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

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Abstract

N-2-(4-picolyl)-*N*′-4-chlorophenylthiourea, 4PicTu4ClPh, triclinic, *P*-1, $a = 7.5235(3)$, $b = 9.1585(5)$, $c = 10.5158(7)$ Å, $\alpha = 76.015(3)$, $\beta = 70.015(4)$, $\gamma = 82.010(4)°$, $V = 1309.8(2)$ Å^3 and $Z = 2$; *N*-2-(5-picolyl)-*N*′-4-chlorophenylthiourea, 5PicTu4ClPh, monoclinic, $P2_1/c$, $a = 15.139(2)$, $b = 4.8386(3)$, $c = 17.338(2)$ Å, $\beta = 90.661(4)°$, $V = 1270.0(2)$ Å^3 and $Z = 4$ and *N*-2-(6-picolyl)-*N*′-4-chlorophenylthiourea, 6PicTu4ClPh, monoclinic, $C2/c$, $a = 33.520(6)$, $b = 4.0750(3)$, $c = 18.658(4)$ Å, $\beta = 97.500(6)°$, $V = 2526.8(7)$ Å^3 and $Z = 8$. The most striking difference between the structures of the three thioureas is the difference in planarity, and among the four *N*-2-(picolyl)-*N*′-4-chlorophenylthioureas, their values for ΔH_{fus}. © 2002 Elsevier Science B.V. All rights reserved.

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

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1. Introduction

After a ^{1}H NMR study of the intramolecular hydrogen bonding between the pyridyl nitrogen and N′H for *N*-(2-pyridyl)thioureas [1], the structure of *N*-(2-pyridyl)-*N*′-phenylthiourea was reported [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 recent structural studies of substituted *N*-(2-pyridyl)-*N*′-arylthioureas including *N*-(2-pyridyl)-*N*′-tolylthioureas [3] and the *N*-2-(4,6-lutidyl)-*N*′-tolylthioureas [4]. In addition, the structures of *N*-(5-bromo-2-pyridyl)-*N*′-2-(2,5-dimethoxyphenyethyl)thiourea [5] and four thioureas with *p*-substituted aryl groups including *N*-(2-pyridyl)-*N*′-(4-chlorophenyl)thiourea [6] have been reported. A study of the structural, thermal and spectral properties of the *N*-2-(picolyl)- and *N*-(4,6-lutidyl)-*N*′-phenylthioureas has been communicated [7]. A recent report showed that some members of a series of *N*-(2-pyridyl)-*N*′-2-methoxyphenylthioureas possess bifurcated intramolecular hydrogen bonding involving the pyridyl nitrogen and methoxy oxygen [8]. A similar study of the *N*-2-(4,6-lutidyl)-*N*′-chlorophenylthioureas showed that *N*-2-(4,6-lutidyl)-*N*′-4-chlorophenylthiourea has interaction between methyl hydrogens and the chlorine atoms (i.e. H···Cl distances <3.00 Å), as well as the expected intramolecular and intermolecular hydrogen bonding [9]. Since C–H···Cl interactions in *N*-2-(pyridyl)-*N*′-4-

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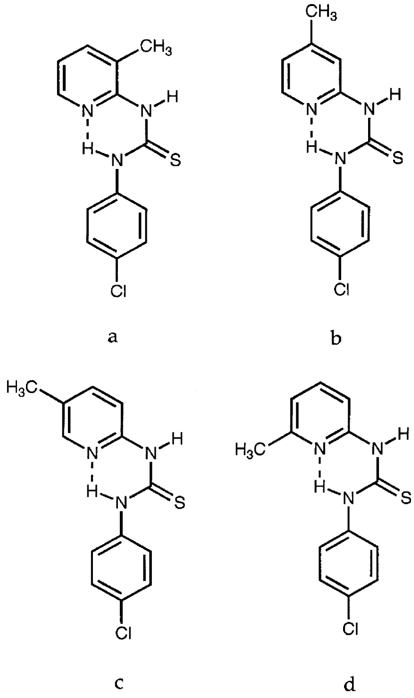

Fig. 1. Representation of (a) *N*-2-(3-picolyl)-; (b) *N*-2-(4-picolyl)-; (c) *N*-2-(5-picolyl)- and (d) *N*-2-(6-picolyl)-*N*′-4-chlorophenylthiourea.

chlorophenylthiourea are weaker (i.e. H···Cl distances >3.00 Å) [6], we decided to extend our study to the four *N*-2-picolyl-*N*′-4-chlorophenylthioureas, 3PicTu4ClPh, 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh, Fig. 1.

