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Synthesis, structural and vibrational properties of 1-(adamantane-1-carbonyl)-3-halophenyl thioureas

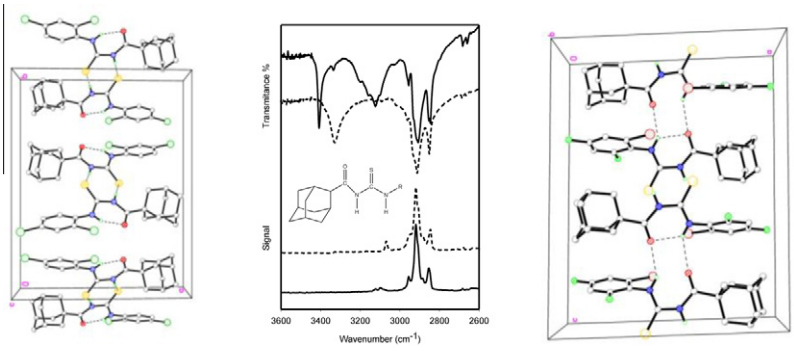
Aamer Saeed [a,*], Mauricio F. Erben [b], Michael Bolte [c]

[a] Department of Chemistry, Quaid-I-Azam University, Islamabad 45320, Pakistan
[b] CEQUINOR (UNLP, CONICET-CCT La Plata), Departamento de Química, Facultad de Ciencias Exactas, Universidad Nacional de La Plata, C.C. 962, 1900 La Plata, Argentina
[c] Institut für Anorganische Chemie, J.W.-Goethe-Universität, Max-von-Laue-Str. 7, D-60438 Frankfurt/Main, Germany

HIGHLIGHTS

- Two adamantane-1-carbonyl thiourea were prepared for the first time.
- Crystal and molecular structures were determined.
- Both intra- and inter-molecular hydrogen bonds are found in the crystal.
- Conformational aspects are discussed in terms of the vibrational spectra.

GRAPHICAL ABSTRACT



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ABSTRACT

1-(Adamantane-1-carbonyl)-3-(2,4-dichlorophenyl)thiourea (**1**) and 1-(adamantane-1-carbonyl)-3-(2-bromo-4,6-difluorophenyl)thiourea (**2**) were synthesized by the reaction of adamantane-1-carbonyl chloride with ammonium thiocyanate to afford the adamantane-1-carbonylisothiocyanate *in situ* followed by treatment with suitable halogenated anilines. The structures of the products were established by elemental analyses, Fourier transform infrared spectroscopy (FTIR), ^{1}H, ^{13}C nuclear magnetic resonance (NMR), mass spectroscopy and single crystal X-ray diffraction study. Bond lengths and angles show the usual values. All of three condensed cyclohexane rings of the adamantane residues adopt the usual chair conformation. The molecular conformation of **1** and **2** is stabilized by an intramolecular (N—H···O=C) hydrogen bond which forms a pseudo-six-membered ring. Structural features have been complemented with the joint analysis of the FTIR and FT-Raman spectra along with quantum chemical calculations at the B3LYP/6-311++G** level.

Introduction

Thiourea derivatives are exceptionally versatile building blocks for the synthesis of a diversity of heterocyclic compounds and exhibit a broad spectrum of bioactivities [1]. Examples include 1-acyl-3-(2′-aminophenyl)thioureas exhibiting anti-nematodal [2], fluorinated thioureas antimicobial [3], N-phenyl-N′-[4-(5-cyclohexylamino-1,3,4-thiadiazole-2-yl)phenyl]thioureas [4] and N-pentofuranosyl-N″-[*p*-isoamyloxy)phenyl] thioureas potent anti-tubercular [5] and 6-thioureido-4-anilinoquinazolines antimalarial activities [6]. Similarly, N-4-substituted benzyl-N′-terbutylbenzylthioureas show analgesic activity [7] and 3,4-dimethoxy phenylethyl-1,3,5-triazinylthioureas exhibit anti-HIV activity [8].

Adamantane molecule consisting of four linked cyclohexane rings arranged in the chair configuration is exceptional in that it is both inflexible and almost stress-free. It posses high T_d symmetry

* Corresponding author. Tel.: +92 51 9064 2128; fax: +92 51 9064 2241.
E-mail address: aamersaeed@yahoo.com (A. Saeed).

