References

Boehlow, T. R. & Spilling, C. D. (1995). *Nat. Prod. Lett.* **7**, 1–6

Brown, R. F. C., Jackson, W. R., McCarthy, T. D. & Fallon, G. D. (1992). *Aust. J. Chem.* **45**, 1833–1843.

Plantier-Royon, R., Anker, D., Robert-Baudouy, J. (1991). *J. Carbohydr. Chem.* **10**, 239–249.

Sheldrick, G. M. (1994). *SHELXTL.* Version 5. Siemens Analytical Instruments Inc., Madison, Wisconsin, USA.

Siemens (1994). *SMART. Data Collection Software.* Siemens Analytical Instruments Inc., Madison, Wisconsin, USA.

Siemens (1994). *SAINT. Data Reducion Software.* Siemens Analytical Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1997). C**53**, 95–97

5-(4-Fluorophenyl)-1,8-dimethyl-2-(*p*-toluoylaminomethyl)-2,3-dihydro-1*H*-1,4-benzodiazepine Monohydrate

OSWALD M. PEETERS, NORBERT M. BLATON AND CAMIEL J. DE RANTER

Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Faculteit Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium. E-mail: maurice.peeters@farm.kuleuven.ac.be

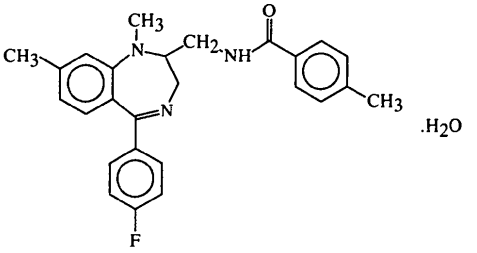
(*Received 1 July 1996; accepted 20 September 1996*)

Abstract

The title compound, *N*-{[5-(4-fluorophenyl)-1,8-dimethyl-2,3-dihydro-1*H*-1,4-benzodiazepin-2-yl]methyl}-*p*-toluamide monohydrate, $C_{26}H_{26}FN_3O.H_2O$, a benzodiazepine derivative with κ-opioid activity, crystallizes as a hydrate with two almost identical molecules in the asymmetric unit. The observed conformation, stabilized by two hydrogen bonds involving the H_2O molecule, is common for the 2-(acylaminomethyl)benzodiazepines. Hydrogen bonds between H_2O molecules and amidic O atoms link the non-equivalent molecules, with formation of endless chains in the **a** direction.

Comment

The title compound, (I), belongs to a series of 2-(acylaminomethyl)benzodiazepine derivatives with κ-opioid activity. Apart from the structure of tifluadom hydrate (Codding, Zeugner & Finner, 1987), the structures of this series were obtained as either chloride or toluenesulfonate salts (Petcher, Widmer, Maetzel & Zeugner, 1985; Blaton, Peeters & De Ranter, 1996, and references therein). As both the protonation and the ionic crystalline environment may influence the conformation of the molecule, the structure determination of a free base seems worthwhile. Unfortunately, the title compound crystallizes as a hydrate and the H_2O molecule mimics the anion site of the salts, as can be seen from the conformation given in Fig. 1.



(I)

The two molecules in the asymmetric unit of (I) are almost identical, as can be deduced from the geometric parameters of the molecules (Table 2) and the puckering parameters of the diazepine rings [q_2 = 0.798 (5) and 0.797 (5); q_3 = 0.226 (6) and 0.233 (6); Q_T = 0.829 (6) and 0.830 (6) Å; φ_2 = −26.0 (4) and 154.9 (3); φ_3 = −128 (1) and 51 (1); θ_2 = 74.2 (4) and 73.8 (4)°, for molecules *A* and *B*, respectively, considering the atomic sequence N1—C2—C3—N4—C5—C5*A*—C9*A*]. The geometric and puckering parameters are close to those of tifluadom hydrate. The main difference between the hydrates and the salts is the value of the endocyclic angle of the N4 atom. Protonation causes the angle to open by *ca* 8°. The diazepine ring exhibits a boat conformation flattened at the stern with a pseudosymmetry plane through the C3 atom [asymmetry parameter ΔC_s(C3) = 0.004 (2) and 0.008 (2) for *A* and *B*, respectively]. Each H_2O molecule is hydrogen bonded with the N4 and N12 atoms of the same

