Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl19

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Version of record first published: 24 Sep 2006.

To cite this article: M. A. Sridhar , A. Indira , S. B. Bellad , A. M. Babu , M. M. M. Abdoh , J. S. Prasad , P. G. Ramappa & K. G. Somasekharappa (1994): Crystal Structure of Diphenyl Pyraline Hydrochloride-Cobalt Chloride Complex, Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals, 250:1, 285-291

To link to this article: http://dx.doi.org/10.1080/10587259408028213

Mol. Cryst. Liq. Cryst., 1994, Vol. 250, pp. 285–291
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Printed in the United States of America

Crystal Structure of Diphenyl Pyraline Hydrochloride—Cobalt Chloride Complex

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(Received October 8, 1993; in final form November 8, 1993)

The crystal structure of the complex of diphenyl pyraline hydrochloride with cobalt chloride, $C_{38}H_{46}O_2N_2Cl_4Co$ has been determined by X-ray diffraction using CuKα radiation. The compound crystallizes in monoclinic space group $P2_1/n$ with the cell parameters $a = 18.120(4)$Å, $b = 8.179(3)$Å, $c = 26.805(3)$Å, $\beta = 102.60(1)°$ with $Z = 4$, $V = 3876(1)$Å^3. The structure was solved using 5484 reflections. The stucture was refined to a final wR of 0.2944 ($R = 0.1130$). The packing of the molecules shows a layered arrangement.

Keywords: Crystal structure, diphenyl pyraline hydrochloride, cobalt chloride

INTRODUCTION

In this paper, we report the results of the structural analysis of a complex of an antihistamine drug, diphenyl pyraline hydrochloride (DPH). The chemical name of DPH is 4-diphenylmethoxy-1-methyl-piperdine hydrochloride. It has found extensive use as a medical agent[1–7] and as an antioxidant in industry.[8–9] Its fungicidal activity has been studied.[10,11] The crystal structure of DPH is reported earlier.[12] In this paper the crystal structure of the complex of cobalt chloride with DPH is reported. The complexes of Co(II) with DPH are found to have increased fungicidal activity beyond a certain concentration level.[13] The fungicidal activity of these complexes were checked on *A alternaria alternata*, a fungus isolated from sunflower plants.

EXPERIMENTAL

The complex was synthesized by mixing hot ethanolic solutions of Co(II) salt with DPH in 1:2 stoichiometry. The solid formed under slow evaporation was filtered and washed in ethanol and dried completely in vacuum. Good single crystals of blue color were obtained from ethanol with a meager amount of water by slow evaporation. The

TABLE I

Crystal Data

Empirical formula	$C_{38}H_{46}O_2N_2Cl_4Co$
Formula weight	763.5
Temperature	293 (2) K
Radiation and wavelength	CuKα, 1.54060 Å
Crystal system	Monoclinic
Space group	P $2_1/n$
Unit cell dimensions	$a = 18.120\ (4)$ Å $b = 8.179\ (2)$ Å, $\beta = 102.60\ (1)°$ $c = 26.805\ (3)$ Å
Volume	3876 (1) Å^3
Z	4
Density (calculated)	1.308 Mg/m^3
μ	6.269 mm^{-1}
F_{000}	1596
Crystal size	0.50 × 0.40 × 0.30 mm
θ range for data collection	2.69° – 62.21°
Index ranges	$-20 \leq h \leq 19, 0 \leq k \leq 8, 0 \leq l \leq 29$
Independent reflections	5484

cell dimensions were refined using 25 reflections with $40° \leq 2\theta \leq 50°$ measured using CuKα radiation on a CAD4 diffractometer. The crystal data are given in Table I.

