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Structural studies of *N*-2-(6-aminopyridine)-*N′*-arylthioureas

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Abstract

N-2-(6-aminopyridine)-*N′*-2-methoxyphenylthiourea, 6AmTu2OMe, monoclinic, *C*2/*c*, $a = 16.3150(17)$, $b = 11.3000(13)$, $c = 14.8970(9)$ Å, $\beta = 101.219(7)°$, $V = 2693.9(4)$ Å^3 and $Z = 8$; *N*-2-(6-aminopyridine)-*N′*-4-chlorophenylthiourea, 6AmTu4Cl, monoclinic, $P2_1/n$, $a = 14.2080(9)$, $b = 9.8060(7)$, $c = 19.041(1)$ Å, $\beta = 104.616(4)°$, $V = 2567.0(3)$ Å^3 and $Z = 8$ and *N*-2-(6-aminopyridine)-*N′*-4-nitrophenylthiourea, 6AmTu4NO_2, monoclinic, $P2_1/n$, $a = 10.809(8)$, $b = 10.427(8)$, $c = 12.303(7)$ Å, $\beta = 110.47(7)°$, $V = 1299.1(16)$ Å^3 and $Z = 4$. The intramolecular hydrogen bonding common to 2-pyridylthioureas between N′H and the pyridine nitrogen is present as well as the intermolecular hydrogen bonding involving the thione sulfur and the NH hydrogen, which is often found for thioureas. In addition, the presence of an amino group in the 6-position of the pyridine ring results in additional intermolecular hydrogen bonding as do the functional groups on the aryl ring of these thioureas.

Keywords: 2-Pyridylthioureas; 6-Aminopyridine; Thiourea; Crystal structures; Hydrogen bonding

1. Introduction

Numerous structural studies of *N*-2-pyridyl-*N′*-arylthioureas with substituents on either or both rings have recently appeared [1–11]. Although there are no large differences in bond distances and angles among these thioureas, other structural parameters have shown substantial variation. Intramolecular *N′*–H···N and intermolecular N–H···S hydrogen bonding, as well as the planarity of the molecules, are representative of structural parameters that are affected by substitution on either or both rings. Substituents on the rings that have affected the intramolecular and intermolecular hydrogen bonding and the planarity of the molecules communicated to date have been bromo [7,11], chloro [7,9–11], methoxy [5,7,14], methyl [2,4,5,8–15] and thiomethoxy [15] functions. Here we report the structural nature of three thioureas with an amino group on the 6-position of the pyridine ring, *N*-2-(6-aminopyridine)-*N′*-2-methoxyphenylthiourea, 6AmTu2OMe; *N*-2-(6-aminopyridine)-*N′*-4-chlorophenylthiourea, 6AmTu4Cl; and *N*-2-(6-aminopyridine)-*N′*-4-nitrophenylthiourea, 6AmTu4NO_2, Fig. 1. The methoxy,

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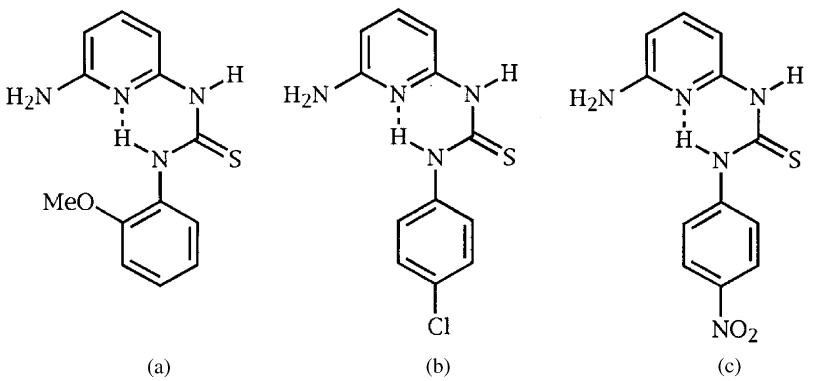

Fig. 1. Representations of 6AmTu2OMe, 6AmTu4Cl and 6AmTu4NO_2.

chloro and nitro substituents on the aryl ring, as well as the amino group in these three thioureas, are expected to provide additional hydrogen bonding interactions.