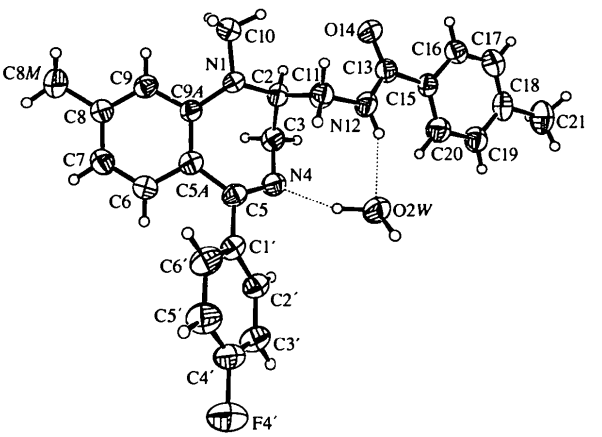


Fig. 1. Perspective view of the title compound (molecule *A*) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

molecule, stabilizing the observed conformation of the acylaminomethyl side chain. A third hydrogen bond between the H_2O molecule and the amidic O atom of a non-equivalent molecule forms endless chains in the **a** direction.

Experimental

Crystals of the title compound were obtained by slow evaporation at room temperature from a methanol/H_2O solution.

Crystal data

$C_{26}H_{26}FN_3O.H_2O$
M_r = 433.51
Monoclinic
$P2_1/n$
a = 13.6293 (6) Å
b = 10.364 (1) Å
c = 33.859 (2) Å
β = 100.236 (3)°
V = 4706.5 (5) Å^3
Z = 8
D_x = 1.224 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 38 reflections
θ = 10–24°
μ = 0.680 mm^{-1}
T = 293 K
Plate
0.60 × 0.35 × 0.05 mm
Pale yellow

