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|  | Single Crystal X-Ray DiffractionSouthampton UniversityContact: Dr S.K.Callear, S.K.Callear@soton.ac.uk |

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**Table 1.** Crystal data and structure refinement details.

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Identification code **06skc0010p212121**

Empirical formula C7H8N2O2

Formula weight 152.15

Temperature 120(2) K

Wavelength 0.71073 Å

Crystal system Orthorhombic

Space group *P*212121

Unit cell dimensions *a* = 3.8043(11) Å *α* = 90°

 *b* = 12.987(4) Å *β* = 90°

 *c* = 14.401(3) Å *γ* = 90°

Volume 711.5(3) Å3

*Z* 4

Density (calculated) 1.420 Mg / m3

Absorption coefficient 0.107 mm−1

*F(000)* 320

Crystal plate; yellow

Crystal size 0.09 × 0.08 × 0.01 mm3

*θ* range for data collection 3.14 − 27.47°

Index ranges −4 ≤ *h* ≤ 4, −16 ≤ *k* ≤ 16, −18 ≤ *l* ≤ 18

Reflections collected 8327

Independent reflections 985 [*Rint* = 0.3099]

Completeness to *θ* = 27.47° 99.5 %

Absorption correction Semi−empirical from equivalents

Max. and min. transmission 0.9989 and 0.9905

Refinement method Full-matrix least-squares on *F*2

Data / restraints / parameters 985 / 2 / 107

Goodness-of-fit on *F*2 1.158

Final *R* indices [*F*2 > 2*σ*(*F*2)] *R1* = 0.1223, *wR2* = 0.1605

*R* indices (all data) *R1* = 0.2282, *wR2* = 0.1960

Absolute structure parameter −10(10)

Largest diff. peak and hole 0.328 and −0.473 e Å**−3**

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**Diffractometer**: *Nonius KappaCCD* area detector (*φ* scans and *ω* scans to fill *asymmetric unit*). **Cell determination:** DirAx (Duisenberg, A.J.M.(1992). J. Appl. Cryst. 25, 92-96.) **Data collection:** Collect (Collect: Data collection software, R. Hooft, Nonius B.V., 1998). **Data reduction and cell** **refinement**: *Denzo* (Z. Otwinowski & W. Minor, *Methods in Enzymology* (1997) Vol. **276**: *Macromolecular Crystallography*, part A, pp. 307−326; C. W. Carter, Jr. & R. M. Sweet, Eds., Academic Press). **Absorption correction**: Sheldrick, G. M. SADABS - Bruker Nonius area detector scaling and absorption correction - V2.10 **Structure solution**: *SHELXS97* (G. M. Sheldrick, Acta Cryst. (1990) A**46** 467−473). **Structure refinement**: *SHELXL97* (G. M. Sheldrick (1997), University of Göttingen, Germany). **Graphics:** ORTEP-3 (L.G. Farrugia, J. Appl. Cryst. (1997) **30**, 565).

**Special details**:

All hydrogens except amine hydrogens fixed using a riding model. The position of the amine hydrogens and their thermal parameters were left to refine while their N-H distances were fixed at standard values.

As the crystals were very small and weakly diffracting, the Rint is higher than usual and there is a lower C-C bond precision. In spite of this the data are perfectly fine for viewing the gross connectivity and interactions in the structure.

 Merge 4 was used as there are no heavy atoms in the structure.**Table 2.** Atomic coordinates [× 104], equivalent isotropic displacement parameters [Å2 × 103] and site occupancy factors. *Ueq* is defined as one third of the trace of the orthogonalized *Uij* tensor.

Atom *x* *y* *z* *Ueq* *S.o.f.*

C1 −270(30) 4131(7) 4371(6) 30(3) 1

C2 890(20) 4762(7) 3644(6) 24(2) 1

C3 610(30) 5817(7) 3785(6) 30(3) 1

C4 −550(30) 6252(7) 4612(6) 30(2) 1

C5 −1590(30) 5609(8) 5309(7) 35(3) 1

C6 −1470(20) 4559(7) 5197(6) 26(2) 1

C7 2410(20) 4294(6) 2778(6) 27(2) 1

N1 −10(20) 3060(6) 4282(5) 28(2) 1

N3 1430(20) 6546(6) 3049(5) 33(2) 1

O3A 370(20) 6349(5) 2261(4) 43(2) 1

O3B 3101(19) 7329(5) 3254(5) 40(2) 1

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**Table 3.** Bond lengths [Å] and angles [°].

