(4)
N1	C1	N2	115.8(3)	O2B	C2B	C3B	120.8(3)
O2	C2	N2	121.4(3)	N2B	C2B	C3B	118.7(3)
O2	C2	C3	120.3(3)	C2B	C3B	C4B	114.1(3)
N2	C2	C3	118.3(3)	C2B	C3B	C5B	107.2(3)
C2	C3	C5	107.7(3)	C2B	C3B	C7B	109.0(3)
C2	C3	C7	107.6(3)	C4B	C3B	C5B	107.2(3)
C4	C3	C2	114.5(3)	C4B	C3B	C7B	108.0(2)
C4	C3	C5	107.7(3)	C7B	C3B	C5B	111.3(3)
C4	C3	C7	108.1(2)	O3B	C4B	N1B	120.8(3)
C7	C3	C5	111.4(3)	O3B	C4B	C3B	120.4(3)
O3	C4	N1	121.2(3)	N1B	C4B	C3B	118.8(3)
O3	C4	C3	120.6(3)	C6B	C5B	C3B	113.4(3)
N1	C4	C3	118.1(3)	C8B	C7B	C3B	112.5(3)
C6	C5	C3	113.1(3)	N4	C9	C10	176.8(4)
C8	C7	C3	112.9(3)	C11	C10	C9	113.2(3)
C4B	N1B	C1B	126.1(3)	N5	C11	C10	176.5(4)
C2B	N2B	C1B	126.0(4)				

Table 5: Hydrogen Atom Coordinates (Å$\times 10^4$) and Isotropic Displacement Parameters (Å$^2 \times 10^3$) for **barbitalnitrile**.

Atom	x	y	z	U(eq)
H1A	5135	10756	6770	22
H2	5078	7605	6748	23
H5A	4188	8491	9376	27
H5B	4227	9807	9406	27
H6A	2971	8603	8135	45
H6B	2952	9914	8238	45
H6C	2357	9150	8946	45
H7A	6425	9760	9337	25
H7B	6441	8445	9277	25
H8A	7512	9887	8066	42
H8B	7489	8576	7967	42
H8C	8211	9130	8727	42
H1BA	4873	7393	2760	24
H2B	4832	4218	2716	23
H5BA	3671	6504	209	26
H5BB	3713	5189	175	26
H6BA	2631	5084	1452	40
H6BB	1916	5810	779	40
H6BC	2631	6398	1528	40
H7BA	5921	5255	65	26
H7BB	5920	6570	122	26
H8BA	7157	6461	1346	45
H8BB	7775	5923	534	45
H8BC	7195	5151	1243	45
H1B	4017	6835	4808	35
H1	4046	8147	4728	35

Table 6: Hydrogen Bonds for **barbitalnitrile**.

D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
N1	H1A	O3B[1]	0.87	1.95	2.815(4)	173.6
N2	H2	O2B[2]	0.87	2.01	2.874(4)	173.0
N1B	H1BA	O3[3]	0.88	2.00	2.870(4)	170.1
N2B	H2B	O2[4]	0.88	1.93	2.805(4)	175.5

Symmetry Code:[1]1-X,2-Y,1/2+Z; [2]1-X,1-Y,1/2+Z; [3]1-X,2-Y,-1/2+Z; [4]1-X,1-Y,-1/2+Z