95.22 (2)°, $V = 428.4$ (2) Å^3, $Z = 2$, $D_x = 1.47$ Mg m^{-3}, $R = 0.147$ for 339 unique reflections.

Related literature. Crystals studied in the present work are part of a series of compounds prepared to explore new nonlinear optical materials with high transparencies for blue light (Okazaki, Shishido & Kubodera, 1987). The internal C—C—C angle of the benzene ring to which NO_2 is bonded ranges from 121.1 (4) to 124.8 (9)°. For the series, the σ-electron-withdrawing character of the NO_2 group (Di Rienzo, Domenicano & Di Sanseverino, 1980) is suggested.

References

Di Rienzo, F., Domenicano, A. & Di Sanseverino, L. R. (1980). *Acta Cryst.* **B36**, 586–591.

Furusaki, A. (1979). *Acta Cryst.* **A35**, 220–224.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Okazaki, M., Shishido, T. & Kubodera, S. (1987). *Jpn. Kokai Tokkyo Koho*, JP62 **210**, 432.

Sakurai, T. & Kobayashi, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

Acta Cryst. (1992). C**48**, 188–190

Structure of 9-Amino-2,7-di-*tert*-butylacridine

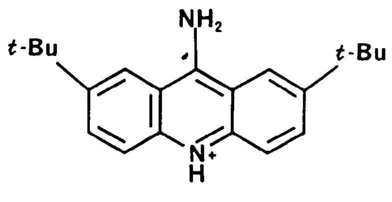
By R. McKenna,* M. Agbandje* and S. Neidle†

Cancer Research Campaign Biomolecular Structure Unit, The Institute of Cancer Research, Sutton, Surrey SM2 5NG, England

(*Received* 11 *March* 1991; *accepted* 24 *July* 1991)

Abstract. $C_{21}H_{28}N_2^+.Cl^-.H_2O$, $M_r = 361.94$, trigonal, $P3_1$, $a = 11.086$ (2), $c = 14.367$ (4) Å, $V = 1529.2$ Å^3, $F(000) = 585$, $Z = 3$, $D_x = 1.179$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 17.34$ cm^{-1}, $T = 293$ K, final $R = 0.067$ for 3252 reflections. There is an extensive network of hydrogen-bonding interactions centered around the chloride ions positioned on the threefold axes. This is effectively a channel into which the 9-amino groups of the title compound are directed and hydrogen bond to the chloride ions. These groups and the protonated ring N atoms also form hydrogen bonds to water molecules present in the channel.

Experimental. The title compound (shown in the scheme below) was crystallized from ethanol as deep-yellow hexagonal tablets. A crystal of dimensions 0.3 × 0.3 × 0.1 mm was used in the analysis. Accurate cell dimensions were obtained by least-squares analysis of 25 reflections ($8 < \theta < 17°$) measured on an Enraf–Nonius CAD-4 diffractometer. Intensity data were collected at room temperature with graphite-monochromated Cu $K\alpha$ radiation in the range $1.5 \leq \theta \leq 70°$ ($0 \leq h \leq 13$, $-17 \leq l \leq 17$) using an ω–2θ scan technique and a maximum scan time of 120 s per reflection. The intensities of three strong reflections were monitored at regular intervals during the data collection and showed no decay (< 3%). A unique data set of 6009 reflections was collected, 3252 had $I \geq 1.5\sigma(I)$. Lorentz, polarization and a (post refinement) absorption correction (Walker & Stuart, 1983) were applied to the data. Maximum and minimum transmission coefficients were 1.23 and 0.81. Systematic absences $00l$, $l = 3n$ were found. Direct methods (*MULTAN*82; Main *et al.*, 1982) were applied to both enantiomorphic space groups $P3_1$, and $P3_2$. The best E map for the latter had a combined figure of merit (CFOM) of 1.452; the map could not be interpreted. The best E map in $P3_1$ had a CFOM of 2.911 and showed all the non-H atoms in the structure together with a chloride ion and a water molecule of crystallization, the former on the crystallographic threefold axis. Refinement was by full-matrix least squares based on F. The positions of some H atoms, including those on the water molecule, were located in difference Fourier maps. Others, especially on the butyl groups, were gener-



---

* Now at: Department of Biology, Purdue University, Lafayette, Indiana 47907, USA.