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and can be described by only two sites, designated as 1 (4 equivalent sites) and 2 (6 equivalent sites). The significance of the adamantyl group in drug design is multidimensional. The hydrophobic substituent constant for the adamantyl group indicates that the log*P* value of a compound with high water solubility ($\log P \ll 0$) could be moved with an adamantyl-based modification to a region that is more clinically useful. The steric bulk of the adamantyl group can restrict or modulate intramolecular reactivity; and impede the access of hydrolytic enzymes, thereby increasing drug stability and plasma half life. Presently there are almost forty adamantyl-based compounds used to treat viral infections, neurodegenerative disorders, acne vulgaris and type 2 diabetes mellitus [9].

There are a few examples of ureas/thioureas containing the adamantyl group in literature. Accordingly, thioureas containing the bulkier 1-adamantyl group have been used as organo-catalysts for synthesis of enantiomerically pure α- and β-amino acids [10] and N-(1-adamantyl)-N9-(4-guanidino-benzyl)urea is a highly selective non-peptidic uPA inhibitor and a lead structure for the development of potent antimetastatic drugs [11].

The aforesaid biological and synthetic significance of thioureas on one hand and the multifunctional value of the adamantyl group in drug design on the other, prompted us to synthesize some new hitherto unknown adamantane-1-carbonylthioureas hybrid compounds derived from adamantane-1-carbonylisothiocyanate (rather than most of the known adamantyl thioureas which are derived from 1-adamantylisothiocyanate) to combine their valuable effects in a single structural entity.

Experimental

Adamantane carboxylic acid, 2,4-dichloroaniline and 2-bromo-4,6-difluoroaniline were the commercial products from Aldrich. Analytical grade acetone (E. Merck) was dried and freshly distilled prior to use. Melting points were recorded using a digital Gallenkamp (SANYO) model MPD.BM 3.5 apparatus and are uncorrected. ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra were determined in $CDCl_3$ at 300 MHz and 75.4 MHz respectively using a Bruker spectrophotometer. Fourier transform infrared spectroscopy (FTIR), spectra were recorded on an FTS 3000 MX spectrophotometer (Pakistan). Mass Spectra (EI, 70 eV) on a gas chromatography–mass spectrometry (GC–MS) instrument Agilent technologies, and elemental analyses were conducted using a LECO-183 CHNS analyzer.

Furthermore, solid-phase infrared spectra were recorded with a resolution of 2 cm^{-1} in the 4000–400 cm^{-1} range on a Bruker EQUINOX 55 FTIR spectrometer (Argentina). The FT-Raman spectra were recorded in the region 4000–100 cm^{-1} using a Bruker IFS 66v spectrometer equipped with Nd:YAG laser source operating at 1.064 μm line with 200 mW power of spectral width 2 cm^{-1}. Quantum chemical calculations were performed with the GAUSSIAN 03 program package. The calculated vibrational properties corresponded in all cases to potential energy minima for which no imaginary frequency was found.

X-ray data collection and structure refinement

Data for **1** and **2** were collected on a STOE IPDS II two-circle diffractometer with graphite-monochromated Mo Kα radiation. Empirical absorption corrections were performed using the MULABS [12] option in PLATON [13]. The structures were solved by direct methods using the program SHELXS and refined against F^2 with full-matrix least-squares techniques using the program SHELXL-97 [14]. H atoms bonded to C were geometrically positioned and refined using a riding model. H atoms bonded to N were isotropically refined. Table 1 shows the main crystal data and structure refinement for compounds **1** and **2**.

Table 1
Crystal data and structure refinement for compounds **1** and **2**.[a]

