

different in all other compounds (Table 3). The inequality of the lengths of S—C(sp²) and S—C(sp³) is easily understood and is due to a difference in hybridization of the carbon atoms. The approximate equality of these two bonds in the sulfones is possibly correlated with the electron-withdrawing effect of the oxygen atoms.

The molecular packing in the crystal is due to van der Waals forces. The shortest intermolecular distance is 2.31 Å between H(22)b and H'(22)b (−x, −y, −z).

References

- ANDREETTI, G. D., BOCELLI, G. & SGARABOTTO, P. (1978). *Cryst. Struct. Commun.* **7**, 543–546.
- BLESSING, R. H. (1987). *Cryst. Rev.* **1**, 3–58.
- DAWSON, M. I., HOBBS, P. D., CHAN, R. L., CHAO, W. & FUNG, V. A. (1981). *J. Med. Chem.* **24**, 583–592.
- DAWSON, M. I., HOBBS, P. D., DERDZINSHKI, K., CHAN, R. L.-S., GRUBER, J., CHAO, W.-R., SMITH, S., THIES, R. W. & SCHIFF, L. J. (1984). *J. Med. Chem.* **27**, 1516–1531.
- DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, edited by W. L. DUAX & D. A. NORTON, Vol. 1, pp. 16–22. New York, Washington, London: IFI/Plenum.
- EALICK, S. E., VAN DER HELM, D., BAKER, J. R. & BERLIN, K. D. (1979). *Acta Cryst.* **B35**, 495–497.
- HARDY, A. D. U., MCKENDRICK, J. J. & MCNICOL, D. D. (1977). *J. Chem. Soc. Perkin Trans.* **2**, pp. 1145–1147.
- HOSSAIN, M. B., VAN DER HELM, D. & BERLIN, K. D. (1987). *Acta Cryst.* **C43**, 1764–1766.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- KHARCHENKO, V. G., KRIVEN'KO, A. P., FEDOTOVA, O. V., EVTUSHENKO, I. YA., SHCHERBAKOV, A. A., ALEKSANDROV, G. G. & STRUCHKOV, YU. T. (1980). *Khim. Geterotsikl. Soedin. Latv. SSSR*, pp. 1337–1342.
- MCNICOL, D. D., MILLS, H. H. & WILSON, F. B. (1969). *Chem. Commun.* pp. 1332–1333.
- METRA, R. G., SCHIFF, L. J., MOORE, S. J., BUCKLEY, A. M. & DAWSON, M. I. (1986). *In Vitro Cell. Dev. Biol.* **22**, 164–168.
- PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.
- RUBAN, G., ZOBEL, D., KOSSMANN, G. & NUCK, R. (1980). *Chem. Ber.* **113**, 3384–3388.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination, Univ. of Cambridge, England.
- SPRUCE, L. W., BERLIN, K. D., VERMA, A. K., BREITMAN, T. R., JI, X. & VAN DER HELM, D. (1989). *J. Med. Chem.* Submitted.
- WAUGH, K. M., BERLIN, K. D., FORD, W. T., HOLT, E. M., CARROL, J. P., SCHOMBER, P. R., THOMPSON, M. D. & SCHIFF, L. J. (1985). *J. Med. Chem.* **28**, 116–124.

Acta Cryst. (1989). **C45**, 754–757

Structures of Monoethanolamine (MEAM), Diethanolamine (DEAM) and Triethanolamine (TEAM)

BY DIETRICH MOOTZ, DIETER BRODALLA AND MICHAEL WIEBCKE

Institut für Anorganische Chemie und Strukturchemie, Universität Düsseldorf, Universitätsstraße 1, D-4000 Düsseldorf, Federal Republic of Germany

(Received 26 May 1988; accepted 7 November 1988)