† Author for correspondence.

0108-2701/92/010188-03$03.00

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* ($\AA^2$) *with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\AA^2)$
C1	0.6007 (4)	−0.1097 (4)	0.5204 (3)	2.9 (1)
C2	0.5085 (4)	−0.1662 (4)	0.5927 (3)	2.8 (1)
C3	0.3830 (4)	−0.1638 (5)	0.5855 (3)	3.4 (1)
C4	0.3506 (4)	−0.1110 (5)	0.5120 (4)	3.5 (1)
C5	0.4612 (4)	0.1110 (4)	0.2122 (4)	3.4 (1)
C6	0.5470 (5)	0.1643 (5)	0.1391 (3)	3.5 (1)
C7	0.6742 (4)	0.1661 (4)	0.1308 (3)	2.9 (1)
C8	0.7108 (4)	0.1098 (4)	0.2030 (3)	2.9 (1)
C9	0.6653 (4)	0.0004 (4)	0.3617 (3)	2.40 (9)
N9	0.7846 (3)	−0.0003 (4)	0.3619 (3)	3.46 (8)
N10	0.4129 (3)	−0.0003 (4)	0.3622 (3)	3.19 (8)
C11	0.5729 (4)	−0.0529 (4)	0.4405 (3)	2.6 (1)
C12	0.4458 (4)	−0.0553 (4)	0.4370 (3)	2.7 (1)
C13	0.6258 (4)	0.0530 (4)	0.2831 (3)	2.45 (9)
C14	0.4992 (4)	0.0531 (4)	0.2869 (3)	2.7 (1)
C15	0.7674 (5)	0.2295 (5)	0.0457 (4)	4.1 (1)
C16	0.9074 (6)	0.2439 (7)	0.0560 (5)	7.7 (2)
C17	0.7870 (7)	0.3709 (7)	0.0255 (7)	10.4 (7)
C18	0.6960 (10)	0.1490 (20)	0.0391 (7)	14.5 (5)
C19	0.5380 (5)	−0.2289 (5)	0.6787 (4)	4.2 (1)
C20	0.6650 (6)	−0.2421 (7)	0.6689 (5)	7.8 (2)
C21	0.4165 (7)	−0.3711 (8)	0.6984 (6)	10.0 (2)
C22	0.5490 (10)	−0.1468 (9)	0.7626 (6)	15.1 (3)
Cl	0	0	0.8620	3.79 (2)
O*W*	0.1680 (3)	0	0.3618 (4)	9.3 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

C1—C2	1.371 (6)	C9—N9	1.326 (6)
C1—C11	1.414 (7)	C9—C11	1.440 (6)
C2—C3	1.408 (7)	C9—C13	1.436 (7)
C2—C19	1.531 (8)	N10—C12	1.372 (7)
C3—C4	1.341 (8)	N10—C14	1.367 (6)
C4—C12	1.417 (6)	C11—C12	1.397 (7)
C5—C6	1.339 (7)	C15—C16	1.486 (9)
C6—C7	1.406 (8)	C15—C17	1.503 (10)
C7—C8	1.372 (7)	C19—C20	1.492 (10)
C7—C15	1.526 (7)	C19—C21	1.501 (7)
C8—C13	1.420 (6)	C19—C22	1.479 (11)

C2—C1—C11	122.6 (4)	C4—C12—C11	120.4 (4)
C1—C2—C3	117.0 (4)	N10—C12—C11	120.6 (4)
C1—C2—C19	122.6 (4)	C8—C13—C9	123.3 (4)
C3—C2—C19	120.4 (4)	C9—C13—C14	118.5 (4)
C2—C3—C4	123.3 (4)	C8—C13—C14	118.2 (4)
C3—C4—C12	119.1 (4)	C5—C14—N10	119.6 (4)
C6—C5—C14	119.2 (4)	C5—C14—C13	119.5 (4)
C5—C6—C7	124.0 (5)	C13—C14—N10	120.9 (5)
C6—C7—C8	116.9 (4)	C7—C15—C16	113.5 (5)
C6—C7—C15	121.2 (4)	C7—C15—C17	110.3 (5)
C8—C7—C15	122.0 (4)	C16—C15—C17	107.7 (5)
C7—C8—C13	122.3 (4)	C16—C15—C18	110.9 (8)
N9—C9—C11	120.2 (4)	C17—C15—C18	103.3 (10)
N9—C9—C13	120.7 (4)	C2—C19—C20	113.3 (5)
C11—C9—C13	119.1 (4)	C2—C19—C21	110.2 (5)
C12—N10—C14	121.9 (4)	C2—C19—C22	110.4 (6)
C1—C11—C9	123.5 (4)	C20—C19—C21	108.1 (6)
C1—C11—C12	117.6 (4)	C20—C19—C22	110.3 (6)
C9—C11—C12	118.9 (4)	C21—C19—C22	104.1 (6)
C4—C12—N10	119.0 (4)		