Compound	1	2
Empirical formula	C_{18} H_{20} Cl_2 N_2 O S	C_{18} H_{19} Br F_2 N_2 O S
Formula weight	383.32	429.32
Crystal system	Monoclinic	Monoclinic
Space group	P 2_1/c	P 2/c
a (Å)	14.4926(11)	14.0594(9) (Å)
b (Å)	18.3737(12)	6.8628(6) (Å)
c (Å)	6.6101(5)	17.7672(12) (Å)
β°	93.526(6)	92.086(5)°
V (Å^3)	1756.8(2)	1713.2(2)
Z	4	4
Dc (Mgm^{-3})	1.449	1.665
Absorp. coeff. (mm^{-1})	0.496	2.551
F(000)	800	872
Crystal size (mm^3)	0.38 × 0.36 × 0.28	0.35 × 0.31 × 0.28
h	$17 \le h \le 17$	−17/17
k	$-22 \le k \le 20$	−7/8
l	$-8 \le l \le 7$	−21/21
Data collected	9358	10635
Unique reflections	3262	3206
R(int)	0.042	0.075
Max./min. transm.	0.8736/0.8339	0.5353/0.4688
Parameters	226	235
GooF	1.05	1.02
R[*I* > 2sigma(*I*)]	*R*1 = 0.034, *wR*2 = 0.087	*R*1 = 0.048, *wR*2 = 0.118
R (all data)	*R*1 = 0.041, *wR*2 = 0.090	*R*1 = 0.057, *wR*2 = 0.123
max/min Δ*F* (e Å^{-3})	0.493 and −0.330 (e Å^{-3})	0.993/−0.985
CCDC deposition numbers	882902	882903

[a] Further conditions and refinement comments: temperature 170(2) K, wavelength 0.71073 Å, theta ranges (°) = 3.49 to 25.66, 3.30 to 25.72. Absorption correction: semi-empirical from equivalents, refinement method: full-matrix least-squares on F^2.

Synthesis of 1-(adamantane-1-carbonyl)-3-halophenyl thioureas

A freshly prepared solution of adamantane-1-carbonyl chloride (10 mmol) in dry acetone (50 ml) was added dropwise to a suspension of ammonium thiocyanate (10 mmol) in acetone (30 ml) and the reaction mixture was refluxed for 30 min under nitrogen. After cooling to room temperature, a solution of the 2,4-dichloroaniline or 2-bromo-4,6-difluoroaniline (10 mmol) in acetone (10 ml) was added and the resulting mixture refluxed for 4 h. The reaction mixture was poured into cold water and the precipitated thioureas were recrystallized from ethanol and ethyl acetate.

1-(Adamantane-1-carbonyl)-3-(2,4-dichlorophenyl)thiourea

(**1**); Yield 70%, mp 196 °C. FT-IR (ν cm^{-1}): 3336 (NH), 3034 (Ar—CH), 2909, 2849 (CH_2, CH), 1675, 1575, 1457, 1370 (C=S),. ^{1}H NMR (300 MHz, $CDCl_3$): δ 12.74 (br s, 1H, NH, D_2O exchangeable); 8.70 (br s, 1H, NH, D_2O exchangeable); 8.03 (d, 1H, *J* = 8.6 Hz Ar), 7.96 (d, 1H, *J* = 8.6 Hz Ar), 7.90 (d, 1H, *J* = 8.6 Hz Ar), 7.83 (d, 1H, *J* = 8.6 Hz Ar), 7.57 (m, 3H, Ar), 2.1 (brs, 3H, adamantane–CH), 2.03 (s, 6H, adamantane–CH_2), 1.81 (q, 6H, adamantane–CH_2, *J* = 8.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): 178.9 (C=S); 170.3 (C=O), 134.10 (Ar), 128.6, 126.9, 125.3, 123.64, 121.67 (ArCs), 41.94, 41.90, 39.2, 38.6, 36.1, 36.0, 31.6, 28.0, 27.8, (adamantane–C); Anal. Calcd for $C_{18}H_{2o}$ Cl_2N_2OS (383.34): C, 56.40; H, 5.26; N, 7.31; S, 8.36%; Found: C, 56.51; H, 5.21; N, 7.26; S, 8.39%.

1-(Adamantane-1-carbonyl)-3-(2-bromo-4,6-difluorophenyl)thiourea

(**2**) Yield 70%, mp 176 °C. FT-IR (ν cm^{-1}): 3336 (NH), 3034 (Ar—CH), 2909, 2849 (CH_2, CH), 1675, 1575, 1457, 1370 (C=S),. ^{1}H

NMR (300 MHz, $CDCl_3$): δ 12.74 (br s, 1H, NH, D_2O exchangeable); 8.70 (br s, 1H, NH, D_2O exchangeable); 7.13 (s, 2H, Ar), 2.1 (brs, 3H, adamantane–CH), 2.03 (s, 6H, adamantane–CH_2), 1.81 (q, 6H, adamantane–CH_2, J = 8.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): 178.9 (C=S); 170.1 (C=O), 134.10 (Ar), 181.7, 128.6, 124.2, 119.7, 114.9 (ArCs), 41.94, 41.90, 39.2, 38.6, 36.1, 36.0, 31.6, 28.0, 27.8, (adamantane–C); Anal. Calcd for $C_{18}H_{19}F_2BrN_2OS$ (429.32): C, 50.36; H, 4.46; N, 6.53; S, 7.47; Found: C, 50.24; H, 4.51; N, 6.57; S, 7.36%.

