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^{ii}	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 ethanolamines 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.)

SHELDRICK, G. M. (1983). *SHELXTL User's Manual*, revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.

Syntex (1976). *XTL/XTLE Structure Determination System.* Syntex Analytical Instruments, Cupertino, California, USA.

Acta Cryst. (1989). C**45**, 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, $C_8H_{12}N_5O_3S^+.Cl^-$, $M_r = 293{\cdot}71$, monoclinic, $P2_1/c$, $a = 10{\cdot}148$ (1), $b = 7{\cdot}881$ (1), $c = 15{\cdot}286$ (3) Å, $\beta = 94{\cdot}77$ (1)°, $V = 1218{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}601$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 4{\cdot}838$ cm^{-1}, $F(000) = 608$, $T = 294$ K, $R = 0{\cdot}069$ 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.

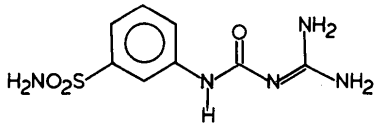
0108-2701/89/050757-04$03.00

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters for* 1-*amidino*-3-(3-*sulfamoylphenyl*)*urea*.HCl

	x	*y*	*z*	B_{eq}*
C(2)	1·1074 (4)	0·8167 (5)	0·9326 (2)	2·79 (10)
C(4)	1·2752 (4)	0·6338 (5)	1·0090 (2)	2·78 (10)
C(7)	0·9872 (3)	1·0622 (5)	0·8624 (2)	2·67 (10)
C(8)	0·8728 (3)	0·9788 (5)	0·8295 (2)	2·99 (10)
C(9)	0·7732 (3)	1·0735 (5)	0·7854 (2)	2·70 (10)
C(10)	0·7823 (4)	1·2465 (5)	0·7750 (2)	3·13 (11)
C(11)	0·8967 (4)	1·3268 (5)	0·8074 (2)	3·32 (11)
C(12)	0·9983 (4)	1·2358 (5)	0·8494 (2)	3·15 (11)
N(1)	1·0962 (3)	0·9797 (4)	0·9071 (2)	3·27 (9)
N(3)	1·2317 (3)	0·7853 (4)	0·9766 (2)	2·84 (8)
N(5)	1·1993 (3)	0·5004 (4)	1·0057 (2)	4·03 (10)
N(6)	1·3966 (3)	0·6284 (4)	1·0467 (2)	3·46 (9)
N(9C)	0·5148 (3)	0·9912 (4)	0·8028 (2)	3·71 (9)
O(2)	1·0235 (2)	0·7081 (4)	0·9210 (2)	3·98 (8)
O(9*A*)	0·6601 (3)	0·7920 (4)	0·7369 (2)	5·15 (9)
O(9*B*)	0·5874 (3)	1·0564 (4)	0·6599 (2)	4·84 (9)
S(9)	0·6296 (1)	0·9687 (1)	0·7395 (1)	3·54 (3)
Cl	1·3580 (1)	1·1496 (1)	1·0041 (1)	3·62 (3)
H(1)	1·155 (4)	1·038 (6)	0·919 (3)	
H(3)	1·286 (4)	0·873 (5)	0·986 (2)	
H(5*A*)	1·235 (4)	0·394 (5)	1·021 (3)	
H(5*B*)	1·122 (4)	0·515 (6)	0·983 (3)	
H(6*A*)	1·421 (4)	0·550 (6)	1·067 (3)	
H(6*B*)	1·458 (4)	0·712 (5)	1·034 (3)	
H(8)	0·862 (4)	0·867 (5)	0·835 (2)	
H(N9*A*)	0·474 (5)	1·094 (6)	0·800 (3)	
H(N9*B*)	0·504 (4)	0·951 (5)	0·838 (2)	
H(10)	0·712 (4)	1·306 (5)	0·747 (2)	
H(11)	0·900 (4)	1·441 (5)	0·803 (2)	
H(12)	1·076 (4)	1·291 (5)	0·870 (2)	

* $B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}(a_i a_j)$.

which not only have antineoplastic and antimalarial activity (Walls, Goodford, Norrington & Richards, 1973; Skowronska-Serafin & Urbanski, 1960), but also have gastrointestinal properties which include antisecretory and antimotility activity (Douglas *et al.*, 1978). The biological activity of this series of compounds has been discussed with respect to its tautomeric configuration. The crystal structure of 1-amidino-3-(3-sulfamoylphenyl)urea (I) provides information regarding the geometry and conformational characteristics of amidinoureas.



