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Structural, spectral and thermal studies of *N*-2-(4,6-lutidyl)-*N*′-chlorophenylthioureas

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Abstract

N-2-(4,6-lutidyl)-*N*′-2-chlorophenylthiourea, 4,6LutTu2ClPh, triclinic, *P*-1, $a = 7.512(1)$, $b = 7.695(2)$, $c = 12.870(3)$ Å, $\alpha = 82.50(2)$, $\beta = 77.07(2)$, $\gamma = 83.50(2)°$, $V = 716.2(3)$ Å^3 and $Z = 2$; *N*-2-(4,6-lutidyl)-*N*′-3-chlorophenylthiourea, 4,6LutTu3ClPh, monoclinic, *C*2/*c*, $a = 25.162(3)$, $b = 7.220(4)$, $c = 16.272(2)$ Å, $\beta = 94.71(1)°$, $V = 2946.0(2)$ Å^3 and $Z = 8$ and *N*-2-(4,6-lutidyl)-*N*′-4-chlorophenylthiourea, 4,6LutTu4ClPh, monoclinic, *C*2/*c*, $a = 33.1254(15)$, $b = 4.3025(1)$, $c = 20.3298(9)$ Å, $\beta = 107.070(10)°$, $V = 2769.80(19)$ Å^3 and $Z = 8$. The strength and nature of the intramolecular hydrogen bonding between N′H and the pyridine nitrogen and intermolecular hydrogen bonding involving the thione sulfur and the NH hydrogen are affected by the position of the chloro substituent on the phenyl ring. N′H is bifurcated in 4,6LutTu2Cl interacting with the chlorine (and the pyridine nitrogen), and the aromatic hydrogens interact weakly with sulfur and chlorine in neighboring molecules in these thioureas. The enthalpies of fusion and melting points are in the order $4,6\text{-LutTu4Cl} \gg 4,6\text{LutTu2Cl} > 4,6\text{LutTu3Cl}$. ^{1}H NMR studies in $CDCl_3$ show the N′H hydrogen resonance considerably downfield from other resonances in the spectrum for each thiourea. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: 2-pyridylthioureas; Chlorophenylthioureas; Crystal structures; Hydrogen bonding; Enthalpy of fusion

1. Introduction

The structure of *N*-(2-pyridyl)-*N*′-phenylthiourea [1] was reported following a ^{1}H NMR study of the intramolecular hydrogen bonding between the pyridyl nitrogen and N′H for *N*-(2-pyridyl)thioureas [2]. Intramolecular N′–H···N and intermolecular N–H···S hydrogen bonding, as well as the planarity of the molecules, has been the focus in more recent structural studies of substituted *N*-(2-pyridyl)-*N*′-arylthioureas including *N*-(2-pyridyl)-*N*′-tolylthioureas [3], the three *N*-2-(4,6-lutidyl)-*N*′-tolylthioureas [4], *N*-(5-bromo-2-pyridyl)-*N*′-2-(2,5-dimethoxyphenylethyl)thiourea [5] and four thioureas with *p*-substituted aryl groups, *N*-(2-pyridyl)-*N*′-4-bromophenyl-, *N*-(2-pyridyl)-*N*′-4-chlorophenyl-, *N*-(2-pyridyl)-*N*′-4-methoxyphenyl- and *N*-(2-pyridyl)-*N*′-4-nitrophenylthiourea [6]. A study of structural, thermal and spectral properties of the *N*-2-(picolyl)-*N*′-phenylthioureas and *N*-(4,6-lutidyl)-*N*′-phenylthiourea has been communicated [7]. A recent communication showed that some members of a series of *N*-(2-pyridyl)-*N*′-2-methoxyphenylthioureas possess

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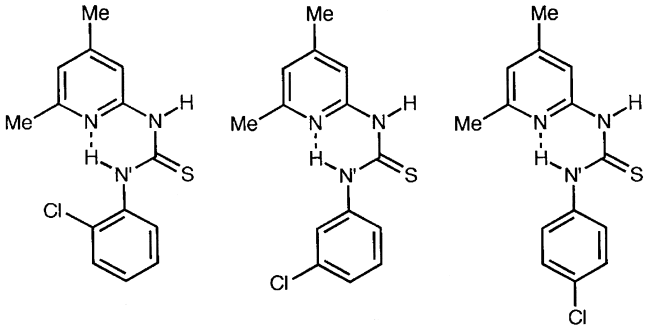