Abstract. MEAM: 2-aminoethanol, C₂H₇NO, *M_r* = 61.08, monoclinic, *Cc*, *a* = 5.026 (3), *b* = 8.818 (6), *c* = 8.319 (7) Å, β = 107.44 (6)°, *V* = 351.74 Å³, *Z* = 4, *D_x* = 1.15 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 0.085 mm⁻¹, *F*(000) = 136, *T* = 263 K, *R* = 0.044 for 494 unique observed reflections. DEAM: 2,2'-imino-diethanol, C₄H₁₁NO₂, *M_r* = 105.14, monoclinic, *P*₂₁/*c*, *a* = 4.464 (1), *b* = 13.052 (2), *c* = 9.812 (2) Å, β = 93.73 (2)°, *V* = 570.47 Å³, *Z* = 4, *D_x* = 1.22 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 0.090 mm⁻¹, *F*(000) = 232, *T* = 192 K, *R* = 0.030 for 921 unique observed reflections. TEAM: 2,2',2''-nitrilotriethanol, C₆H₁₅NO₃, *M_r* = 149.19, rhombohedral, *R* $\bar{3}$, *a* = 11.491 (1), *c* = 10.070 (1) Å {the *c*-axis length was incorrectly given in a preliminary report of the TEAM structure [Brodalla & Mootz (1981). *Angew. Chem.* **93**, 824; *Angew. Chem. Int. Ed. Engl.* **20**, 791–792]}, *V* = 1151.53 Å³, *Z* = 6, *D_x* = 1.29 Mg m⁻³, λ(Mo *K*α)

= 0.71069 Å, μ = 0.095 mm⁻¹, *F*(000) = 492, *T* = 140 K, *R* = 0.033 for 721 unique observed reflections. The crystal structures are governed by the geometry of the molecules and extensive intermolecular hydrogen bonding. The MEAM molecules are arranged in a three-dimensional network with strong N—H...O as well as weak N—H...O and O—H...N bonds. Rings of two O—H...O-bridged DEAM molecules are linked *via* strong O—H...N bonds into one-dimensional tube-like stacks; weak N—H...O bonds across the rings are also present. The TEAM molecules are linked to form discrete cage-like dimers with point symmetry $\bar{3}$ *via* rings of six strong O—H...O bonds with chair conformation.

Introduction. In the course of our investigations of hydrogen-bonded solids we have determined the crystal structures of the ethanolamines MEAM, DEAM and

TEAM by low-temperature single-crystal X-ray diffractometry. The investigations and their results are described in the following.

Experimental. Crystal growth for the structure determinations of MEAM, DEAM and TEAM was performed on a Syntex $P2_1$ diffractometer equipped with a modified Syntex LT-1 low-temperature device. Samples of commercially available products sealed in thin-walled glass capillaries with approximate diameters of 0.3 mm were cooled to appropriate temperatures in the adjustable gas stream of the diffractometer. A miniature zone-melting technique using focused heat radiation was applied (Brodalla, Mootz, Boese & Osswald, 1985).

The X-ray measurements were performed after further lowering the temperature of the single crystals obtained. Graphite monochromator. Lattice parameters from setting angles of 15 strong reflections. Intensities by variable ω -scan technique. Three standard reflections every 50 reflections showed only small random variations. No correction for absorption. Criterion for observed reflections $I > 1.96\sigma(I)$. MEAM: lattice parameters from reflections with $25 < 2\theta < 46^\circ$. 1663 reflections measured up to $(\sin\theta)/\lambda = 0.704 \text{ \AA}^{-1}$ in the hemisphere $0 \leq h \leq 7$, $-12 \leq k \leq 12$, $-11 \leq l \leq 11$. Systematically extinct reflections measured only in some segments. 531 unique reflections after merging equivalent ones, $R_{\text{int}} = 0.059$. 14 unobserved reflections. Seven reflections with too large intensities and 16 reflections with distinctly asymmetric background excluded from structure refinement. DEAM: lattice parameters from reflections with $26 < 2\theta < 36^\circ$. 2315 reflections measured up to $(\sin\theta)/\lambda = 0.595 \text{ \AA}^{-1}$ in the hemisphere $0 \leq h \leq 5$, $-15 \leq k \leq 15$, $-11 \leq l \leq 11$. 1015 unique reflections after merging equivalent ones, $R_{\text{int}} = 0.016$. 94 unobserved reflections. TEAM: lattice parameters from reflections with $26 < 2\theta < 38^\circ$. 2420 reflections measured up to $(\sin\theta)/\lambda = 0.704 \text{ \AA}^{-1}$ in the hemisphere $0 \leq h \leq 10$, $-10 \leq k \leq 10$, $-10 \leq l \leq 10$, using a rhombohedral setting with $a = 7.435 \text{ \AA}$ and $\alpha = 101.21^\circ$. Transformation to hexagonal axes. 762 unique reflections after merging equivalent ones, $R_{\text{int}} = 0.015$. 35 unobserved reflections. Six reflections with too large intensities excluded from structure refinement.