STRUCTURE DETERMINATION AND REFINEMENT

Intensity data were collected on an Enraf Nonius CAD4 diffractometer within a 2θ limit of 125°. A total of 5758 counter intensities were measured employing $\omega - 2\theta$ scan mode out of which 5484 were unique. Lorentz and polarization corrections were

TABLE II

Details of the Final Least-Squares Cycle

Refinement	Full-matrix least-squares on F^2
Function minimized	$\sqrt{\frac{\sum w(F_o^2 - F_c^2)^2}{\sum w(F_o^2)^2}}$
Least-squares weight	Counting statistics
Number of reflections	5484 reflections
Number of atoms	93
Number of parameters	439
R (all reflections)	0.1956
R (observed reflections)	0.1130
Observation criterion	$I > 2\sigma(I)$
wR (all reflections)	0.3327
wR (observed reflections)	0.2944
Observation criterion	$I > -3\sigma(I)$
Goodness of fit (all)	0.970
Maximum shift/sigma	4.421
Extinction coefficient	0.0002 (2)
Maximum and minimum peak in the final difference map	1.14 and -0.81 e Å^{-3}

TABLE III

Atomic coordinates and equivalent thermal parameters (Å^2) for non-hydrogens. *Ueq* is defined as one third of the trace of the Orthogonalized *Uij* tensor

Atom	*x*	*y*	*z*	*Ueq*
Co1	0.05757 (11)	0.6432 (3)	0.14215 (7)	0.0535 (7)
Cl1	0.0273 (2)	0.3833 (4)	0.11328 (12)	0.0689 (11)
Cl2	−0.0390 (2)	0.7327 (4)	0.17624 (13)	0.0688 (10)
Cl3	0.0656 (2)	0.8135 (4)	0.07724 (13)	0.0691 (11)
Cl4	0.1688 (2)	0.6245 (5)	0.19932 (13)	0.0755 (12)
C1	−0.0802 (11)	0.113 (3)	0.4488 (9)	0.108 (7)
C2	−0.0375 (10)	0.249 (3)	0.4586 (7)	0.104 (6)
C3	−0.0362 (8)	0.359 (2)	0.4183 (6)	0.078 (5)
C4	−0.0814 (8)	0.326 (2)	0.3703 (5)	0.064 (4)
C5	−0.1247 (9)	0.191 (2)	0.3642 (8)	0.095 (6)
C6	−0.1241 (13)	0.081 (3)	0.4044 (10)	0.112 (8)
C7	−0.2550 (9)	0.815 (2)	0.3083 (7)	0.091 (5)
C8	−0.2038 (9)	0.809 (2)	0.2756 (7)	0.083 (5)
C9	−0.1508 (9)	0.688 (2)	0.2808 (6)	0.077 (5)
C10	−0.1457 (8)	0.573 (2)	0.3212 (6)	0.074 (4)
C11	−0.1978 (9)	0.584 (2)	0.3510 (6)	0.088 (5)
C12	−0.2513 (10)	0.703 (2)	0.3442 (7)	0.102 (6)
C13	−0.0874 (14)	0.442 (2)	0.3256 (7)	0.126 (11)
O1	−0.0681 (7)	0.401 (2)	0.2867 (5)	0.122 (5)
C14	−0.0084 (7)	0.298 (2)	0.2809 (5)	0.069 (4)
C15	−0.0326 (9)	0.223 (2)	0.2282 (5)	0.086 (5)
C16	0.0258 (7)	0.120 (2)	0.2132 (6)	0.073 (4)
N1	0.0985 (6)	0.2133 (12)	0.2181 (3)	0.050 (3)
C17	0.1239 (7)	0.277 (2)	0.2708 (5)	0.061 (4)
C18	0.0653 (7)	0.388 (2)	0.2850 (5)	0.066 (4)
C19	0.1570 (8)	0.116 (2)	0.2015 (5)	0.080 (5)
C20	0.4340 (9)	0.320 (2)	0.1731 (6)	0.069 (4)
C21	0.3619 (9)	0.322 (2)	0.1802 (5)	0.061 (4)
C22	0.3048 (8)	0.4031 (13)	0.1426 (5)	0.054 (4)
C23	0.3228 (7)	0.4771 (13)	0.1023 (4)	0.039 (3)
C24	0.3963 (7)	0.473 (2)	0.0959 (5)	0.060 (4)
C25	0.4539 (8)	0.395 (2)	0.1319 (6)	0.073 (4)
C26	0.3340 (10)	1.043 (3)	0.0356 (9)	0.109 (7)
C27	0.3395 (8)	0.929 (2)	0.0023 (7)	0.089 (6)
C28	0.3156 (7)	0.771 (2)	0.0112 (5)	0.061 (4)
C29	0.2841 (7)	0.737 (2)	0.0519 (5)	0.050 (3)
C30	0.2759 (8)	0.860 (2)	0.0861 (6)	0.078 (5)
C31	0.3000 (10)	1.015 (2)	0.0779 (8)	0.115 (7)
C32	0.2612 (7)	0.5668 (14)	0.0624 (4)	0.051 (3)
O2	0.2448 (4)	0.4618 (10)	0.0188 (3)	0.050 (2)
C33	0.1730 (6)	0.495 (2)	−0.0160 (4)	0.059 (4)
C34	0.1762 (7)	0.424 (2)	−0.0671 (5)	0.058 (4)
C35	0.1835 (7)	0.240 (2)	−0.0652 (5)	0.064 (4)
N2	0.1163 (6)	0.1697 (12)	−0.0481 (4)	0.057 (3)
C36	0.1149 (8)	0.230 (2)	0.0051 (5)	0.063 (4)
C37	0.1088 (7)	0.421 (2)	0.0038 (5)	0.060 (4)
C38	0.1187 (9)	−0.014 (2)	−0.0481 (6)	0.088 (5)

