

Data collection

Nicolet P3F diffractometer	$\theta_{\max} = 24^\circ$
ω scans	$h = 0 \rightarrow 9$
Absorption correction:	$k = 0 \rightarrow 12$
none	$l = -16 \rightarrow 15$
2166 measured reflections	2 standard reflections
2017 independent reflections	monitored every 98
1600 observed reflections	reflections
[$I > 4\sigma(I)$]	intensity variation: 2%
$R_{\text{int}} = 0.022$	

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.0002F^2]$
$R = 0.039$	$(\Delta/\sigma)_{\max} = 0.018$
$wR = 0.043$	$\Delta\rho_{\max} = 0.25 \text{ e } \text{Å}^{-3}$
$S = 1.61$	$\Delta\rho_{\min} = -0.23 \text{ e } \text{Å}^{-3}$
1600 reflections	Atomic scattering factors
145 parameters	from <i>International Tables</i>
Riding model, C—H 0.96 Å,	for <i>X-ray Crystallography</i>
fixed isotropic $U = 0.08 \text{ Å}^2$	(1974, Vol. IV)

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

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
P(1)	0.1715 (1)	0.1768 (1)	0.2207 (1)	0.037 (1)
O(1)	0.1395 (2)	0.0437 (1)	0.2298 (1)	0.054 (1)
C(1)	0.3326 (3)	0.2377 (2)	0.3187 (2)	0.048 (1)
C(2)	0.3015 (4)	0.2205 (3)	0.4164 (2)	0.076 (1)
C(11)	-0.0085 (3)	0.2680 (2)	0.2118 (2)	0.034 (1)
C(12)	-0.0032 (3)	0.3912 (2)	0.2362 (2)	0.047 (1)
C(13)	-0.1464 (3)	0.4558 (2)	0.2265 (2)	0.058 (1)
C(14)	-0.2959 (3)	0.3988 (3)	0.1923 (2)	0.056 (1)
C(15)	-0.3023 (3)	0.2775 (3)	0.1664 (2)	0.056 (1)
C(16)	-0.1594 (3)	0.2117 (2)	0.1764 (2)	0.045 (1)
C(21)	0.2283 (3)	0.2101 (2)	0.1125 (2)	0.040 (1)
C(22)	0.2385 (4)	0.1140 (3)	0.0514 (2)	0.069 (1)
C(23)	0.2809 (5)	0.1372 (3)	-0.0322 (2)	0.093 (2)
C(24)	0.3094 (4)	0.2543 (3)	-0.0563 (2)	0.076 (1)
C(25)	0.2991 (3)	0.3496 (3)	0.0027 (2)	0.062 (1)
C(26)	0.2594 (3)	0.3283 (2)	0.0873 (2)	0.049 (1)

Table 2. Selected geometric parameters (Å , $^\circ$)

P(1)—O(1)	1.484 (2)	P(1)—C(1)	1.796 (2)
P(1)—C(11)	1.800 (2)	P(1)—C(21)	1.800 (3)
C(1)—C(2)	1.520 (4)	C(11)—C(16)	1.384 (3)
O(1)—P(1)—C(1)	114.1 (1)	O(1)—P(1)—C(11)	111.6 (1)
C(1)—P(1)—C(11)	107.5 (1)	O(1)—P(1)—C(21)	111.9 (1)
C(11)—P(1)—C(21)	105.2 (1)	C(1)—P(1)—C(21)	106.1 (1)
P(1)—C(1)—C(2)	113.0 (2)		

Data collection and cell refinement: Nicolet P3F diffractometer software. All other calculations and molecule graphics: *SHELXTL-Plus* (Sheldrick, 1990) program system. The strong reflections, 200, 020 and 002, were not used in the calculations due to an error in the intensity measurements.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, coordinates of equivalent atoms bonded to unique atoms and associated geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71627 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1090]