(I)

Experimental. Crystals of 1-amidino-3-(3-sulfamoylphenyl)urea.HCl were grown from an ethanol/HCl solution. The crystal used for data collection had dimensions 0·08 × 0·08 × 0·28 mm. Nicolet *P*3 diffractometer, Mo $K\alpha$ radiation, Nb filter. Unit-cell dimensions and orientation matrix were determined from 25 reflections with 2θ values in the range 19·39 to 27·72°. 3004 total data with θ between 4 and 50° ($0 < h < 13$, $-1 < k < 10$, $-19 < l < 19$). Intensities of 5 standard reflections (142, 42$\bar{5}$, 008, 61$\bar{1}$, 413) were monitored after every 91 measurements and showed no decline through data collection. Diffraction data were corrected for Lorentz and polarization effects, but not for extinction or absorption effects. Of the 2166 unique data, 1779 had $I > 2\sigma(I)$ $\{\sigma^2(F) = (k/4\mathrm{Lp}I)[\sigma^2(I) + (0{\cdot}01I)^2]$ (Stout & Jensen, 1968)$\}$ and were considered observed. The structure was solved using the direct-methods programs *MULTAN* (Germain, Main & Woolfson, 1971) and *NQEST* (De Titta, Edmonds, Langs & Hauptman, 1975). The structure was refined using full-matrix least-squares methods, minimizing $\sum[w(F_o - F_c)^2]$, where $w = 1/\sigma^2(F_o)$. Non-H atoms were refined with anisotropic thermal parameters. The H atoms were located on a difference map and their positional parameters were refined after full refinement of the heavy atoms. H-atom thermal parameters were assigned isotropic values one unit greater than the heavy atoms to which they were bound; their thermal parameters were held constant during refinement. The maximum value of the shift/e.s.d. during the last cycle of refinement was 0·00. The final R values are $R = 0{\cdot}069$ and $wR = 0{\cdot}043$ for 1779 observed data and $R = 0{\cdot}085$ for all 2166 data; $S = 1{\cdot}973$. The final difference map has maximum and minimum densities of 0·518 and −0·318 e Å^{-3}. Atomic scattering factors and dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a VAX 8600 computer using a locally modified version of the Enraf–Nonius *SDP* crystallographic package and plots were prepared with *ORTEP*II (Johnson, 1976).

Discussion. Final positional and isotropic thermal parameters for 1-amidino-3-(3-sulfamoylphenyl)urea.HCl are listed in Table 1.* The molecular structure of the title compound is planar (Fig. 1) where the r.m.s. deviation from the best plane (all heavy atoms with the exception of the sulfamoyl O and N atoms) is 0·020 Å. The sulfamoyl O and N atoms deviate from this plane by more than 0·05 Å.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51603 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

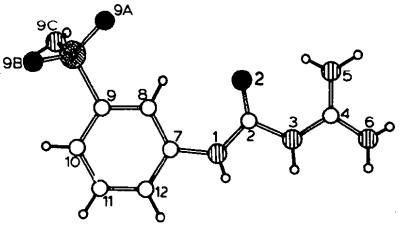


Fig. 1. Atomic numbering and molecular conformation of 1-amidino-3-(3-sulfamoylphenyl)urea.HCl. The solid atoms are O, the stippled atom is S and the hashed atoms are N.

The amidinourea chain can be drawn in many tautomeric configurations, some of which form intramolecular hydrogen bonds. The molecular geometry of this compound (Fig. 2) shows that it adopts tautomer (I) with protonation at N(3) and forms an intramolecular hydrogen bond between O(2) and N(5). The planarity of the molecule leads to the conclusion that all the C and N atoms have sp^2 hybridization and indicates the presence of a delocalized π system over the entire molecule. The bond lengths involving C(4) indicate that the amidino moiety participates in the delocalization of the double bond.