Fig. 1. Representations of 4,6LutTu2Cl, 4,6LutTu3Cl and 4,6LutTu4Cl showing expected intramolecular hydrogen bonding.

bifurcated intramolecular hydrogen bonding to the pyridyl nitrogen and methoxy oxygen [8]. We prepared and acquired crystals of the three *N*-2-(4,6-lutidyl)-*N*′-chlorophenylthioureas, 4,6LutTu2ClPh, 4,6LutTu3ClPh and 4,6LutTu4ClPh, Fig. 1, in order to determine whether the position of the chloro substituent on the phenyl ring alters the nature or strength of the hydrogen bonding interactions, as well as the overall planarity of the molecule. Also, the possibility of the 4,6LutTu2ClPh involving the chloro substituent in a bifurcated hydrogen bond or its participation in hydrogen bonding in any of these thioureas prompted this study.

Table 1
Crystallographic data and methods of data collection, solution and refinement for 4,6LutTu2Cl, 4,6LutTu3Cl and 4,6LutTu4Cl

Crystal data	4,6LutTu2Cl	4,6LutTu3Cl	4,6LutTu4Cl
Empirical formula	$C_{14}H_{14}ClN_3S$	$C_{14}H_{14}ClN_3S$	$C_{14}H_{14}ClN_3S$
Crystal color, habit	Colorless, prism	Colorless, prism	Colorless, needle
Crystal size (mm)	0.30 × 0.25 × 0.16	0.25 × 0.18 × 0.14	0.96 × 0.07 × 0.05
Crystal system	Triclinic	Monoclinic	Monoclinic
Space group	*P*-1	*C*2/*c*	*C*2/*c*
a (Å)	7.5120(14)	25.162(3)	33.1254(15)
b (Å)	7.6950(16)	7.220(4)	4.30250(10)
c (Å)	12.870(3)	16.272(2)	20.3298(9)
α (°)	82.497(17)		
β (°)	77.072(17)	94.71(1)	107.070(2)
γ (°)	83.504(16)		
Volume (Å^3)	716.2(2)	2946.2(18)	2769.80(19)
Z	2	8	8
Formula weight	291.8	291.8	291.8
Density (calcd) (g cm^{-3})	1.353	1.316	1.399
Absorption coeff. (mm^{-1})	0.402	0.391	0.415
F(000)	304	1216	1216
Reflections for indexing	25	25	306
Index ranges	$0 \le h \le 9$	$0 \le h \le 32$	$-45 \le h \le 45$
	$-10 \le k \le 10$	$0 \le k \le 9$	$-5 \le k \le 5$
	$-16 \le l \le 16$	$-21 \le l \le 21$	$-27 \le l \le 26$
Reflections collected	3267	2255	5955
Independent reflections, R_{int}	2255, 0.0128	1256, N/A	3356, 0.0663
Absorption correction	Ψ-scan	Ψ-scan	HKL-Scalepack
Goodness-of-Fit	1.118	1.163	0.992
Largest diff. peak, hole (e Å^{-3})	0.419, −0.341	0.299, −0.314	0.190, −0.210
R_1, *wR*	0.0444, 0.0870	0.0489, 0.0739	0.0429, 0.0937
R_1, *wR* (all reflections)	0.0811, 0.1147	0.1277, 0.1142	0.1154, 0.1107

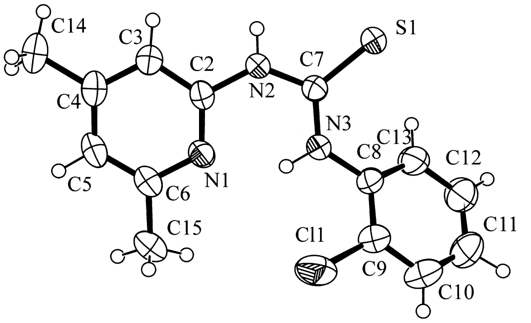

Fig. 2. ORTEP diagram of 4,6LutTu2Cl with atom numbering scheme and displacement ellipsoids at 50% probability level.