2. Experimental

2,6-diaminopyridine, 2-methoxyphenyl isothiocyanate, 4-chlorophenyl isothiocyanate, and 4-nitrophenyl isothiocyanate were purchased from Aldrich and used as received. 2,6-diaminopyridine was mixed in a 1:1 molar ratio (i.e. 2 mmol) with the different isothiocyanates in 95% ethanol (40 ml), and the mixture stirred with warming (55 °C) 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 and dried on a warm plate. The yields are ca. 65% for each of these thioureas and their melting points are all above 200 °C.

Crystals of 6AmTu2OMe, 6AmTu4Cl, and 6AmTu4NO_2 were grown by slow evaporation of 1:1 by volume acetone-anhydrous ethanol mixtures at room temperature. The thiourea crystals were mounted in random orientation on a glass fiber on a Nonius Kappa CCD or Nonius MACH3 diffractometer (6AmTu4NO_2), Mo Kα ($\lambda = 0.71073$ Å). Cell constants and an orientation matrix for data collections were obtained by least squares refinements of the diffraction data from more than 500 reflections or 25 well-centered reflections (6AmTu4NO_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 the hydrogens were found on a difference Fourier map and refined isotropically except for the methoxy hydrogens, which were placed and refined with a riding model. Scattering factors are from Waasmaier and Kirfel [16]. The structures were solved with MaXus [17], structure refinement was carried out with SHELXL-97 [18] and the graphics used are Zortep [19]. Table 1 summarizes the crystal data, collection information, and refinement data for 6AmTu2OMe, 6AmTu4Cl, and 6AmTu4NO_2.

3. Results and discussion

Bond distances and angles are listed in Table 2, the hydrogen bonding parameters are listed in Table 3 and the mean plane data in Table 4. Ortep drawings for 6AmTu2OMe, 6AmTu4Cl and 6AmTu4NO_2 are shown in Figs. 2–4, respectively.

3.1. 6AmTu2OMe

N-2-pyridylthioureas with a methoxy group in the 2-position of the aryl ring are of special interest in that several thioureas including *N*-2-(6-picolyl)-*N*′-2-methoxy-phenylthiourea, 6PicTu2OMe [5], have bifurcated hydrogen bonding involving the methoxy group and pyridine nitrogen. However, the hydrogen

Table 1
Crystallographic data and methods of data collection, solution and refinement for 6AmTu2OMe, 6AmTu4Cl, and 6AmTu4NO_2

Crystal data	6AmTu2OMe	6AmTu4Cl	6AmTu4NO_2
Empirical formula	$C_{13}H_{14}N_4OS$	$C_{12}H_{11}ClN_4S$	$C_{12}H_{11}N_5O_2S$
Crystal color, habit	Colorless, prism	Colorless, prism	Yellow, prism
Crystal size (mm)	0.17 × 0.17 × 0.17	0.53 × 0.29 × 0.14	0.30 × 0.22 × 0.20
Temperature (K)	293(2)	293(2)	293(2)
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$C2/c$(#15)	$P2_1/n$(#14)	$P2_1/n$(#14)
a, (Å)	16.3150(17)	14.2080(9)	10.809(8)
b, (Å)	11.3000(13)	9.8060(7)	10.427(8)
c, (Å)	14.8970(9)	19.041(1)	12.303(7)
α,°	90	90	90
β,°	101.219(7)	104.616(4)	110.47(7)
γ,°	90	90	90
Volume ($Å^3$)	2693.9(4)	2567.0(3)	1299.1(16)
Z	8	8	4
Formula weight	274.34	278.76	289.32
Density (calcd.) (g/cm^3)	1.353	1.443	1.479
Absorp. coeff. (mm^{-1})	0.238	0.447	0.259
F(000)	1152	1152	600
Index ranges	$0 \le h \le 19$	$-17 \le h \le 17$	$0 \le h \le 12$
	$-13 \le k \le 11$	$-11 \le k \le 12$	$0 \le k \le 11$
	$-15 \le l \le 14$	$-23 \le l \le 22$	$-13 \le l \le 12$
2θ range for data collection	2.47–24.70	2.55–26.35	2.16–23.20
Total reflections	3403	8662	1894
Independent reflects (R_{int})	2087, 0.0491	5207, 0.0598	1790, 0.0539
Absorption correction	HKL-SCALEPACK	HKL-SCALEPACK	Ψ-scan
Max. and min. transmissions	0.9607 and 0.9607	0.9401 and 0.7977	0.9501 and 0.9264
Extinction coefficient	none	0.0016(5)	None
Goodness-of-fit	0.905	0.835	1.097
Largest diff. peak, hole (e $Å^{-3}$)	0.136, −0.157	0.223, −0.246	0.361, −0.325
R_1, wRM	0.0418, 0.0821	0.0419, 0.0796	0.0478, 0.1208
R_1, wR (all reflections)	0.1042, 0.0821	0.1547, 0.1019	0.0685, 0.1448