Results and discussion

Synthesis and characterization

The reaction sequence leading to the formation of thioureas is depicted in Scheme 1. The starting material 1-adamantane carbonyl chloride was obtained by the reaction of 1-adamantane carboxylic acid with thionyl chloride [15]. A solution of adamantane-1-carbonyl chloride in dry acetone was treated with an equimolar quantity of potassium thiocyanate in dry acetone to afford the adamantane-1-carbonylisothiocyanate as intermediate. Treatment of the latter with an equimolar quantity of the two substituted halogenated anilines in acetone furnished the title thiourea derivatives (**1**) and (**2**).

The adamantane-1-carbonylthioureas were characterized by IR absorptions (see Section 3.3) at 3350–3325, 3154–3125 (free and associated NH), 3031–3024 (Ar—CH), 2902–2926 (CH_2), 2847–2850 (CH), 1672–1685 (carbonyl groups). In the ^{1}H-NMR the characteristic signals of adamantyl moiety: a 6H quartet at δ 1.75–1.79 (adamantane–CH_2), a 6H, singlet at 1.95–1.98 (adamantane–CH_2) and a 3H, singlet around 2.08 (adamantane–CH), besides singlets at δ 8.5–8.7 and 12.7–13.0 ppm were observed for HN(1) and HN(2) respectively. In the ^{13}C NMR characteristic signals at for adamantyl moiety at δ 27.7, 36.1–36.4, 38.6–38.5 and 41.5–41.5 as well those at δ around 170.1 (C=O) for carbonyl and δ 178–179 for thiocarbonyl carbons were observed. However, mass spectra of most of compounds did not show the molecular ion peaks; the base peak at m/z 135 is derived from the adamantyl cation which on further fragmentation results in weaker signals at m/z = 93, 80, 79, 67, 41 and 39. The other major fragments correspond to those of the N-McLafferty rearrangement.

X-ray crystal structure

1-(Adamantane-1-carbonyl)-3-(2,4-dichlorophenyl)thiourea (**1**) and 1-(adamantane-1-carbonyl)-3-(2-bromo-4,6-difluorophenyl)thiourea (**2**) were further characterized by single crystal X-ray diffraction study. The molecular structure of thioureas **1** and **2** are shown in Fig. 1. The molecular conformation of **1** and **2** is stabilized by an intramolecular (N2—H2···O1) hydrogen bond which forms a pseudo-six-membered ring. Whereas the crystal packing of **1** shows centrosymmetric dimers connected by N—H···S hydrogen bonds, there is no hydrogen bond for H2 in **2**. The molecular conformation of both compounds is rather similar. A least-squares fit of the carbonyl-thiourea moiety of both molecules (r.m.s. deviation 0.022 Å) shows only minor differences: The torsion of the adamantyl residue differs by 20.7 degrees (**1**: O1—C2—C21—C26 – 94.7°; **2**: O1—C2—C21—C27 – 74.0°) and the dihedral angle of the halogen substituted ring with respect to the thiourea moiety differs by only 3° (**1**: Cl1—C12—C11—N1 – 1.5°; **2**: Br1—C12—C11—N1 – 4.5°).

