Table 2. *Molecular geometry*

Bond distances (Å)

O(1)—N(1)	1·396 (2)	N(21)—C(9)	1·462 (3)
O(1)—C(6)	1·395 (3)	C(1)—C(2)	1·356 (3)
O(2)—N(21)	1·227 (3)	C(2)—C(3)	1·421 (3)
O(3)—N(21)	1·221 (3)	C(3)—C(4)	1·417 (3)
N(1)—C(1)	1·354 (3)	C(4)—C(5)	1·355 (3)
N(1)—C(5)	1·346 (3)	C(6)—C(7)	1·397 (3)
N(2)—C(6)	1·307 (3)	C(7)—C(8)	1·370 (4)
N(2)—C(10)	1·343 (3)	C(8)—C(9)	1·393 (3)
N(11)—C(3)	1·324 (3)	C(9)—C(10)	1·375 (3)

Bond angles (°)

N(1)—O(1)—C(6)	113·3 (2)	N(11)—C(3)—C(4)	121·1 (2)
O(1)—N(1)—C(5)	118·4 (2)	C(3)—C(4)—C(5)	120·2 (2)
O(1)—N(1)—C(1)	117·7 (2)	N(1)—C(5)—C(4)	119·3 (2)
C(1)—N(1)—C(5)	123·8 (2)	O(1)—C(6)—N(2)	119·4 (2)
C(6)—N(2)—C(10)	116·6 (2)	N(2)—C(6)—C(7)	126·9 (2)
O(2)—N(21)—O(3)	123·6 (2)	O(1)—C(6)—C(7)	113·7 (2)
O(3)—N(21)—C(9)	118·9 (2)	C(6)—C(7)—C(8)	116·3 (2)
O(2)—N(21)—C(9)	117·6 (2)	C(7)—C(8)—C(9)	117·7 (2)
N(1)—C(1)—C(2)	118·9 (2)	N(21)—C(9)—C(8)	119·5 (2)
C(1)—C(2)—C(3)	120·2 (2)	C(8)—C(9)—C(10)	121·4 (2)
N(11)—C(3)—C(2)	121·3 (2)	N(21)—C(9)—C(10)	119·1 (2)
C(2)—C(3)—C(4)	117·6 (2)	N(2)—C(10)—C(9)	121·1 (2)

Hydrogen-bond parameters (Å, °)

A—H···*B*	*A*—H	H···*B*	*A*···*B*	*A*—H···*B*
C(5)—H···Cl^{i}	0·94 (4)	2·48 (3)	3·380 (3)	159 (4)
C(4)—H···Cl^{ii}	0·88 (5)	2·73 (4)	3·494 (3)	146 (4)
N(11)—H(11)···Cl^{ii}	0·85 (4)	2·41 (4)	3·251 (3)	169 (4)
N(11)—H(12)···Cl^{iii}	1·04 (3)	2·23 (3)	3·260 (3)	172 (3)

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

CRYSRULER programs (Rizzoli, Sangermano, Calestani, Andreetti, 1986). The final atomic parameters are listed in Table 1,* bond distances, angles and hydrogen bonds in Table 2. Fig. 1 shows a view of the molecule with the numbering of the atoms.

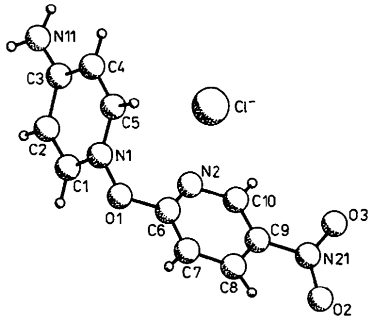

Fig. 1. A view of the molecule with the numbering of the atoms.

Related literature. Preparation of similar compounds is given in Kalinowski, Rykowski & Nantka-Namirski (1984).

Intensities were measured at the Institute of Technical Biochemistry, Technical University of Łódź. This work was supported by project R.P.II.10 from MEN.

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* 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 53490 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Kalinowski, J., Rykowski, A. & Nantka-Namirski, P. (1984). *Pol. J. Chem.* **58**, 125–134.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Rizzoli, C., Sangermano, V., Calestani, G. & Andreetti, G. D. (1986). *CRYSRULER.* Version 1.1, Polish version. Univ. degli Studi di Parma, Italy.