bonding data in Table 3 indicates that bifurcated N1···H3···O1 bonding is not present in 6AmTu2OMe even though the H3···O1 distance, ca. 2.8 Å, and the ∠N3–H3···O1 angle of ca. 130(2)° would suggest interaction. Therefore, it is of interest to compare bond distances and angles of 6AmTu2OMe with those of the above-mentioned 6PicTu2OMe, which has two crystallographically different molecules [5]. There is no significant difference in the bond distances of 6AmTu2OMe and the average bond distances of 6PicTu2OMe. However, there are small differences (i.e. 2 − 3°) in the bond angles around C7 for the two molecules: ∠N2–C7–N3, ∠S1–C7–N3, and ∠C7–N3–C8 are 118.1(2), 123.8(2) and 125.0(2)°, respectively, in 6AmTu2OMe, but average 116.3(4), 126.1(3), and 128.9(3)° in 6PicTu2OMe [5].

6AmTu2OMe has the intramolecular hydrogen bond from N3H (i.e. N') to the pyridine nitrogen that is present in all N-2-pyridylthioureas studied to date [1–15]. The potential bifurcation participant, O1, is instead involved in a strong intermolecular hydrogen bond with one of the 6-amino hydrogens, as well as a weaker intermolecular hydrogen bond with N3H. The other strong intermolecular hydrogen bond is between N2H2 and S1 to form a dimer, that is often found for N(2)-pyridylthioureas [1–15], as well as other aryl thioureas [20]. In addition, it is common among N-2-pyridylthioureas for the hydrogen on C3, H3, to also interact weakly with the same sulfur [8–15] and for the H3···S1 distance to be 0.2 − 0.4 Å longer than the H2···S1 distance for the N2–H2···S1 interaction. Values with these differences are shown for 6AmTu2OMe in Table 3.

Table 2
Selected bond distances (Å) and angles (°) for 6AmTu2OMe, 6AmTu4Cl, and 6AmTu4NO$_2$

	6AmTu2Ome	6AmTu4Cl	6AmTu4NO$_2$
Distances (Å)			
S1–C7	1.683(3)	1.683(3) 1.680(3)	1.679(4)
N1–C2	1.329(3)	1.331(3) 1.329(3)	1.334(4)
N1–C6	1.353(3)	1.344(3) 1.350(3)	1.343(4)
C2–N2	1.397(3)	1.397(4) 1.411(3)	1.407(4)
N2–C7	1.366(3)	1.359(3) 1.353(3)	1.363(4)
N3–C7	1.328(3)	1.329(3) 1.343(3)	1.346(4)
N3–C8	1.426(3)	1.415(3) 1.420(3)	1.402(4)
C9–O1	1.375(3)	1.737(3) 1.746(3) (C11–Cl1)	1.453(4) (C11–N5)
C6–N4	1.363(4)	1.374(4) 1.365(4)	1.357(5)
Angles (°)			
C2–N1–C6	117.4(3)	117.5(3) 116.9(2)	118.2(3)
N1–C2–C3	124.1(3)	123.3(3) 124.5(3)	123.3(3)
N1–C2–N2	118.0(3)	118.2(3) 117.3(3)	118.1(3)
N2–C2–C3	117.9(3)	118.5(3) 118.2(3)	118.6(3)
C2–N2–C7	131.5(3)	131.8(3) 132.3(3)	132.0(3)
S1–C7–N2	118.1(2)	119.8(2) 119.4(2)	118.4(2)
N2–C7–N3	118.1(2)	116.7(3) 115.9(3)	115.0(3)
S1–C7–N3	123.8(2)	123.5(3) 124.7(2)	126.6(3)
C7–N3–C8	125.0(2)	127.4 (3) 126.5(3)	132.7(3)
N1–C6–N4	115.8(3)	116.4(3) 115.7(3)	116.2(3)