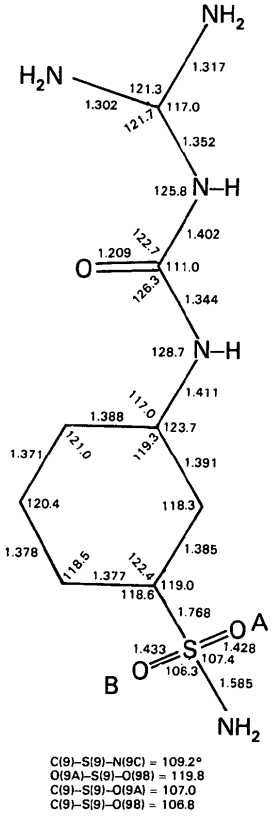


Fig. 2. Bond distances and angles for 1-amidino-3-(3-sulfamoylphenyl)urea.HCl. The average e.s.d.'s for bond lengths and angles are ±0·003 Å and ±0·2°, respectively.

Table 2. *Hydrogen-bond geometry* (Å,°) *for* 1-*amidino*-3-(3-*sulfamoylphenyl*)*urea*.HCl

D–H···*A*	*D*···*A*	*D*–H	H···*A*	*D*–H ···*A*	Symmetry
N(1)–H(1)···Cl	3·224 (3)	0·76 (4)	2·50 (4)	158 (3)	1/000
N(3)–H(3)···Cl	3·158 (3)	0·89 (4)	2·30 (4)	160 (3)	1/000
N(5)–H(5*A*)···O(2)	3·080 (4)	0·94 (4)	2·95 (4)	89 (2)	2/212
N(5)–H(5*A*)···Cl	3·200 (3)	0·94 (4)	2·32 (4)	156 (3)	1/010
N(5)–H(5*B*)···O(2)	3·080 (4)	0·84 (4)	2·79 (4)	102 (3)	2/212
N(5)–H(5*B*)···O(2)*	2·674 (4)	0·84 (4)	2·01 (4)	135 (3)	1/000
N(6)–H(6*A*)···N(6)	3·322 (4)	0·73 (4)	2·99 (5)	111 (3)	2/322
N(6)–H(6*A*)···O(9*B*)	2·883 (4)	0·73 (4)	2·28 (4)	142 (3)	3/110
N(6)–H(6*B*)···Cl	3·192 (3)	0·94 (4)	2·28 (4)	164 (3)	2/322
N(9C)–H(9C*A*)···O(9*A*)	2·993 (4)	0·91 (5)	2·12 (5)	162 (3)	4/101
N(9C)–H(9C*B*)···Cl	3·313 (3)	0·65 (4)	2·80 (4)	139 (3)	2/222

* Intramolecular hydrogen bond.

The molecular conformation of this protonated arylamidinourea is similar to that previously reported for amidinoureas (Begley, Hubberstey & More, 1985; Zaman & Darlow, 1986) which reveal that protonation of the central N atom leads to a decrease in the π character of the bridging C–N bonds and a corresponding increase in that of the terminal C–N bonds of the non-protonated parent compound. The geometry of the sulfamoyl moiety in the title compound is consistent with other reported sulfamoylphenyls (Foresti, Riva di Sanseverino & Sabatino, 1985; Karapetyan, Khajakyan, Andrianov, Lindeman & Struchkov, 1981).

The molecular packing (Fig. 3 and Table 2) shows that all of the N atoms make intermolecular contacts to the Cl^- ion and that the protonated N(3) atom has the shortest contact.

We thank Corwin Hansch for supplying the parent compound 4,6-diamino-1,2-dihydro-2,2-dimethyl-1-(3-sulfamoylphenyl)-*s*-triazine. This work was supported in part by grants from NCI-CA-34714 and from the American Cancer Society Faculty Research Award FRA-287 (VC).