2. Experimental

2.1. Preparative, spectral and thermal methods

2-amino-4,6-lutidine and the three chlorophenyl isothiocyanates were purchased from Aldrich and used as received. 2-amino-4,6-lutidine was mixed in a 1:1 molar ratio with the desired chlorophenyl isothiocyanate in 95% ethanol and the mixture stirred with gentle warming for a minimum of 1 h. On cooling and slowly evaporating the reactant mixture (35°C), the thioureas crystallized from solution. The solids were filtered, washed with cold isopropanol, dried on a warm plate and then stored until required for spectral characterization. The yields are ca. 75% for each of the thioureas and the melting points are as follows: 4,6LutTu2ClPh, 193–195°C; 4,6LutTu3ClPh, 174–175°C; and 4,6LutTu4ClPh, 226–227°C. Their ^{1}H NMR spectra were recorded in CD_2Cl_2 with a Bruker 200 MHz spectrometer and the enthalpies of fusion were obtained with approximately 1 mg samples at a heating rate of 10°C min^{-1} using a Perkin–Elmer Differential Scanning Calorimeter, DSC7.

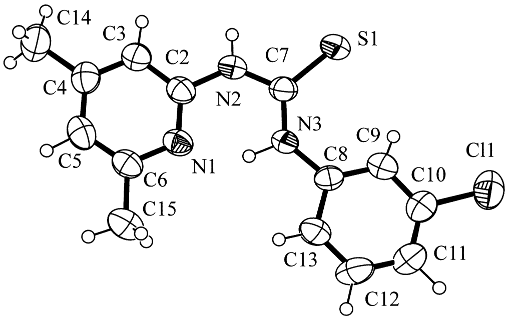

Fig. 3. ORTEP diagram of 4,6LutTu3Cl with atom numbering scheme and displacement ellipsoids at 50% probability level.

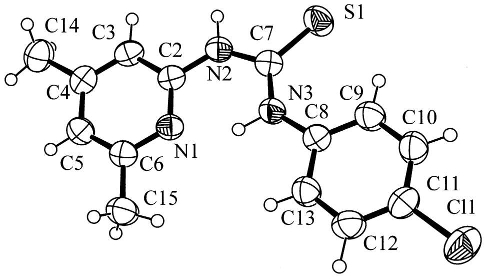

Fig. 4. ORTEP diagram of 4,6LutTu4Cl with atom numbering scheme and displacement ellipsoids at 50% probability level.

2.2. X-ray data collection, structure solution and refinement

Crystals of the three thioureas were grown by slow evaporation of 1:1 by volume acetone–ethanol mixtures at room temperature. The colorless thiourea crystals were mounted in random orientation on a glass fiber and data acquired with a Nonius MACH3 Four Circle Diffractometer (4,6LutTu2ClPh and 4,6LutTu3ClPh) or a Nonius Kappa CCD Diffractometer (4,6LutTu4ClPh), the latter at 130(2)°. The structures were solved by direct methods and missing atoms were found by difference-Fourier synthesis. The non-hydrogen atoms were refined with anisotropic temperature factors and all hydrogens attached to nitrogens and carbons, except for methyl groups, were found on a difference-Fourier map and refined isotropically. For 4,6LutTu4ClPh, DENZO [9] was used for data reduction and

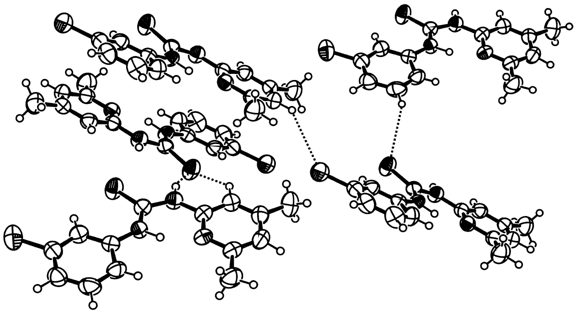

Fig. 5. Packing diagram for 4,6LutTu3Cl showing the various intermolecular interactions listed in Table 3.

Table 2
Selected bond distances (Å) and angles (°) for 4,6LutTu2Cl, 4,6LutTu3Cl and 4,6LutTu4Cl