References

- Churchill, M. R., See, R. F., Randall, S. L. & Atwood, J. D. (1993). *Acta Cryst. C* **49**, 345–347.
- Orama, O., Hietala, J. & Koskinen, J. T. (1990). XIVth International Conference on Organometallic Chemistry, Wayne State Univ., Detroit, Michigan, USA. Abstract 229.
- Orama, O., Karhu, M., Näsäkkälä, M., Sundberg, M. & Uggla, R. (1979). *Cryst. Struct. Commun.* **8**, 409–413.
- Sheldrick, G. M. (1990). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1994). **C50**, 609–613

Diastereomeric Octahydronaphthoquinolizines

BING CAI, YANPING PAN, HANGXIA QIU,
JOHN C. DEWAN, DONALD J. WINK,
RANDALL B. MURPHY AND DAVID I. SCHUSTER*

Department of Chemistry and Center for Neural
Science, New York University, New York, NY 10003,
USA

(Received 17 February 1993; accepted 31 August 1993)

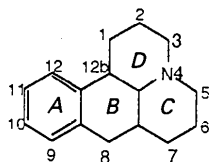
Abstract

Crystal structures of three diastereoisomers of the 2,3,6,7,7a,8,12b,12c-octahydro-4*H*⁺,1*H*,5*H*-naphtho-[1,2,3-*ij*]quinolizinium cation, $\text{C}_{16}\text{H}_{22}\text{N}^+$ (as the chloride dichloromethane solvate and as two perchlorate salts), $\text{C}_{16}\text{H}_{22}\text{N}$, are reported. The ring junctions *B/C*, *B/D* and *C/D* are *cis,cis,trans* (1), *trans,trans,trans* (2) and *cis,trans,cis* (3), respectively. In all three isomers, rings *C* and *D* of the quinolizine moiety adopt an almost perfect chair conformation while the fused ring *B* of the naphtho moiety adopts a half-chair conformation. These structural features are consistent with the presence of Bohlmann bands in the IR spectra of (1) and (2), but not (3).

Comment

In the course of preparation of new types of neuroreceptor ligands with high affinity to dopamine, serotonin and sigma receptor sites in the central nervous system (Largent, Wikström, Gundlach & Snyder, 1987), we have discovered a new tetracyclic ring system, the octahydronaphthoquinolizine compounds (OHNQ). These compounds have been synthesized in three steps from corresponding 2-tetralones (Cai, Pan, Dewan, Wink, Murphy & Schuster, 1993) and have been shown to have activity at selected neuroreceptor binding sites. Since

OHNQs possess three stereogenic centers, they exist as four diastereomers: *cis,cis*; *cis,trans*; *trans,cis*; *trans,trans*. The various diastereomers of each OHNQ have been separated and characterized by IR and NMR spectroscopy.



The structures of compounds (1), (2) and (3) have been confirmed by their X-ray crystal analyses, which are reported here. The data show that rings C and D each adopt a perfect chair conformation while ring B has a half-chair conformation in all three diastereomers. In addition, rings C and D are found to be *trans* fused in compounds (1) and (2), and *cis* fused in compound (3). This feature is consistent with the IR spectra, which show Bohlmann bands (Crabb & Katritzky, 1984) for compounds (1) and (2) but not for (3). The N4—C12c—C12b—C12a torsion angles are -79 (1), 175.4 (3) and 171.9 (3) $^\circ$

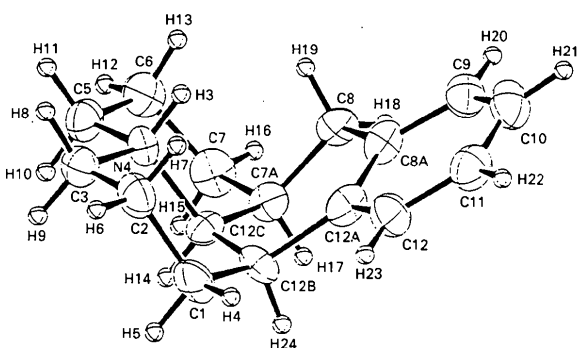


Fig. 1. Drawing of (1) produced using ORTEP (Johnson, 1965).