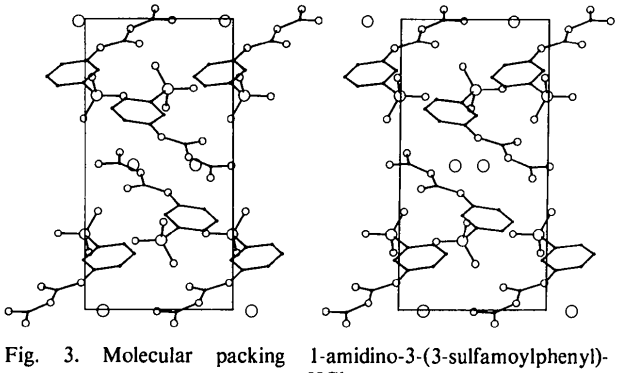


Fig. 3. Molecular packing 1-amidino-3-(3-sulfamoylphenyl)-urea.HCl.

References

Begley, M. J., Hubberstey, P. & More, C. H. M. (1985). *J. Chem. Res.* (*M*), pp. 4035–4068.

Blaney, J. M., Hansch, C., Silipo, C. & Vittoria, A. (1984). *Chem. Rev.* **84**, 333–404.

De Titta, G. T., Edmonds, J. W., Langs, D. A. & Hauptman, H. A. (1975). *Acta Cryst.* A**31**, 472–479.

Douglas, G. H., Diamond, J., Studt, W. L., Mir, G. N., Alioto, R. L., Auyang, K., Burns, B. J., Cias, J., Darkes, P. R., Dodson, S. A., O'Connor, S., Santora, N. J., Tsuei, C. T., Zalipsky, J. J. & Zimmerman, H. K. (1978). *Arzneim.-Forsch.* **28**(II), 1435–1441.

Foresti, E., Riva di Sanseverino, L. & Sabatino, P. (1985). *Acta Cryst.* C**41**, 240–243.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–147. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KARAPETYAN, H. A., KHAJAKYAN, L. V., ANDRIANOV, V. G., LINDEMAN, S. V. & STRUCHKOV, Y. T. (1981). *Cryst. Struct. Commun.* **10**, 445–450.

SKOWRONSKA-SERAFIN, B. & URBANSKI, T. (1960). *Tetrahedron*, **10**, 12–25.

STOUT, G. H. & JENSEN, L. H. (1968). *X-ray Structure Determination*. New York: Macmillan.

WALLS, L. P., GOODFORD, P. S., NORRINGTON, F. E. & RICHARDS, W. H. G. (1973). *Br. J. Pharmacol.* **48**, 650–654.

ZAMAN, N. & DARLOW, S. F. (1986). *J. Bangladesh Acad. Sci.* **10**, 79.

Acta Cryst. (1989). C**45**, 760–763

Die Struktur zweier 3,7-Dioxa-2,6-diazabicyclo[3.3.0]octane

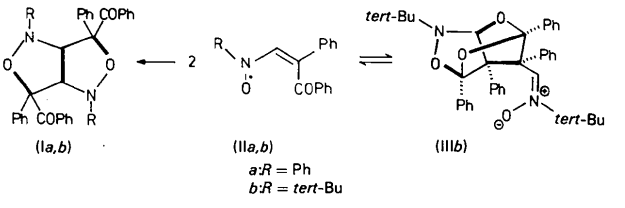
VON HANS GÜNTER AURICH,* GERHARD BAUM, WERNER MASSA* UND KLAUS-DIETER MOGENDORF

Fachbereich Chemie der Universität Marburg, Hans-Meerwein-Straße, D-3550 Marburg, Bundesrepublik Deutschland

(*Eingegangen am* 27. *Juni* 1988; *angenommen am* 28. *September* 1988)