A convenient measure of planarity for *N*-2-pyridyl-*N*′-arylthioureas is the angle between the mean planes of the two rings. For thioureas that are relatively planar with a 2-substituent on the aryl ring capable donating electrons (i.e. OMe, Cl) bifurcation of hydrogen on N3, H3, of the thiourea moiety occurs. However, in 6AmTu2OMe the angle between these mean planes is 70.83(0.10)° consistent with the lack of bifurcation, but averages ca. 25° in the bifurcated 6PicTu2OMe. Like previously studied *N*-2-pyridylthioureas [1–15], the angle between the mean planes of the pyridine ring and thiourea moiety is less than 15°, which is in part due to the intramolecular N3H3···N1 hydrogen bond. Thus, the absence of bifurcation in 6AmTu2OMe can be ascribed to the lack of planarity, as well as the involvement of the OMe function in the strong intermolecular hydrogen bond with the 6-amino group.

3.2. 6AmTu4Cl

In contrast to 6AmTu2OMe, there are two crystallographically different molecules for 6AmTu4Cl. There are marginal differences in the bond distances between these two molecules, as well as between these molecules and the bond distances of 6AmTu2OMe, Table 2. The structure of *N*-2-(6-picolyl)-*N*′-4-chlorophenylthiourea, 6PicTu4Cl, has recently been reported [10], and the only bond that has a significant difference is N2–C7 (i.e. 1.376(4) Å in 6PicTu4Cl [10] and an average of 1.356(3) Å in 6AmTu4Cl). Although the bond angles of 6AmTu4Cl are similar to the analogous angles of 6AmTu2OMe, the bond angles involving C7 in 6PicTu4Cl [10] are significantly different. For example, ∠S1–C7–N3 averages 124.1(3) ° in 6AmTu4Cl, but is 127.7(2)° in 6PicTu4Cl, and ∠C7–N3–C8 averages 127.0(3)° in 6AmTu4Cl, but is 131.0(3)° in 6PicTu4Cl.

The intramolecular hydrogen bonding parameters of the two different molecules of 6AmTu4Cl are similar to each other, as well as to the parameters of 6AmTu2OMe, Table 3. In addition, the intermolecular N2H2···S2 (and N22H22–S1), which are between crystallographically different molecules, are similar to the interaction of 6AmTu2OMe. However, the C3H3···S2 and C23H23···S1 have H···S distances of 3.08(3) and 3.01(3) Å, which are weaker than found for 6AmTu2OMe, but similar to the distance

Table 3
Intramolecular and intermolecular hydrogen bond distances (Å) and angles (°) for 6AmTu2OMe, 6AmTu4Cl, and 6AmTu4NO_2, 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						
6AmTu2Ome	N3	N1	0.89(3)	1.98(3)	2.698(3)	136(2)
6AmTu4Cl	N3	N1	0.79(2)	1.98(2)	2.674(4)	146(2)
	N23	N21	0.83(3)	2.00(3)	2.680(3)	138(3)
6AmTu4NO_2	N3	N1	0.84(4)	1.91(4)	2.650(5)	146(4)
Intermolecular						
6AmTu2OMe[a]	N2	S1#1	0.88(3)	2.65(3)	3.454(3)	153(2)
	C3	S1#1	0.97(3)	2.87(3)	3.634(4)	136(2)
	N3	O1#2	0.89(3)	2.55(3)	3.202(3)	130(2)
	N4	O1#2	0.88(4)	2.13(4)	2.983(4)	164(3)
6AmTu4Cl[b]	N2	S2#1	0.75(2)	2.70(2)	3.450(3)	177(2)
	C10	N2#2	0.93(3)	2.86(3)	3.437(4)	122(2)
	N4	S1#2	0.70(2)	2.95(3)	3.638(4)	167(3)
	C13	N1#3	0.90(3)	2.84(3)	3.372(4)	119(2)
	N22	S1#4	0.76(2)	2.68(2)	3.425(3)	169(2)
	N24	Cl2#5	0.92(4)	2.94(4)	3.680(4)	138(3)
6AmTu4NO_2[c]	N2	S1#1	0.94(4)	2.57(4)	3.455(4)	157(3)
	C3	S1#1	0.92(3)	2.95(4)	3.714(5)	141(3)
	C5	N2#2	0.90(3)	2.96(3)	3.419(5)	113(2)
	C9	O1#3	0.89(4)	2.71(4)	3.418(5)	137(3)
	N4	O1#3	0.88(4)	2.52(4)	3.324(6)	153(3)
	C10	O2#3	0.89(4)	2.46(4)	3.272(5)	152(3)
	C12	O2#4	0.94(4)	2.61(4)	3.450(6)	150(3)
	N2	N4#5	0.94(4)	2.88(4)	3.293(6)	108(3)
	N4	S1#6	0.82(5)	2.87(5)	3.624(5)	153(4)
	N4	O2#7	0.82(5)	2.95(5)	3.354(5)	113(4)