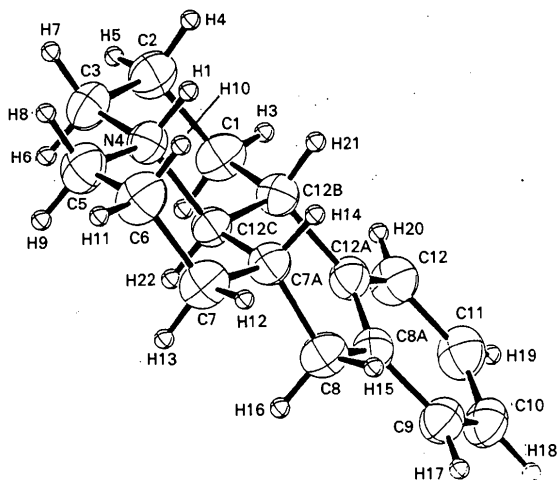


Fig. 2. Drawing of (2) produced using ORTEP (Johnson, 1965).

in compounds (1), (2) and (3), respectively. This means that the C—N bond between rings C and D is almost coplanar with the plane of the phenyl ring in compounds (2) and (3), but not in compound (1). Due to the structural characteristics and rigidity of these compounds, the biological properties of OHNQs at selected neuroreceptor sites are dependent on their stereochemistry. These new receptor ligands are expected to play a very important role in topological analysis of ligand/receptor interactions at a selected group of neuroreceptor binding sites.

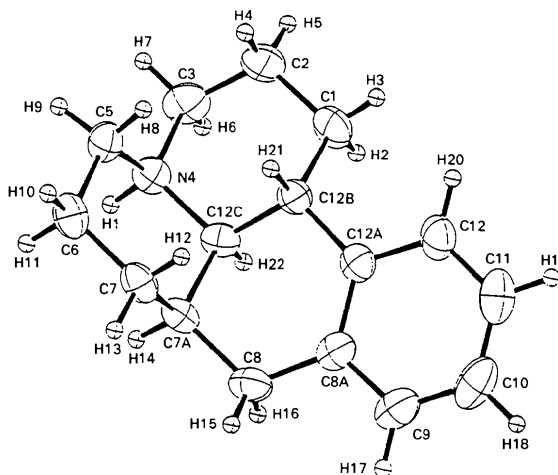


Fig. 3. Drawing of (3) produced using ORTEP (Johnson, 1965).

Experimental

Compound (1)

Crystal data

C₁₆H₂₂N⁺.Cl⁻.CH₂Cl₂

$M_r = 348.75$

Monoclinic

$P2_1/c$

$a = 10.597$ (3) Å

$b = 11.704$ (4) Å

$c = 14.390$ (6) Å

$\beta = 103.28$ (3) $^\circ$

$V = 1737$ Å³

$Z = 4$

$D_x = 1.333$ Mg m⁻³

Cu K α radiation

$\lambda = 1.54178$ Å

Cell parameters from 25 reflections

$\theta = 15.2$ – 17.5°

$\mu = 4.812$ mm⁻¹

$T = 298$ K

Plate

$0.52 \times 0.43 \times 0.13$ mm

Colorless

Data collection

Rigaku AFC-6R diffractometer

$\omega/2\theta$ scans

Absorption correction:

empirical (DIFABS;

Walker & Stuart, 1983)

$T_{\min} = 0.57$, $T_{\max} = 1.26$

2772 measured reflections

2621 independent reflections

1177 observed reflections

$[I > 2\sigma(I)]$

$R_{\text{int}} = 0.050$

$\theta_{\text{max}} = 60.0^\circ$

$h = 0 \rightarrow 10$

$k = 0 \rightarrow 13$

$l = -16 \rightarrow 14$

3 standard reflections

monitored every 100

reflections

intensity variation: 9.0%

(linear correction)