Direct methods. Least-squares refinement, function minimized $\sum w(\Delta F)^2$, observed reflections only, weighted according to $w = 1/[\sigma^2(F) + 0.0001|F_o|^2]$. H atoms located by difference-Fourier method. Complex atomic scattering factors from *International Tables for X-ray Crystallography* (1974). In final refinement variation of scale factor, coordinates of all atoms, and anisotropic (non-H atoms) and isotropic (H atoms) thermal parameters. MEAM: 63 parameters varied, $R = 0.044$, $wR = 0.053$, $S = 2.601$, all Δ/σ in last cycle 0.00. $\Delta\rho_{\text{min}}$ and $\Delta\rho_{\text{max}}$ in final difference-

Table 1. Atomic coordinates and (equivalent) isotropic thermal parameters (\AA^2) of MEAM, DEAM and TEAM with e.s.d.'s in parentheses

	x	y	z	U^\dagger
MEAM				
N	0.3418	0.3879 (2)	0.3516	0.0399 (6)
C1	0.3349 (5)	0.2457 (3)	0.4376 (3)	0.0376 (6)
C2	0.1661 (5)	0.2508 (3)	0.5613 (3)	0.0387 (7)
O	0.1764 (3)	0.1067 (2)	0.6387 (2)	0.0425 (5)
H11	0.221 (5)	0.164 (3)	0.335 (3)	0.035 (6)
H12	0.537 (5)	0.242 (3)	0.513 (3)	0.031 (5)
H21	0.007 (8)	0.309 (5)	0.497 (6)	0.073 (10)
H22	0.200 (8)	0.326 (5)	0.621 (5)	0.064 (9)
H3	0.480 (8)	0.375 (5)	0.320 (5)	0.075 (9)
H4	0.426 (4)	0.455 (3)	0.434 (3)	0.033 (5)
H5	0.354 (8)	0.081 (6)	0.738 (5)	0.075 (10)
DEAM				
N	0.3040 (2)	0.4025 (1)	0.3456 (1)	0.0225 (3)
C1	0.1192 (3)	0.3095 (1)	0.3511 (1)	0.0249 (3)
C2	-0.0066 (3)	0.2975 (1)	0.4891 (1)	0.0263 (3)
C3	0.3793 (2)	0.4278 (1)	0.2062 (1)	0.0273 (3)
C4	0.5450 (3)	0.5284 (1)	0.2006 (1)	0.0306 (4)
O1	-0.1934 (2)	0.3820 (1)	0.5216 (1)	0.0266 (3)
O2	0.3541 (2)	0.6143 (1)	0.2127 (1)	0.0357 (3)
H11	-0.048 (3)	0.310 (1)	0.279 (1)	0.035 (3)
H12	0.248 (3)	0.252 (1)	0.332 (1)	0.032 (3)
H21	-0.121 (3)	0.233 (1)	0.491 (1)	0.032 (3)
H22	0.153 (3)	0.294 (1)	0.559 (1)	0.027 (3)
H31	0.504 (3)	0.374 (1)	0.173 (1)	0.033 (3)
H32	0.198 (3)	0.432 (1)	0.144 (1)	0.034 (3)
H41	0.632 (3)	0.534 (1)	0.113 (2)	0.038 (3)
H42	0.709 (3)	0.532 (1)	0.274 (1)	0.032 (3)
H5	0.201 (3)	0.453 (1)	0.379 (1)	0.027 (3)
H6	-0.341 (4)	0.385 (1)	0.465 (2)	0.056 (4)
H7	0.311 (3)	0.618 (1)	0.293 (2)	0.048 (4)
TEAM				
N	0	0	0.27460 (10)	0.0127 (2)
C1	0.11118 (7)	0.12941 (7)	0.22915 (7)	0.0152 (3)
C2	0.24020 (7)	0.17671 (7)	0.30760 (7)	0.0164 (3)
O	0.22483 (5)	0.20042 (6)	0.44419 (5)	0.0195 (2)
H11	0.084 (1)	0.201 (1)	0.239 (1)	0.017 (2)
H12	0.130 (1)	0.127 (1)	0.134 (1)	0.021 (3)
H21	0.271 (1)	0.111 (1)	0.301 (1)	0.021 (2)
H22	0.309 (1)	0.261 (1)	0.270 (1)	0.022 (3)
H3	0.214 (2)	0.134 (1)	0.485 (1)	0.041 (4)