[a] (a) #1: 0.5 − x, −0.5 − y, −z; #2: 1.5 − x, 0.5 + y, 0.5 − z.
[b] (b) #1: 0.5 + x, −1.5 − y, 0.5 + z; #2: 1 − x, −0.5 + y, 0.5 − z; #3: 1.5 − x, −0.5 + y, 0.5 − z; #4: −0.5 + x, −1.5 − y, −0.5 + z; #5: 0.5 − x, 0.5 + y, 0.5 − z.
[c] (c) #1: 1 − x, −y, 1 − z; #2: 1.5 − x, −0.5 + y, 1.5 − z; #3: 1.5 − x, −0.5 + y, 2.5 − z; #4: 1 − x, 1 − y, 2 − z; #5: −0.5 + x, −0.5 − y; −0.5 + z; #6: 0.5 + x, −0.5 − y, 0.5 + z; #7: x, −1 + y, z.

Table 4
Mean plane data and angles between planes for 6AmTu2OMe, 6AmTu4Cl and 6AmTu4NO_2

Compound plane	Plane angle	Plane	Mean plane deviation	Atom with larges deviation	Plane/plane	Angle
6AmTu2OMe	N1–C2–C3–C4–C5–C6	1	0.0042	C2, 0.0061(0.0017)	2/1	4.92(0.09)
	N2–C7–S1–N3	2	0.0061	C7, 0.0105(0.0019)	3/2	69.20(0.09)
	C8–C9–C10–C11–C12–C13	3	0.0024	C13, 0.0033(0.0022)	1/3	70.83(0.10)
6AmTu4Cl	N1–C2–C3–C4–C5–C6	1	0.0055	C6, 0.0085(0.0021)	2/1	4.66(0.15)
	N2–C7–S1–N3	2	0.0060	C7, 0.0104(0.0021)	3/2	67.94(0.10)
	C8–C9–C10–C11–C12–C13	3	0.0142	C8, 0.0224(0.0022)	1/3	70.15(0.10)
	N21–C22–C23–C24–C25–C26	4	0.0033	C26, 0.0056(0.0020)	5/4	12.25(0.15)
	N22–C27–S2–N3	5	0.0029	C27, 0.0050(0.0023)	6/5	48.20(0.09)
	C28–C29–C30–C31–C32–C33	6	0.0134	C28, 0.0184(0.0020)	4/6	41.17(0.10)
6AmTu4NO_2	N1–C2–C3–C4–C5–C6	1	0.0067	C3, 0.0096(0.0029)	2/1	3.18(0.11)
	N2–C7–S2–N3	2	0.0003	C7, 0.0005(0.0026)	3/2	7.42(0.13)
	C8–C9–C10–C11–C12–C13	3	0.0055	C12, 0.0096(0.0025)	1/3	9.06(0.13)

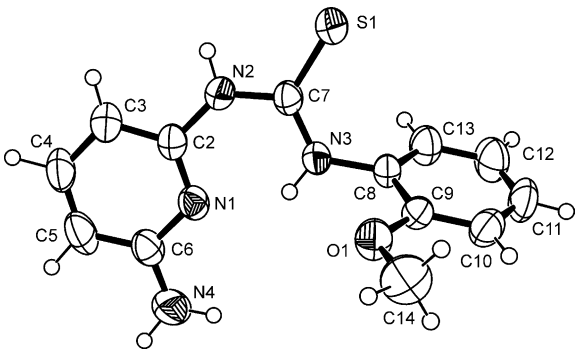

Fig. 2. ORTEP drawing showing 6AmTu2OMe with atom numbering scheme and displacement ellipsoids at 50% probability level.