Refinement

Refinement on F $R = 0.085$ $wR = 0.080$ $S = 2.26$

1177 reflections

190 parameters

All H-atom parameters refined

$$w = 4F_o^2/\sigma^2(F_o^2)$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) $P2_1/n$

$$a = 8.922 (2) \text{ \AA}$$

$$b = 11.522 (5) \text{ \AA}$$

$$c = 15.734 (2) \text{ \AA}$$

$$\beta = 97.51 (1)^\circ$$

$$V = 1603.5 \text{ \AA}^3$$

$$Z = 4$$

Cell parameters from 25

reflections

$$\theta = 15.5\text{--}17.4^\circ$$

$$\mu = 2.277 \text{ mm}^{-1}$$

$$T = 298 \text{ K}$$

Block

$$0.26 \times 0.25 \times 0.23 \text{ mm}$$

Colorless

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for compound (1)
$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j$$

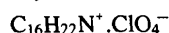
	x	y	z	B_{eq}
N4	0.9347 (7)	-0.4444 (7)	0.7443 (5)	3.6 (4)
C1	0.866 (1)	-0.381 (1)	0.5440 (7)	5.1 (6)
C2	0.998 (1)	-0.359 (1)	0.6062 (8)	5.1 (6)
C3	1.034 (1)	-0.446 (1)	0.6843 (7)	4.5 (6)
C5	0.970 (1)	-0.535 (1)	0.8196 (8)	4.9 (6)
C6	0.877 (1)	-0.535 (1)	0.8855 (7)	5.2 (6)
C7	0.737 (1)	-0.546 (1)	0.8309 (7)	5.3 (6)
C7A	0.7043 (9)	-0.456 (1)	0.7503 (7)	4.0 (5)
C8	0.6971 (9)	-0.338 (1)	0.7899 (6)	4.1 (5)
C8A	0.6888 (9)	-0.243 (1)	0.7175 (7)	4.4 (5)
C9	0.645 (1)	-0.135 (1)	0.7354 (8)	4.9 (6)
C10	0.637 (1)	-0.045 (1)	0.675 (1)	5.9 (7)
C11	0.670 (1)	-0.067 (1)	0.5895 (9)	5.4 (7)
C12	0.715 (1)	-0.171 (1)	0.5677 (8)	5.0 (6)
C12A	0.723 (1)	-0.261 (1)	0.6295 (7)	4.2 (5)
C12B	0.762 (1)	-0.382 (1)	0.6027 (7)	4.2 (5)
C12C	0.7994 (9)	-0.4631 (9)	0.6863 (7)	3.5 (5)
C111	0.9493 (3)	0.2482 (3)	0.6225 (2)	5.2 (1)
C121	0.5659 (3)	0.3050 (4)	0.5818 (2)	9.5 (2)
C122	0.6354 (3)	0.3758 (4)	0.4082 (3)	9.3 (2)
C21	0.627 (1)	0.265 (1)	0.4850 (8)	6.3 (7)

Table 2. Selected geometric parameters (\AA , $^\circ$) for compound (1)

N4—C3	1.51 (1)	C12B—C12C	1.51 (1)
N4—C5	1.50 (1)	C7A—C8	1.51 (1)
N4—C12C	1.50 (1)	C8A—C12A	1.41 (1)
C7A—C12C	1.52 (1)	C10—C11	1.37 (1)
C3—N4—C5	108.2 (8)	C8A—C12A—C12B	120 (1)
C3—N4—C12C	112.5 (7)	C1—C12B—C12A	114 (1)
C5—N4—C12C	110.5 (8)	C1—C12B—C12C	110.4 (9)
C7—C7A—C8	111.4 (8)	C12A—C12B—C12C	114.3 (9)
C7—C7A—C12C	110.9 (9)	N4—C12C—C7A	109.8 (7)
C8—C7A—C12C	112.0 (9)	N4—C12C—C12B	113.4 (8)
C7A—C8—C8A	114.5 (9)	C7A—C12C—C12B	111.1 (9)
C8—C8A—C12A	122 (1)		
N4—C3—C2—C1	-59 (1)		
N4—C5—C6—C7	54 (1)		
N4—C12C—C7A—C7	-58 (1)		
N4—C12C—C7A—C8	67 (1)		
N4—C12C—C12B—C1	50 (1)		
N4—C12C—C12B—C12A	-79 (1)		
C2—C1—C12B—C12A	77 (1)		
C3—C2—C1—C12B	58 (1)		
C7—C7A—C8—C8A	169.9 (8)		

