

- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
- Shimizu, S., Kashino, S. & Haisa, M. (1975). *Acta Cryst.* **B31**, 1287–1292.
- Thappa, R. K. (1989). Personal communication.

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## Bis[4-(dimethylamino)phenyl](*N*-methylcarbamoyl)amine, C<sub>18</sub>H<sub>24</sub>N<sub>4</sub>O

ISAO FUJII AND NORIAKI HIRAYAMA

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

NORIHITO AOYAMA AND AKIRA MIIKE

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

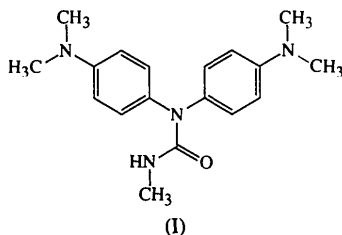
(Received 5 January 1995; accepted 10 May 1995)

### Abstract

The two phenyl rings in the title compound, *N,N*-bis[4-(dimethylamino)phenyl]-*N'*-methylurea, are almost perpendicular to one another, with dihedral angles of 79.9 (4) and 89.2 (4)° for the two crystallographically independent molecules in the asymmetric unit.

### Comment

In the presence of peroxidase and hydrogen peroxide, the title compound, (I), is converted into 4,4'-bis(dimethylamino)diphenylamine (Bindshedler's green leuco base) (Cheng, Ueno & Imamura, 1982; Tichý & Petter, 1984) and a blue colour is developed. Therefore, the molecule can be applicable clinically as a functional dye used to measure the activity of monoamine oxidase



in blood. Effective conversion is essential for the sensitivity and accuracy of the clinical diagnosis. The structure analysis of the title compound was undertaken in order to discover the structure–function relationships.

The two crystallographically independent molecules in the asymmetric unit have similar conformations. The molecules as a whole have propeller forms as opposed to the butterfly form observed in a similar functional dye (MCDP; Fujii, Hirayama & Miike, 1993). The angles at the hinge N atoms total 359.3 (9) and 359.8 (9)°, indicating a very planar structure at the N atom. The N–C(=O) distances of 1.400 (4) and 1.383 (4) Å in the independent molecules are significantly longer than the corresponding distances in the *N*-methylcarbamoyl group, indicating that this group may be a good leaving group due to the bond difference. The sum of the bond angles around atoms N4 and N4' indicates that the terminal dimethylamino groups are very planar even though they are not coplanar with the phenyl rings to which they are attached. The conjugated system in the molecule can be schematically represented by formula (II) below.

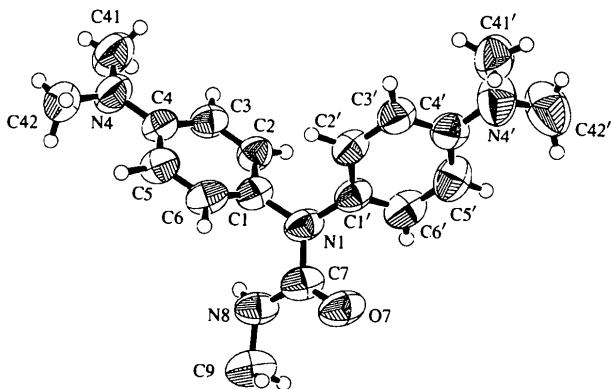
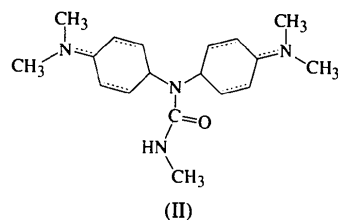


Fig. 1. ORTEPII drawing (Johnson, 1976) of molecule A, showing heavy atoms with 50% probability ellipsoids and H atoms as circles of arbitrary radii.

### Experimental

The crystals were grown from an ethanol solution at 281 (2) K. A crystal sealed in a glass capillary filled with nitrogen gas was used for the diffraction experiments.