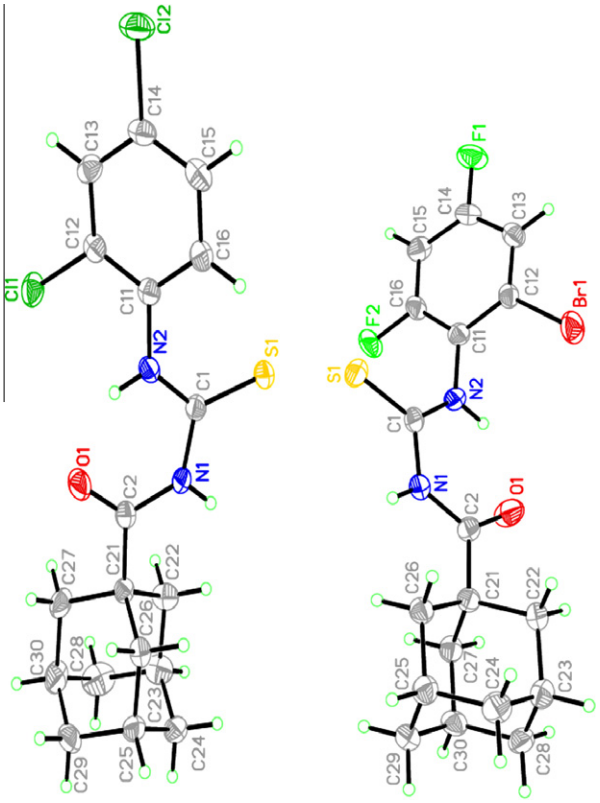
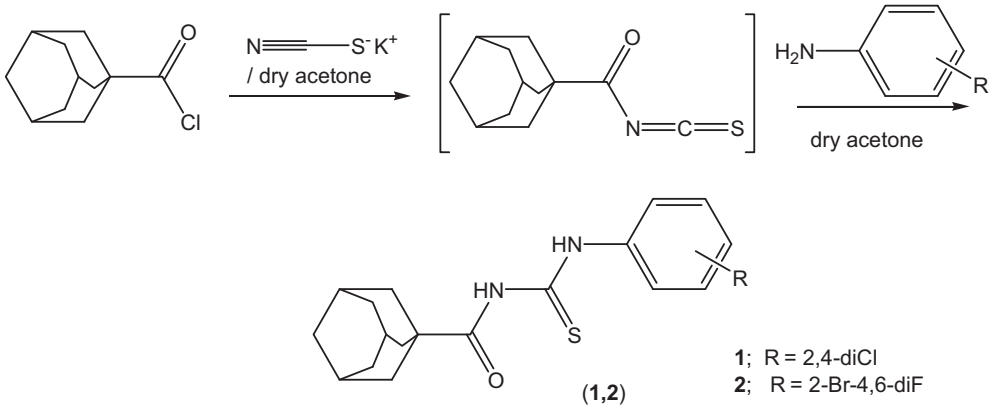


Fig. 1. Molecular structure of **1** and **2** with displacement ellipsoids plotted at 50% probability level.

The crystal packing diagram for **1** is shown in Fig. 2. It shows centrosymmetric dimers connected by N—H···S hydrogen bonds. The crystal packing diagram for **2** is shown in Fig. 3. It shows a bifurcated N—H hydrogen bond: the H atom form an intramolecular and



Scheme 1. Synthetic route to 1-(adamantane-1-carbonyl)-3-halophenylthioureas.