Sheldrick, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1991). C47, 1101–1103

Structure of 2,9-Dimethyl-1,10-phenanthroline Hemihydrate

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(*Received* 20 *June* 1990; *accepted* 9 *October* 1990)

Abstract. $C_{14}H_{12}N_2.\frac{1}{2}H_2O$, $M_r = 217·27$, tetragonal, $I4_1/a$, $a = 14·258$ (3), $c = 22·286$ (4) Å, $V = 4531$ (3) Å^3, $Z = 16$, $D_x = 1·274$ (1) g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71073$ Å, $\mu = 0·74$ cm^{-1}, $F(000) = 1840$, $T = 297$ K, $R = 0·041$ for 1196 unique observed reflections with $I > 2\sigma(I)$. Pairs of dimethylphenanthroline molecules related by a two-fold axis are bridged by water molecules lying on the

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
N1	0·1364 (1)	0·2438 (1)	0·84922 (7)	4·3 (1)
N2	0·1042 (1)	0·4310 (1)	0·86844 (8)	4·5 (1)
C1	0·1511 (2)	0·1535 (2)	0·8399 (1)	4·8 (1)
C2	0·1528 (2)	0·1141 (2)	0·7822 (1)	5·6 (1)
C3	0·1398 (2)	0·1700 (2)	0·7338 (1)	5·7 (1)
C4	0·1242 (2)	0·2658 (2)	0·7416 (1)	4·6 (1)
C5	0·1110 (2)	0·3287 (2)	0·6925 (1)	5·9 (1)
C6	0·0961 (2)	0·4204 (2)	0·7017 (1)	6·0 (1)
C7	0·0928 (2)	0·4578 (2)	0·7611 (1)	5·0 (1)
C8	0·0763 (2)	0·5528 (2)	0·7726 (1)	6·2 (2)
C9	0·0734 (2)	0·5842 (2)	0·8298 (1)	6·1 (1)
C10	0·0878 (2)	0·5213 (2)	0·8775 (1)	5·2 (1)
C11	0·1060 (1)	0·3989 (2)	0·8109 (1)	4·1 (1)
C12	0·1223 (1)	0·2999 (2)	0·80104 (9)	4·0 (1)
C13	0·1655 (2)	0·0933 (2)	0·8946 (1)	6·7 (2)
C14	0·0853 (2)	0·5531 (2)	0·9417 (1)	7·1 (2)
O11	0	0·25	0·9528 (1)	8·7 (2)
H1	0·051 (3)	0·269 (4)	0·921 (2)	19 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

N1—C1	1·322 (3)	C6—C7	1·427 (4)
N1—C12	1·353 (3)	C7—C8	1·399 (4)
N2—C10	1·325 (3)	C7—C11	1·404 (3)
N2—C11	1·362 (3)	C8—C9	1·352 (4)
C1—C2	1·405 (4)	C9—C10	1·406 (4)
C1—C13	1·504 (4)	C10—C14	1·501 (4)
C2—C3	1·355 (4)	C11—C12	1·448 (3)
C3—C4	1·394 (4)	O11—H1	1·06 (4)
C4—C12	1·411 (3)	N1—H1	2·03 (4)
C4—C5	1·427 (4)	N1—O11	3·020 (3)
C5—C6	1·341 (5)		

C1—N1—C12	118·3 (2)	C9—C8—C7	119·9 (3)
C10—N2—C11	118·3 (2)	C8—C9—C10	119·8 (2)
N1—C1—C2	122·4 (2)	N2—C10—C9	122·1 (2)
N1—C1—C13	116·8 (2)	N2—C10—C14	116·3 (2)
C2—C1—C13	120·8 (2)	C9—C10—C14	121·6 (2)
C3—C2—C1	119·4 (2)	N2—C11—C7	122·7 (2)
C2—C3—C4	120·0 (2)	N2—C11—C12	118·3 (2)
C3—C4—C12	117·2 (2)	C7—C11—C12	119·0 (2)
C3—C4—C5	122·7 (2)	N1—C12—C4	122·6 (2)
C12—C4—C5	120·1 (2)	N1—C12—C11	118·6 (2)
C6—C5—C4	121·0 (2)	C4—C12—C11	118·7 (2)
C5—C6—C7	120·8 (2)	H1—O11—H1′	95 (3)
C8—C7—C11	117·2 (2)	O11—H1—N1	154 (3)
C8—C7—C6	122·5 (2)	N1—O11—N1	80·3 (2)
C11—C7—C6	120·3 (2)		

