Table 4. *Selected torsion angles* (°)

C(2)–C(1)–C(7)–C(8)	74·1 (8)
C(1)–C(7)–C(8)–C(9)	−1·5 (9)
C(7)–C(8)–C(9)–C(10)	−141·9 (6)
C(7)–C(8)–C(13)–C(12)	142·3 (6)
C(8)–C(9)–C(10)–C(11)	−6·4 (9)
C(9)–C(10)–C(11)–C(12)	−16·7 (8)
C(10)–C(11)–C(12)–C(13)	17·3 (8)
C(11)–C(12)–C(13)–C(8)	5·2 (9)

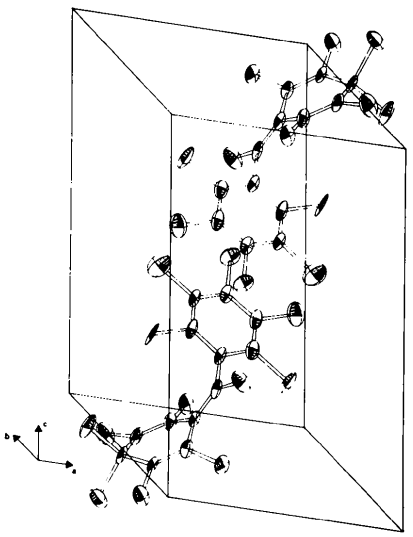

Fig. 4. A perspective view of the unit cell.

providing the crystals, and to Enraf–Nonius, who kindly supplied us with a CAD-4 automatic diffractometer.

References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1966). NRC Crystallographic Programs for the IBM/360 System. National Research Council, Ottawa, Canada.

BALLESTER, M., REIRA, J., CASTAÑER, J., BADIA, C. & MONSO, J. M. (1971). *J. Am. Chem. Soc.* **93** (3), 2215–2225.

BALLESTER, M., RIERA, J. & DE LA FUENTE, C. (1970). Unpublished.

BROWN, G. M. & STRYDOM, O. A. W. (1974). *Acta Cryst.* B**30**, 801–804.

GALÍ, S. (1975). *Crystal and Molecular Structures of Perchlorinated Organic Compounds.* Thesis. Univ. of Barcelona, Spain.

GALÍ, S., MIRAVITLLES, C. & FONT-ALTABA, M. (1975). *Acta Cryst.* B**31**, 2510–2512.

HOWELLS, E. R., PHILLIPS, D. C. & ROGERS, D. (1950). *Acta Cryst.* **3**, 210–214.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

JOHNSON, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.

PEDERSEN, B. F. (1975). *Acta Cryst.* B**31**, 2931–2933.

Acta Cryst. (1978). B**34**, 1741–1744

Choline *O*-Sulphate*

BY MICHAEL L. POST†

Division of Biological Sciences, National Research Council of Canada, Ottawa K1A 0R6, Canada

(*Received* 18 *November* 1977; *accepted* 23 *December* 1977)

Abstract. $C_5H_{13}NO_4S$, M_r = 183·22, monoclinic, $P2_1/c$, a = 8·391 (2), b = 8·674 (2), c = 11·317 (2) Å, β = 97·29 (1)°, D_m = 1·50, D_c = 1·49 g cm^{-3}, Z = 4, R = 0·034 on 1240 observed data. The molecule exists as a zwitterion with a *gauche* conformation in the choline chain; electrostatic stability is achieved through intra- and intermolecular interactions.

* Issued as NRCC No. 16585.

† Present address: Division of Chemistry, NRCC.

Introduction. $(CH_3)_3NCH_2CH_2OSO_3$ was prepared by the method of Stevens & Vohra (1955), and good air-stable crystals were grown by vapour-diffusion methods in an ethanol/water system. Preliminary photographic work showed that the cell dimensions and space group were in agreement with those previously reported (Okaya, 1966); the presence of pseudo-symmetry was indicated with the majority of the *l*-odd reflections weak in intensity.

