

Table 2 (*cont.*)

N(4)—C(5)—C(5a)	108.4 (2)	N(3)—C(21)—C(22)	120.0 (2)
N(4)—C(5)—C(51)	111.2 (1)	N(3)—C(21)—C(26)	120.5 (1)
C(5a)—C(5)—C(51)	115.9 (2)	C(22)—C(21)—C(26)	119.5 (1)
C(5)—C(5a)—C(6)	123.4 (2)	C(21)—C(22)—C(23)	119.4 (2)
C(5)—C(5a)—C(9a)	117.0 (1)	C(22)—C(23)—C(24)	121.3 (2)
C(6)—C(5a)—C(9a)	119.5 (2)	C(23)—C(24)—C(25)	119.1 (2)
C(5a)—C(6)—C(7)	121.3 (2)	C(24)—C(25)—C(26)	121.0 (2)
C(6)—C(7)—C(8)	119.4 (2)	C(21)—C(26)—C(25)	119.7 (2)
C(6)—C(7)—O(71)	124.3 (2)	C(3a)—C(31)—O(32)	126.3 (2)
C(8)—C(7)—O(71)	116.3 (2)	C(3a)—C(31)—O(33)	109.3 (1)
C(7)—C(8)—C(9)	119.7 (2)	O(32)—C(31)—O(33)	124.4 (1)
C(7)—C(8)—O(81)	115.7 (2)	C(31)—O(33)—C(34)	116.3 (1)
C(9)—C(8)—O(81)	124.6 (2)	O(33)—C(34)—C(35)	106.7 (2)
C(8)—C(9)—C(9a)	120.8 (2)	C(7)—O(71)—C(72)	117.5 (1)
C(5a)—C(9a)—C(9)	119.2 (1)	C(8)—O(81)—C(82)	117.7 (1)

41 $\bar{3}$, 420. ω scans; amplitude $s = (0.95 + 0.14 \tan \theta)^\circ$. Intensity variations during measurements not significant. No absorption correction. 3923 unique reflections measured; 820 unobserved reflections [$I < 3\sigma(I)$]. Direct method (symbolic addition method), program *SIMPEL* (Schenk, Overbeek, van der Putten, Olthof, Schagen, Peschar, Seignette, Driessen & Kiers, 1982). H atoms located from difference Fourier maps. Full-matrix refinements based on *F*. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). Refined parameters: x, y, z for all atoms and β_{ij} values for C, N and O atoms. For H atoms B was chosen equal to 1.30 times the value of B_{eq} of the neighbouring heavy atom. $R = 0.037$, $wR = 0.052$, $w = 1/\sigma^2(F)$, $S = 1.95$. Secondary-extinction coefficient, $g = 2.6(3) \times 10^{-6}$. Maximum shift to e.s.d. ratio $(\Delta/\sigma)_{\max} < 0.04$. $\Delta\rho_{\max} = 0.17(3)$, $\Delta\rho_{\min} = -0.14(3) \text{ e } \text{Å}^{-3}$. Computer programs of the *SDP* System (B. A. Frenz & Associates, Inc., 1982). Program *ORTEPII* (Johnson, 1976) used to represent

the molecule (Fig. 1). Atomic coordinates are listed in Table 1, bond lengths and angles in Table 2.*

Related literature. A comparison of experimental torsion angles and data given by Bucourt & Hainaut (1965) shows that ring *B* adopts a quasi 1,3-diplanar conformation. Details of the synthesis are given by Moustaid (1991) and Moustaid, Nguyen Dinh An, Mercier, Sedqui & Laude (1992).

* Lists of structure factors, coordinates of H atoms, anisotropic thermal parameters for non-H atoms, bond distances involving H atoms, average planes of rings and torsion angles, and an *ORTEPII* plot of the molecular packing have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54867 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA0252]

References

- B. A. FRENZ & ASSOCIATES, INC. (1982). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands.
- BU COURT, R. & HAINAUT, D. (1965). *Bull. Soc. Chim. Fr.* pp. 1366–1378.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- MOUSTAID, K. (1991). DSc Thesis. Univ. of Franche-Comté, France.
- MOUSTAID, K., NGUYEN DINH AN, MERCIER, M. F., SEDQUI, A. & LAUDE, B. (1992). *Can. J. Chem.* In the press.
- SCHENK, H., OVERBEEK, O., VAN DER PUTTEN, N., OLTHOF, G., SCHAGEN, J. D., PESCHAR, R., SEIGNETTE, P., DRIESSEN, R. & KIERS, C. (1982). *SIMPEL*. Crystallography Department, Univ. of Amsterdam.