twofold axis and H bonded to one of the N atoms in each molecule. The H bonds are long and far from linear: O—H 1·06 (4), H···N 2·03 (4), O···N 3·020 (3) Å; O—H···N 154 (3)°. This is presumably a consequence of the approximately parallel arrangement of the two phenanthroline molecules in the (phen)$_2$.H$_2$O complex, which are tilted 4·7 (1)° with respect to each other; the atoms in one molecule are 3·50 to 3·81 Å from the plane of the other molecule. On the other side of the phenanthroline is another phenanthroline related by a center of symmetry with the atoms of one molecule 3·41 to 3·45 Å from the plane of the other molecule. The phenanthroline molecule has close to 2*mm* symmetry, but the individual C$_6$ rings are tilted about 1° with respect to each other.

Experimental. High-quality crystals of the title compound were obtained as a by-product in the synthesis of metal complexes with the compound as a ligand. A regular octahedron, 0·50 × 0·50 × 0·50 mm, was used for the data collection. Data were collected on an Enraf–Nonius CAD-4 diffratometer equipped with a graphite monochromator. 25 reflections with $11 < \theta < 19°$ were used to determine the cell parameters. Systematic extinctions (*hkl*, $h+k+l$ odd; *hk*0, h odd; 00*l*, $l \neq 4n$) uniquely determined the space group. Data were collected, using ω scans, in the range $0 < \theta < 25°$ for one quadrant (ranges: h −16 to 16, k 0 to 16, l 0 to 26). No absorption corrections were made. 4255 reflections were measured and averaged to give 2048 independent reflections ($R_{\rm int}$ = 0·041) of which the 1196 with $I > 2\sigma(I)$ were used in the calculations. Three check reflections measured every 6000 s of exposure time showed no systematic change with time. A trial structure was found using the direct-methods program *MITHRIL* (Gilmore, 1984). All atoms except H were given anisotropic thermal parameters. The aromatic H atoms were included at idealized positions with isotropic thermal parameters 20% larger than the isotropic equivalent of the attached atoms. Since idealized positions could not be predicted for the H$_2$O and CH$_3$ H atoms; they were located from difference Fourier maps. The H$_2$O H atoms were refined as independent atoms with an isotropic thermal parameter. The CH$_3$ H atoms were refined as H$_3$ groups with idealized distances but with variable orientations and group isotropic thermal parameters. All parameters were refined by full-matrix least-squares techniques on F's. Refinement converged with R = 0·041, wR =

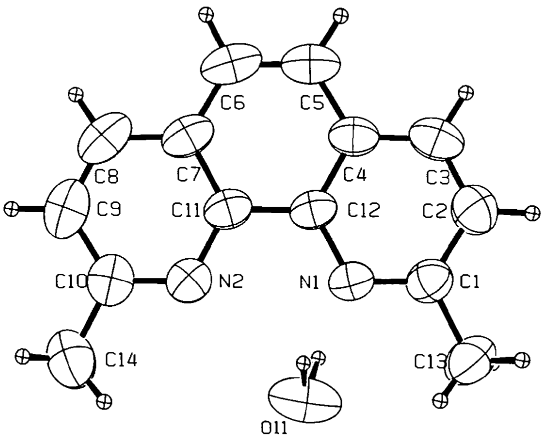

Fig. 1. (CH$_3$)$_2$C$_{12}$H$_6$N$_2$.$\frac{1}{2}$H$_2$O. Thermal ellipsoids are shown at the 50% probability level. The O11 atom lies on a twofold axis that in projection passes above the C4 atom. H atoms are shown with arbitrary size.