	4,6LutTu2Cl	4,6LutTu3Cl	4,6LutTu4Cl
Distances (Å)			
S1–C7	1.687(2)	1.678(4)	1.6774(19)
N1–C2	1.327(3)	1.333(5)	1.328(2)
N1–C6	1.349(3)	1.355(5)	1.350(2)
C2–N2	1.400(3)	1.408(5)	1.399(2)
N2–C7	1.366(3)	1.378(5)	1.365(2)
N3–C7	1.339(3)	1.338(5)	1.343(2)
N3–C8	1.416(3)	1.415(5)	1.404(2)
C–Cl1	1.744(3)	1.745(5)	1.739(2)
Angles (°)			
C2–N1–C6	118.5(2)	117.4(5)	117.86(16)
N1–C2–C3	123.3(2)	123.4(5)	123.45(18)
N1–C2–N2	118.5(2)	119.1(5)	118.84(17)
N2–C2–C3	118.3(2)	117.5(5)	117.70(17)
C2–N2–C7	130.3(2)	130.4(4)	131.17(17)
S1–C7–N2	118.17(17)	117.2(4)	117.35(15)
N2–C7–N3	116.3(2)	115.9(4)	115.29(17)
S1–C7–N3	125.50(17)	126.9(4)	127.26(16)
C7–N3–C8	128.4(2)	132.1(4)	131.87(17)
N3–C8–C9	118.7(2)	114.2(5)	115.95(18)
N3–C8–C13	123.0(2)	125.8(4)	125.69(18)

scattering factors were from Wassmaire and Kirfel [10]. The structures were solved with MaXus [11], structure refinement for the three thioureas was carried out with SHELXL-97 and the graphics used are ZORTEP [12]. Table 1 summarizes the crystal data, collection information and refinement data for these thioureas.

3. Results and Discussion

3.1. Structural studies

The ORTEP diagrams of 4,6LutTu2ClPh, 4,6LutTu3ClPh and 4,6LutTu4ClPh are shown in Figs. 2–4, respectively, and a packing diagram for 4,6LutTu3ClPh in Fig. 5. These three thioureas crystallize with one unique molecule in the cell. Like other pyridyl thioureas [1,3–8], 4,6LutTu2ClPh, 4,6LutTu3ClPh and 4,6LutTu4ClPh are found in a conformation resulting from intramolecular hydrogen bonding of N3*H* (N′H) to the pyridine nitrogen, N1, in a manner similar to the benzoyl and acyl thioureas [13–20] and '*cis–cis*' like *N*-phenyl-*N*′-phenylthiourea [21]. The three thioureas have essentially the same bond distances, and most of the bond angles are also similar, Table 2. However, C7–N3–C8, N3–C8–C9, and N3–C8–C13 for 4,6LutTu2ClPh are significantly different than the analogous angles in the other two thioureas of this study. This variation suggests a difference in the hydrogen bonding interactions of N3*H* (vide infra), as does the position of the chloro substituent in 4,6LutTu2ClPh and 4,6LutTu3ClPh, Figs. 2 and 3.

Table 3
Intramolecular and intermolecular hydrogen bond distances (Å) and angles (°) for 4,6LutTu2Cl, 4,6LutTu3Cl and 4,6LutTu4Cl

Thiourea	D	A	D–H	H···A (Å)	D–H···A (Å)	∠(D–H···A) (°)
Intramolecular						
4,6LutTu2Cl	N3	N1	0.86(3)	1.91(3)	2.630(3)	141(2)
	N3	Cl1	0.86(3)	2.59(2)	2.945(2)	105.8(19)
4,6LutTu3Cl	N3	N1	0.96(4)	1.78(4)	2.643(4)	147(3)
4,6LutTu4Cl	N3	N1	0.94(2)	1.82(2)	2.652(2)	145.8(19)
Intermolecular						
4,6LutTu2Cl	N2	S1#1	0.83(3)	2.60(3)	3.398(2)	162(2)
	C3	S1#1	0.83(3)	2.85(2)	3.361(3)	147.1(9)
	C13	Cl1#2	0.93(3)	3.07(3)	3.796(3)	137(2)
4,6LutTu3Cl	N2	S1#1	0.83(4)	2.72(4)	3.466(4)	150(4)
	C3	S1#1	0.92(3)	2.92(4)	3.663(5)	139(3)
	C12	S1#2	0.96(5)	3.02(5)	3.745(6)	133(4)
	C5	Cl1#3	0.96(4)	3.06(4)	3.859(5)	142(3)
4,6LutTu4Cl	N2	S1#1	0.93(2)	2.56(2)	3.465(2)	163.2(19)
	C3	S1#1	0.959(19)	3.011(17)	3.488(2)	112.2(12)

Table 4
Mean plane data and angles between planes for 4,6LutTu2Cl, 4,6LutTu3Cl and 4,6LutTu4Cl

