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Structures of Complexes of 2,3,3a,5,6,6a-Hexahydro-3,6-diphenyl-1H,4H-pyrrolo[3,4-c]pyrrole-1,4-dithione with N-Methyl-2-pyrrolidone and Dimethyl Sulfoxide

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Abstract. Two complexes of the title compound (DPPT) have been formed with N-methyl-2-pyrrolidone (NMP) and dimethyl sulfoxide (Me$_2$SO): DPPT/(NMP)$_2$ and DPPT/(Me$_2$SO)$_2$. The crystal data for DPPT/(NMP)$_2$ are: $C_{18}H_{12}N_2S_2.2C_5H_9NO$, $M_r = 518.6907$, triclinic, $P\bar{1}$, $a = 7.527$ (1), $b = 9.157$ (1), $c = 9.988$ (1) Å, $\alpha = 96.39$ (1), $\beta = 110.40$ (1), $\gamma = 93.24$ (1)°, $V = 637.9$ (3) Å^3, $Z = 1$, $D_m = 1.345$, $D_x = 1.350$ Mg m^{-3}, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.70926$ Å, $\mu = 2.32$ mm^{-1}, $F(000) = 274$, $T = 193$ K, $R = 0.077$ for 1822 reflections. The crystal data for DPPT/(Me$_2$SO)$_2$ are: $C_{18}H_{12}N_2S_2.2C_2H_6OS$, $M_r = 476.6838$, monoclinic, $P2_1/c$, $a = 12.674$ (1), $b = 10.318$ (1), $c = 8.761$ (1) Å, $\beta = 100.32$ (1)°, $V = 1127.1$ (4) Å^3, $Z = 2$, $D_m = 1.398$, $D_x = 1.404$ Mg m^{-3}, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.70926$ Å, $\mu = 4.25$ mm^{-1}, $F(000) = 500$, $T = 188$ K, $R = 0.052$ for 2146 reflections. In both complexes, two solvent molecules are bonded to one DPPT molecule through intermolecular hydrogen bonds between the NH group of DPPT and the O atom of the solvent. Both complexes belong to point group C_i. The phenyl rings of DPPT are twisted in the same direction out of plane of the heterocyclic system by 24.7 (4)° in DPPT/(NMP)$_2$ and by 21.2 (5)° in DPPT/(Me$_2$SO)$_2$. The DPPT molecule is entirely enclosed by six solvent molecules in DPPT/(NMP)$_2$,

whereas the environment is quite different in DPPT/(Me$_2$SO)$_2$.

Introduction. The optical properties of 3,6-diphenyl-pyrrolo[3,4-c]pyrrole-1,4-dithione (DPPT) have been extensively studied in connection with applications to electrophotographic photoreceptors for laser printers (Mizuguchi & Rochat, 1988; Mizuguchi & Homma, 1989; Mizuguchi, 1989) and to optical information storage (Mizuguchi & Rochat, 1991). Both of the applications are based on the near-IR optical absorption of DPPT.

We have previously reported that there are three crystal modifications, (I), (II) and (III), of DPPT, all of which are monoclinic. Single crystals have been grown from the vapor phase (Mizuguchi, Rochat & Rihs, 1990; Mizuguchi, Arita & Rihs, 1991). Electronic properties of DPPT have been systematically studied in solution and in the solid state from the standpoint of deprotonation and intermolecular hydrogen bonding (Mizuguchi, Rochat & Rihs, 1992). The polarization experiments using single crystals have shown that the near-IR optical absorption is attainable only with modification (III). The optical transition is interpreted as being due to $\pi-\pi$ interactions between neighboring molecules along the stacking axis, although a weak charge-transfer interaction is also partly operative.

In contrast to the optical properties of DPPT, little is known about the phase of DPPT powders as synthesized. The powder phase is very similar to modification (III) according to the X-ray diffraction diagrams, but still quite different in optical properties (Mizuguchi & Rochat, 1988). An attempt was therefore made to grow single crystals from N-methyl-2-pyrrolidone (NMP) in order to characterize the powder phase. The crystals obtained from solution were found to contain solvent molecules.