A crystal (0·48 × 0·36 × 0·10 mm) was mounted for cell refinement and data collection with **b** parallel to

the φ axis of a Picker card-automated diffractometer equipped with a scintillation detector and pulse-height discriminator. Intensity data were recorded throughout the range $2\theta \leq 130°$ with Ni-filtered Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54178$ Å) employing the θ–2θ scanning mode operating at $2°$ min^{-1} in 2θ, with scan widths from 1·8 to 2·4° in 2θ, dependent upon 2θ. Background counts of 10 s duration were recorded at the higher scan limit. Of 1387 independent reflections, 147 had I_{net} < predetermined threshold criteria (0·1 × background or 80 counts), and these were excluded during refinement of the structure. Scaling, with respect to three check reflections, Lorentz, polarization and absorption corrections [μ(Cu $K\alpha$) = 32·80 cm^{-1}] were applied and F_o's derived. At a later stage a correction was made for secondary extinction.

The position of the $-O-SO_3$ group was determined from a sharpened Patterson map and the remaining heavy atoms were chosen in a difference Fourier synthesis. Ten of the eleven non-H atoms were related by a pseudo mirror plane coincident with the *c* glide. The whole molecule then required a shift of $\frac{1}{4}$ along *z* to bring it to the correct position, the originally incorrect placement being a result of ambiguity due to pseudosymmetry. Refinement was accomplished by block-diagonal least squares. The strong pseudosymmetry present in the original model slowly relaxed during the early cycles, such that the *l*-odd reflexions began to receive a realistic contribution. From that point, the refinement proceeded smoothly, with non-H atoms thermally anisotropic, and H atoms, all of which were found in a difference Fourier synthesis, being allotted isotropic thermal motion. Convergence was attained at an *R* of 0·034 and an R_w* of 0·046 for 1240 reflections (R = 0·039 for all data) and no shift was >0·13σ in the last cycle. A final difference map showed no physically interpretable features, the largest peaks being <0·2 e Å^{-3}. The function minimized throughout was $\sum w(|F_o| - |F_c|)^2$ with weights, *w*, in the form: $w = 1{\cdot}0$ if $F_o <$ 12·7, $w^{1/2} = 12{\cdot}7/F_o$ if $F_o \geq 12{\cdot}7$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974) and Stewart, Davidson & Simpson (1965), and all computations were carried out with the NRC program set (Ahmed, Hall, Pippy & Huber, 1973). Final atomic parameters are presented in Tables 1 and 2.*

* $R_w = [\sum w(|F_o| - |F_c|)^2/\sum wF_o^2]^{1/2}$.

Table 1. *Final positional coordinates* (×10^4) *for the non-hydrogen atoms, with estimated standard deviations in parentheses*

	x	*y*	*z*
S(1)	2871 (1)	2414 (1)	762 (1)
O(1)	4356 (2)	1943 (2)	1752 (1)
O(2)	1569 (2)	2740 (3)	1419 (2)
O(3)	2664 (2)	1038 (2)	67 (1)
O(4)	3404 (3)	3710 (2)	159 (2)
N(1)	7837 (2)	2689 (2)	2753 (1)
C(1)	4795 (2)	3091 (2)	2653 (2)
C(2)	6354 (2)	2680 (2)	3387 (2)
C(3)	9278 (3)	2737 (4)	3686 (2)
C(4)	7877 (3)	4062 (3)	1960 (2)
C(5)	7936 (2)	1275 (2)	2015 (2)

Table 2. *Final hydrogen-atom positional coordinates* (×10^3) *and isotropic thermal parameters* [(Å^2 ×10^3), *with estimated standard deviations in parentheses*

	x	*y*	*z*	U_{iso}
H(11)	484 (3)	401 (3)	225 (2)	49 (7)
H(12)	406 (3)	322 (3)	315 (2)	48 (7)
H(21)	655 (3)	343 (2)	399 (2)	36 (6)
H(22)	627 (2)	170 (2)	370 (2)	30 (5)
H(31)	1024 (3)	264 (3)	324 (2)	53 (7)
H(32)	915 (3)	182 (3)	411 (2)	46 (6)
H(33)	921 (3)	359 (3)	410 (3)	72 (9)
H(41)	889 (3)	408 (3)	168 (2)	49 (7)
H(42)	689 (4)	397 (4)	134 (3)	76 (9)
H(43)	783 (3)	497 (3)	249 (2)	56 (7)
H(51)	895 (3)	131 (3)	167 (2)	46 (7)
H(52)	705 (3)	130 (3)	150 (2)	49 (7)
H(53)	800 (3)	34 (3)	257 (2)	46 (6)