Acta Cryst. (1992). **C48**, 1331–1333

N-Benzylmethanesulfonamide

BY DONALD T. ELMORE, JOHN H. JONES AND MARYAM RAISSI

The Dyson Perrins Laboratory, The University of Oxford, OX1 3QY, England

AND KEITH PROUT

Chemical Crystallography Laboratory, The University of Oxford, OX1 3PD, England

(Received 3 March 1991; accepted 25 November 1991)

Abstract. $\text{C}_8\text{H}_{11}\text{NO}_2\text{S}$, $M_r = 185.24$, orthorhombic, *Pbc*₂₁, $a = 5.473(2)$, $b = 8.627(4)$, $c = 19.425(5)$ Å, $V = 917.3$ Å³, $Z = 4$, $D_x = 1.341$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 27.7$ cm⁻¹, $F(000) = 392$, $T = 293$ K, $wR = 4.52$, $R = 3.94\%$ for 824 reflections with $I > 3\sigma(I)$. This is the first crystal structure

determination reported for a simple sulfonamide containing the structural element $\text{C}(sp^3)\text{—SO}_2\text{NH—C}(sp^3)$.

Experimental. The title compound was prepared according to Johnston, Kussner & Holum (1960),

and crystallized by dissolving it in hot ether and allowing the solvent to evaporate slowly, giving diamond-shaped crystals suitable for X-ray single-crystal diffraction. A summary of the data collection and structure refinement is given in Table 1. The structure was solved using *SHELXS86* (Sheldrick, 1986). Isotropic refinement followed by difference Fourier synthesis gave the H-atom positions, which were refined using a 'riding' algorithm. The origin was fixed by the method of Flack & Schwarzenbach (1988). The weighting scheme is according to Prince (1982). Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 202). All computations, other than direct methods, were performed using *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Atomic parameters are given in Table 2,* bond lengths and some important bond angles are given in Table 3, and atom labelling is indicated in Fig. 1.

Related literature. No other crystal structure for a simple sulfonamide containing the structural element C(sp³)—SO₂NH—C(sp³) has been reported. At the time the present determination was undertaken, the only example of the class in the crystallographic literature was 7,8-dihydroxy-4-methyl-5-(4-methylbenzylsulfonamido)-10-phenylselenomethyl-3-oxabicyclo[4.4.0]decan-2-one (Yoshioka, Nakai & Ohno, 1984), but the structures of two pseudopeptide derivatives of taurine (2-aminoethanesulfonic acid) have since been described: *N*-acetyltauryl-L-phenylalanine methyl ester (Calcagni, Gavuzzo, Lucente, Mazza, Pochetti & Rossi, 1989) and *cyclo*-(*N*-methyltauryl-L-phenylalanyl-D-proline) (Calcagni, Gavuzzo, Lucente, Mazza, Pinnen, Pochetti & Rossi, 1989).

* Full lists of anisotropic displacement parameters, H-atom coordinates, bond distances and angles including H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54890 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

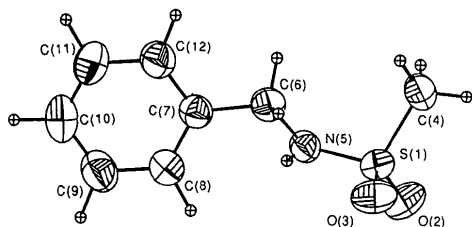


Fig. 1. Structure of C₆H₅CH₂NHSO₂CH₃ with atom-labelling scheme.

Table 1. Summary of the data collection and structure refinement for CH₃SO₂NHCH₂C₆H₅

Crystal shape, dimensions (mm)	Tablet, 0.8 × 0.9 × 0.1
Volume (mm ³)	7.2 × 10 ⁻²
Diffractometer	Enraf-Nonius CAD-4, graphite-monochromated Cu radiation
Determination of cell parameters	
Number of reflections used	25
θ range (°)	16.5–23.5
Intensity-data collection	
Max. sin θ/λ (Å ⁻¹)	0.61
Range of hkl	-1, -1, -1 to 6, 10, 23
Scan mode	ω-2θ
Min. scan angle (°)	1.2
Max. scan time (s)	60
Standard reflections, intensity variation (%)	117, 221, 128, < 3
Measured reflections	1545
Unique reflections	902
Observed reflections [I > 3σ(I)]	824
R _{int}	0.44
Numeric absorption correction	
Structure refinement (on F)	
R, wR (%)	3.94, 4.52
Number of parameters	120
R.m.s. shift/e.s.d.	< 0.0312
Final Δρ (e Å ⁻³)	-0.25 ≤ Δρ ≤ 0.18