2. Experimental

2.1. Preparative, spectral and thermal methods

4-Chlorophenyl isothiocyanate and the four 2-aminopicolines were purchased from Aldrich and used as received. The desired 2-aminopicoline was mixed in a 1:1 molar ratio with 4-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, the thioureas crystallized from solution and were filtered and stored until required for spectral and thermal characterization. The yields are ca. 70% for each of the thioureas and their melting points are listed in Table 5 with the enthalpies of fusion. Their ^{1}H NMR spectra were recorded in $CDCl_3$ with a Varian 300 MHz spectrometer and the enthalpies of fusion were obtained with approximately 3 mg samples at a heating rate of 10 °C/min using a Perkin–Elmer Differential Scanning Calorimeter, DSC7.

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

Crystals of the 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 (4PicTu4ClPh) or a Nonius Kappa CCD Diffractometer (5PicTu4ClPh and 6PicTu4ClPh), both 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 hydrogens attached to nitrogens and carbons, except for methyl groups, were found on a difference-Fourier map and refined isotropically for 4PicTu4ClPh and 6PicTu4ClPh. The hydrogens on carbons for 5PicTu4ClPh were fixed and allowed to ride on their parent atoms with a fixed isotropic temperature factor, $U = 0.05$ Å^2.

Table 5
Melting points (°C), ΔH_{fus} values (kJ/mol) and selected ^{1}H NMR assignments for the *N*-2-pyridyl-*N*′-4-chlorophenylthioureas

Thiourea	mp	ΔH_{fus}	N3H (N′H)	NH (N2H)	C6H	CH_3	References
PyTu4ClPh	188–190	34.3	13.91	8.32	7.84		[6]
3PicTu4ClPh	117–119	16.8	13.96	8.25	8.06	2.36	This work
4PicTu4ClPh	186–187	35.2	13.73	8.30	8.04	2.36	This work
5PicTu4ClPh	200–201	51.1	14.60	8.18	7.93	2.42	This work
6PicTu4ClPh	190–192	40.1	14.04	8.36		2.51	This work
4,6LutTu4ClPh	226–227	66.5	14.11	8.26		2.39, 2.23	[9]

Table 1
Crystallographic data and methods of data collection, solution and refinement for 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh

Crystal data	4PicTu4Cl	5PicTu4Cl	6PicTu4Cl
Empirical formula	$C_{13}H_{12}ClN_3S$	$C_{13}H_{12}ClN_3S$	$C_{13}H_{12}ClN_3S$
Crystal color, habit	Colorless, prism	Colorless, needle	Colorless, needle
Crystal size (mm^3)	0.25 × 0.20 × 0.12	0.65 × 0.05 × 0.05	0.16 × 0.07 × 0.05
Temperature (K)	293(2)	130(2)	130(2)
Crystal system	Triclinic	Monoclinic	Monoclinic
Space group	*P*-1(#2)	$P2_1/c$(#14)	*C*2/*c*(#15)
a (Å)	7.5235(3)	15.139(2)	33.520(6)
b (Å)	9.1585(5)	4.8386(3)	4.0750(3)
c (Å)	10.5158(7)	17.338(2)	18.658(4)
α (°)	76.015(3)	90	90
β (°)	70.015(4)	90.661(4)	97.500(6)
γ (°)	82.010(4)	90	90
Volume (Å^3)	1309.8(2)	1270.0(2)	2526.8(7)
Z	2	4	8
Formula weight	277.77	277.77	277.77
Density (calcd) (g/cm^3)	1.399	1.453	1.460
Absorp. coeff. (mm^{-1})	0.432	0.449	0.451
F(000)	288	576	1152
Index ranges	$-8 \le h \le 8$	$-19 \le h \le 19$	$0 \le h \le 38$
	$-10 \le k \le 10$	$-4 \le k \le 4$	$-4 \le k \le 4$
	$-12 \le l \le 12$	$-22 \le l \le 22$	$-21 \le l \le 21$
θ range for data collection (°)	2.64–24.71	3.55–28.27	3.07–24.79
Total reflections	3954	5026	2076
Independent reflects, R_{int}	2183, 0.0303	3776, 0.1269	1328, 0.0852
Absorption correction	Ψ-scan	HKL-Scalepack	HKL-Scalepack
Max and min transmissions	0.9499 and 0.8996	0.9770 and 0.7590	0.9788 and 0.6711
Goodness-of-fit	1.017	0.855	0.867
Largest diff. peak (e/Å^3)	0.176	0.293	0.291
Largest diff. hole (e/Å^3)	−0.214	−0.284	−0.291
R_1, wR	0.0365, 0.0897	0.0471, 0.0709	0.0454, 0.0891
R_1, wR (all reflections)	0.0594, 0.0993	0.1461, 0.0879	0.0836, 0.1000