Discussion. The conformation of the choline chain in various classes of small molecules has been studied in the solid state (Sundaralingam, 1968; Baker, Chothia, Pauling & Petcher, 1971; Viswamitra, Seshadri, Post & Kennard, 1975; Jagner & Jensen, 1977, and references therein), in solution (Dufourcq & Lussan, 1972; Aslanian, Balkanski & Lautie, 1977; Koyama, Toda & Kyogoku, 1977), and by theoretical calculations (Pullman, 1976). Attention has also been directed toward macromolecules containing the choline moiety,

* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles for the hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33316 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

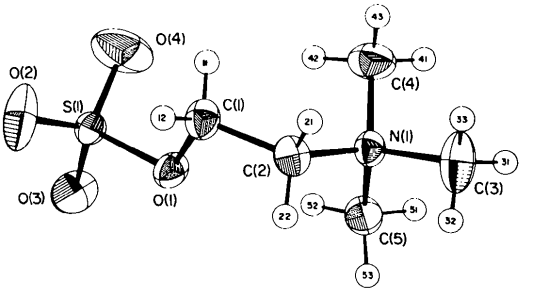

Fig. 1. A perspective view of choline *O*-sulphate. Non-hydrogen atoms are represented by 50% probability thermal ellipsoids and hydrogen atoms by spheres of arbitrary radius.

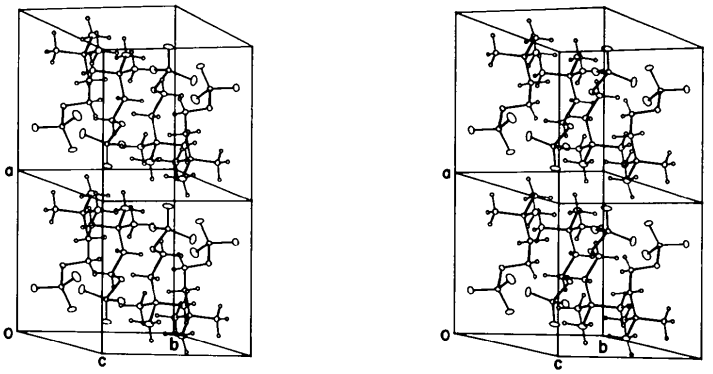

Fig. 2. Stereoscopic view of the contents of two unit cells.

e.g. the biomembrane constituents such as phospholipids (Sundaralingam, 1972; Dufourcq & Lussan, 1972; Gupta & Govil, 1972; McAlister, Yathindra & Sundaralingam, 1973), which contain phosphorylcholine as a head-group. Generally, the diverse physical approaches lead to the identification of certain favoured conformers, and particularly the indication of a *gauche* arrangement of the O—C—C—N bonds, as is found in the present structure.

The constitution of choline *O*-sulphate ensures that no hydrogen bonding is possible and that the molecule possesses net zero charge – in contrast to the great majority of structures previously reported. For crystalline choline *O*-sulphate, weaker forces of association in the lattice will predominate, with a concomitant low perturbation of molecular conformation. It is likely that the structural features reported here resemble closely those which exist for the molecule in some solvents.