Data collection

Siemens $P4$ four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (*XEMP*; Siemens, 1989)
T_{min} = 0.86, T_{max} = 0.97
8769 measured reflections
6386 independent reflections
2130 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.0381
θ_{max} = 57.19°
$h = -1 \rightarrow 14$
$k = -1 \rightarrow 11$
$l = -36 \rightarrow 36$
3 standard reflections monitored every 100 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R(F)$ = 0.0532
$wR(F^2)$ = 0.1799
S = 0.853
6384 reflections
584 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2)]$
$(\Delta/\sigma)_{max}$ = 0.109
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.00087 (9)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
N1*A*	0.8237 (3)	0.1517 (5)	0.3396 (1)	0.057 (2)
C2*A*	0.8589 (4)	0.0719 (5)	0.3090 (2)	0.056 (2)
C3*A*	0.9139 (4)	−0.0472 (6)	0.3261 (2)	0.061 (2)
N4*A*	1.0132 (3)	−0.0207 (5)	0.3487 (1)	0.059 (2)
C5*A*	1.0160 (4)	0.0308 (5)	0.3837 (2)	0.053 (2)
C5*AA*	0.9283 (4)	0.0653 (5)	0.4014 (2)	0.053 (2)
C6*A*	0.9363 (4)	0.0472 (6)	0.4432 (2)	0.061 (2)
C7*A*	0.8614 (4)	0.0773 (6)	0.4633 (2)	0.065 (2)
C8*A*	0.7753 (4)	0.1342 (5)	0.4438 (2)	0.054 (2)
C8*MA*	0.6931 (5)	0.1693 (7)	0.4654 (2)	0.079 (3)
C9*A*	0.7646 (4)	0.1535 (5)	0.4029 (2)	0.052 (2)
C9*AA*	0.8394 (4)	0.1203 (5)	0.3806 (1)	0.048 (2)
C10*A*	0.7361 (4)	0.2291 (6)	0.3233 (2)	0.076 (3)
C11*A*	0.9204 (4)	0.1572 (6)	0.2858 (2)	0.061 (2)
N12*A*	0.9535 (3)	0.0902 (5)	0.2528 (1)	0.064 (2)
C13*A*	0.8925 (4)	0.0692 (6)	0.2175 (2)	0.055 (2)
O14*A*	0.8054 (3)	0.1058 (4)	0.2115 (1)	0.071 (2)
C15*A*	0.9370 (4)	−0.0016 (5)	0.1867 (1)	0.050 (2)
C16*A*	0.8905 (4)	0.0097 (6)	0.1472 (2)	0.061 (2)
C17*A*	0.9299 (5)	−0.0491 (6)	0.1168 (2)	0.069 (2)
C18*A*	1.0161 (5)	−0.1220 (6)	0.1255 (2)	0.065 (2)
C19*A*	1.0614 (4)	−0.1352 (6)	0.1650 (2)	0.066 (2)
C20*A*	1.0228 (4)	−0.0756 (6)	0.1954 (2)	0.065 (2)
C21*A*	1.0607 (5)	−0.1852 (6)	0.0922 (2)	0.090 (3)
C1′*A*	1.1184 (4)	0.0501 (5)	0.4078 (2)	0.054 (2)
C2′*A*	1.1911 (4)	−0.0420 (6)	0.4058 (2)	0.059 (2)
C3′*A*	1.2860 (4)	−0.0284 (6)	0.4282 (2)	0.065 (2)
C4′*A*	1.3064 (4)	0.0769 (6)	0.4521 (2)	0.063 (2)
F4′*A*	1.3994 (2)	0.0893 (4)	0.4741 (1)	0.089 (1)
C5′*A*	1.2381 (4)	0.1712 (6)	0.4543 (2)	0.070 (2)
C6′*A*	1.1425 (4)	0.1568 (6)	0.4324 (2)	0.066 (2)
N1*B*	1.2520 (3)	0.6483 (4)	0.3380 (1)	0.058 (2)
C2*B*	1.1903 (4)	0.5692 (5)	0.3071 (1)	0.053 (2)
C3*B*	1.1482 (4)	0.4494 (6)	0.3240 (2)	0.059 (2)
N4*B*	1.0687 (3)	0.4779 (4)	0.3462 (1)	0.058 (2)
C5*B*	1.0969 (4)	0.5274 (5)	0.3813 (2)	0.052 (2)
C5*AB*	1.2003 (4)	0.5611 (5)	0.3996 (1)	0.047 (2)
C6*B*	1.2281 (4)	0.5382 (5)	0.4408 (1)	0.056 (2)
C7*B*	1.3207 (4)	0.5701 (5)	0.4618 (2)	0.059 (2)
C8*B*	1.3888 (4)	0.6303 (5)	0.4428 (2)	0.055 (2)
C8*MB*	1.4893 (4)	0.6693 (7)	0.4648 (2)	0.079 (3)
C9*B*	1.3645 (4)	0.6508 (5)	0.4016 (1)	0.051 (2)
C9*AB*	1.2720 (3)	0.6174 (5)	0.3789 (1)	0.046 (2)
C10*B*	1.3249 (4)	0.7268 (6)	0.3219 (2)	0.074 (2)
C11*B*	1.1088 (4)	0.6545 (6)	0.2839 (2)	0.060 (2)
N12*B*	1.0473 (3)	0.5881 (4)	0.2504 (1)	0.060 (2)
C13*B*	1.0788 (4)	0.5640 (5)	0.2159 (2)	0.055 (2)
O14*B*	1.1623 (3)	0.5992 (4)	0.2104 (1)	0.069 (2)
C15*B*	1.0072 (4)	0.4933 (5)	0.1843 (1)	0.050 (2)
C16*B*	1.0219 (4)	0.5063 (6)	0.1451 (2)	0.060 (2)
C17*B*	0.9569 (5)	0.4478 (6)	0.1141 (2)	0.068 (2)
C18*B*	0.8772 (5)	0.3785 (6)	0.1214 (2)	0.068 (3)
C19*B*	0.8637 (4)	0.3641 (6)	0.1606 (2)	0.067 (2)
C20*B*	0.9283 (4)	0.4224 (6)	0.1922 (2)	0.061 (2)
C21*B*	0.8032 (5)	0.3155 (6)	0.0881 (2)	0.090 (3)
C1′*B*	1.0172 (4)	0.5478 (5)	0.4057 (2)	0.053 (2)
C2′*B*	0.9423 (4)	0.4589 (6)	0.4044 (2)	0.065 (2)
C3′*B*	0.8686 (4)	0.4740 (7)	0.4279 (2)	0.072 (2)
C4′*B*	0.8735 (5)	0.5808 (7)	0.4521 (2)	0.071 (3)
F4′*B*	0.8002 (3)	0.5933 (4)	0.4744 (1)	0.098 (2)
C5′*B*	0.9438 (5)	0.6710 (7)	0.4535 (2)	0.077 (3)
C6′*B*	1.0172 (4)	0.6551 (6)	0.4304 (2)	0.071 (2)
O1*W*	0.8842 (3)	0.5053 (5)	0.2876 (1)	0.092 (2)
O2*W*	1.1473 (3)	0.0041 (5)	0.2904 (1)	0.095 (2)