The single crystals grown from NMP were lustrous, but they soon became matt and brittle when kept in air, or even under high vacuum in the dark. We assumed that the single crystals contained some solvent. In fact, thermogravimetric analysis (TGA) revealed that the loss of weight commenced at 336 K and finished at 402 K. The weight loss in this temperature range was 38.4%. This corresponds exactly to the proportion DPPT/NMP = 1/2. In order to suppress solvent evaporation, the present structure analysis has been carried out at low temperature.

This paper also describes similar complexes with dimethyl sulfoxide (Me₂SO).

Experimental. DPPT was prepared from 2,3,3a,5,6,6a-hexahydro-3,6-diphenyl-1H,4H-pyrrolo[3,4-c]pyrrole-1,4-dione (Iqbal, Cassar, Rochat, Pfenninger & Wallquist, 1988) by using a Lawesson's reagent as the thionation agent (Mizuguchi & Rochat, 1988). The product synthesized was identified by mass spectrometry, NMR and elemental analysis. The blue-black DPPT powder was then recrystallized from N-methyl-2-pyrrolidone for purification.

Single crystals were grown from DPPT solution in NMP and Me₂SO. In both cases, lustrous crystals were obtained in the form of platelets. According to the TGA measurements, the weight loss of the former complex was 38.4% in the temperature range 336–402 K and 30.9% in the range 343–387 K in the latter complex. These results suggest the formation of complexes of DPPT/(NMP)₂ and DPPT/(Me₂SO)₂, respectively.

The dimensions of the single crystal under investigation were $0.9 \times 0.9 \times 0.1$ mm in the NMP complex and $1.0 \times 1.0 \times 0.1$ mm in the Me₂SO complex. The crystal structures were analyzed at 193 and 188 K, respectively, with a four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation (Philips PW1100 diffractometer with $\lambda = 0.70926$ Å). θ–2θ scans. Unit-cell dimensions from 25 reflections (10 < θ < 15°) for the NMP complex, from 25 reflections (10 < θ < 15°) for the Me₂SO complex. 3926 independent reflections obtained in the range 6 < 2θ < 60° for the NMP complex and 3360 in the range 6 < 2θ < 58° for the Me₂SO complex. Of these, 1822 and 2146 reflections with $I > 3\sigma(I)$ were used for

structure refinement for the NMP and Me₂SO complexes, respectively. Corrections for absorption and secondary extinction were not applied. Three standard reflections monitored every 2 h during data collection showed no significant change in intensity. The density of the single crystals was measured by flotation in a mixture of benzene and carbon tetrachloride.

The structure was determined by direct methods using MULTAN82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The structures were refined on $|F|$ by full-matrix least-squares calculations using the SDP package (Frenz, 1985) with anisotropic atomic displacement parameters, neglecting H atoms, to $R = 0.101$ for the NMP complex and $R = 0.089$ for the Me₂SO complex. 11 of 15 H atoms in the NMP complex were then located in difference electron density maps and the remaining H atoms were positioned by calculation. In the Me₂SO complex, all 12 H atoms were located in difference electron density maps. Final $R = 0.077$, $wR = 0.096$, $S = 2.423$ for the NMP complex and $R = 0.052$, $wR = 0.068$, $S = 1.821$ for the Me₂SO complex. Weights of each reflection in the refinement were $w = 1/\sigma^2(F_o)$, $\sigma(F_o)$ being the e.s.d. based on counting statistics. $(\Delta/\sigma)_{max} = 0.06$ for the NMP complex and 0.04 for the Me₂SO complex. The maximum heights in the final difference electron density maps were 0.74 e Å⁻³ for the NMP complex and 0.69 e Å⁻³ for the Me₂SO complex. Atomic scattering factors for C, N and S were taken from International Tables for X-ray Crystallography (1974, Vol. IV). The final atomic positional and equivalent isotropic displacement parameters appear in Tables 1 and 2.*

Discussion.