The structure (Fig. 1) is zwitterionic with formal chain representation $>\overset{\oplus}{N}-C-C-O\overset{\ominus}{=}S<$, but with charge almost certainly distributed over atoms bonded at N and S. The choline moiety exhibits a *gauche* conformation, with a torsion angle N(1)—C(2)—C(1)—O(1) of 66·1° [other torsion angles: C(3)—N(1)—C(2)—C(1) = 162·9°; C(2)—C(1)—O(1)—S(1) = −169·6°; C(1)—O(1)—S(1)—O(2) = −57·3°], and intramolecular distances N(1)···O(1) and C(5)···O(1) of 3·065 (2) and 3·037 (2) Å, respectively, each atom pair imparting partial electrostatic stabilization. Bond lengths and angles for the 'heavy' atoms (Table 3) are as expected. The distorted tetrahedral geometry around S(1), with all O(1)—S(1)—O(*n*) angles less than sp^3 values, has been previously observed for *O*-sulphate esters (Fries & Sundaralingam, 1971). The average C—H distance is 0·95 Å with an average associated angle of 109°.

Lattice packing (Fig. 2) is partly achieved through a number of head-to-tail approaches of the zwitterion, the closer of these being across centres of symmetry with a cell translation in the **a** [O(3)···C(5), 3·087 (2) Å] and **b** [O(4)···C(4), 3·160 (2) Å] directions. Additionally, there are some longer approaches between O(2) and **a**-translated $-\overset{+}{N}(CH_3)_3$ groups [O(2)···N(1), C(4), C(5); at 3·645 (2), 3·430 (2) and 3·447 (2) Å respectively]. All these interactions are of an electrostatic character such that, overall, the charge on the molecule is accommodated both inter- and intramolecularly. Other contacts in the lattice, none of which are unusually short, are of the van der Waals type.

Table 3. *Bond lengths* (Å) *and angles* (°) *for the non-hydrogen atoms*

Estimated standard deviations are in parentheses.

S(1)—O(1)	1·619 (1)	C(1)—C(2)	1·501 (3)
S(1)—O(2)	1·425 (2)	N(1)—C(2)	1·513 (3)
S(1)—O(3)	1·427 (2)	N(1)—C(3)	1·501 (3)
S(1)—O(4)	1·417 (2)	N(1)—C(4)	1·494 (3)
O(1)—C(1)	1·439 (3)	N(1)—C(5)	1·493 (3)
O(1)—S(1)—O(2)	105·2 (1)	C(1)—C(2)—N(1)	116·6 (2)
O(1)—S(1)—O(3)	101·5 (1)	C(2)—N(1)—C(3)	107·7 (2)
O(1)—S(1)—O(4)	105·7 (1)	C(2)—N(1)—C(4)	111·8 (2)
O(2)—S(1)—O(3)	113·9 (1)	C(2)—N(1)—C(5)	111·6 (2)
O(2)—S(1)—O(4)	113·8 (1)	C(3)—N(1)—C(4)	108·8 (2)
O(3)—S(1)—O(4)	114·9 (1)	C(3)—N(1)—C(5)	108·6 (2)
S(1)—O(1)—C(1)	114·9 (1)	C(4)—N(1)—C(5)	108·1 (2)
O(1)—C(1)—C(2)	110·8 (2)		

I thank Dr Martin Young, of this laboratory, for supplying the material and the National Research Council of Canada for the award of a Research Associateship.

References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1973). NRC Crystallographic Programs for the IBM/360 System. Accession numbers 133–147 in *J. Appl. Cryst.* **6**, 309–346.

Aslanian, D., Balkanski, M. & Lautie, A. (1977). *J. Am. Chem. Soc.* **99**, 1974–1976.

Baker, R. W., Chothia, C. H., Pauling, P. & Petcher, T. J. (1971). *Nature (London)*, **230**, 439–445.

Dufourcq, J. & Lussan, C. (1972). *FEBS Lett.* **26**, 35–38.