Table 2. *Geometric parameters (Å, °)*

N1*A*—C2*A*	1.470 (7)	N1*B*—C2*B*	1.471 (6)
N1*A*—C9*AA*	1.406 (6)	N1*B*—C9*AB*	1.399 (6)
N1*A*—C10*A*	1.462 (7)	N1*B*—C10*B*	1.464 (8)
C2*A*—C3*A*	1.506 (8)	C2*B*—C3*B*	1.522 (8)
C2*A*—C11*A*	1.530 (8)	C2*B*—C11*B*	1.523 (7)
C3*A*—N4*A*	1.458 (6)	C3*B*—N4*B*	1.456 (7)
N4*A*—C5*A*	1.294 (7)	N4*B*—C5*B*	1.287 (7)
C5*A*—C5*AA*	1.472 (8)	C5*B*—C5*AB*	1.477 (7)
C5*A*—C1′*A*	1.499 (7)	C5*B*—C1′*B*	1.492 (8)
C5*AA*—C6*A*	1.412 (7)	C5*AB*—C6*B*	1.400 (7)
C5*AA*—C9*AA*	1.408 (7)	C5*AB*—C9*AB*	1.425 (7)
C6*A*—C7*A*	1.362 (8)	C6*B*—C7*B*	1.375 (7)
C7*A*—C8*A*	1.373 (7)	C7*B*—C8*B*	1.372 (8)
C8*A*—C8*MA*	1.487 (9)	C8*B*—C8*MB*	1.494 (7)