Molecular geometry

The conformations of the NMP and Me₂SO complexes are given in Figs. 1 and 2, where the atoms are represented by displacement ellipsoids (ORTEP; Johnson, 1971). The interatomic distances and bond angles and their e.s.d.'s are listed for both complexes in Tables 3 and 4.

Two solvent molecules are bonded to one DPPT, in both complexes, through intermolecular hydrogen bonds between the NH group of DPPT and the O atom of the solvent. In DPPT/(NMP)₂, the angles N1—H1···O1 and C10—O1···H1 are 165 (8) and

Table 1. *Positional and equivalent isotropic displacement parameters with e.s.d.'s in parentheses for DPPT/(NMP)$_2$*

	x	y	z	U_{eq}* (Å^2)
S1	0.3073 (3)	0.2148 (2)	0.2941 (2)	0.036 (1)
O1	0.2962 (8)	0.4922 (6)	0.0225 (5)	0.041 (1)
N1	0.1159 (9)	0.2191 (6)	0.0146 (5)	0.025 (1)
N2	0.4078 (9)	0.7338 (7)	0.0688 (6)	0.031 (1)
C1	0.154 (1)	0.1406 (7)	0.1322 (7)	0.025 (1)
C2	0.050 (1)	0.0000 (7)	0.0765 (6)	0.022 (1)
C3	-0.007 (1)	0.1346 (7)	-0.1098 (6)	0.022 (1)
C4	-0.062 (1)	0.1982 (7)	-0.2457 (6)	0.027 (1)
C5	-0.059 (1)	0.3496 (8)	-0.2461 (7)	0.032 (1)
C6	-0.114 (1)	0.4078 (8)	-0.3758 (8)	0.038 (1)
C7	-0.173 (1)	0.3111 (9)	-0.5057 (8)	0.042 (1)
C8	-0.180 (1)	0.1615 (9)	-0.5060 (7)	0.042 (1)
C9	-0.125 (1)	0.1016 (8)	-0.3780 (7)	0.033 (1)
C10	0.396 (1)	0.6010 (8)	0.1027 (7)	0.030 (1)
C11	0.534 (1)	0.6010 (9)	0.2578 (8)	0.037 (1)
C12	0.585 (1)	0.7634 (9)	0.3156 (8)	0.042 (1)
C13	0.547 (1)	0.8418 (9)	0.1812 (8)	0.038 (1)
C14	0.323 (1)	0.7664 (9)	-0.0779 (7)	0.039 (1)

* Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Positional and equivalent isotropic displacement parameters with e.s.d.'s in parentheses for DPPT/(Me$_2$SO)$_2$*

	x	y	z	U_{eq}* (Å^2)
S1	0.9948 (1)	0.2756 (2)	0.2799 (2)	0.032 (1)
S2	0.7438 (1)	0.3496 (2)	0.4354 (2)	0.037 (1)
O1	0.7052 (3)	0.3884 (5)	0.2693 (4)	0.037 (1)
N1	0.8680 (3)	0.4404 (4)	0.0948 (5)	0.023 (1)
C1	0.9703 (4)	0.3876 (5)	0.1437 (5)	0.021 (1)
C2	0.8629 (3)	0.5303 (5)	-0.0179 (5)	0.020 (1)
C3	1.0335 (3)	0.4563 (5)	0.0493 (5)	0.020 (1)
C4	0.7614 (3)	0.5936 (5)	-0.0853 (5)	0.024 (1)
C5	0.6633 (4)	0.5325 (6)	-0.0723 (6)	0.029 (1)
C6	0.5672 (4)	0.5951 (7)	-0.1366 (6)	0.036 (1)
C7	0.5675 (4)	0.7107 (7)	-0.2092 (7)	0.036 (1)
C8	0.6641 (4)	0.7722 (6)	-0.2222 (7)	0.039 (1)
C9	0.7598 (4)	0.7096 (6)	-0.1616 (7)	0.033 (1)
C10	0.6411 (6)	0.404 (1)	0.5378 (8)	0.070 (1)
C11	0.8411 (5)	0.4653 (8)	0.5190 (8)	0.046 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

148 (3)°, respectively. The N1—H1, H1—O1 and N1—O1 distances are 0.90 (9), 1.88 (9) and 2.757 (8) Å, respectively. In DPPT/(Me$_2$SO)$_2$, the angles N1—H1···O1 and S2—O1···H1 are 156 (8) and 117 (2)°, respectively. The N—H1, H1—O1 and N1—O1 distances are 0.84 (9), 2.04 (9) and 2.831 (5) Å, respectively.