FRIES, D. C. & SUNDARALINGAM, M. (1971). *Acta Cryst.* B27, 401–410.
GUPTA, S. P. & GOVIL, G. (1972). *FEBS Lett.* **27**, 68–70.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
JAGNER, S. & JENSEN, B. (1977). *Acta Cryst.* B**33**, 2757–2762.
KOYAMA, Y., TODA, S. & KYOGOKU, Y. (1977). *Chem. Phys. Lipids*, **19**, 74–92.
MCALISTER, J., YATHINDRA, N. & SUNDARALINGAM, M. (1973). *Biochemistry*, **12**, 1189–1195.
OKAYA, Y. (1966). *Acta Cryst.* **21**, A124–A125.
PULLMAN, B. (1976). *Proceedings of the Eighth Jerusalem Symposium on Quantum Chemistry and Biochemistry*, edited by B. PULLMAN, pp. 55–80. Holland: Reidel.
STEVENS, C. M. & VOHRA, P. (1955). *J. Am. Chem. Soc.* **77**, 4935–4936.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
SUNDARALINGAM, M. (1968). *Nature (London)*, **217**, 35–37.
SUNDARALINGAM, M. (1972). *Ann. NY Acad. Sci.* **195**, 324–355.
VISWAMITRA, M. A., SESHADRI, T. P., POST, M. L. & KENNARD, O. (1975). *Nature (London)*, **258**, 497–501.

Acta Cryst. (1978). B**34**, 1744–1746

2-Hydroxy-3-phenyl-3-pyrrolidinylpropionamide

BY A. KIRFEL

Lehrstuhl für Mineralogie und Kristallographie am Mineralogischen Institut der Universität Bonn, Poppelsdorfer Schloss, D 5300 Bonn, Federal Republic of Germany

(*Received* 16 *December* 1977; *accepted* 14 *January* 1978)

Abstract. $C_{13}H_{18}N_2O_2$, monoclinic, $P2_1/c$, a = 7·844 (3), b = 16·619 (7), c = 20·802 (12) Å, β = 103·2 (3)°, V = 2640 (2) Å^3, Z = 8, D_x = 1·179 g cm^{-3}. The compound is an *erythro* phenylisoserine derivative. One molecule of the asymmetric unit forms an N···O hydrogen-bonded dimer with its inversion image; the second is connected to two different dimers by O···O hydrogen bonds. The result is a network of molecules within a layer parallel to (100) of thickness d_{100} = 7·636 Å. The layers are held together by packing forces only.

Introduction. The title compound (Fig. 1) was synthesized by Dr W. Tack and Professor Zymalkowski (Pharmazeutisches Institut der Universität Bonn). A crystal, 0·2 × 0·1 × 0·1 mm, was selected for the diffraction experiments. Intensity measurements were carried out in the ω mode on an automatic Syntex $P2_1$ four-circle diffractometer with Mo $K\alpha$ radiation (λ = 0·71069 Å) monochromatized by a graphite crystal. 3406 reflexions were recorded, resulting in a set of 2921 unique reflexions, of which 887 were regarded as unobserved ($I < 3\sigma$).

No absorption correction was applied (μ = 0·90 cm^{-1}). The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971), which fixed the positions of 33 non-hydrogen atoms in the asymmetric unit. The completion of the structure solution was achieved by Fourier methods and calculation of H atom positions. Refinement was by full-matrix least squares with weights $w = 1/\sigma^2(F)$ and anisotropic temperature factors. The H atoms were allocated the isotropic temperature factors of their carrier atoms, but their positions were refined. Owing to the large number of parameters (416) refinement had to take place in overlapping cycles. An isotropic extinction factor g {Zachariasen, 1963; $F_c = kF_o[1 + \beta(2\theta)gI_c]$} was included in the list of variables (final value 7 × 10^{-7}). With six reflexions excluded from refinement the final R

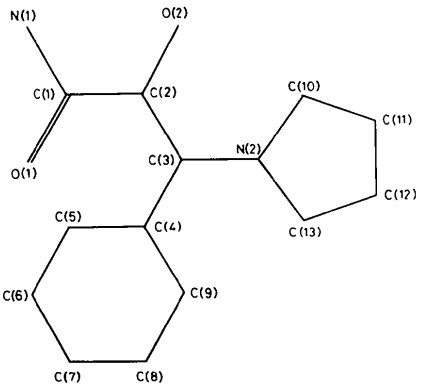

Fig. 1. The numbering scheme.