C8*A*—C9*A*	1.382 (7)	C8*B*—C9*B*	1.391 (7)
C9*A*—C9*AA*	1.415 (8)	C9*B*—C9*AB*	1.398 (6)
C11*A*—N12*A*	1.453 (7)	C11*B*—N12*B*	1.457 (6)
N12*A*—C13*A*	1.347 (6)	N12*B*—C13*B*	1.339 (7)
C13*A*—O14*A*	1.229 (7)	C13*B*—O14*B*	1.241 (7)
C13*A*—C15*A*	1.488 (8)	C13*B*—C15*B*	1.503 (7)
C15*A*—C16*A*	1.381 (7)	C15*B*—C16*B*	1.386 (7)
C15*A*—C20*A*	1.385 (7)	C15*B*—C20*B*	1.368 (8)
C16*A*—C17*A*	1.386 (9)	C16*B*—C17*B*	1.385 (7)
C17*A*—C18*A*	1.383 (9)	C17*B*—C18*B*	1.361 (9)
C18*A*—C19*A*	1.376 (8)	C18*B*—C19*B*	1.382 (9)
C18*A*—C21*A*	1.521 (9)	C18*B*—C21*B*	1.521 (8)
C19*A*—C20*A*	1.384 (9)	C19*B*—C20*B*	1.396 (8)
C1′*A*—C2′*A*	1.386 (8)	C1′*B*—C2′*B*	1.370 (8)
C1′*A*—C6′*A*	1.388 (8)	C1′*B*—C6′*B*	1.393 (8)
C2′*A*—C3′*A*	1.385 (7)	C2′*B*—C3′*B*	1.397 (9)
C3′*A*—C4′*A*	1.356 (9)	C3′*B*—C4′*B*	1.370 (9)
C4′*A*—F4′*A*	1.358 (6)	C4′*B*—F4′*B*	1.363 (8)
C4′*A*—C5′*A*	1.362 (9)	C4′*B*—C5′*B*	1.334 (9)
C5′*A*—C6′*A*	1.388 (8)	C5′*B*—C6′*B*	1.383 (9)
C9*AA*—N1*A*—C10*A*	118.0 (4)	C9*AB*—N1*B*—C10*B*	118.1 (4)
C2*A*—N1*A*—C10*A*	113.0 (4)	C2*B*—N1*B*—C10*B*	112.6 (4)
C2*A*—N1*A*—C9*AA*	123.7 (4)	C2*B*—N1*B*—C9*AB*	124.3 (4)
N1*A*—C2*A*—C11*A*	108.3 (4)	N1*B*—C2*B*—C11*B*	108.4 (4)
N1*A*—C2*A*—C3*A*	113.1 (4)	N1*B*—C2*B*—C3*B*	113.1 (4)
C3*A*—C2*A*—C11*A*	113.0 (4)	C3*B*—C2*B*—C11*B*	112.3 (4)
C2*A*—C3*A*—N4*A*	113.6 (5)	C2*B*—C3*B*—N4*B*	113.2 (4)
C3*A*—N4*A*—C5*A*	115.5 (4)	C3*B*—N4*B*—C5*B*	115.4 (4)
N4*A*—C5*A*—C1′*A*	115.2 (4)	N4*B*—C5*B*—C1′*B*	116.0 (4)
N4*A*—C5*A*—C5*AA*	125.3 (5)	N4*B*—C5*B*—C5*AB*	126.1 (5)
C5*AA*—C5*A*—C1′*A*	119.4 (4)	C5*AB*—C5*B*—C1′*B*	117.9 (4)
C5*A*—C5*AA*—C9*AA*	125.8 (4)	C5*B*—C5*AB*—C9*AB*	125.2 (4)
C5*A*—C5*AA*—C6*A*	116.8 (4)	C5*B*—C5*AB*—C6*B*	116.6 (4)
C6*A*—C5*AA*—C9*AA*	117.4 (5)	C6*B*—C5*AB*—C9*AB*	118.2 (4)
C5*AA*—C6*A*—C7*A*	122.8 (5)	C5*AB*—C6*B*—C7*B*	122.4 (5)
C6*A*—C7*A*—C8*A*	120.6 (5)	C6*B*—C7*B*—C8*B*	120.1 (5)
C7*A*—C8*A*—C9*A*	118.3 (5)	C7*B*—C8*B*—C9*B*	118.7 (5)