The DPPT molecule is not entirely planar; the phenyl rings are twisted out of the plane of the planar heterocyclic system by 24.7 (4)° in DPPT/(NMP)$_2$ and by 21.2 (5)° in DPPT/(Me$_2$SO)$_2$. Both complexes as well as the DPPT molecule belong to point group C_i.

Arrangement of molecules

Figs. 3 and 4 show stereodiagrams of the crystal structures of DPPT/(NMP)$_2$ and DPPT/Me$_2$SO)$_2$, respectively. In DPPT/(NMP)$_2$, the five-membered ring of the solvent and heterocyclic ring system align, more or less, in the same plane and these repeating

units lie parallel to each other with an interplanar distance of 3.00 (1) Å. The DPPT molecule is entirely surrounded by six NMP molecules. In DPPT/(Me$_2$SO)$_2$, the Me$_2$SO molecules are hydrogen bonded to DPPT at an angle of about 10° to the heterocyclic plane of the DPPT molecule. The repeating units are stacked in a herringbone fashion along the c axis.

Intermolecular hydrogen bonding

No single crystals of DPPT could be grown from solution in NMP and Me$_2$SO; instead, complexes of DPPT with the solvents were formed. This can be explained in the following way.

In the solid state of DPPT, there are intermolecular hydrogen bonds between the HN group of one DPPT molecule and the S atom of another [HN···S 3.435 (3), 3.440 (5) and 3.359 (5) Å in modifications (I), (II) and (III), respectively] (Mizuguchi, Rochat & Rihs, 1990; Mizuguchi, Arita & Rihs, 1991). The NH···S hydrogen bond is, however, known to be much weaker than NH···O because the S atom is less electronegative (Pauling, 1967). A strong intermolecular NH···O bond is generally linear (to within
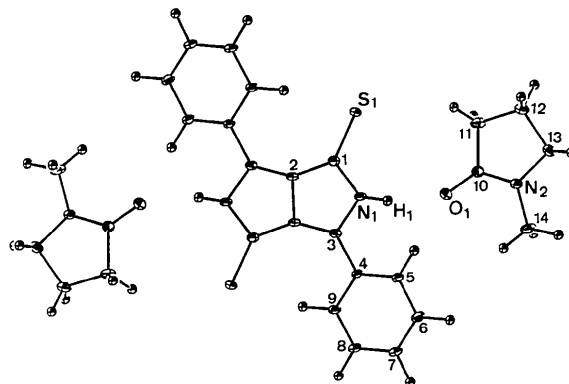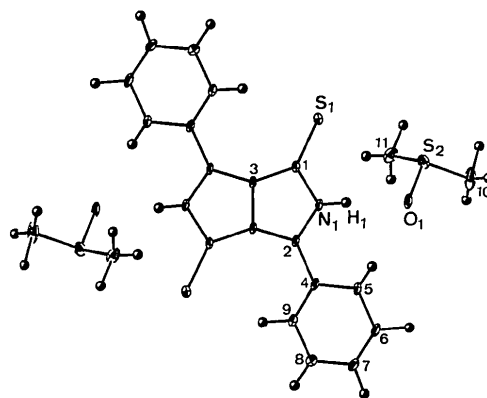

Fig. 1. Conformation of DPPT/(NMP)$_2$.

Fig. 2. Conformation of DPPT/(Me$_2$SO)$_2$.