found or 6PicTu4Cl [10]. Interestingly, the 6-amino function, $N4H_2$, has one of its hydrogens weakly interacting with S1, but the strongest interaction for $N24H_2$ is with Cl2; both of these interactions are with crystallographically identical molecules. Another important difference between the two different molecules of 6AmTu4Cl is the angle between the mean planes of their pyridine and aryl rings of 70.15(10) and 41.17(10), respectively. The angle between the mean planes of the pyridine rings of the two molecules is 70.80(0.09)° (Table 4).

3.3. 6AmTu4NO$_2$

Table 2 shows that the bond distance for N3–C8 in 6AmTu4NO$_2$, which is marginally shorter than found for 6AmTu2OMe and 6AmTu4Cl, Table 2, may be influenced by the inductive effect of the nitro group. To date *N*-2-pyridyl-*N'*-4-nitrophenylthiourea, PyTu4NO$_2$ is the only *N*-2-pyridylthiourea with a nitro group that has a solved structure [10]. The bond distance for N3–C8, 1.406(5) Å, in PyTuNO$_2$ [10], is essentially the same as the present 6AmTu4NO$_2$, and it is

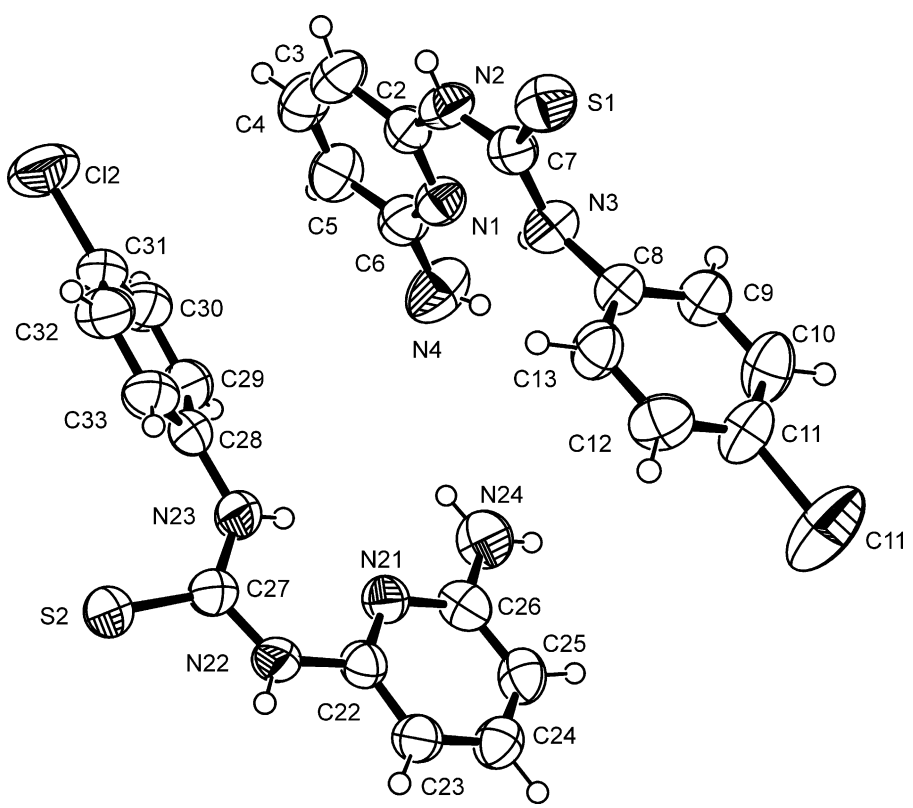

Fig. 3. ORTEP drawing showing the two unique molecules of 6AmTu4Cl with atom numbering scheme and displacement ellipsoids at 50% probability level.