applied. Absorption correction was not applied. The number of reflections with $I > 2\sigma(I)$ was 2479. The Wilson plot[14] gave the value of the mean-square amplitude of the atomic vibration as 0.059 Å^2. The structure was solved using SHELXS-86.[15] The hydrogens atoms were placed in calculated positions and refined using the riding

model. Weights based on counting statistics were used. Extinction coefficient was also refined.[17] Refinement was carried out on F^2 for all the 5484 reflections. Weighted residual factors wR and all goodnesses of fit are based on F^2. The conventional R factors are based of F, with F set to zero for negative F^2. The details of the last least-squares refinement and the subsequent difference Fourier map are given in Table II.

TABLE IV

Anisotropic Displacement Parameters (Å^2) for Non-Hydrogen

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
Co1	0.0574 (13)	0.0556 (14)	0.0433 (11)	0.0037 (10)	0.0016 (9)	0.0071 (11)
Cl1	0.099 (3)	0.049 (2)	0.052 (2)	0.000 (2)	0.001 (2)	0.004 (2)
Cl2	0.060 (2)	0.079 (2)	0.066 (2)	−0.012 (2)	0.010 (2)	0.003 (2)
Cl3	0.066 (2)	0.075 (3)	0.063 (2)	0.020 (2)	0.006 (2)	−0.008 (2)
Cl4	0.056 (2)	0.102 (3)	0.059 (2)	−0.005 (2)	−0.007 (2)	0.011 (2)
C1	0.083 (14)	0.12 (2)	0.13 (2)	0.04 (2)	0.039 (13)	0.006 (13)
C2	0.079 (12)	0.16 (2)	0.072 (13)	0.015 (14)	0.007 (11)	0.031 (13)
C3	0.056 (9)	0.090 (13)	0.098 (12)	−0.024 (11)	0.037 (9)	−0.022 (9)
C4	0.065 (9)	0.069 (10)	0.058 (9)	0.000 (8)	0.015 (8)	0.003 (8)
C5	0.67 (10)	0.10 (2)	0.11 (2)	−0.004 (14)	0.004 (11)	−0.014 (11)
C6	0.09 (2)	0.12 (2)	0.14 (2)	0.00 (2)	0.06 (2)	−0.003 (14)
C7	0.065 (11)	0.088 (13)	0.110 (14)	0.014 (11)	−0.003 (11)	0.008 (10)
C8	0.079 (11)	0.059 (9)	0.100 (13)	0.027 (9)	−0.003 (10)	0.018 (9)
C9	0.088 (11)	0.074 (11)	0.062 (10)	0.017 (9)	0.001 (8)	0.001 (9)
C10	0.060 (9)	0.082 (10)	0.079 (11)	−0.006 (9)	0.011 (9)	0.019 (8)
C11	0.081 (11)	0.084 (12)	0.103 (3)	0.012 (10)	0.026 (10)	0.029 (10)
C12	0.093 (13)	0.12 (2)	0.090 (13)	0.017 (12)	0.019 (11)	0.032 (12)