Table 2. Positional and equivalent isotropic displacement (Å²) parameters for the non-H atoms with e.s.d.'s in parentheses

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$, where U_{11} , U_{22} and U_{33} are the mean-square displacements (Å²) along each of the principal axes of the thermal ellipsoid.

	x	y	z	U_{eq}
S(1)	0.3864 (1)	0.72705 (9)	0.04683 (8)	0.0443 (3)
O(2)	0.6274 (5)	0.7102 (3)	0.0746 (2)	0.059 (3)
O(3)	0.2603 (6)	0.5900 (3)	0.0258 (2)	0.060 (3)
C(4)	0.2003 (9)	0.8231 (6)	0.1066 (2)	0.058 (3)
N(5)	0.4128 (6)	0.8390 (4)	-0.0200 (2)	0.046 (2)
C(6)	0.1869 (8)	0.8875 (5)	-0.0541 (2)	0.055 (3)
C(7)	0.2279 (7)	0.9343 (5)	-0.1279 (2)	0.049 (3)
C(8)	0.4179 (9)	0.8781 (6)	-0.1679 (2)	0.060 (3)
C(9)	0.4405 (9)	0.9240 (7)	-0.2361 (3)	0.069 (4)
C(10)	0.278 (1)	1.0279 (7)	-0.2647 (3)	0.073 (4)
C(11)	0.088 (1)	1.0838 (7)	-0.2246 (2)	0.075 (5)
C(12)	0.0632 (9)	1.0369 (7)	-0.1571 (2)	0.064 (4)

Table 3. Bond lengths (Å) and bond angles (°) for the non-H atoms, with e.s.d.'s in parentheses

S(1)—O(2)	1.432 (3)	C(7)—C(8)	1.386 (5)
S(1)—O(3)	1.429 (3)	C(7)—C(12)	1.385 (6)
S(1)—C(4)	1.753 (4)	C(8)—C(9)	1.390 (7)
S(1)—N(5)	1.625 (3)	C(9)—C(10)	1.379 (8)
N(5)—C(6)	1.463 (5)	C(10)—C(11)	1.388 (8)
C(6)—C(7)	1.506 (5)	C(11)—C(12)	1.379 (7)
O(3)—S(1)—O(2)	117.9 (2)	C(8)—C(7)—C(6)	123.5 (4)
C(4)—S(1)—O(2)	109.5 (2)	C(12)—C(7)—C(6)	117.7 (4)
C(4)—S(1)—O(3)	107.5 (2)	C(12)—C(7)—C(8)	118.9 (4)
N(5)—S(1)—O(2)	106.2 (2)	C(9)—C(8)—C(7)	120.1 (5)
N(5)—S(1)—O(3)	107.8 (2)	C(10)—C(9)—C(8)	120.8 (5)
N(5)—S(1)—C(4)	107.5 (2)	C(11)—C(10)—C(9)	118.9 (5)
C(6)—N(5)—S(1)	117.2 (3)	C(12)—C(11)—C(10)	120.4 (5)
C(7)—C(6)—N(5)	112.4 (3)	C(11)—C(12)—C(7)	120.9 (5)

We are grateful to Mr D. Kinna for a literature survey, and to Dr J. M. Peach for advice.

References

- CALCAGNI, A., GAVUZZO, E., LUCENTE, G., MAZZA, F., PINNEN, F., POCHETTI, G. & ROSSI, D. (1989). *Int. J. Pept. Protein Res.* **34**, 471–479.
- CALCAGNI, A., GAVUZZO, E., LUCENTE, G., MAZZA, F., POCHETTI, G. & ROSSI, D. (1989). *Int. J. Pept. Protein Res.* **34**, 319–324.

- FLACK, H. D. & SCHWARZENBACH, D. (1988). *Acta Cryst.* **A44**, 499–506.
- JOHNSTON, T. P., KUSSNER, C. L. & HOLUM, L. B. (1960). *J. Org. Chem.* **25**, 399–402.
- PRINCE, E. (1982). *Mathematical Techniques in Crystallography and Mathematical Science*. Berlin: Springer-Verlag.
- SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
- WATKIN, D. J., CARRUTHERS, J. R. & BETTERIDGE, P. W. (1985). *CRYSTALS User's Guide*. Chemical Crystallography Laboratory, Univ. of Oxford, England.
- YOSHIOKA, M., NAKAI, H. & OHNO, M. (1984). *J. Am. Chem. Soc.* **106**, 1133–1135.