Compound (2)

Crystal data

 $M_r = 327.81$

Monoclinic

$$D_x = 1.358 \text{ Mg m}^{-3}$$

Cu $K\alpha$ radiation

$$\lambda = 1.54178 \text{ \AA}$$

Data collection

Rigaku AFC-6R diffractometer

 ω/θ scans

Absorption correction:

empirical (DIFABS; Walker & Stuart, 1983)

$$T_{\min} = 0.64, T_{\max} = 1.26$$

2655 measured reflections

2477 independent reflections

1833 observed reflections

$$[I > 2\sigma(I)]$$

Refinement

Refinement on F $R = 0.066$ $wR = 0.075$ $S = 2.59$

1833 reflections

199 parameters

All H-atom parameters refined

$$\theta_{\max} = 59.5^\circ$$

$$h = 0 \rightarrow 9$$

$$k = 0 \rightarrow 12$$

$$l = -17 \rightarrow 17$$

3 standard reflections monitored every 250 reflections

intensity variation: none

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for compound (2)
$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j$$

	x	y	z	B_{eq}
N4	-0.1156 (3)	-0.3311 (3)	0.6225 (2)	4.6 (1)
C1	0.0378 (4)	-0.1069 (4)	0.6407 (3)	5.7 (2)
C2	0.1236 (4)	-0.2199 (4)	0.6308 (3)	5.7 (2)
C3	0.0472 (4)	-0.3220 (4)	0.6651 (3)	5.7 (2)
C5	-0.1894 (5)	-0.4370 (4)	0.6529 (3)	5.9 (2)
C6	-0.3465 (5)	-0.4512 (4)	0.6070 (3)	6.4 (2)
C7	-0.4398 (4)	-0.3456 (4)	0.6202 (3)	5.7 (2)
C7A	-0.3665 (4)	-0.2343 (3)	0.5931 (2)	4.6 (2)
C8	-0.4544 (4)	-0.1291 (4)	0.6158 (3)	5.3 (2)
C8A	-0.3739 (4)	-0.0140 (3)	0.6105 (2)	4.6 (2)
C9	-0.4576 (4)	0.0872 (4)	0.6121 (3)	5.5 (2)
C10	-0.3905 (6)	0.1939 (4)	0.6116 (3)	5.9 (2)
C11	-0.2377 (6)	0.2008 (4)	0.6088 (3)	6.3 (2)
C12	-0.1524 (5)	0.1019 (4)	0.6062 (3)	5.8 (2)
C12A	-0.2197 (4)	-0.0075 (3)	0.6067 (2)	4.6 (2)
C12B	-0.1255 (4)	-0.1181 (3)	0.5999 (2)	4.6 (2)
C12C	-0.2015 (4)	-0.2209 (3)	0.6357 (2)	4.2 (2)
C111	0.1843 (1)	0.3478 (1)	0.62595 (7)	5.56 (5)
O11	0.1131 (4)	0.3715 (4)	0.6983 (2)	8.9 (2)
O12	0.0810 (4)	0.3779 (4)	0.5525 (2)	9.0 (2)
O13	0.2178 (4)	0.2286 (3)	0.6215 (3)	10.1 (3)
O14	0.3192 (4)	0.4081 (4)	0.6278 (3)	10.1 (2)

Table 4. Selected geometric parameters (\AA , $^\circ$) for compound (2)

N4—C3	1.522 (5)	C12B—C12C	1.510 (5)
N4—C5	1.496 (5)	C7A—C8	1.512 (5)
N4—C12C	1.511 (4)	C8A—C12A	1.386 (5)
C7A—C12C	1.542 (5)	C10—C11	1.372 (7)