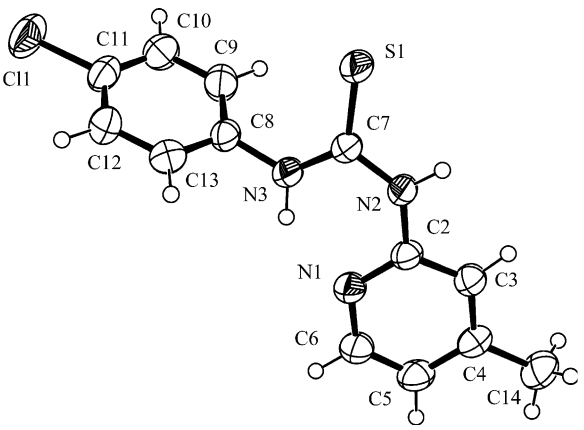

Fig. 2. ORTEP drawing showing 4PicTu4Cl with atom numbering scheme and displacement ellipsoids at 50% probability level.

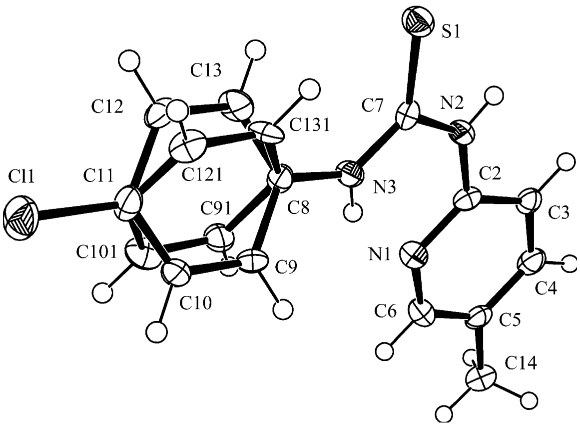

Fig. 3. ORTEP drawing showing 5PicTu4Cl with atom numbering scheme and displacement ellipsoids at 50% probability level. The disorder in the aryl ring is equally populated.

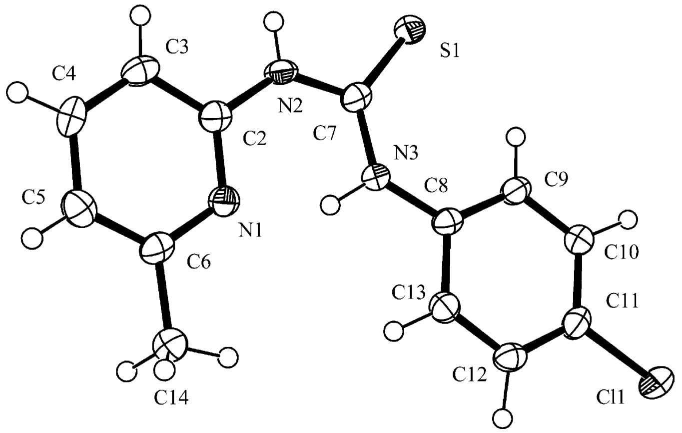

Fig. 4. ORTEP diagram showing 6PicTu4Cl with atom numbering scheme and displacement ellipsoids at 50% probability level.

DENZO [10] was used for data reduction and scattering factors are from Wassmaire and Kirfel [11]. The structures were solved with MaXus [12], structure refinement for the three thioureas was carried out with SHELXL-97 and the graphics used are Zortep [13]. Table 1 summarizes the crystal data, collection information and refinement data for these thioureas.