Acta Cryst. (1992). **C48**, 1333–1335

Structure of 4-Amino-6,8-dimethylimidazo[1,5-*a*][1,3,5]triazine Monohydrate

BY EWA CISZAK

Institute of Chemistry, Pedagogical University, ul. Oleska 48, 45-052 Opole, Poland

AND BOZENNA GOLANKIEWICZ AND PIOTR JANUSZCZYK

Polish Academy of Sciences, ul. Noskowskiego 12/14, 61-704 Poznań, Poland

(Received 3 October 1991; accepted 11 November 1991)

Abstract. $C_7H_9N_5 \cdot H_2O$, $M_r = 181.20$, monoclinic, $P2_1/a$, $a = 8.895$ (1), $b = 13.463$ (2), $c = 7.805$ (1) Å, $\beta = 108.84$ (9)°, $V = 884.6$ (2) Å³, $Z = 4$, $D_m = 1.36$, $D_x = 1.36$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 0.725$ mm⁻¹, $F(000) = 384$, $T = 293$ K, $R = 0.041$ for 1040 observed reflexions. The imidazo[1,5-*a*]-[1,3,5]triazine system is planar ($\chi^2 = 38$) within 3σ with average and maximum out-of-mean-plane deviations of 0.004 (6) and 0.009 (3) Å, respectively. Although N(5) can be formally sp^3 hybridized, it has a perfect trigonal configuration: the sum of bond angles around N(5) is 360.0 (4)°. The endocyclic N—C bonds can be divided into two classes: (i) bonds in the range 1.303 (3)–1.326 (3) Å which show strong double-bond character and (ii) bonds in the range 1.365 (3)–1.426 (3) Å which reflect some double-bond character, but to a much lesser extent than the first class. All protons available from the *exo*-amino group and from the water molecule, as well as all acceptors in the ring system and the water O atom, take part in intermolecular hydrogen bonding. The crystal packing is thus governed by hydrogen bonds which form a three-dimensional network.

Experimental. Colourless crystals obtained from chloroform–methanol 95/5. D_m by flotation. Approximate unit-cell dimensions and space group from Weissenberg photographs. Crystal 0.25×0.45

$\times 0.30$ mm. Syntex $P2_1$ diffractometer, graphite-monochromated Cu $K\alpha$ radiation, $\theta/2\theta$ scan mode; precise lattice parameters from 15 reflexions with $6 < \theta < 15^\circ$. No absorption correction. 1181 unique reflexions measured in the range $h -9 \rightarrow 9$, $k 0 \rightarrow 14$, $l 0 \rightarrow 8$, maximum $\sin\theta/\lambda = 0.546$ Å⁻¹ at $T = 293$ K. No significant intensity variation ($\pm 2.5\%$) for two standard reflexions ($\bar{1}\bar{2}\bar{2}$, $\bar{1}12$) checked every 100 reflexions. Peak-profile analysis according to Lehmann & Larsen (1974). Lorentz and polarization correction. 1040 observed reflexions with $I \geq 1.96\sigma(I)$. Structure solved by direct methods using *SHELXS86* (Sheldrick, 1985). All H atoms from $\Delta\rho$ map. Full-matrix least-squares refinement on F , anisotropic thermal parameters for non-H atoms and isotropic for H atoms. F_c values multiplied by $(1 - xF_c^2/\sin^2\theta)$ where x is the empirical isotropic extinction parameter refined to $4.04(3) \times 10^{-6}$. 163 parameters in final cycle. $R = 0.041$, $wR = 0.067$, $w = 1/[\sigma^2(F_o) + 0.00015F_o^2]$, $S = 4.30$, $(\Delta/\sigma)_{\max} = 0.002$, $(\Delta\rho)_{\min} = -0.18$, $(\Delta\rho)_{\max} = 0.15$ e Å⁻³. Programs used: *SHELXS86* (Sheldrick, 1985), *SHELX76* (Sheldrick, 1976) and other local programs (Jaskólski, 1982). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on an IBM PC computer. The final atomic coordinates are given in Table 1, bond lengths, bond angles and