C3—N4—C5	110.5 (3)	C8A—C12A—C12B	120.8 (3)	C7A	0.0460 (6)	-0.1969 (4)	0.4811 (7)	4.7 (2)
C3—N4—C12C	110.7 (3)	C1—C12B—C12A	113.8 (3)	C8	-0.0320 (6)	-0.2823 (4)	0.5593 (7)	5.3 (2)
C5—N4—C12C	112.9 (3)	C1—C12B—C12C	111.2 (3)	C8A	0.0843 (6)	-0.3611 (4)	0.6429 (7)	4.7 (2)
C7—C7A—C8	110.7 (3)	C12A—C12B—C12C	110.4 (3)	C9	0.0426 (8)	-0.4279 (5)	0.7601 (8)	6.1 (2)
C7—C7A—C12C	112.5 (3)	N4—C12C—C7A	109.2 (3)	C10	0.143 (1)	-0.5011 (4)	0.8408 (9)	7.0 (3)
C8—C7A—C12C	108.2 (3)	N4—C12C—C12B	110.3 (3)	C11	0.293 (1)	-0.5090 (4)	0.8096 (9)	6.8 (3)
C7A—C8—C8A	115.1 (3)	C7A—C12C—C12B	111.5 (3)	C12	0.3383 (7)	-0.4435 (4)	0.6950 (8)	5.7 (2)
C8—C8A—C12A	122.0 (3)			C12A	0.2360 (6)	-0.3700 (3)	0.6080 (7)	4.3 (2)
N4—C3—C2—C1	55.6 (5)	N4—C12C—C12B—C12A	175.4 (3)	C12B	0.2908 (6)	-0.2987 (3)	0.4858 (7)	4.3 (2)
N4—C5—C6—C7	-58.0 (5)	C2—C1—C12B—C12A	-179.7 (3)	C12C	0.1429 (5)	-0.2423 (3)	0.3657 (6)	4.1 (2)
N4—C12C—C7A—C7	50.9 (4)	C3—C2—C1—C12B	-54.5 (5)	C111	0.2604 (1)	0.1587 (1)	0.0732 (2)	5.16 (5)
N4—C12C—C7A—C8	173.6 (3)	C7—C7A—C8—C8A	166.8 (3)	O11	0.3190 (6)	0.1604 (4)	0.2745 (6)	8.7 (2)
N4—C12C—C12B—C1	-57.3 (4)			O12	0.1126 (6)	0.0924 (4)	-0.0020 (8)	11.0 (3)
				O13	0.2200 (9)	0.2523 (4)	0.011 (1)	13.5 (3)
				O14	0.382 (1)	0.1249 (7)	0.015 (1)	16.6 (5)

Compound (3)*Crystal data*C₁₆H₂₂N⁺.ClO₄⁻M_r = 327.81

Triclinic

P $\bar{1}$

a = 8.427 (2) Å

b = 13.509 (1) Å

c = 7.467 (3) Å

α = 92.06 (2)°

β = 111.18 (2)°

γ = 94.11 (1)°

V = 788.7 Å³

Z = 2

Data collection

Rigaku AFC-6R diffractometer

ω/θ scans

Absorption correction:

empirical (DIFABS;

Walker & Stuart, 1983)

T_{min} = 0.65, T_{max} = 1.18

2495 measured reflections

2301 independent reflections

1964 observed reflections

[I > 2σ(I)]

Refinement

Refinement on F

R = 0.08

wR = 0.103

S = 3.96

1964 reflections

199 parameters

All H-atom parameters

refined

D_x = 1.38 Mg m⁻³

Cu Kα radiation

λ = 1.54178 Å

Cell parameters from 25

reflections

θ = 15.0–17.4°

μ = 2.314 mm⁻¹

T = 298 K

Block

0.27 × 0.23 × 0.22 mm

Colorless

θ_{max} = 59.5°

h = -6 → 9

k = -15 → 15

l = -8 → 7

3 standard reflections

monitored every 250

reflections

intensity variation: none

w = 4F_o²/σ²(F_o²)(Δ/σ)_{max} = 0.001Δρ_{max} = 0.57 e Å⁻³Δρ_{min} = -0.38 e Å⁻³

Atomic scattering factors

from *International Tables*

for X-ray Crystallography

(1974, Vol. IV)