Compound	Plane	No.	Mean plane deviation	Atom with greatest deviation	Plane/Plane	Angle
4,6LutTu2Cl	N1–C2–C3–C4–C5–C6	1	0.0059	C2, 0.0090(0.0016)	1/2	2.57(0.10)
	N2–C7–S1–N3	2	0.00440	C7, 0.0075(0.0019)	2/3	39.37(0.07)
	C8–C9–C10–C11–C12–C13	3	0.0132	C8, 0.0203(0.0020)	1/3	38.44(0.08)
4,6LutTu3Cl	N1–C2–C3–C4–C5–C6	1	0.0051	C5, 0.0078(0.0037)	1/2	5.14(0.15)
	N2–C7–S1–N3	2	0.0049	C7, 0.0084(0.0039)	2/3	7.14(0.16)
	C8–C9–C10–C11–C12–C13	3	0.0048	C13, 0.0077(0.0038)	1/3	3.01(0.15)
4,6LutTu4Cl	N1–C2–C3–C4–C5–C6	1	0.0027	N1, 0.0040(0.0012)	1/2	8.21(0.11)
	N2–C7–S1–N3	2	0.0005	C7, 0.0008(0.0015)	2/3	8.57(0.11)
	C9–C10–C11–C12–C13–C14	3	0.0064	C9, 0.0103(0.0016)	1/3	4.58(0.12)

The intermolecular N2H2···S1 interaction, which is present in thioureas with intramolecular hydrogen bonding to either an oxygen [13–20] or nitrogen [1,3–8], as well as thioureas without intramolecular hydrogen bonding [21–25], appears to be strongest for 4,6LutTu4ClPh and weakest for 4,6LutTu3ClPh based on the H2···S1 distances, Table 3. Among the series of *N*-2-(4,6-lutidyl)-*N*′-tolylthioureas (i.e. 4,6LutTu2T, 4,6LutTu3T, and 4,6LutTu4T), 4,6LutTu2T has the shortest H2···S distance, 2.48(1) Å compared to 2.53(1) and 2.59(1) Å for 4,6LutTu3T, and 4,6LutTu4T [4], and is significantly shorter than 2.60(3) Å found for 4,6LutTu2ClPh. The H2···S distance for 4,6LutTu4ClPh, 2.56(2) Å, is comparable to 2.59(3) Å found for 4,6LutTu4T, which suggests that the chloro atom in 4,6LutTu2ClPh and 4,6LutTu3ClPh significantly weakens the N2–H2···S1 interaction. Similarly, *N*-(3-picolyl)-*N*′-phenylthiourea, 3PicTuPh, in which the methyl group in the 3-position sterically hinders the N2–H2···S1 interaction, has a very long H2···S1 distance, an average of 3.09 Å [7]. Each of the present thioureas appears to have a weak interaction, C3–H3···S1; S1 is positioned almost equally between C3 and N2 of another molecule.

The intramolecular N3–H3···N1 interaction has a longer H3···N1 distance for 4,6LutTu2ClPh than 4,6LutTu3ClPh and 4,6LutTu4ClPh, the latter two being essentially equal, Table 3. The weak interaction with the chlorine (i.e. bifurcation) in 4,6LutTu2ClPh may contribute to the weakness of the N3–H3···N1 interaction, but a series of 2-pyridylthioureas with a 2-methoxy group [8], some with bifurcation and others not, showed that the H3···N1 distance was not affected by bifurcation. There is considerable difference in the N3–H3···N1 angle with 4,6LutTu2ClPh being substantially smaller than the other two, possibly because of the weak N3–H3···Cl1 interaction. The H3···Cl1 distance is comparable to the H2···S1 distance of the intermolecular hydrogen bonding interaction, which suggests that this is a meaningful interaction. The bifurcated hydrogen bond for *N*-2-(4,6-lutidyl)-*N*′-2-methoxyphenylthiourea, 4,6LutTu2-MeOPh [8], has the following distances and angles: N3–H3, 0.966 Å; H3···O1, 2.275(3) Å; N3···O1, 2.612(4) Å and ∠N3–H3···O1, 99.5(2)°. Therefore, the N3–H3···Cl angle, 105.8(19) °, is comparable, and we consider H3 to be bifurcated and interacting with N1 and Cl1 in 4,6LutTu2ClPh. The chloro substituent in 4,6LutTu3ClPh is weakly interacting with H5 of the pyridine ring of an adjacent molecule, and in the lattice of 4,6LutTu4ClPh the hydrogens in the disordered