C13	0.19 (3)	0.10 (2)	0.10 (2)	0.067 (14)	0.09 (2)	0.11 (2)
O1	0.135 (10)	0.161 (12)	0.097 (9)	0.072 (9)	0.086 (8)	0.086 (9)
C14	0.072 (9)	0.077 (10)	0.067 (10)	0.033 (8)	0.034 (8)	0.018 (8)
C15	0.072 (11)	0.12 (2)	0.063 (9)	0.021 (10)	0.010 (8)	−0.016 (11)
C16	0.077 (9)	0.068 (10)	0.66 (10)	0.011 (8)	−0.004 (8)	−0.018 (8)
N1	0.050 (7)	0.055 (7)	0.041 (6)	−0.002 (5)	0.001 (5)	0.001 (6)
C17	0.052 (8)	0.043 (9)	0.078 (9)	0.005 (7)	−0.005 (7)	−0.003 (7)
C18	0.076 (9)	0.064 (10)	0.058 (9)	0.004 (8)	0.015 (8)	0.001 (8)
C19	0.092 (10)	0.060 (11)	0.083 (11)	−0.005 (9)	0.012 (9)	0.019 (8)
C20	0.081 (12)	0.040 (8)	0.074 (10)	−0.010 (7)	−0.011 (9)	0.000 (8)
C21	0.097 (11)	0.043 (8)	0.041 (8)	0.007 (6)	0.008 (8)	−0.004 (8)
C22	0.071 (10)	0.032 (7)	0.059 (8)	−0.013 (6)	0.018 (8)	−0.017 (7)
C23	0.049 (8)	0.034 (7)	0.037 (7)	−0.011 (5)	0.018 (6)	−0.006 (6)
C24	0.069 (9)	0.057 (9)	0.053 (8)	0.001 (7)	0.013 (7)	−0.004 (7)
C25	0.060 (10)	0.083 (12)	0.071 (10)	0.002 (9)	0.006 (8)	−0.002 (9)
C26	0.090 (13)	0.08 (2)	0.13 (2)	0.019 (14)	−0.045 (12)	−0.039 (11)
C27	0.060 (10)	0.072 (12)	0.11 (2)	0.025 (12)	−0.029 (10)	−0.014 (9)
C28	0.049 (7)	0.036 (8)	0.090 (10)	−0.003 (7)	0.001 (7)	−0.001 (6)
C29	0.048 (7)	0.040 (8)	0.053 (8)	−0.010 (7)	−0.007 (6)	−0.005 (6)
C30	0.083 (10)	0.058 (10)	0.073 (10)	−0.023 (8)	−0.028 (8)	0.024 (8)
C31	0.109 (14)	0.040 (11)	0.15 (2)	−0.042 (11)	−0.067 (13)	0.009 (9)
C32	0.042 (7)	0.051 (8)	0.056 (7)	−0.022 (6)	0.001 (6)	0.007 (6)
O2	0.051 (5)	0.046 (5)	0.050 (5)	−0.008 (4)	0.001 (4)	−0.003 (4)
C33	0.057 (8)	0.053 (9)	0.055 (8)	0.007 (7)	−0.016 (6)	0.009 (7)
C34	0.048 (8)	0.064 (9)	0.057 (8)	0.009 (7)	0.002 (7)	−0.003 (7)
C35	0.071 (9)	0.066 (9)	0.052 (9)	−0.009 (7)	0.009 (7)	0.016 (7)
N2	0.061 (7)	0.037 (6)	0.070 (7)	−0.003 (6)	0.009 (6)	−0.011 (6)
C36	0.058 (10)	0.079 (10)	0.054 (8)	−0.004 (7)	0.019 (7)	−0.010 (7)
C37	0.051 (8)	0.080 (10)	0.045 (8)	−0.018 (7)	0.004 (7)	0.003 (7)
C38	0.100 (13)	0.043 (9)	0.108 (13)	−0.006 (9)	−0.007 (10)	0.004 (8)