Table 3. *Intermolecular hydrogen-bond geometries*

Symmetry translation		Angle (°)	Donor-acceptor distance (Å)	Acceptor-hydrogen distance (Å)
$y-x+1,\ -x,\ z-\frac{1}{3}$	Cl···H91—N9	146	3.382 (4)	2.55 (3)
$1-y,\ x-y,\ z-\frac{2}{3}$	Cl···H92—N9	149	3.379 (4)	2.53 (3)
$y-x,\ -x,\ z-\frac{1}{3}$	Cl···HO*W*1—O*W*	152	3.036 (5)	2.09 (5)
$-y,\ x-y,\ z-\frac{2}{3}$	Cl···HO*W*2—O*W*	158	3.031 (5)	1.93 (6)
$x,\ y,\ z$	N10—H101···O*W*	175 (5)	2.717 (5)	1.96 (6)

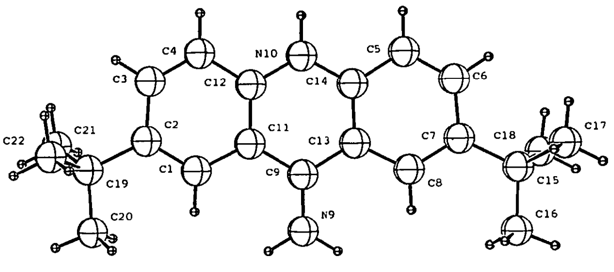


Fig. 1. Molecular structure of 9-amino-2,7-di-*tert*-butylacridine.

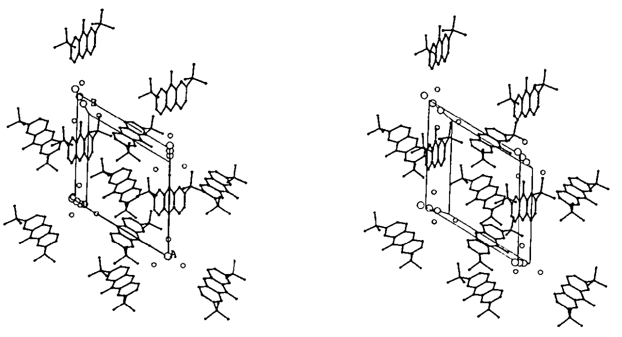


Fig. 2. A view of the crystal packing, viewed down the 3_1 screw axes.

ated geometrically. All H atoms except those directly attached to the acridine ring were assigned temperature factors of 5 $\AA^2$, apart from those at the terminal of the butyl groups, which were set at 10.0–15.0 $\AA^2$. Positional parameters for H atoms directly attached to the acridine ring and those on the located water molecule were included in the refinements; others were kept fixed. All non-H atoms were refined anisotropically. The final $R = 0.067$ and $wR = 0.084$ with weighting $w = 1/[\sigma^2(F) + (0.04F)^2]$. The maximum shift/e.s.d. was < 0.02 and the limits of residual electron density in the final difference Fourier map were 0.74, -0.68 e $\AA^{-3}$. These peaks were around the butyl groups. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed with the *SDP* system (Frenz, 1980). Final atomic coordinates are given in Table 1, bond lengths and angles in Table 2 and intermolecular geometry in Table 3.* Figs. 1 and 2 show the molecular structure and the crystal-packing arrangement.

---

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54551 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0074]

Related literature. Acridines and 9-aminoacridines crystallize as salts [for example: proflavine hemisulfate hydrate (Jones & Neidle, 1975); 9-aminoacridine hydrochloride monohydrate (Talacki, Carrell & Glusker, 1974)] and invariably have the protonated ring nitrogen atom involved in intermolecular hydrogen bonding with anions and water molecules.