C7*A*—C8*A*—C8*MA*	121.6 (5)	C7*B*—C8*B*—C8*MB*	121.6 (5)
C8*MA*—C8*A*—C9*A*	120.1 (5)	C8*MB*—C8*B*—C9*B*	119.6 (5)
C8*A*—C9*A*—C9*AA*	122.8 (5)	C8*B*—C9*B*—C9*AB*	123.0 (4)
C5*AA*—C9*AA*—C9*A*	118.1 (4)	C5*AB*—C9*AB*—C9*B*	117.4 (4)
N1*A*—C9*AA*—C9*A*	118.3 (4)	N1*B*—C9*AB*—C9*B*	119.0 (4)
N1*A*—C9*AA*—C5*AA*	123.5 (4)	N1*B*—C9*AB*—C5*AB*	123.4 (4)
C2*A*—C11*A*—N12*A*	113.1 (4)	C2*B*—C11*B*—N12*B*	113.1 (4)
C11*A*—N12*A*—C13*A*	122.2 (4)	C11*B*—N12*B*—C13*B*	122.5 (4)
N12*A*—C13*A*—C15*A*	115.9 (4)	N12*B*—C13*B*—C15*B*	116.4 (4)
N12*A*—C13*A*—O14*A*	121.5 (5)	N12*B*—C13*B*—O14*B*	121.5 (5)
O14*A*—C13*A*—C15*A*	122.6 (5)	O14*B*—C13*B*—C15*B*	122.2 (5)
C13*A*—C15*A*—C20*A*	123.9 (4)	C13*B*—C15*B*—C20*B*	123.8 (4)
C13*A*—C15*A*—C16*A*	117.5 (5)	C13*B*—C15*B*—C16*B*	116.7 (5)
C16*A*—C15*A*—C20*A*	118.6 (5)	C16*B*—C15*B*—C20*B*	119.5 (5)
C15*A*—C16*A*—C17*A*	120.7 (5)	C15*B*—C16*B*—C17*B*	120.0 (5)
C16*A*—C17*A*—C18*A*	120.6 (5)	C16*B*—C17*B*—C18*B*	121.2 (5)
C17*A*—C18*A*—C21*A*	120.9 (5)	C17*B*—C18*B*—C21*B*	122.5 (5)
C17*A*—C18*A*—C19*A*	118.6 (6)	C17*B*—C18*B*—C19*B*	118.6 (6)
C19*A*—C18*A*—C21*A*	120.5 (6)	C19*B*—C18*B*—C21*B*	118.9 (5)
C18*A*—C19*A*—C20*A*	121.0 (6)	C18*B*—C19*B*—C20*B*	121.1 (5)
C15*A*—C20*A*—C19*A*	120.5 (5)	C15*B*—C20*B*—C19*B*	119.5 (5)
C5*A*—C1′*A*—C6′*A*	122.1 (5)	C5*B*—C1′*B*—C6′*B*	121.6 (5)
C5*A*—C1′*A*—C2′*A*	119.1 (5)	C5*B*—C1′*B*—C2′*B*	119.9 (5)
C2′*A*—C1′*A*—C6′*A*	118.8 (5)	C2′*B*—C1′*B*—C6′*B*	118.4 (5)
C1′*A*—C2′*A*—C3′*A*	120.7 (5)	C1′*B*—C2′*B*—C3′*B*	120.9 (6)
C2′*A*—C3′*A*—C4′*A*	118.7 (5)	C2′*B*—C3′*B*—C4′*B*	117.8 (6)
C3′*A*—C4′*A*—C5′*A*	122.7 (6)	C3′*B*—C4′*B*—C5′*B*	123.1 (6)
C3′*A*—C4′*A*—F4′*A*	118.5 (5)	C3′*B*—C4′*B*—F4′*B*	116.5 (6)
F4′*A*—C4′*A*—C5′*A*	118.8 (5)	F4′*B*—C4′*B*—C5′*B*	120.4 (6)
C4′*A*—C5′*A*—C6′*A*	118.7 (6)	C4′*B*—C5′*B*—C6′*B*	118.9 (6)
C1′*A*—C6′*A*—C5′*A*	120.4 (5)	C1′*B*—C6′*B*—C5′*B*	120.8 (6)