Table 2
Selected bond distances (Å) and angles (°) for 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh

Distances (Å)	4PicTu4Cl	5PicTu4Cl	6PicTu4Cl
S1–C7	1.674(2)	1.682(3)	1.674(3)
N1–C2	1.331(3)	1.332(4)	1.334(4)
N1–C6	1.344(3)	1.343(3)	1.358(4)
C2–N2	1.401(3)	1.404(3)	1.394(4)
N2–C7	1.370(2)	1.369(4)	1.376(4)
N3–C7	1.332(3)	1.344(4)	1.350(4)
N3–C8	1.434(3)	1.414(3)	1.410(4)
C11–Cl1	1.745(2)	1.746(3)	1.742(3)
Angles (°)			
C2–N1–C6	116.68(18)	117.3(3)	118.7(3)
N1–C2–C3	123.36(19)	123.0(3)	123.0(3)
N1–C2–N2	118.23(17)	118.9(3)	118.6(3)
N2–C2–C3	118.42(19)	118.1(3)	118.4(3)
C2–N2–C7	130.15(18)	130.7(3)	131.9(3)
S1–C7–N2	119.63(16)	118.4(2)	118.1(2)
N2–C7–N3	116.46(18)	115.0(3)	114.2(3)
S1–C7–N3	123.90(15)	126.5(2)	127.7(2)
C7–N3–C8	125.30(18)	130.8(3)	131.0(3)
N3–C8–C9	121.10(19)	117.7(4), 114.0(4)[a]	126.7(3)
N3–C8–C13	119.0(2)	122.9(4), 127.5(4)[b]	114.7(3)

[a] N3–C8–C91 angle.

[b] N3–C8–C131 angle.

3. Results and discussion

3.1. Structural studies

The ORTEP diagrams of 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh are shown in Figs. 2–4, respectively. Unfortunately, we were unable to obtain suitable crystals for 3PicTu4ClPh. 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh all crystallize with one unique molecule in the unit cell, but even at low temperature there is disorder (i.e. equal population) in the aryl ring of 5PicTu4ClPh. The previously reported *N*-2-pyridyl-*N*′-4-chlorophenylthiourea, PyTu4ClPh, crystallizes *P*-1 with two different molecules in the unit cell [6] and *N*-2-(4,6-lutidyl)-*N*′-4-chlorophenylthiourea, 4,6LutTu4ClPh, *C*2/*c* with one unique molecule [9] like 6PicTu4ClPh. Like other pyridyl thioureas [2–9], 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh 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 [14–21] and 'cis–cis' like *N*-phenyl-*N*′-phenylthiourea [22]. The three thioureas have essentially the same bond distances, Table 2. There are more differences among the bond angles, particularly close to the thiourea moiety;

Table 3
Intramolecular and intermolecular hydrogen bond distances (Å) and angles (°) for 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh, as well as C–H···X interactions with H···X distances of less than 3.00 Å

Thiourea	D	A	D–H	H···A	D–H···A	∠(D–H···A)
Intramolecular						
4PicTu4Cl	N3	N1	0.90(3)	1.92(2)	2.650(2)	137(2)
5PicTu4Cl	N3	N1	0.86(3)	1.88(3)	2.633(3)	145(3)
6PicTu4Cl	N3	N1	0.93(3)	1.80(3)	2.647(4)	149(3)
Intermolecular						
4PicTu4Cl[a]	N2	S1#1	0.92(3)	2.49(3)	3.383(2)	165.0(19)
	C3	S1#1	0.92(2)	2.97(2)	3.729(2)	139.9(16)
	C6	Cl1#2	0.98(2)	2.96(2)	3.585(2)	122.5(16)
5PicTu4Cl[b]	N2	S1#1	0.83(3)	2.61(3)	3.411(3)	163(3)
	C3	S1#1	0.95	2.81	3.612(3)	143.2
	C91	Cl1#2	0.95	2.80	3.594(6)	142.1
6PicTu4Cl[c]	N2	S1#1	0.82(3)	2.60(3)	3.403(3)	164(3)

[a] #1: $1-x, -y, -z$; #2: $2-x, -y, 1-z$.