Abstract. 4,8-Dibenzoyl-2,4,6,8-tetraphenyl-3,7-dioxa-2,6-diazabicyclo[3.3.0]octane, $C_{42}H_{32}N_2O_4$ (I*a*), M_r = 628·73, triclinic, $P\bar{1}$, a = 14·232 (4), b = 11·991 (2), c = 10·293 (3) Å, α = 107·38 (3), β = 88·75 (7), γ = 98·37 (3)°, V = 1658·1 (7) Å^3, Z = 2, D_x = 1·259 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·76 cm^{-1}, F(000) = 660, T = 295 (1) K, R = 0·045 for 3262 observed reflections. [Reduced cell for (I*a*): a = 10·293 (3), b = 11·991 (2), c = 14·232 (4) Å, α = 81·63 (3), β = 88·75 (7), γ = 72·66 (3)°.] 4,8-Dibenzoyl-2,6-di-*tert*-butyl-4,8-diphenyl-3,7-dioxa-2,6-diazabicyclo[3.3.0]octane, $C_{38}H_{40}N_2O_4$ (I*b*), M_r = 588·75, monoclinic, *Cc*, a = 9·524 (2), b = 18·681 (5), c = 17·994 (5) Å, β = 91·02 (5)°, V = 3201 (1) Å^3, Z = 4, D_x = 1·222 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·74 cm^{-1}, F(000) = 1256, T = 295 (1) K, R = 0·050 for 1977 observed reflections. The bicyclic compounds (I*a*) and (I*b*) are formed by two tetrahydroisoxazole units. In (I*a*) the O atom protrudes out of the plane which is approximately formed by the other four ring atoms, whereas in (I*b*) it is the N atom which projects from the plane. The dihedral angles between the two planes are 121·5 and 113·8°, respectively.

Einleitung. Im Gegensatz zum *N-tert*-Butylvinylaminyloxid (*N-tert*-Butylvinylnitroxid) (II*b*), das bei Raumtemperatur in das tricyclische Dimere (III*b*) übergeht (Aurich, Baum, Massa, Mogendorf & Schmidt, 1984), dimerisiert das *N*-phenylsubstituierte Radikal (II*a*) unter Bildung des 4,8-Dibenzoyl-2,4,-6,8-tetraphenyl-3,7-dioxa-2,6-diazabicyclo[3.3.0]-octans (I*a*) (Aurich, Mogendorf & Schmidt, 1984). Aus (III*b*), das in Lösung im Gleichgewicht mit (II*b*) vorliegt, entsteht beim Refluxieren in Benzol neben dem 5,10-Di-*tert*-butyl-3,8,11,12-tetraphenyl-2,4,7,9-tetraoxa-5,10-diazatetracyclo[4.4.2.0^{3,12}.0^{8,11}]dodecan das 4,8-Dibenzoyl-2,6-di-*tert*-butyl-4,8-diphenyl-3,7-dioxa-2,6-diazabicyclo[3.3.0]octan (I*b*) (Aurich, Bubenheim, Kessler & Mogendorf, 1988).



(I*a*,*b*) (II*a*,*b*) (III*b*)

a:R = Ph
b:R = *tert*-Bu

Wir haben von den beiden bicyclischen Dimeren (I) eine Röntgenstrukturanalyse durchgeführt, um Aufschluß über den räumlichen Aufbau dieses neuen heterocyclischen Systems und den Einfluß des Substituenten am Stickstoff auf die Struktur zu erhalten.

Experimentelles. Von einer Lösung von 200 mg (I*a*) bzw. (I*b*) in 10 ml Methylenchlorid ließ man bei Raumtemperatur das Lösungsmittel langsam verdunsten. Nach zwei bis drei Wochen schieden sich farblose Kristalle ab. Kristallformate: (I*a*) 0,75 × 0,25 × 0,15 mm; (I*b*) 0,85 × 0,60 × 0,48 mm. Die Kristalle wurden auf einem Enraf–Nonius CAD-4 Diffraktometer mit Mo $K\alpha$-Strahlung (Graphitmonochromator) vermessen, die genauen Gitterkonstanten durch Verfeinerung auf der Basis der durch Messung im positiven und negativen Winkelbereich bestimmten Beugungswinkel von 25 Reflexen (θ = 9–18°) (I*a*) bzw. 22 Reflexen (θ = 9–20°) (I*b*) ermittelt. Auf eine

* Korrespondenzautoren.

0108-2701/89/050760-04$03.00