#### Crystal data

C<sub>18</sub>H<sub>24</sub>N<sub>4</sub>O  
M<sub>r</sub> = 312.41

Cu Kα radiation  
λ = 1.54184 Å

Monoclinic  
 $P2_1/n$   
 $a = 21.811 (7) \text{ \AA}$   
 $b = 10.177 (1) \text{ \AA}$   
 $c = 16.006 (2) \text{ \AA}$   
 $\beta = 99.91 (2)^\circ$   
 $V = 3500 (1) \text{ \AA}^3$   
 $Z = 8$   
 $D_x = 1.186 \text{ Mg m}^{-3}$

Cell parameters from 25 reflections  
 $\theta = 30\text{--}35^\circ$   
 $\mu = 0.602 \text{ mm}^{-1}$   
 $T = 298 (2) \text{ K}$   
Prism  
 $0.5 \times 0.4 \times 0.3 \text{ mm}$   
Yellow

C2B	0.2721 (2)	-0.0840 (4)	0.9079 (2)	5.7 (1)
C2'B	0.3039 (2)	-0.2341 (4)	0.7228 (2)	6.3 (1)
C3B	0.2149 (2)	-0.0615 (4)	0.9313 (2)	5.8 (1)
C3'B	0.3052 (2)	-0.2070 (4)	0.6375 (2)	6.7 (1)
C4B	0.1765 (2)	-0.1633 (4)	0.9471 (2)	6.0 (1)
C4'B	0.3574 (2)	-0.1549 (4)	0.6124 (2)	6.2 (1)
C5B	0.1984 (2)	-0.2921 (5)	0.9382 (2)	6.4 (1)
C5'B	0.4079 (2)	-0.1265 (4)	0.6762 (2)	6.6 (1)
C6B	0.2545 (2)	-0.3126 (5)	0.9139 (2)	6.3 (1)
C6'B	0.4058 (2)	-0.1514 (4)	0.7607 (2)	6.0 (1)
C7B	0.4009 (2)	-0.2880 (4)	0.9251 (2)	5.6 (1)
C9B	0.4435 (2)	-0.3574 (6)	1.0689 (3)	8.0 (1)
C41B	0.1002 (2)	-0.0109 (8)	0.9835 (4)	11.9 (2)
C41'B	0.3067 (3)	-0.1624 (7)	0.4643 (3)	9.9 (2)
C42B	0.0806 (2)	-0.2508 (7)	0.9850 (4)	11.8 (2)
C42'B	0.4188 (3)	-0.1201 (7)	0.5010 (3)	10.7 (2)

## Data collection

Enraf-Nonius CAD-4 Turbo diffractometer  
 $\omega/2\theta$  scans  
Absorption correction: none  
5558 measured reflections  
5493 independent reflections  
3051 observed reflections  
 $[F > 3\sigma(F)]$

$R_{\text{int}} = 0.073$   
 $\theta_{\text{max}} = 59.98^\circ$   
 $h = 0 \rightarrow 24$   
 $k = -11 \rightarrow 0$   
 $l = -17 \rightarrow 17$   
3 standard reflections  
frequency: 50 min  
intensity decay: 10.8%

## Refinement

Refinement on  $F$   
 $R = 0.055$   
 $wR = 0.068$   
 $S = 2.85$   
3051 reflections  
575 parameters  
 $w = 1/\sigma^2(F)$   
 $(\Delta/\sigma)_{\text{max}} = 0.02$   
 $\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

Extinction correction:  
 $|F_c|/(1 + gI_c)$   
Extinction coefficient:  
 $3.86 \times 10^{-6}$