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C1−C6 1.389(11)

C1−C2 1.400(11)

C1−N1 1.401(12)

C2−C3 1.389(11)

C2−C7 1.504(11)

C3−C4 1.388(12)

C3−N3 1.456(11)

C4−C5 1.364(12)

C4−H4 0.9500

C5−C6 1.374(12)

C5−H5 0.9500

C6−H6 0.9500

C7−H7A 0.9800

C7−H7B 0.9800

C7−H7C 0.9800

N1−H1A 0.87(2)

N1−H1B 0.88(2)

N3−O3A 1.231(9)

N3−O3B 1.236(9)

C6−C1−C2 120.6(8)

C6−C1−N1 120.0(9)

C2−C1−N1 119.3(9)

C3−C2−C1 116.3(8)

C3−C2−C7 123.3(8)

C1−C2−C7 120.3(8)

C4−C3−C2 123.4(9)

C4−C3−N3 115.3(8)

C2−C3−N3 121.2(8)

C5−C4−C3 118.3(9)

C5−C4−H4 120.9

C3−C4−H4 120.9

C4−C5−C6 120.7(9)

C4−C5−H5 119.6

C6−C5−H5 119.6

C5−C6−C1 120.5(9)

C5−C6−H6 119.7

C1−C6−H6 119.7

C2−C7−H7A 109.5

C2−C7−H7B 109.5

H7A−C7−H7B 109.5

C2−C7−H7C 109.5

H7A−C7−H7C 109.5

H7B−C7−H7C 109.5

C1−N1−H1A 112(7)

C1−N1−H1B 119(6)

H1A−N1−H1B 116(10)

O3A−N3−O3B 124.1(8)

O3A−N3−C3 117.7(8)

O3B−N3−C3 118.2(8)

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Symmetry transformations used to generate equivalent atoms:

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**Table 4.** Anisotropic displacement parameters [Å2× 103]. The anisotropic displacement

factor exponent takes the form: −2*π* 2[*h*2*a*\*2*U*11 + ... + 2 *h k a*\* *b*\* *U*12 ].

Atom *U*11 *U*22 *U*33 *U*23 *U*13 *U*12

C1 35(7) 34(6) 22(5) 3(5) 2(5) −7(5)

C2 18(5) 34(6) 20(5) −4(4) −6(4) 1(5)

C3 39(7) 26(6) 25(5) 9(4) 2(5) 10(5)

C4 33(6) 29(6) 28(5) 10(4) −3(5) 7(5)

C5 27(6) 49(7) 29(6) −9(5) 3(5) 0(5)

C6 33(6) 34(6) 12(5) 13(4) −1(4) −5(5)

C7 29(6) 31(6) 22(5) −8(4) −3(4) −2(5)

N1 27(5) 31(5) 24(5) 0(4) −10(4) −5(4)

N3 34(5) 33(5) 32(5) −6(4) −9(4) 0(5)

O3A 60(5) 37(4) 33(4) 3(4) 0(4) 0(4)

O3B 51(5) 25(4) 45(4) 8(4) 9(4) −8(4)

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**Table 5.** Hydrogen bonds [Å and °].

 *D*−H···*A* *d*(*D*−H) *d*(H···*A*) *d*(*D*···*A*) ∠(*D*H*A*)

 N1−H1A...N1i 0.87(2) 2.30(3) 3.165(12) 173(9)

 N1−H1B...O3Aii 0.88(2) 2.35(5) 3.145(10) 151(8)

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Symmetry transformations used to generate equivalent atoms:

(i) x−1/2,−y+1/2,−z+1 (ii) −x,y−1/2,−z+1/2

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