\dagger For the non-H atoms calculated as $U_{\text{eq}} = \frac{1}{3}(U_{11}a^*{}^2a^2 + U_{22}b^*{}^2b^2 + \dots)$.

Fourier synthesis -0.16 and $+0.21 e \text{ \AA}^{-3}$, respectively. DEAM: 108 parameters varied, $R = 0.030$, $wR = 0.040$, $S = 2.960$, all Δ/σ in last cycle 0.00. $\Delta\rho_{\text{min}}$ and $\Delta\rho_{\text{max}}$ in final difference-Fourier synthesis -0.19 and $+0.14 e \text{ \AA}^{-3}$, respectively. TEAM: 51 parameters varied, $R = 0.033$, $wR = 0.048$, $S = 3.389$, all Δ/σ in last cycle 0.00. $\Delta\rho_{\text{min}}$ and $\Delta\rho_{\text{max}}$ in final difference-Fourier synthesis -0.14 and $+0.44 e \text{ \AA}^{-3}$, respectively. Final atomic parameters of MEAM, DEAM and TEAM are listed in Table 1.*

The calculations were performed with the program systems *XTL/XTLE* (Syntex, 1976) and *SHELXTL* (Sheldrick, 1983). The latter was used for the final calculations and generation of the drawings.

* Lists of anisotropic thermal parameters, less important interatomic distances and angles involving the H atoms, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51580 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. The crystal structures of MEAM, DEAM and TEAM are governed by the geometry of the molecules (one independent molecule in each structure, in TEAM with crystallographic threefold rotational symmetry) and extensive intermolecular hydrogen bonding. The molecules with the labeling of the atoms are illustrated in Fig. 1; in the cases of DEAM and TEAM characteristic dimeric units are shown. The crystal structures are illustrated in Fig. 2. Selected intramolecular and intermolecular distances and angles are listed in Table 2. The values are not corrected for thermal motion and the systematic error of the X-ray method to produce N—H and O—H bond lengths which are shorter than the internuclear distances. In the hydrogen bonds, therefore, most of the distances between the H and the acceptor atom appear as too large, but could nevertheless be used, along with the angles at the H atom, for the respective assignment.

In crystalline MEAM the molecules (point symmetry 1) are linked *via* strong (short) N—H...O

hydrogen bonds into angular chains parallel to the longer diagonal of the *ac* plane (direction $[10\bar{1}]$). Significantly weaker (longer) N—H...O and O—H...N hydrogen bonds exist between these chains. In the three-dimensional network formed the molecules are hydrogen-bonded to six neighbouring ones. The DEAM molecules (point symmetry 1) are linked in pairs *via* strong O—H...O hydrogen bonds to form centrosymmetric rings, which in turn are further linked *via* strong O—H...N bridges to form one-dimensional tubular stacks parallel to the *a* axis. The main planes of the dimers are inclined against this axis. The imino groups point with the H atoms into the interior of the tubes and form weak hydrogen bonds to O atoms of the inverse molecule in the respective rings. In the structure of TEAM the molecules (point symmetry 3) form discrete cage-like dimers (point symmetry $\bar{3}$), in which the 2-hydroxyethyl groups enclose the lone pair at the N atoms and the O atoms are linked *via* strong hydrogen bonds to form six-membered rings with chair