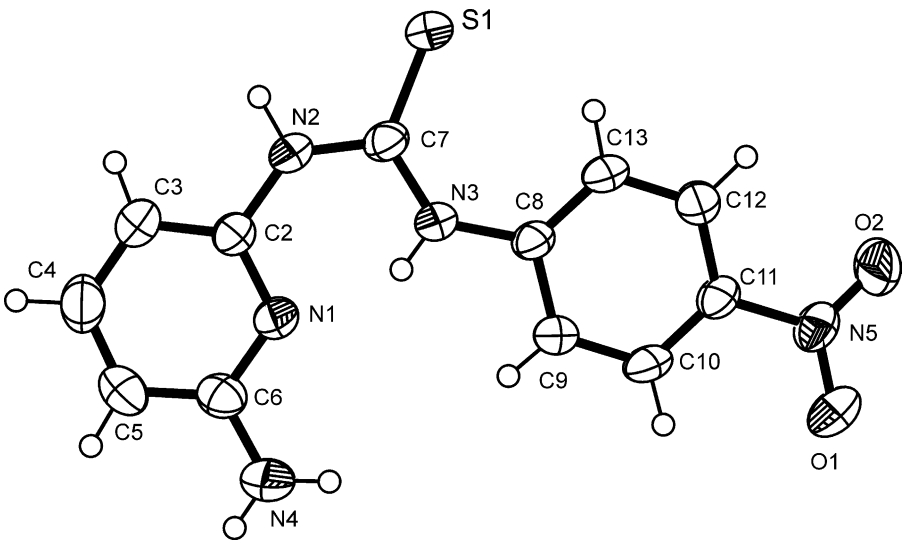

Fig. 4. ORTEP drawing showing $6AmTu4NO_2$ with atom numbering scheme and displacement ellipsoids at 50% probability level.

significantly shorter than in *N*-2-pyridyl-*N′*-(4-methoxyphenyl)thiourea, -(4-chlorophenyl)thiourea and -(4-bromophenyl)thiourea, PyTu4OMe, PyTu4Cl and PyTu4Br, respectively [10]. The S1–C7–N3 and C7–N3–C8 angles of $6AmTu4NO_2$ are larger than in 6AmTu2OMe and 6AmTu4Cl, and similar differences are present for these angles in $PyTu4NO_2$ compared to PyTu4OMe, PyTu4Cl and PyTu4Br [10] indicating that the inductive effect of the nitro group significantly affects bond distances and angles.

The intramolecular hydrogen bonding parameters of $6AmTu4NO_2$ are similar to those of 6AmTu2OMe and 6AmTu4Cl. As might be expected, $6AmTu4NO_2$ has a larger number of intermolecular interactions with the NH_2 and NO_2 functions participating. Like most thioureas [1–15,20] the intermolecular N2–H2···S1 hydrogen bond is present with normal parameters. Like 6AmTu2OMe, the C3–H3···S1 interaction in $6AmTuNO_2$ is significant. One of the H's on N4 interacts strongly with a NO_2 oxygen based on H···O distance, but the C10 hydrogen has the shortest H···O distance, Table 3.

$6AmTuNO_2$ is considerably more planar than the other two thioureas of this study. The angle between the mean planes of the pyridine and aryl rings, 9.06(0.13)°, is also smaller than the analogous angle in $PyTu4NO_2$, 19.4(0.6)°, and it in turn is much smaller than PyTu4OMe, PyTu4Cl and PyTu4Br [10]. Therefore, the hydrogen bonding interactions promoted by the nitro function, as well as the inductive effect, play a significant part in the structure of $6AmTu4NO_2$.

4. Conclusion

The three 2-pyridylthioureas, whose structures have been described here, represent the first examples in which the pyridine ring has a substituent capable of hydrogen bonding interactions. Our previous studies [2,4–15] have focused on thioureas with functional groups on the pyridine and aryl rings that could produce a steric effect, as well as groups that provide sites for hydrogen bonding on the aryl ring [5,7,9–11, 14,15]. Therefore, more meaningful hydrogen bonding interactions are present, and these interactions along with the planarity of the molecules represent the important structural properties. Since these molecules, or these functions in larger molecules, have potential medical importance, structural findings on the various 2-pyridylthioureas could prove useful in structure/activity relationships.

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-183125 for 6AmTu2OMe, CCDC-183126 for 6AmTu4Cl, and CCDC-183127 for 6AmTu4NO_2. 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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