[b] #1: $1-x, 1-y, 2-z$; #2: $2-x, 1-y, 2-z$.

[c] #1: $1-x, y, 0.5-z$; #2: $0.5-x, -0.5+y, 0.5-z$.

for example, C7–N3–C8 is 125.30(18)° for 4PicTu4ClPh, 130.8(3)° for 5PicTu4ClPh and 131.0(3)° for 6PicTu4ClPh.

The non-bonding N3···N1 distance for 5PicTu4ClPh, 2.633(3) Å, is less than for the other two thioureas of this study, which are both closer to the average of 2.669(6) Å for PyTu4ClPh [6] and 2.652(2) Å for 4LutTu4ClPh [9]. These non-bonding distances of 4PicTu4ClPh and 6PicTu4ClPh are somewhat longer than the analogous *N*-2-(4-picolyl)- [23] and *N*-2-(6-picolyl)-N'-4-tolylthioureas [24], 2.630(3) and 2.623(2) Å, indicating a difference in the electronic effect by the aryl substituent on this interaction. However, the N3···N1 distances for the corresponding *N*-2-(4-picolyl)- and *N*-2-(6-picolyl)-N'-phenylthioureas, 2.622(4) and 2.647(3) Å [7], indicate that other factors influence this interaction. For thioureas with pyridine rings the angle for the N3–H3···N1 interaction ranges between 135 and 150° [2–4,6–9,23,24] as found for 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh.

The intermolecular N2H2···S1 interaction is present in thioureas with intramolecular hydrogen bonding to an oxygen [14–21] or nitrogen atom [2–9], as well as thioureas without intramolecular hydrogen bonding [22,25–28]. The N2···S1 non-bonding distances show

Table 4
Mean plane data and angles between planes for 4PicTu4ClPh, 5PicTu4ClPh and 6PicTu4ClPh

Compound	Plane	Plane	Mean plane deviation	Atom with greatest deviation	Plane/plane	Angle
4PicTu4Cl	N1–C2–C3–C4–C5–C6	1	0.0081	C5, 0.0098(0.0016)	2/1	12.77(0.21)
	N2–C7–S1–N3	2	0.0051	N2, 0.0067(0.0013)	3/2	67.79(0.10)
	C8–C9–C10–C11–C12–C13	3	0.0076	C10, 0.0118(0.0018)	1/3	62.30(0.07)
5PicTu4Cl	N1–C2–C3–C4–C5–C6	1	0.0066	C2, 0.0098(0.0019)	2/1	4.81(0.14)
	N2–C7–S1–N3	2	0.0015	C7, 0.0025(0.0023)	3/2	28.98(0.22)
	C8–C9–C10–C11–C12–C13	3	0.0255	C11, 0.0426(0.0041)	1/3	30.24(0.21)
	C8–C91–C101–C11–C121–C131	4	0.0023	C121, 0.0034(0.0048)	3/4	47.85(0.23)
6PicTu4Cl	N1–C2–C3–C4–C5–C6	1	0.0040	C6, 0.0054(0.0019)	2/1	7.55(0.13)
	N2–C7–S1–N3	2	0.0018	C7, 0.0032(0.0021)	3/2	7.98(0.14)
	C8–C9–C10–C11–C12–C13	3	0.0014	C9, 0.0023(0.0015)	1/3	7.12(0.15)

small differences for the present thioureas, but all three are larger than PyTu4ClPh, 3.413(5) Å [6], and smaller than 3.465(2) Å for 4,6LutTu4ClPh [9], suggesting a steric effect due to the methyl group(s) on the pyridine ring.

Like other *N*-2-pyridyl-*N*′-arylthioureas [6–9,27,28], 4PicTu4ClPh and 5PicTu4ClPh have a weak interaction, C3–H3···S1; the interaction is weaker in 6PicTu4ClPh with a H3···S distance of 3.08(3) Å. S1 is positioned almost equally between C3 and N2 of another molecule and the C3···S1 and H3···S1 distances are often ca. 0.2 Å longer than the corresponding distances of the N2H2···S1 interaction. Of note is that *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]. The comparatively low melting point and ΔH_{fus} (vide infra) for 3PicTu4ClPh suggests weak intermolecular interaction and probably contributes to our inability to acquire a suitable crystal. Other C–H···X interactions in which a hydrogen is within less than 3.00 Å of X and the angle is above 110° are listed in Table 3.

