Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2,5-Dihydro-2,5-dimethyl-3,6-diphenylpyrrolo[3,4-*c*]pyrrole-1,4-(diylidene)-bis(cyanamide) chloroform disolvate

Jin Mizuguchi

Department of Applied Physics, Graduate School of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya-ku, 240-8501 Yokohama, Japan

Correspondence e-mail: mizu-j@ynu.ac.jp

Received 26 August 2003
Accepted 1 September 2003
Online 11 September 2003

The title compound, $C_{22}H_{16}N_6 \cdot 2CHCl_3$, a diketopyrrolopyrrole analogue (red pigment), has C_i symmetry and the phenyl rings are at an angle of 53.5 (1)° to the planar heterocylic ring system.

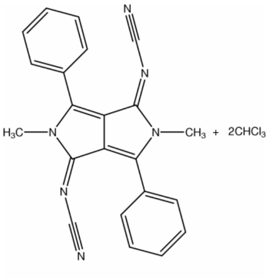
Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.118
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

3,6-Diphenylpyrrolo[3,4-*c*]pyrrole-1,4-dione (DPP) and its derivatives are well known red pigments on the market (Mizuguchi *et al.*, 1992). The title compound, (I), is a 1:2 solvated complex of a DPP analogue with chloroform. The unsolvated analogue, obtained by recrystallization from methylene chloride, exhibits intense orange luminescence (Mizuguchi, 2003), whereas the present solvated analogue is red in color and has some luminescent properties.



(I)

The title pigment molecule has a centre of symmetry at the mid-point of the central bond of the heterocyclic ring system. The latter is entirely planar. Each aryl ring is twisted from the heterocyclic system by 53.5 (1)° [N1/C7/C9/C10/C10^i; symmetry code (i): $-x$, $1 - y$, $-z$]. The molecular conformation is quite similar to that of the unsolvated compound (Mizuguchi, 2003). It is also of interest to note that the angle N2—C11—N3 is not exactly 180° [because of the repulsion between atoms N3 and C6^i at 3.125 (4) Å] and that the plane composed of atoms C9/N2/C11/N3 is at an angle of 10.7 (1)° to the heterocyclic ring. Fig. 2 shows the projection on to the *ab* plane. All other bond parameters agree well with those of the unsolvated compound (Mizuguchi, 2003) and also of DPP (Mizuguchi *et al.*, 1992). The chloroform molecules are stacked in channels along the *c* axis (Fig. 2).

Experimental

The title compound was prepared by reacting 1,4-diketo-3,6-diphenylpyrrolo[3,4-*c*]pyrrole with bis(trimethylsilyl)carbodiimide in the presence of $TiCl_4$ (Zambounis *et al.*, 1994). The product was purified by sublimation under argon at about 575 K, using a two-zone furnace (Mizuguchi, 1981). Single crystals of (I) were grown from a chloroform solution by slow evaporation.

Crystal data

$C_{22}H_{16}N_6\cdot 2CHCl_3$
$M_r = 603.16$
Monoclinic, $P2_1/c$
$a = 8.656\ (3)$ Å
$b = 13.768\ (7)$ Å
$c = 11.670\ (5)$ Å
$\beta = 110.61\ (2)^\circ$
$V = 1301.8\ (10)$ Å^3
$Z = 2$

$D_x = 1.539$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 6142 reflections
$\theta = 4.0$–67.6°
$\mu = 6.25$ mm^{-1}
$T = 93.2$ K
Platelet, red
$0.30 \times 0.15 \times 0.02$ mm

Data collection

Rigaku RAXIS-RAPID Imaging Plate diffractometer
ω scans
Absorption correction: multi-scan (Higashi, 1995)
$T_{\min} = 0.396$, $T_{\max} = 0.883$
11504 measured reflections

2361 independent reflections
1924 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.074$
$\theta_{\max} = 68.2^\circ$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.118$
$S = 1.29$
2235 reflections
163 parameters

