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1,3,5-Trichloro-2,4,6-tripyrrol-1-ylbenzene

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The title compound, $C_{18}H_{12}Cl_3N_3$, has two molecules in the asymmetric unit, related by an approximate inversion center. C—Cl distances are in the range 1.718 (2)–1.7234 (19) Å, C(phenyl)—N distances are in the range 1.412 (2)–1.419 (2) Å and C(pyrrole)—N distances are in the range 1.366 (3)–1.387 (2) Å.

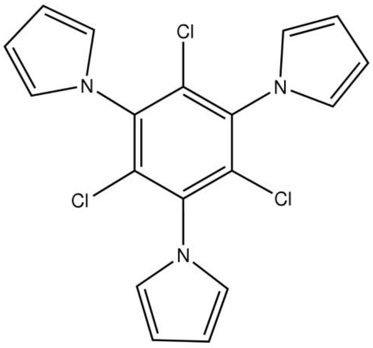
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.099
Data-to-parameter ratio = 19.2

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

Comment

In the course of a research program aimed at the synthesis of highly conjugated polycyclic molecules, the title molecule (I) was prepared from 1,3,5-trichloro-2,4,6-trifluorobenzene for further amination of the chlorine atoms. The structure determination was performed to confirm its identity.



(I)

There are two molecules in the asymmetric unit of (I), as illustrated in Fig. 1. They are related by an approximate inversion center near (0.41, 0.35, 0.35), but deviations are substantial, with the two Cl_3Ph groups forming a dihedral angle of 14.89 (8)°. The pyrrole rings are not coplanar with the phenyl rings, having C—C—N—C torsion angles in the range 61.9 (3)–97.6 (3)° (Table 1). The pyrrole rings are more nearly perpendicular to the phenyl ring than those seen in the corresponding compound lacking the Cl substituents (Thallapally *et al.*, 2001). That molecule lies on a position of site symmetry 32 and has a 31.6° C—C—N—C torsion angle. Hexapyrrolebenzene (Biemans *et al.*, 1996; Fronczek *et al.*, 1996) has torsion angles in the range 47.8 (1)–67.9 (1)°.

Experimental

A 250 ml round-bottom flask under Ar was charged with 2.4 g 1,3,5-trichloro-2,4,6-trifluorobenzene and 100 ml dry THF. Through a solid addition funnel, 5.0 g potassium pyrrole was added stepwise with stirring. The reaction was carried out in an ice bath for 2.5 h. The reaction was then quenched with 6 *N* HCl, adjusting the pH to 6. The

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solvent was removed under reduced pressure, and the remaining solid was taken up with 300 ml diethyl ether, washed with water (3 *x* 75 ml), dried over $MgSO_4$ and filtered. The filtrate was concentrated under reduced pressure. With a vacuum column, the residue was chromatographed over silica gel (50 g, 200 mesh) using hexane:DCM (3:2) as the eluent, affording a white solid (3.41 g, 92%), which was crystallized from benzene.

Crystal data

$C_{18}H_{12}Cl_3N_3$
$M_r = 376.66$
Triclinic, $P\bar{1}$
$a = 11.167$ (3) Å
$b = 11.534$ (3) Å
$c = 15.799$ (3) Å
$\alpha = 86.02$ (2)°
$\beta = 69.58$ (2)°
$\gamma = 65.19$ (2)°
$V = 1723.9$ (8) Å^3
$Z = 4$
$D_x = 1.451$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.54$ mm^{-1}
$T = 100$ K
Fragment, colorless
0.50 × 0.40 × 0.35 mm

Data collection

Enraf–Nonius CAD-4 diffractometer with an Oxford Cryosystems Cryostream cooler
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.733$, $T_{max} = 0.835$
8712 measured reflections
8304 independent reflections
6579 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 28.0°$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.099$
$S = 1.04$
8304 reflections
433 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.6927P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1*A*—C1*A*	1.718 (2)	N1*A*—C2*A*	1.412 (2)
Cl1*B*—C1*B*	1.7234 (19)	N1*B*—C2*B*	1.417 (2)
Cl2*A*—C3*A*	1.7209 (19)	N2*A*—C4*A*	1.419 (2)
Cl2*B*—C3*B*	1.7201 (18)	N2*B*—C4*B*	1.414 (2)
Cl3*A*—C5*A*	1.7208 (19)	N3*A*—C6*A*	1.412 (2)
Cl3*B*—C5*B*	1.7206 (19)	N3*B*—C6*B*	1.419 (2)
C7*A*—N1*A*—C2*A*—C1*A*	97.6 (3)	C11*B*—N2*B*—C4*B*—C3*B*	61.9 (3)
C7*B*—N1*B*—C2*B*—C3*B*	75.6 (2)	C15*A*—N3*A*—C6*A*—C1*A*	74.4 (3)
C11*A*—N2*A*—C4*A*—C3*A*	87.6 (2)	C15*B*—N3*B*—C6*B*—C5*B*	82.8 (3)

All H atoms were placed in idealized positions, with C—H = 0.95 Å, and thereafter treated as riding, $U_{iso}(H) = 1.2U_{eq}(C)$.

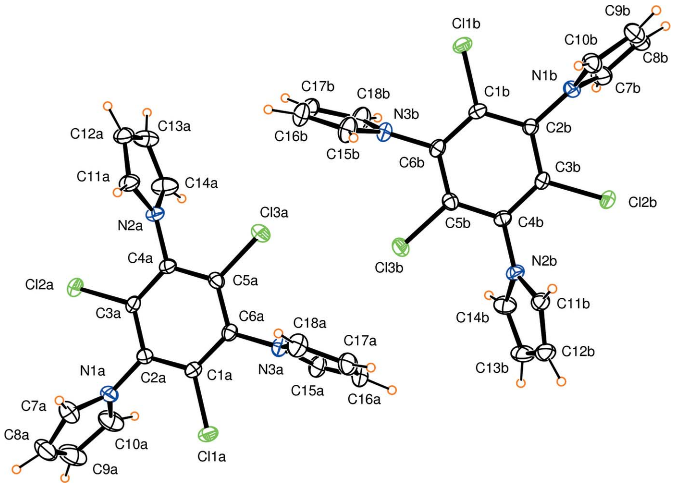

Figure 1
The asymmetric unit of (I) with displacement ellipsoids drawn at the 50% level. H atoms are represented by spheres of arbitrary radius.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1994); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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