Table 6. Selected geometric parameters (Å, °) for compound (3)

N4—C3	1.512 (6)	C12B—C12C	1.521 (6)
N4—C5	1.507 (6)	C7A—C8	1.524 (7)
N4—C12C	1.505 (6)	C8A—C12A	1.407 (6)
C7A—C12C	1.527 (6)	C10—C11	1.372 (9)
C3—N4—C5	112.6 (4)	C8A—C12A—C12B	121.2 (4)
C3—N4—C12C	111.0 (4)	C1—C12B—C12A	115.1 (4)
C5—N4—C12C	112.4 (3)	C1—C12B—C12C	108.5 (4)
C7—C7A—C8	115.6 (4)	C12A—C12B—C12C	111.2 (4)
C7—C7A—C12C	109.9 (4)	N4—C12C—C7A	109.0 (4)
C8—C7A—C12C	107.3 (4)	N4—C12C—C12B	110.7 (3)
C7A—C8—C8A	115.2 (4)	C7A—C12C—C12B	113.7 (4)
C8—C8A—C12A	122.2 (4)		
N4—C3—C2—C1	52.1 (7)	N4—C12C—C12B—C12A	171.9 (3)
N4—C5—C6—C7	52.6 (6)	C2—C1—C12B—C12A	-178.0 (5)
N4—C12C—C7A—C7	-60.2 (5)	C3—C2—C1—C12B	-53.9 (7)
N4—C12C—C7A—C8	173.4 (4)	C7—C7A—C8—C8A	-78.7 (5)
N4—C12C—C12B—C1	-60.5 (5)		

The structures were solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refinement was by full-matrix least squares using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985).

This work was supported in part by grants from the Research Challenge Fund and the Arts and Science Technology Transfer Fund at New York University. We are deeply grateful to Professor Martin Kuehne of the University of Vermont for bringing Mandell's cyclization studies to our attention.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71622 (106 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1066]

Table 5. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for compound (3)

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	x	y	z	B _{eq}
N4	0.2044 (5)	-0.1615 (3)	0.2655 (5)	4.6 (2)
C1	0.3703 (8)	-0.3461 (4)	0.3513 (8)	5.9 (2)
C2	0.4260 (8)	-0.2665 (4)	0.241 (1)	6.7 (3)
C3	0.2877 (8)	-0.2042 (5)	0.1361 (7)	6.0 (2)
C5	0.3164 (6)	-0.0786 (4)	0.4045 (7)	4.9 (2)
C6	0.2275 (7)	-0.0375 (4)	0.5315 (8)	5.6 (2)
C7	0.1632 (6)	-0.1187 (4)	0.6312 (7)	4.9 (2)

References

- Cai, B., Pan, Y. P., Dewan, J. C., Wink, D. J., Murphy, R. B. & Schuster, D. I. (1993). *Tetrahedron Lett.* **34**, 2067–2070.
- Crabb, T. A. & Katritzky, A. R. (1984). *Adv. Heterocycl. Chem.* **36**, 42–49.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Largent, B. L., Wikström, H., Gundlach, A. L. & Snyder, S. H. (1987). *Mol. Pharmacol.* **32**, 772–784.
- Molecular Structure Corporation (1985). *TEXSAN*. *TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A39, 158–166.

Acta Cryst. (1994). C50, 613–614

A Triazine Derivative

G. D. NIGAM*

*Department of Physics and Meteorology,
Indian Institute of Technology, Kharagpur 721302, India*

S. KARAK AND C. R. SAHA

*Department of Chemistry, Indian Institute of
Technology, Kharagpur 721302, India*

S. SRINIVASAN

*Department of Physics, Indian Institute of Technology,
Madras 600036, India*

(Received 25 February 1993; accepted 8 September 1993)

Abstract

In 4,6-diamino-1-ethyl-2-(2-hydroxyphenyl)-1,2-dihydro-3*H*⁺,5*H*⁺-1,3,5-triazinium sulfate monohydrate, C₁₁H₁₇N₅O²⁺·SO₄²⁻·H₂O, the sulfate group and the triazine ring are bridged via N—H···O hydrogen bonds. The O atom of the water molecule forms O—H···O hydrogen bonds with the sulfate moieties of two neighbouring units and O···H—N hydrogen bonds with two other neighbouring units.