TABLE V

Bond Lengths (Å) and angles (°) of Non-Hydrogens and their esd's

Co1–C14	2.255 (4)	C8–C9	1.36 (2)	C17–C18	1.51 (2)	C29–C32	1.49 (2)
Co1–C13	2.258 (4)	C9–C10	1.42 (2)	C20–C25	1.38 (2)	C30–C31	1.38 (2)
Co1–C12	2.265 (4)	C10–C11	1.37 (2)	C20–C21	1.36 (2)	C32–O2	1.43 (1)
Co1–C11	2.287 (4)	C10–C13	1.49 (2)	C21–C22	1.44 (2)	O2–C33	1.45 (1)
C1–C6	1.31 (3)	C11–C12	1.36 (2)	C22–C23	1.34 (2)	C33–C34	1.50 (2)
C1–C2	1.35 (2)	C13–O1	1.22 (2)	C23–C24	1.38 (2)	C33–C37	1.51 (2)
C2–C3	1.41 (2)	O1–C14	1.40 (2)	C23–C32	1.55 (2)	C34–C35	1.52 (2)
C3–C4	1.39 (4)	C14–C18	1.51 (2)	C24–C25	1.41 (2)	C35–N2	1.50 (2)
C4–C5	1.34 (2)	C14–C15	1.51 (2)	C26–C27	1.31 (2)	N2–C38	1.50 (2)
C4–C13	1.52 (2)	C15–C16	1.47 (2)	C26–C31	1.42 (2)	N2–C36	1.52 (2)
C5–C6	1.41 (3)	C16–N1	1.50 (2)	C27–C28	1.40 (2)	C36–C37	1.56 (2)
C7–C12	1.32 (2)	N1–C19	1.47 (1)	C28–C29	1.37 (2)		
C7–C8	1.41 (2)	N1–C17	1.48 (1)	C29–C30	1.39 (2)		

C14–Co1–C13	111.6 (2)	C14–Co1–C12	113.7 (2)	C13–Co1–C12	107.2 (2)
C14–Co1–C11	106.3 (2)	C13–Co1–C11	111.3 (1)	C12–Co1–C11	106.7 (2)
C6–C1–C2	124 (2)	C1–C2–C3	119 (2)	C4–C3–C2	119 (2)
C5–C4–C3	119 (2)	C5–C4–C13	119 (2)	C3–C4–C13	122 (2)
C4–C5–C6	122 (2)	C1–C6–C5	118 (2)	C12–C7–C8	120 (2)
C9–C8–C7	121 (2)	C8–C9–C10	119 (2)	C11–C10–C9	118 (2)
C11–C10–C13	125 (2)	C9–C10–C13	118 (2)	C10–C11–C12	122 (2)
C7–C12–C11	121 (2)	O1–C13–C10	118 (2)	O1–Co13–C4	121 (1)
C10–C13–C4	116 (1)	C13–O1–C14	129 (1)	O1–C14–C18	113 (1)
O1–C14–C15	106 (1)	C18–C14–C15	110 (1)	C16–C15–C14	114 (1)
C15–C16—N1	111 (1)	C19–N1–C17	112.6 (9)	C19–N1–C16	112 (1)
C17–N1–C16	110 (1)	N1–C17–C18	111.2 (10)	C14–C18–C17	110 (1)
C25–C20–C21	122 (1)	C20–C21–C22	118 (1)	C23–C22–C21	121 (1)
C22–C23–C24	120 (1)	C22–C23–C32	120 (1)	C24–C23–C32	120 (1)
C23–C24–C25	121 (1)	C20–C25–C24	118 (1)	C27–C26–C31	123 (2)
C26–C27–C28	118 (2)	C29–C28–C27	122 (2)	C28–C29–C30	120 (1)
C28–C29–C32	122 (1)	C30–C29–C32	118 (1)	C31–C30–C29	119 (2)
C30–C31–C26	119 (2)	O2–C32–C29	115 (1)	O2–C32–C23	105.7 (9)
C29–C32–C23	112.5 (9)	C32–O2–C33	114.2 (9)	O2–C33–C34	108 (1)
O2–C33–C37	110 (1)	C34–C33–C37	111 (1)	C33–C34–C35	112 (1)
N2–C35–C34	109 (1)	C38–N2–C35	111 (1)	C38–N2–C36	1090 (1)
C35–N2–C36	110.0 (9)	N2–C36–C37	109 (1)	C33–C37–C36	111 (1)