C2*A*—N1*A*—C9*AA*—C5*AA*	38.6 (7)
C10*A*—N1*A*—C9*AA*—C9*A*	6.5 (7)
C9*AA*—N1*A*—C2*A*—C11*A*	−126.2 (5)
C9*AA*—N1*A*—C2*A*—C3*A*	−0.1 (7)
N1*A*—C2*A*—C11*A*—N12*A*	−176.1 (4)
N1*A*—C2*A*—C3*A*—N4*A*	−73.2 (6)
C2*A*—C3*A*—N4*A*—C5*A*	74.1 (6)
C3*A*—N4*A*—C5*A*—C5*AA*	−1.0 (8)
N4*A*—C5*A*—C1′*A*—C2′*A*	−37.1 (7)
N4*A*—C5*A*—C5*AA*—C9*AA*	−39.7 (8)
C5*A*—C5*AA*—C9*AA*—N1*A*	−1.0 (8)
C2*A*—C11*A*—N12*A*—C13*A*	78.5 (6)
C11*A*—N12*A*—C13*A*—C15*A*	−179.6 (4)
N12*A*—C13*A*—C15*A*—C16*A*	−160.0 (5)
C2*B*—N1*B*—C9*AB*—C5*AB*	−38.1 (7)
C10*B*—N1*B*—C9*AB*—C9*B*	−6.6 (7)
C9*AB*—N1*B*—C2*B*—C11*B*	126.0 (5)
C9*AB*—N1*B*—C2*B*—C3*B*	0.8 (7)
N1*B*—C2*B*—C11*B*—N12*B*	175.8 (4)
N1*B*—C2*B*—C3*B*—N4*B*	72.7 (6)
C2*B*—C3*B*—N4*B*—C5*B*	−74.8 (6)
C3*B*—N4*B*—C5*B*—C5*AB*	2.4 (8)
N4*B*—C5*B*—C1′*B*—C2′*B*	37.8 (7)
N4*B*—C5*B*—C5*AB*—C9*AB*	39.4 (8)
C5*B*—C5*AB*—C9*AB*—N1*B*	0.3 (8)
C2*B*—C11*B*—N12*B*—C13*B*	−76.1 (6)
N12*B*—C13*B*—C15*B*—C16*B*	158.3 (5)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12*A*—H12*A*···O2*W*	0.860	2.07	2.863 (5)	153
O2*W*—H2*WB*···N4*A*	1.061	1.92	2.930 (6)	159
O2*W*—H2*WA*···O14*B*^{i}	1.079	1.76	2.781 (5)	155
N12*B*—H12*B*···O1*W*	0.860	2.08	2.873 (6)	152
O1*W*—H1*WB*···N4*B*	1.199	1.97	2.929 (5)	133
O1*W*—H1*WA*···O14*A*^{ii}	1.170	1.74	2.791 (5)	147

Symmetry codes: (i) $\frac{5}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

The title structure was solved by direct methods and refined by full-matrix least squares on F^2. H atoms (except those of the H_2O molecules obtained from the ΔF synthesis) were included at calculated positions riding on their parent atoms.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEX* (McArdle, 1994). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

The authors thank Dr H. Zeugner, Kali-Chemie Pharma GmbH, Hannover, for providing a sample of the title compound.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1261). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A. & Polidori, G. (1994). *J. Appl. Cryst.* **27**, 435.

Blaton, N. M., Peeters, O. M. & De Ranter, C. J. (1996). *Acta Cryst.* C**52**, 2793–2795.

Codding, P. W., Zeugner, H. & Finner, E. (1987). *Acta Cryst.* C**43**, 1394–1397.

McArdle, P. (1994). *J. Appl. Cryst.* **27**, 438–439.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Petcher, T. J., Widmer, A., Maetzel, U. & Zeugner, H. (1985). *Acta Cryst.* C**41**, 909–912.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1989). *XEMP. Empirical Absorption Correction Program*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1994). *XSCANS Users Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.