Comment

Dihydrotriazines are of interest because of their anti-malarial and anticancer activities (Katritzky & Rees, 1984). The Cu^{II} complex of the title compound (I) was prepared by refluxing Cu(*N*-ethylbiguanide) base with salicylaldehyde. The decomposition of the complex with H₂SO₄ (6*N*) yielded (I) which forms interesting complexes with *d*-block metal ions (Saha, Karak & Santra, 1992). Single crystals were obtained by evaporation from an aqueous solution.

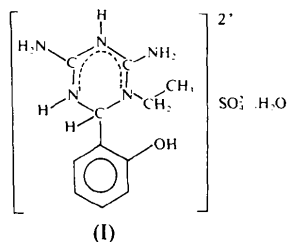


Fig. 1 shows an ORTEPII plot of the molecule (Johnson, 1976). The intramolecular hydrogen bond between N(7) and O(1) has the parameters N(7)—H(7.2)···O(1) = 2.743 (9), O(1)···H(7.2) = 1.859 (73) and N(7)—H(7.2) = 0.960 (68) Å. The intermolecular bonds involving the water of crystallization are

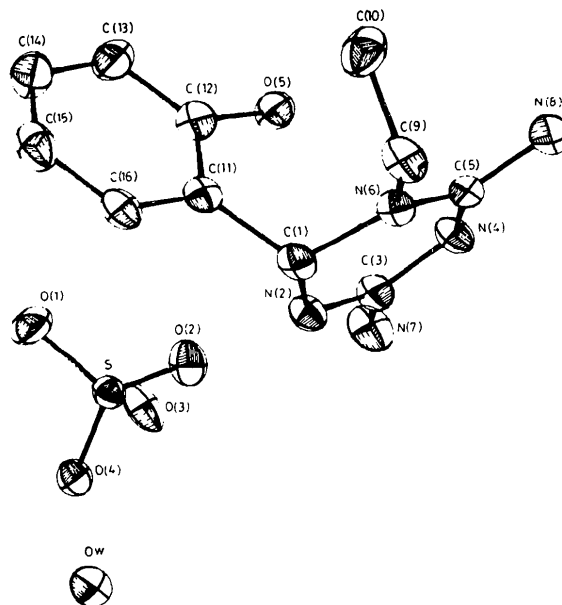


Fig. 1. Numbering scheme and displacement ellipsoids drawn at the 50% probability level.

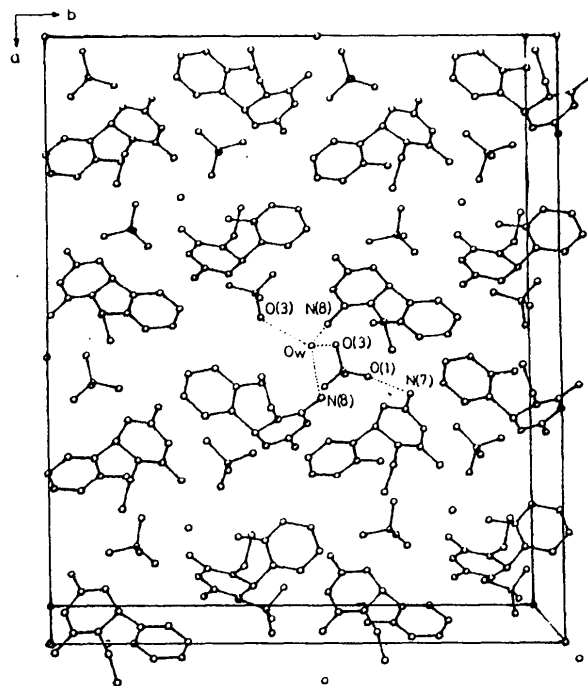


Fig. 2. Packing of molecules in the unit cell with the intermolecular hydrogen bonds marked by dotted lines.