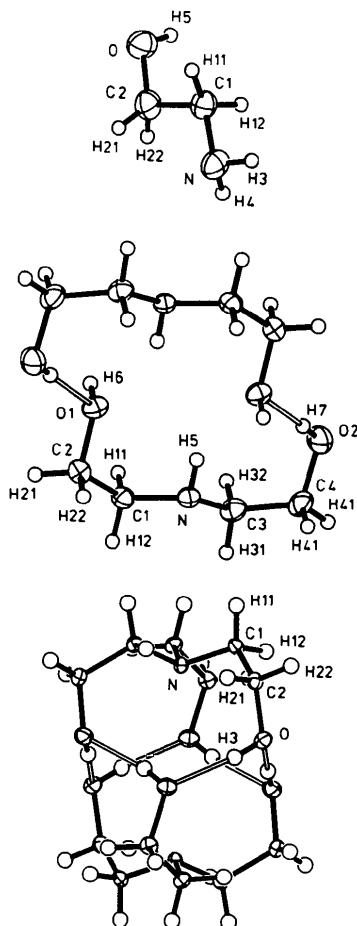


Fig. 1. MEAM molecule (top), ring of two hydrogen-bonded DEAM molecules (center) and cage-like dimer of hydrogen-bonded TEAM molecules (bottom). All ellipsoids correspond to the 50% probability level. H atoms with arbitrary size.

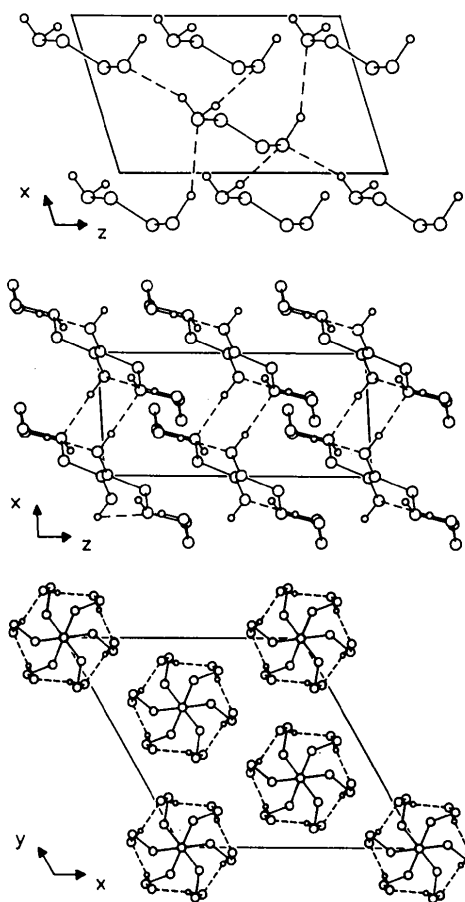


Fig. 2. Projections of the structures of MEAM (top), DEAM (center) and TEAM (bottom). All atoms with arbitrary size. H atoms which are bonded to C atoms are neglected for clarity. Hydrogen bonds with dashed lines.

Table 2. Selected interatomic distances (Å) and angles (°) of MEAM, DEAM and TEAM with *e.s.d.'s* in parentheses

Dotted distances denote hydrogen bonds.