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

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$B_{\text{eq}}$
O7A	0.5753 (1)	-0.1915 (3)	0.6908 (2)	8.2 (1)
N1A	0.5907 (1)	-0.1346 (3)	0.8311 (2)	6.3 (1)
N4A	0.6159 (2)	-0.2878 (4)	1.1737 (2)	7.5 (1)
N4'A	0.7034 (2)	0.3510 (5)	0.7814 (3)	9.8 (1)
N8A	0.5365 (1)	-0.3231 (3)	0.7829 (2)	6.5 (1)
C1A	0.5963 (2)	-0.1769 (4)	0.9180 (2)	5.5 (1)
C1'A	0.6187 (2)	-0.0129 (4)	0.8159 (2)	5.8 (1)
C2A	0.5743 (2)	-0.0983 (4)	0.9764 (2)	5.8 (1)
C2'A	0.6752 (2)	0.0228 (4)	0.8631 (2)	6.3 (1)
C3A	0.5813 (2)	-0.1339 (4)	1.0597 (2)	5.9 (1)
C3'A	0.7030 (2)	0.1411 (5)	0.8528 (2)	6.6 (1)
C4A	0.6099 (2)	-0.2494 (4)	1.0901 (2)	5.7 (1)
C4'A	0.6737 (2)	0.2320 (4)	0.7928 (2)	6.6 (1)
C5A	0.6320 (2)	-0.3297 (4)	1.0305 (2)	6.4 (1)
C5'A	0.6167 (2)	0.1963 (5)	0.7459 (3)	7.5 (1)
C6A	0.6262 (2)	-0.2902 (5)	0.9469 (2)	6.4 (1)
C6'A	0.5895 (2)	0.0783 (5)	0.7582 (2)	7.2 (1)
C7A	0.5673 (2)	-0.2179 (4)	0.7635 (2)	6.5 (1)
C9A	0.5058 (2)	-0.4120 (6)	0.7190 (3)	8.1 (1)
C41A	0.5910 (3)	-0.2087 (6)	1.2331 (3)	9.5 (2)
C41'A	0.7649 (3)	0.3728 (6)	0.8175 (3)	10.9 (2)
C42A	0.6418 (3)	-0.4114 (5)	1.2024 (3)	7.9 (1)
C42'A	0.6700 (3)	0.4511 (7)	0.7324 (5)	11.8 (2)
O7B	0.4470 (1)	-0.3289 (3)	0.8986 (1)	6.6 (1)
N1B	0.3509 (1)	-0.2331 (3)	0.8718 (2)	5.6 (1)
N4B	0.1193 (2)	-0.1413 (4)	0.9674 (2)	9.0 (1)
N4'B	0.3601 (2)	-0.1251 (4)	0.5279 (2)	8.3 (1)
N8B	0.3957 (2)	-0.2977 (4)	1.0071 (2)	6.9 (1)
C1B	0.2929 (1)	-0.2096 (4)	0.8992 (2)	5.3 (1)
C1'B	0.3540 (2)	-0.2058 (3)	0.7847 (2)	5.3 (1)

