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Structure of 3,7-Bis(dimethylamino)-10-(*N*-methylcarbamoyl)phenothiazine (MCDP)–Ethanol (1/1)

ISAO FUJII AND NORIAKI HIRAYAMA*

Department of Biological Science and Technology, Tokai University, 317 Nishino, Numazu, Shizuoka 410-03, Japan

AKIRA MIIKE

Diagnostics Research Laboratories Kyowa Medex Co. Ltd, 600-1 Minami-Ishiki, Nagaizumi-cho, Sunto-gun, Shizuoka 411, Japan

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Abstract

In the title structure, 3,7-bis(dimethylamino)-*N*-methylphenothiazine-10-carboxamide, the phenothiazine ring adopts a boat conformation with the S and N atoms occupying the bow and stern positions, respectively. The dihedral angle between the two phenyl rings is 138.4 (1)°. The conjugate system in the molecule is remarkably different from those in methylene blue molecules.

Comment

The title compound is one of the functional dyes which are used clinically as diagnostics. MCDP is used to measure the activity of monoamine oxidase in blood. In the presence of peroxidase and hydrogen peroxide, MCDP converts into methylene blue and the blue color is developed. The effective conversion is essential for the sensitivity and accuracy of diagnosis. To understand the relationship between the efficiency of conversion and the molecular structure, structure analysis of the title compound was undertaken.

The molecule as a whole takes a butterfly form. The angles at the N atom in the phenothiazine ring sum to 357 (1)° indicating a nearly planar conformation at the atom. The N—C(=O) bond distance of 1.390 (4) Å, however, reveals that electron delocalization between the N atom and the carbonyl group of the methylcarbamoyl group is not so significant. Therefore, breaking this N—C bond is relatively easy and results in blue coloration. The amino moiety of the methylcarbamoyl group is almost parallel to the phenothiazine ring. The torsion angles N12—C11—N10—C1*a* and O11—C11—N10—C5*a* are −5.0 (4) and 15.0 (4)°, respectively. The sums of the bond angles around N3 and N7 atoms are 356 (1) and 360 (1)°, respectively, but the terminal dimethylamino groups are not coplanar with the attached phenyl plane. Bond distances and angles in the molecule are within the expected range, but those in the conjugate system are quite different from those observed in methylene blue pentahydrate (Marr & Stewart, 1973) and methylene blue thiocyanate (Khan-Harari, Ballard & Norris, 1973).

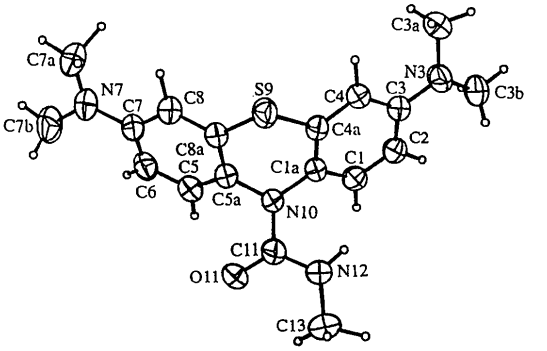

Fig. 1. *ORTEP*II drawing (Johnson, 1976) of the molecule, representing heavy atoms as 30% probability ellipsoids and H atoms as spheres of arbitrary radii.

Experimental

Crystal data

$C_{18}H_{22}N_4OS.C_2H_6O$
M_r = 388.53
Monoclinic
$P2_1/n$
a = 8.6406 (8) Å
b = 17.151 (1) Å
c = 14.542 (1) Å
β = 106.106 (8)°
V = 2070.5 (3) Å^3
Z = 4
D_x = 1.25 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 23 reflections
θ = 30–35°
μ = 1.522 mm^{-1}
T = 298 (2) K
Rod
0.5 × 0.4 × 0.3 mm
Ice blue

Data collection

Enraf–Nonius CAD-4 Turbo diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ-scan
T_{min} = 0.871, T_{max} = 0.998
2910 observed reflections [$F > 3\sigma(F)$]
R_{int} = 0.036
θ_{max} = 75.1°
$h = -9 \rightarrow 10$
$k = -20 \rightarrow 0$
$l = 0 \rightarrow 17$

3934 measured reflections
3638 independent reflections

3 standard reflections
frequency: 50 min
intensity variation: −8.5%

Refinement

Refinement on F
Final R = 0.060
wR = 0.082
S = 2.87
2776 reflections
333 parameters
Refinement of all H atoms except those in ethanol
$w = 1/\sigma$
$(\Delta/\sigma)_{max}$ = 0.01