Table 3. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses for DPPT/(NMP)₂

S1—C1	1.667 (6)	C3—C4	1.472 (9)
O1—C10	1.232 (8)	C4—C5	1.39 (1)
N1—C1	1.399 (9)	C4—C9	1.418 (9)
N1—C3	1.378 (7)	C5—C6	1.39 (1)
N2—C10	1.30 (1)	C6—C7	1.40 (1)
N2—C13	1.467 (8)	C7—C8	1.37 (1)
N2—C14	1.453 (9)	C8—C9	1.39 (1)
C1—C2	1.413 (9)	C10—C11	1.534 (9)
C2—C2	1.450 (8)	C11—C12	1.51 (1)
C2—C3	1.36 (1)	C12—C13	1.54 (1)
C1—N1—C3	110.9 (5)	C3—C4—C9	118.9 (6)
C10—N2—C13	115.1 (5)	C5—C4—C9	120.0 (6)
C10—N2—C14	122.7 (5)	C4—C5—C6	120.4 (6)
C13—N2—C14	120.8 (6)	C5—C6—C7	119.1 (7)
S1—C1—N1	120.4 (5)	C6—C7—C8	120.8 (7)
S1—C1—C2	133.6 (5)	C7—C8—C9	121.0 (6)
N1—C1—C2	105.8 (5)	C4—C9—C8	118.8 (7)
C1—C2—C2	107.1 (6)	O1—C10—N2	125.9 (6)
C1—C2—C3	144.9 (5)	O1—C10—C11	125.4 (7)
C2—C2—C3	107.9 (5)	N2—C10—C11	108.7 (5)
N1—C3—C2	108.2 (5)	C10—C11—C12	103.3 (6)
N1—C3—C4	118.9 (6)	C11—C12—C13	105.1 (6)
C2—C3—C4	132.8 (5)	N2—C13—C12	101.8 (6)
C3—C4—C5	121.2 (5)		

Fig. 3. Stereoview of the crystal structure of DPPT/(NMP)₂. a is horizontal, b is vertical.

Table 4. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses for DPPT/(Me₂SO)₂

S1—C1	1.649 (5)	C2—C4	1.468 (6)
S2—O1	1.504 (4)	C3—C3	1.421 (6)
S2—C10	1.796 (8)	C4—C5	1.417 (7)
S2—C11	1.778 (7)	C4—C9	1.369 (8)
N1—C1	1.401 (6)	C5—C6	1.404 (7)
N1—C2	1.348 (6)	C6—C7	1.352 (9)
C1—C3	1.438 (7)	C7—C8	1.402 (8)
C2—C3	1.396 (6)	C8—C9	1.392 (7)
O1—S2—C10	105.2 (3)	C1—C3—C3	109.2 (4)
O1—S2—C11	107.5 (3)	C2—C3—C3	107.4 (4)
C10—S2—C11	95.5 (4)	C2—C4—C5	119.2 (5)
C1—N1—C2	114.4 (4)	C2—C4—C9	121.4 (4)
S1—C1—N1	122.6 (4)	C5—C4—C9	119.5 (4)
S1—C1—C3	135.1 (3)	C4—C5—C6	118.3 (5)
N1—C1—C3	102.3 (4)	C5—C6—C7	121.3 (5)
N1—C2—C3	106.8 (4)	C6—C7—C8	120.9 (5)
N1—C2—C4	121.7 (4)	C7—C8—C9	118.2 (6)
C3—C2—C4	131.6 (4)	C4—C9—C8	121.8 (5)
C1—C3—C2	143.5 (4)		

Fig. 4. Stereoview of the crystal structure of DPPT/(Me₂SO)₂. b is horizontal, c is vertical.

10°) and the N–O distance is about 2.8 Å. In the present experiment, the hydrogen-bond interaction between DPPT molecules is expected to be much weaker than the hydrogen bond between the NH group of DPPT and the O atom of the solvent [NH···O 2.757 (8) Å in DPPT/(NMP)₂ and 2.831 (5) Å in DPPT/(Me₂SO)₂]. This explains why the complexes recrystallized from DPPT solution.

From the NH···O distance in the above complexes, the hydrogen bond between DPPT and NMP is slightly stronger than that between DPPT and Me₂SO. This is reflected in the sublimation temperatures of NMP and Me₂SO from the corresponding complexes (402 and 387 K, respectively) as shown by our TGA measurements.