Table 5
Partial ^{1}H NMR assignments for 4,6LutTu2Cl, 4,6LutTu3Cl and 4,6LutTu4Cl

Thiourea	N3*H*	N2*H*	aromatic C*H*	C6H_3	C4H_3
4,6LutTu2ClPh	14.09	8.21	8.36d (C10H) 7.38d (C13H)	2.40	2.23
4,6LutTu3ClPh	14.23	8.60	7.78s (C9H) 7.53d (C11H)	2.38	2.23
4,6LutTu4ClPh	14.11	8.26	7.62d (C10H,C12H)	2.39	2.23

$C15H_3$ have H···Cl1 distances between 3.00(2) and 3.29(2) Å (i.e., 3 different Cl's).

The data for the mean planes is shown in Table 4, and there is a considerable difference in the planarity of the three molecules. The angle between the mean planes of the two rings, as a measure of planarity, is a useful parameter since the angle between the mean planes of the pyridine ring and the thiourea moiety are <15° for the 2-pyridyl thioureas studied to date [3–8]. 4,6LutTu3ClPh and 4,6LutTu4ClPh are quite planar with angles between the two rings of 3.01(0.15) and 4.58(0.12)°, respectively. However, 4,6LutTu-2ClPh has an angle of 38.44(0.08)° and this larger angle is presumably due to the steric requirement of Cl1, which would likely interact to a greater extent with H3 in a more planar structure [8]. The angle between the pyridine ring and aromatic ring mean planes in 4,6-LutTu2OMe is 27.2(0.4)° [8].

3.2. Thermal studies

The DSC plots of these thioureas show a sharp peak due to melting with the following values for ΔH_{fus}: 4,6LutTu2ClPh, 42.2 kJ mol^{-1}; 4,6LutTu3ClPh, 32.8 kJ mol^{-1} and 4,6LutTu4ClPh, 66.1 kJ mol^{-1}. The lower values for 4,6LutTu2ClPh and 4,6LutTu3ClPh indicate weaker crystal packing due to the position of the Cl atoms and their steric requirements. The very low value for 4,6LutTu3ClPh is consistent with the even lower value found for 3PicTuPh, 21.3 kJ mol^{-1}. Although *N*-2-(4,6-lutidyl)-*N′*-phenylthiourea, 4,6LutTuPh, has the highest value compared to six previous heterocyclic thioureas measured, 50.9 kJ mol^{-1}, it is far less than found for 4,6LutTu4ClPh indicating that not only is the N2H2···S1 interaction stronger, but the interaction of Cl with hydrogens on other molecules in the lattice must contribute. The melting points listed in Section 2 indicate these same conclusions.

3.3. Spectral Studies

The ^{1}H NMR signals, Table 5, show little change in chemical shift for the three compounds of this study. The downfield position of N3*H* is consistent with its involvement in intramolecular hydrogen bonding in solution. The presence of a methyl group in the 6-position on the pyridyl ring in a previous study (i.e. 6PicTuPh and 4,6LutTuPh) [7]was found to provide shielding to the N3*H* resonance due to the diamagnetic anisotropic effect of the C6–CH_3 bond. Therefore, it is expected that the N3*H* resonance will be found further downfield in thioureas without a 6-methyl substituent, which are under study in our laboratory. We have tentatively assigned some of the phenyl ring *H*'s based on chemical shift and multiplicity of the signal, Table 5.

4. Conclusions

The weak interaction between H3 and Cl1 in 4,6LutTu2ClPh, as well as the N3H3···N1 interaction, is another example of bifurcated intramolecular hydrogen bonding. Other examples of chlorine being involved in bifurcated hydrogen bonding interactions are anticipated with different substitution on the pyridine ring. The low value of the ΔH_{fus} for 4,6LutTu3ClPh shows how the position of a heteroatom on the aromatic ring affects the intermolecular interactions. The unusually large value of ΔH_{fus} for 4,6LutTu4ClPh suggests that the interaction between Cl and a neighboring molecule's $C15H_3$ is significant.

5. Supplementary Material

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no CCDC-158984 for 4,6LutTu2ClPh, CCDC-158985 for 4,6LutTu3ClPh, and CCDC-158986 for 4,6LutTu2ClPh. Copies of available material can be obtained, free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: +44-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

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