0·051, $S = 1{\cdot}72$; $w = 1/\sigma^2(F)$ was calculated from $\sigma^2(I) = \sigma^2(I_c) + (0{\cdot}04I)^2$, where $\sigma(I_c)$ is the standard deviation in I based on counting statistics alone. $(\Delta/\sigma)_{\max}$ in the final cycle was 0·06. $(\Delta\rho)_{\max} = 0{\cdot}17$, $(\Delta\rho)_{\min} = -0{\cdot}13$ e Å^{-3}. The four highest peaks in the final difference Fourier map are near the CH_3 groups suggesting that there is some disorder in their orientations. Atomic scattering factors and anomalous-dispersion corrections for all atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The computer programs used were from *TEXSAN* (Molecular Structure Corporation, 1985).

Atomic coordinates are given in Table 1 and interatomic distances and angles in Table 2.* The atomic labelling and thermal ellipsoids are shown in Fig. 1.

Related literature. Sen (1969) reported the same space group and unit cell for the title compound, dmp.$\frac{1}{2}H_2O$, although he did not recognize the presence of the water molecule. His experimental density, 1·27 g cm^{-3}, is in excellent agreement with our calculated value. Watson, Galloy, Vögtle & Müller (1984) have reported the structure of the 1/1 complex of dmp with resorcinol. The only other reported structure involving free dmp is that of $Zn(dmp)(CN)_2.dmp.3H_2O$ (Monge, Martínez-Ripoll & García-Blanco, 1978). The structure of the dmpH$^+$ ion has been reported in the $FeCl_4^-$ salt (Veidis, Witten, Reiff, Brennan & Garafalo, 1981).

There are also about two dozen structures reported where dmp is a ligand in a metal complex. The most recent of these are $Cu(dmp)(CH_3CN)^+$ (Munakata, Maekawa, Kitagama, Matsuyama & Masuda, 1989), Ru(dmp)(*o*-phen)$_2^{2+}$ (Ichida, Tachigashiki & Sasaki, 1989) and $Cu(dmp)(CN)_2^-$ (Ogura, Shemish, Scott, Pyrka & Fernando, 1988).

LCT would like to thank the Research Corporation for their support of this work.

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* Lists of anisotropic thermal parameters, H-atom parameters, intermolecular distances, least-squares planes, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53642 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

Ichida, H., Tachigashiki, S. & Sasaki, Y. (1989). *Chem. Lett.* pp. 1579–1580.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200A Research Forest Drive, The Woodlands, TX77381, USA.

Monge, A., Martínez-Ripoll, M. & García-Blanco, S. (1978). *Acta Cryst.* B**34**, 2847–2850.

Munakata, M., Maekawa, M., Kitagama, S., Matsuyama, S. & Masuda, H. (1989). *Inorg. Chem.* **289**, 4300–4302.

Ogura, T., Shemish, E., Scott, N., Pyrka, G. J. & Fernando, Q. (1988). *Inorg. Chim. Acta*, **149**, 57–62.

Sen, D. K. (1969). *Acta Cryst.* B**25**, 988.

Veidis, M. V., Witten, E. H., Reiff, W. M. Brennan, J. F. & Garafalo, A. R. (1981). *Inorg. Chim. Acta*, **54**, L133–L135.

Watson, W. H., Galloy, J., Vögtle, F. & Müller, W. M. (1984). *Acta Cryst.* C**40**, 200–202.

Acta Cryst. (1991). C**47**, 1103–1105

Structure of 7-Methoxy-2-(2-methoxyphenyl)-4*H*-1-benzopyran-4-one (2′,7-Dimethoxyflavone)

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(*Received* 23 *May* 1990; *accepted* 17 *September* 1990)

Abstract. $C_{17}H_{14}O_4$, $M_r = 282{\cdot}30$, monoclinic, $P2_1/c$, $a = 12{\cdot}498$ (4), $b = 9{\cdot}490$ (2), $c = 12{\cdot}184$ (2) Å, $\beta = 107{\cdot}87$ (2)°, $V = 1375{\cdot}4$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}36$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 7{\cdot}98$ cm^{-1}, $F(000) = 592$, $T = 291$ K, $R = 0{\cdot}042$ for 1848 observed reflections. The benzopyran heterocycle is

0108-2701/91/051103-03$03.00