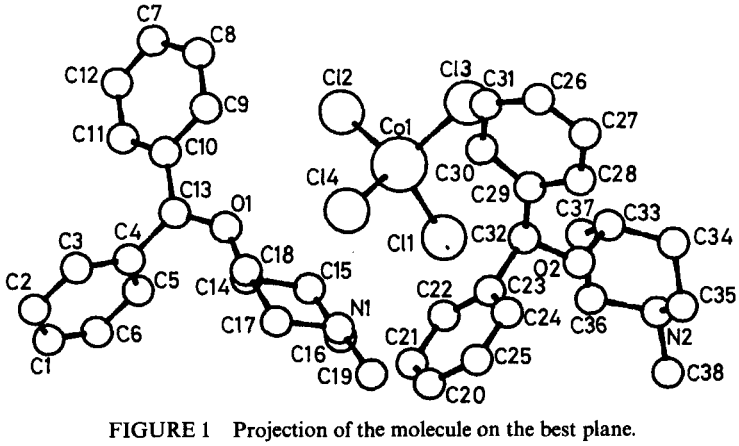

FIGURE 1 Projection of the molecule on the best plane.

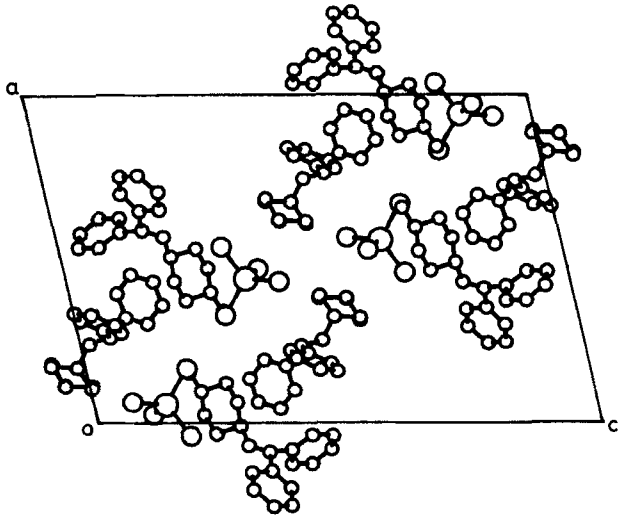

FIGURE 2 Packing of the molecules down 'b'.

RESULTS AND DISCUSSION

The final positional coordinates of all the atoms, thermal parameters, bond distances and angles and are given in Tables III–V. The bond lengths and bond angles are in good agreement with the standard values. The metal to chlorine distances are comparable to the values found in similar complexes of lignocaine hydrochloride with platinum chloride,[18] ferric chloride,[19] cobalt chloride,[20] copper chloride[21] and zinc chloride.[22]

The stoichiometry of this complex is 1:2. The projection of the molecule on the plane with least overlap is shown in Figure 1. The packing of molecules down "b" is shown in Figure 2. The packing of the molecules shows a strongly layered arrangement as is expected of the complexes.

Acknowledgement

The authors (MAS, AI, SBB, AMB and KGS) wish to thank UGC, Government of India for financial assistance. The author (MMMA) wishes to thank International Center for Theoretical Physics, Trieste, Italy for financial assistance for his visit to University of Mysore, India. The author (JSP) wishes to thank DST, Government of India for financial assistance under the project No. SP/S2/M-34/86.

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