Complexes from other solvents

Recrystallization of DPPT has also been carried out from dimethylformamide (DMF) and 2-pyrrolidone. The latter solvent could form two hydrogen bonds with DPPT: with NH from DPPT and C=O from 2-pyrrolidone and vice versa. No satisfactory single crystals have been obtained from solution in DMF. On the other hand, two kinds of single crystals in the form of needles and platelets have been grown from 2-pyrrolidone. The needle and platelet crystals have been identified as modifications (II) and (III) of DPPT, respectively. Surprisingly, no crystalline complexes could be isolated with this solvent. The present results conflict with the interpretation mentioned above. Details are not yet understood.

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The Crystal Packing of 4,7-Dibromobenzofurazan 1-Oxide

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Abstract. $C_6H_2Br_2N_2O_2$, $M_r = 293.90$, monoclinic, $C2/c$, $a = 23.083$ (26), $b = 7.842$ (3), $c = 28.081$ (13) Å, $\beta = 105.97$ (7)°, $V/Z = 203.6$ (5) Å^3, $Z = 24$ (3 molecules in the asymmetric unit), $D_x = 2.396$ (6) g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 98.1$ cm^{-1}, $F(000) = 3312$, $T = 297$ (2) K, $R = 0.079$ for 2187 unique observed reflections with $I > \sigma(I)$. Bond lengths and angles are normal. Each of the six independent Br atoms is close to either an O or an N atom on an adjacent molecule. These interactions organize the molecules into two-dimensional sheets. These sheets are of two different kinds, both involving the same kinds of intermolecular interactions, but with distinctly different overall patterns.

Introduction. Short intermolecular N⋯X distances were found in the crystal structures of 5-chloro- and 5-bromobenzofurazan 1-oxide (Britton & Noland, 1962; Britton, Hardgrove, Hegstrom & Nelson, 1972) and short O⋯I distances were found in the structure of 5-iodobenzofurazan 1-oxide (Gehrz & Britton, 1972). These were interpreted as Lewis acid–base interactions with the halogen atoms as the acids and the N and O atoms as the bases. Seeking another example where this sort of interaction is possible, we report here the crystal structure of one of the isomers of dibromobenzofurazan 1-oxide.

Experimental. A sample of the compound was prepared by Professor W. E. Noland and K. A. N. Juneau of this Department. Crystals suitable for X-ray diffraction were hard to obtain. The first eight crystals examined were of very poor quality: the

errors in the cell constants, measured in the usual way, were of the order of 1/500; the peaks were broad with peak widths of 2–3°; the intensities were low. Lowering the temperature did not improve the quality of the data. A slightly better crystal was eventually found and used for the data collection. It had been recrystallized from acetone and was a yellow prism 0.10 × 0.18 × 0.35 mm. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 23 reflections with $9 < \theta < 13°$ were used to determine the cell parameters. Systematic extinctions (hkl, $h + k$ odd; $h0l$, l odd) indicated the space group to be either Cc or $C2/c$. The latter was chosen on the basis of the eventual solution. Data were collected, using ω scans, in the range $0 < \theta < 24°$ over one hemisphere (ranges: h −26 to 26; k 0 to 8; l −32 to 32). The intensities of 7252 different reflections were measured. Three check reflections measured every 5000 s of exposure time showed a 5% linear decay over the entire data collection; this was corrected for. The structure was solved by direct methods (*MITHRIL*; Gilmore, 1984). After preliminary refinement, absorption corrections were made, using the program *DIFABS* (Walker & Stuart, 1983; transmission factors 0.64 to 1.32), and equivalent reflections were combined to give 3634 independent reflections ($R_{int} = 0.052$), of which the 2187 with $I > \sigma(I)$ were used in the calculations. The structure was refined by full-matrix least squares on F's. Since the amount of data was limited, only the Br, O and N atoms were given anisotropic thermal parameters. The H atoms were included at idealized positions with isotropic B