This work was supported by the Cancer Research Campaign (grants to SN and research studentship to MA).

References

FRENZ, B. A. (1980). *Enraf–Nonius Structure Determination Package*. Version 17. College Station, Texas, USA.

JONES, T. A. & NEIDLE, S. (1975). *Acta Cryst.* **B31**, 1324–1333.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

TALACKI, R., CARRELL, H. L. & GLUSKER, J. P. (1974). *Acta Cryst.* **B30**, 1044–1047.

WALKER, N. L. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1992). C**48**, 190–191

Structure of a Norbornanepolysulfane: *exo*-3,4,5,6,7-Pentathiatricyclo[7.2.1.$0^{2,8}$]dodecane

BY JOACHIM PICKARDT, MONIKA KUSTOS AND RALF STEUDEL*

Institut für Anorganische und Analytische Chemie, Technische Universität Berlin, Straße des 17. *Juni* 135, *D*-1000 *Berlin* 12, *Germany*

(*Received* 3 *March* 1991; *accepted* 15 *July* 1991)

Abstract. $C_7H_{10}S_5$, $M_r = 254.46$, monoclinic, $P2_1/n$, $a = 10.167$ (4), $b = 11.092$ (2), $c = 10.278$ (4) Å, $\beta = 115.22$ (2)°, $V = 1049$ (1) Å^3, $Z = 4$, $D_x = 1.61$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.01$ mm^{-1}, $F(000) = 528$, $T = 293$ K, final $R = 0.0339$ for 1858 observed reflections. The two alpha S atoms in the pentathiepane derivative of norbornane, obtained from the reaction of norbornene with activated sulfur [Bartlett & Ghosh (1987). *J. Org. Chem.* **52**, 4937–4943], are both in *exo* positions.

Experimental. Bright yellow crystals were obtained from a solution of $C_7H_{10}S_5$ in *n*-pentane, saturated at 293 K and kept for 24 h at 275 K. A crystal of approximate dimensions 0.3 × 0.3 × 0.4 mm was used for the X-ray analysis. Intensities measured on a Syntex $P2_1$ diffractometer, Mo $K\alpha$ radiation (graphite monochromator). Cell dimensions determined from least-squares refinement of 25 reflections ($8.7 \leq 2\theta \leq 24.0°$). Reflections measured in the ω-scan mode, $2\theta_{\max} = 55°$ ($-13 \leq h \leq 11$, $0 \leq k \leq 13$, $0 \leq l \leq 13$); two standard reflections (303; $\bar{1}21$) measured every 100 reflections showed only random variations ≤ 1.5%. 2610 reflections collected, 2213 symmetry independent ($R_{\rm int} = 0.019$), 1858 observed with $I \geq 2\sigma(I)$. Lorentz and polarization corrections applied, empirical absorption correction with *DIFABS* (Walker & Stuart, 1983), minimum/maximum corrections 0.780/1.139.

Structure solved by direct methods using the program *SHELX*86 (Sheldrick, 1986), refinement with *SHELX*76 (Sheldrick, 1976), geometrical calculations with *PLATON* (Spek, 1982). Atomic scattering factors taken from Cromer & Mann (1968). H-atom positions calculated assuming sp^3 hybridization and C—H bond lengths of 1.08 Å. Full-matrix least-squares refinements on F with anisotropic thermal parameters, unit weights, 110 parameters, $R = 0.034$; $(\Delta/\sigma)_{\max} = 0.0001$; $\Delta\rho_{\min}$ −0.27, $\Delta\rho_{\max}$ 0.34 e Å^{-3}, goodness of fit = 0.71. Atomic parameters are given in Table 1 and bond lengths and angles are in Table 2.† Fig. 1 shows a view of the molecule with the atom-numbering scheme.

Related literature. Sulfuration of norbornene derivatives gives the trithiolane and pentathiepine derivatives. The position of both alpha S atoms is always determined as the *exo* position (Bartlett & Ghosh, 1987; Watson, Jain, Bartlett & Ghosh, 1985; Emsley & Griffiths, 1979).

---

* Author to whom correspondence should be addressed.

† 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 54468 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0255]

0108-2701/92/010190-02$03.00