$\Delta\rho_{max}$ = 0.47 (7) e Å^{-3}
$\Delta\rho_{min}$ = −0.42 (7) e Å^{-3}
Extinction correction: $|F_c|/(1+gI_c)$
Extinction coefficient: 2.73×10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$B_{eq} = (4/3)[a^2B(1,1)+b^2B(2,2)+c^2B(3,3)+ab(\cos\gamma)B(1,2) +ac(\cos\beta)B(1,3)+bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
C1	1.2153 (4)	0.3426 (2)	0.9046 (2)	4.26 (6)
C1*a*	1.0809 (3)	0.3632 (2)	0.8318 (2)	3.79 (6)
C2	1.2825 (4)	0.3928 (2)	0.9780 (2)	4.19 (6)
C3	1.2226 (3)	0.4682 (2)	0.9796 (2)	3.86 (6)
N3	1.2903 (3)	0.5195 (1)	1.0535 (2)	4.75 (6)
C3*a*	1.2596 (4)	0.6019 (2)	1.0398 (2)	5.43 (8)
C3*b*	1.4331 (4)	0.4970 (2)	1.1267 (2)	5.17 (7)
C4	1.0904 (3)	0.4902 (2)	0.9033 (2)	3.81 (6)
C4*a*	1.0200 (3)	0.4374 (2)	0.8318 (2)	3.66 (6)
C5	0.9981 (4)	0.3043 (2)	0.5842 (2)	4.73 (7)
C5*a*	0.9617 (4)	0.3422 (2)	0.6610 (2)	4.05 (6)
C6	0.9476 (4)	0.3362 (2)	0.4924 (2)	5.03 (7)
C7	0.8636 (4)	0.4059 (2)	0.4748 (2)	5.12 (7)
N7	0.8098 (4)	0.4367 (2)	0.3835 (2)	6.53 (8)
C7*a*	0.7325 (5)	0.5112 (2)	0.3681 (2)	7.1 (1)
C7*b*	0.8211 (5)	0.3927 (3)	0.3031 (2)	7.7 (1)
C8	0.8366 (4)	0.4468 (2)	0.5537 (2)	4.73 (7)
C8*a*	0.8862 (4)	0.4142 (2)	0.6443 (2)	4.10 (6)
S9	0.84843 (9)	0.46599 (4)	0.74065 (5)	4.57 (2)
N10	1.0060 (3)	0.3102 (1)	0.7553 (1)	4.14 (5)
C11	0.9361 (4)	0.2401 (2)	0.7703 (2)	4.33 (6)
O11	0.8658 (3)	0.1990 (1)	0.7022 (1)	5.41 (5)
N12	0.9509 (3)	0.2196 (1)	0.8612 (2)	5.25 (6)
C13	0.8893 (5)	0.1450 (2)	0.8838 (3)	6.7 (1)
O1(solv.)	0.5658 (4)	0.2388 (2)	0.5697 (2)	8.60 (8)
C1(solv.)	0.4956 (8)	0.2949 (4)	0.7008 (4)	14.2 (2)
C2(solv.)	0.4829 (9)	0.2626 (6)	0.6266 (4)	18.8 (3)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

C1—C1*a*	1.383 (4)	C5*a*—N10	1.427 (3)
C1—C2	1.370 (4)	C6—C7	1.385 (4)
C1*a*—C4*a*	1.379 (4)	C7—N7	1.383 (4)
C1*a*—N10	1.443 (3)	C7—C8	1.418 (4)
C2—C3	1.396 (4)	N7—C7*a*	1.431 (5)
C3—N3	1.387 (3)	N7—C7*b*	1.417 (5)
C3—C4	1.405 (3)	C8—C8*a*	1.385 (4)
N3—C3*a*	1.441 (4)	C8*a*—S9	1.763 (3)
N3—C3*b*	1.441 (4)	N10—C11	1.390 (4)
C4—C4*a*	1.385 (3)	C11—O11	1.231 (3)
C4*a*—S9	1.762 (2)	C11—N12	1.339 (4)
C5—C5*a*	1.402 (4)	N12—C13	1.457 (5)
C5—C6	1.395 (4)	C5*a*—C8*a*	1.387 (4)
C1—C1*a*—N10	122.1 (2)	C4*a*—C1*a*—N10	119.5 (2)
C2—C3—N3	121.5 (2)	N3—C3—C4	121.2 (2)
C3—N3—C3*a*	119.6 (2)	C3—N3—C3*b*	119.5 (2)
C3*a*—N3—C3*b*	116.9 (2)	C1*a*—C4*a*—S9	119.9 (2)
C4—C4*a*—S9	119.1 (2)	C5—C5*a*—N10	121.9 (2)
C8*a*—C5*a*—N10	119.9 (2)	C6—C7—N7	121.8 (3)
N7—C7—C8	120.1 (3)	C7—N7—C7*a*	120.6 (3)
C7—N7—C7*b*	120.5 (3)	C7*a*—N7—C7*b*	118.8 (3)
C5*a*—C8*a*—S9	119.4 (2)	C8—C8*a*—S9	118.5 (2)
C4*a*—S9—C8*a*	99.0 (1)	C1*a*—N10—C5*a*	116.3 (2)
C1*a*—N10—C11	123.0 (2)	C5*a*—N10—C11	118.1 (2)
N10—C11—O11	120.5 (3)	N10—C11—N12	117.2 (2)
O11—C11—N12	122.4 (3)	C11—N12—C13	120.8 (2)

The crystals were grown from an ethanol solution at 281 K. A crystal sealed in a glass capillary with N_2 gas was used for the diffraction experiments. An anisotropic decay correction was applied and refinement was by full-matrix least-squares methods.

Program used throughout the analysis: *MolEN* (Fair, 1990). Program used to solve structure: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Program used to draw figure: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71114 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1005]

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Structure of 5,5′,6,6′-Tetraphenyl-3,3′-bi-1,2,4-triazine

J. Breu and K.-J. Range

Institut für Anorganische Chemie der Universität Regensburg, Universitätsstraße 31, D-8400 Regensburg, Germany

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Abstract

The title compound (BDT) crystallizes in an *s-trans* conformation, the inversion center lying on the bond