$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N1A—C1A	1.441 (4)	N1B—C1B	1.428 (4)
N1A—C1'A	1.421 (4)	N1B—C1'B	1.436 (4)
N1A—C7A	1.400 (4)	N1B—C7B	1.383 (4)
N4A—C4A	1.378 (4)	N4B—C4B	1.361 (4)
N4A—C41A	1.423 (6)	N4B—C41B	1.428 (7)
N4A—C42A	1.423 (6)	N4B—C42B	1.455 (6)
N4'A—C4'A	1.400 (5)	N4'B—C4'B	1.397 (4)
N4'A—C41'A	1.385 (6)	N4'B—C41'B	1.458 (6)
N4'A—C42'A	1.409 (7)	N4'B—C42'B	1.421 (6)
C7A—O7A	1.235 (4)	C7B—O7B	1.227 (4)
C7A—N8A	1.329 (5)	C7B—N8B	1.341 (4)
N8A—C9A	1.442 (5)	N8B—C9B	1.441 (5)
C1A—N1A—C1'A	117.3 (3)	C1B—N1B—C1'B	117.3 (3)
C1A—N1A—C7A	121.6 (3)	C1B—N1B—C7B	121.7 (3)
C1'A—N1A—C7A	120.4 (3)	C1'B—N1B—C7B	120.8 (3)
N1A—C1A—C2A	120.0 (3)	N1B—C1B—C2B	120.8 (3)
N1A—C1A—C6A	122.4 (3)	N1B—C1B—C6B	121.2 (3)
N1A—C1'A—C2'A	120.4 (3)	N1B—C1'B—C2'B	119.6 (3)
N1A—C1'A—C6'A	122.4 (3)	N1B—C1'B—C6'B	122.1 (3)
N4A—C4A—C3A	123.2 (3)	N4B—C4B—C3B	121.9 (4)
N4A—C4A—C5A	120.5 (4)	N4B—C4B—C5B	121.1 (4)
C4A—N4A—C41A	120.3 (4)	C4B—N4B—C41B	120.3 (4)
C4A—N4A—C42A	122.1 (4)	C4B—N4B—C42B	120.4 (4)
C41A—N4A—C42A	117.4 (4)	C41B—N4B—C42B	118.7 (4)
N4'A—C4'A—C3'A	119.6 (4)	N4'B—C4'B—C3'B	122.8 (4)
N4'A—C4'A—C5'A	123.2 (4)	N4'B—C4'B—C5'B	120.2 (3)
C41'A—N4'A—C4'A	121.4 (4)	C41'B—N4'B—C4'B	117.2 (4)
C42'A—N4'A—C4'A	119.5 (4)	C42'B—N4'B—C4'B	119.5 (4)
C42'A—N4'A—C41'A	119.2 (5)	C42'B—N4'B—C41'B	116.7 (4)
N1A—C7A—O7A	120.3 (4)	N1B—C7B—O7B	122.0 (3)
N1A—C7A—N8A	116.0 (3)	N1B—C7B—N8B	116.3 (3)
O7A—C7A—N8A	123.7 (4)	O7B—C7B—N8B	121.7 (3)
C7A—N8A—C9A	122.1 (4)	C7B—N8B—C9B	122.0 (3)
C3A—C4A—N4A—C41A			1.5 (6)
C5A—C4A—N4A—C42A			-3.1 (6)
C3'A—C4'A—N4'A—C41'A			10.6 (6)
C5'A—C4'A—N4'A—C42'A			12.5 (7)
O7A—C7A—N1A—C1'A			6.0 (5)
N8A—C7A—N1A—C1A			16.3 (5)
C6A—C1A—N1A—C7A			52.3 (4)
C6'A—C1'A—N1A—C7A			52.2 (4)
O7A—C7A—N8A—C9A			-4.4 (6)
N1A—C7A—N8A—C9A			175.2 (3)
C3B—C4B—N4B—C41B			-7.9 (6)
C5B—C4B—N4B—C42B			3.4 (6)
C3'B—C4'B—N4'B—C41'B			-5.4 (6)
C5'B—C4'B—N4'B—C42'B			26.7 (6)
O7B—C7B—N1B—C1'B			-8.3 (5)
N8B—C7B—N1B—C1B			-10.7 (5)
C6B—C1B—N1B—C7B			-70.1 (4)
C6'B—C1'B—N1B—C7B			-42.5 (5)
O7B—C7B—N8B—C9B			-0.4 (6)
N1B—C7B—N8B—C9B			177.7 (4)

All non-H atoms were located by direct methods using the program SAPI91 (Fan, 1991). Most H atoms were found from difference Fourier maps and the positions of the remaining H atoms were calculated geometrically. All non-H atoms were

refined anisotropically and some of the H atoms were refined isotropically.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SAPI91* (Fan, 1991). Program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1992). Molecular graphics: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1171). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Cheng, K. L., Ueno, K. & Imamura, T. (1982). In *Handbook of Organic Analytical Reagents*. Boca Raton, Florida: CRC Press.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Fan, H.-F. (1991). *SAPI91. Structure Analysis Programs with Intelligent Control*. Rigaku Corporation, Tokyo, Japan.
- Fujii, I., Hirayama, N. & Miike, A. (1993). *Acta Cryst.* C49, 1540–1541.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Molecular Structure Corporation (1992). *TEXSAN. Single Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, TX 77381, USA.
- Tichý, K. & Petter, W. (1984). *Acta Cryst.* C40, 1055–1057.

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