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 pyridyl and aryl ring, as a measure of planarity, is a useful parameter. This is particularly true since the angle between the mean planes of the pyridine ring and the thiourea moiety do not show large variation; they have been found to be less than 15° for the *N*-2-pyridyl-*N*′-arylthioureas studied to date [2–9,23,24]. 6PicTu4ClPh is quite planar although less planar than 4,6LutTu4ClPh, which has an angle between the mean planes of the two rings of less than 5° [9]. In contrast, the angle between the rings in 4PicTu4ClPh is similar to the average for the two unique molecules of PyTu4ClPh, ca. 60° [6] and 5PicTu4Cl falls in between at about 30°. The disorder of the aryl ring in 5PicTu4Cl has 50% occupancy for each form. One of the rings has a C–H within less than 3.0 Å of a Cl1 of neighboring molecule, Table 3, while the other ring has the analogous CH greater than 3.0 Å from a neighboring Cl1.

3.2. Thermal studies

The DSC plots of these thioureas show a sharp peak due to melting, and values for ΔH_{fus} are shown in Table 5 along with their melting points. Data for *N*-(2-pyridyl)-*N*′-4-chlorophenylthiourea, PyTu4ClPh [6], and *N*-2-(4,6-lutidyl)-*N*′-4-chlorophenylthiourea, 4,6LutTu4ClPh [9] are included. The very low value for 3PicTu4ClPh is due to the 3-methyl group reducing the strength of the N–H···S interaction. Similarly, the ΔH_{fus} for *N*-2-(3-picolyl)-*N*′-phenylthiourea is 21.3 kJ/mol compared to 43.5 kJ/mol for *N*-2-(6-picolyl)-*N*′-phenylthiourea [7]. In addition, *N*-2-(3-picolyl)-*N*′-4-tolylthiourea [23], *N*-2-(4-picolyl)-*N*′-4-tolylthiourea [23], and *N*-2-(6-picolyl)-*N*′-4-tolylthiourea [24], have ΔH_{fus} values of 22.5, 44.6 and 47.2 kJ/mol, respectively. These data confirm that blocking the 3-methyl position of the pyridine ring reduces the strength of the intermolecular N2–H···S1 interaction and also, the weak C3–H···S1 interaction is absent.

Since the tolyl analogues [23,24] of 3PicTu4Cl, 4PicTu4Cl and 6PicTu4Cl all have higher ΔH_{fus} values, the C–H···Cl interactions listed in Table 3 do not have much affect on the intermolecular attractions of the present molecules. However, for *N*-2-(4,6-lutidyl)-*N*′-phenylthiourea, 4,6LutTuPh, $\Delta H_{\text{fus}} =$ 50.9 kJ/mol and is far less than found for 4,6LutTu4ClPh, $\Delta H_{\text{fus}} = 66.5$ *kJ/mol* [9]. Therefore, other factors besides the dominant N2–H···S interaction have an effect on the packing of these molecules and their enthalpies of fusion. These other factors include the number of molecules in the unit cell and the planarity of the molecule; in general, the more planar molecules have higher ΔH_{fus} values. The melting points listed in Table 5 are consistent with these same conclusions.

3.3. NMR spectral studies

The ^{1}H NMR signals, Table 5, show little change in chemical shift for the thioureas 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, but this is only true for 5PicTu4Cl of this study.

4. Conclusions

The difference in the planarity of the three molecules is striking, although not unexpected, given the difference between PyTu4Cl [6] and 4,6LutTu4Cl [9] noted earlier. The longer distance for C3H3···S1 in 6PicTu4Cl is in agreement with earlier findings for 4,6LutTu4Cl, which also has the H3···S1 distance above 3.00 Å [9]. The very low values for the ΔH_{fus} and melting point for 3PicTu4Cl is consistent with results found for other 3-substituted *N*-2-pyridyl-N′-phenylthioureas [23] and makes it difficult to acquire suitable crystals for structural study.

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-167228 for 4PicTu4ClPh, CCDC-167229 for 5PicTu4ClPh, and CCDC-167230 for 6PicTu4ClPh. Copies of available material can be obtained, free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: +44-(0) 1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

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