MEAM			
N—C1	1.449 (3)	C1—C2	1.518 (4)
C2—O	1.418 (3)	N...O ⁱ	2.785 (3)
N...O ⁱⁱ	3.138 (3)	O...N ⁱⁱⁱ	3.282 (3)
N—H3	0.82 (5)	N—H4	0.91 (2)
O—H5	1.04 (4)	H3...O ⁱ	2.05 (5)
H4...O ⁱⁱ	2.23 (2)	H5...N ⁱⁱⁱ	2.36 (4)
N—C1—C2	114.2 (2)	C1—C2—O	109.2 (2)
N—H3...O ⁱ	149 (3)	N—H4...O ⁱ	175 (3)
O—H5...N ⁱⁱⁱ	147 (3)		
Symmetry code: (i) $\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$; (ii) $\frac{1}{2} + x, \frac{1}{2} + y, z$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$.			
DEAM			
N—C1	1.471 (1)	N—C3	1.467 (1)
C1—C2	1.507 (2)	C2—O1	1.431 (1)
C3—C4	1.509 (2)	C4—O2	1.418 (1)
N...O1 ⁱ	3.152 (1)	O1...N ⁱⁱ	2.754 (1)
O2...O1 ⁱ	2.749 (1)	N—H5	0.88 (1)
O1—H6	0.83 (2)	O2—H7	0.83 (2)
H5...O1 ⁱ	2.36 (1)	H6...N ⁱⁱ	1.92 (2)
H7...O1 ⁱ	1.92 (2)		
C1—N1—C3	112.5 (1)	N—C1—C2	111.2 (1)
C1—C2—O1	112.1 (1)	N—C3—C4	111.9 (1)
C3—C4—O2	112.7 (1)	N—H5...O1 ⁱ	150 (1)
O1—H6...N ⁱⁱ	180 (2)	O2—H7...O1 ⁱ	177 (1)
Symmetry code: (i) $-x, 1 - y, 1 - z$; (ii) $-1 + x, y, z$.			
TEAM			
N—C1	1.467 (1)	C1—C2	1.520 (1)
C2—O	1.430 (1)	O...O ⁱ	2.700 (1)
O—H3	0.82 (2)	H3...O ⁱ	1.88 (2)
C1—N—C1 ⁱⁱ	110.7 (1)	N—C1—C2	113.5 (1)
C1—C2—O	112.1 (1)	O—H3...O ⁱ	180 (2)
Symmetry code: (i) $x - y, x, 1 - z$; (ii) $y - z, -x, z$.			

conformation. The arrangement of the dimers follows the principle of cubic close packing of equal spheres.

In the crystalline ethanalamines all H atoms of the donor groups (*i.e.* the hydroxy, amino and imino groups) are involved in the hydrogen-bonding systems. Each molecule donates and accepts a total of six hydrogen bonds with the mean strength increasing in the sequence MEAM (two strong and four weak bonds), DEAM (four strong and two weak bonds) and TEAM (six strong bonds). The length of the C—O bonds is influenced by the number of strong hydrogen bonds (one or two) in which the respective O atom participates. The volume enclosed by the TEAM dimer is too small for the enclathration of guest atoms or molecules.

The work was supported by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

References

- BRODALLA, D. & MOOTZ, D. (1981). *Angew. Chem.* **93**, 824; *Angew. Chem. Int. Ed. Engl.* **20**, 791–792.
- BRODALLA, D., MOOTZ, D., BOESE, R. & OSSWALD, W. (1985). *J. Appl. Cryst.* **18**, 316–319.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- SHELDRIK, G. M. (1983). *SHELXTL User's Manual*, revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.
- Syntax (1976). *XTL/XTLE Structure Determination System*. Syntax Analytical Instruments, Cupertino, California, USA.

Acta Cryst. (1989). **C45**, 757–760

Structure of 1-Amidino-3-(3-sulfamoylphenyl)urea Hydrochloride

BY PAUL A. SUTTON AND VIVIAN CODY*

The Medical Foundation of Buffalo, 73 High Street, Buffalo, NY 14203, USA

(Received 29 April 1988; accepted 3 November 1988)

Abstract. 1-(Diaminomethylene)-3-(3-sulfamoylphenyl)uronium chloride, $C_8H_{12}N_5O_3S^+ \cdot Cl^-$, $M_r = 293.71$, monoclinic, $P2_1/c$, $a = 10.148$ (1), $b = 7.881$ (1), $c = 15.286$ (3) Å, $\beta = 94.77$ (1)°, $V = 1218.2$ Å³, $Z = 4$, $D_x = 1.601$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 4.838$ cm⁻¹, $F(000) = 608$, $T = 294$ K, $R = 0.069$ for 1779 data. The molecular conformation of the protonated arylamidinourea is completely planar and all amidinourea N atoms have considerable sp^2

hybridization. The stable tautomeric form has an intramolecular hydrogen bond between the ureido O atom and an amidino N atom. All N atoms in the molecular packing make contact with the Cl⁻ ion.

Introduction. The title compound (I) is a hydrolysis product of 4,6-diamino-1,2-dihydro-2,2-dimethyl-1-(3-sulfamoylphenyl)-s-triazine, which is a dihydrofolate reductase inhibitor with antineoplastic activity (Blaney, Hansch, Silipo & Vittoria, 1984). The hydrolysis product is structurally similar to a class of compounds

* To whom all correspondence should be addressed.