H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.05({\rm Max}(F_o^2,0) + 2F_c^2)/3)^2]$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C7	1.377 (3)	N3—C11	1.160 (3)
N1—C8	1.468 (3)	C1—C7	1.463 (3)
N1—C9	1.401 (3)	C7—C10	1.381 (3)
N2—C9	1.304 (3)	C9—C10$^{\rm i}$	1.434 (3)
N2—C11	1.330 (3)	C10—C10$^{\rm i}$	1.425 (4)
C7—N1—C8	125.4 (2)	C1—C7—C10	130.2 (2)
C7—N1—C9	111.3 (2)	N1—C9—N2	118.4 (2)
C8—N1—C9	122.6 (2)	N1—C9—C10$^{\rm i}$	105.2 (2)
C9—N2—C11	120.7 (2)	N2—C9—C10$^{\rm i}$	136.3 (2)
C2—C1—C7	122.3 (2)	C7—C10—C9$^{\rm i}$	144.0 (2)
C6—C1—C7	118.4 (2)	C7—C10—C10$^{\rm i}$	109.0 (3)
C1—C2—C3	119.7 (2)	C9$^{\rm i}$—C10—C10$^{\rm i}$	107.0 (3)
N1—C7—C1	122.3 (2)	N2—C11—N3	172.9 (3)
N1—C7—C10	107.2 (2)		
N1—C7—C1—C2	−54.3 (3)	N2—C9—C10$^{\rm i}$—C7$^{\rm i}$	5.6 (6)
N1—C7—C1—C6	125.2 (3)	C10—C9$^{\rm i}$—N2$^{\rm i}$—C11$^{\rm i}$	−5.1 (4)
N1—C9—N2—C11	−170.7 (2)		

Symmetry code: (i) $-x, 1-y, -z$.

X-ray intensity data were collected at 93 K, since the solvent molecules (choloroform) sublime quite rapidly at room temperature. All H atoms were positioned by calculation (C—H 0.950–0.951 Å) but not refined.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *TEXSAN*.

The author expresses his sincere thanks to Mr I. Suzuki for experimental assistance.

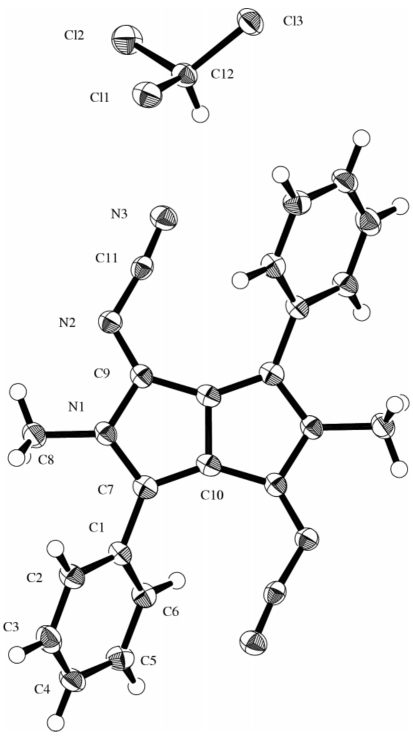


Figure 1
A view of the molecular structure of (I), showing 50% displacement ellipsoids for non-H atoms.

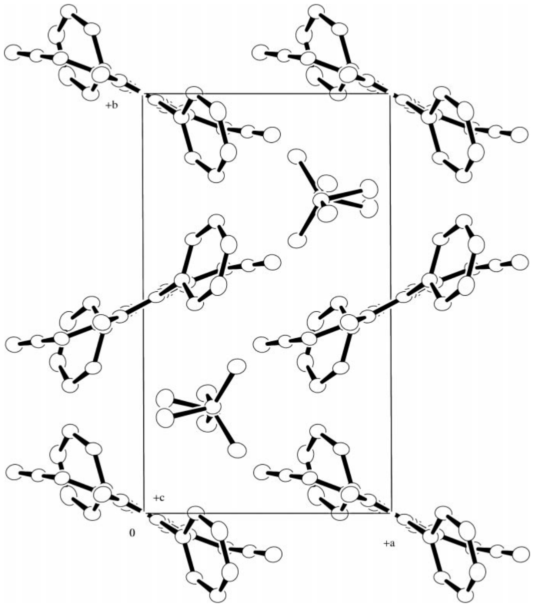


Figure 2
Projection of the crystal structure on to the *ab* plane.

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory. Tennessee, USA.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Mizuguchi, J. (1981). *Cryst. Res. Technol.* **16**, 695–700.
Mizuguchi, J. (2003). *Z. Kristallogr. New Cryst. Struct.* **218**, 141–142.
Mizuguchi, J., Grubenmann, A., Wooden, G. & Rihs, G. (1992). *Acta Cryst.* B**48**, 696–700.
Molecular Structure Corporation (2001). *TEXSAN*. Version 1.11. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1985). *SHELXS*86. University of Göttingen, Germany.
Zambounis, J., Hao, H. & Iqbal, A. (1994). US Patent 5 484 943.