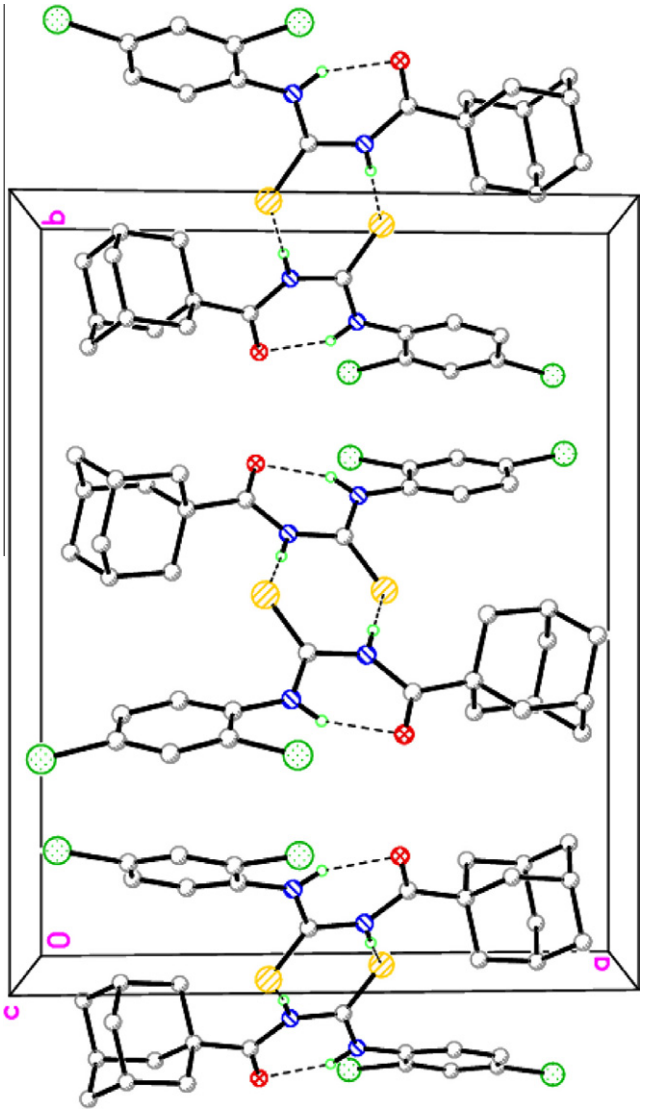


Fig. 2. A packing diagram of the compound **1.**

intermolecular N—H···O hydrogen bond. It is remarkable that the other amino group is not involved in hydrogen bonding. Centrosymmetric dimers – connected by both, N—H···S=C [16,17] and bifurcated N—H···O=C [18,19] hydrogen bonds – have been already reported for acyl-thioureas. Thus, hydrogen bonding clearly *dominates the crystal packing* and subtle differences in the substituent attached to the thiourea group seems to determine the preference of N—H···X=C (X=O or S) network.

Vibrational analysis

The presence of strong inter- and intramolecular interactions observed in the crystalline state for the title species invites to close analyze the infrared and Raman spectra in order to better understand how these features affect the vibrational properties [20–27]. The observed and calculated (B3LYP/6-311++G**) infrared absorptions, the FT-Raman frequencies along with their relative intensities and probable assignments are summarized in Table S1, in the supporting information. A tentative assignment of the observed bands was carried out by comparison with spectra of related molecules [20–27], aided by visualization of the animations for displacement vectors of localized vibrational modes.

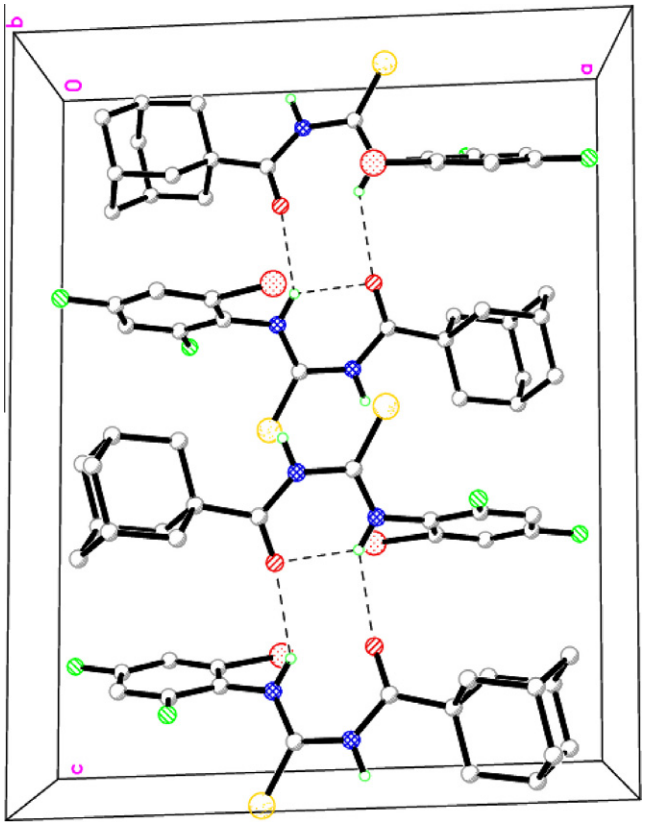


Fig. 3. A packing diagram of the compound **2**.

The intramolecular N_2—H···O=C hydrogen bond has a strong influence in the vibrational properties of the central —C(O)NHS(O)NH— moiety [20–27]. In particular, a noticeable red-shift is observed for the N—H stretching mode of the thioamide-like group involved in the aforementioned six-membered ring [28,29]. The 3600–2600 cm^{-1} region of the infrared and Raman spectra for the title compounds are showed in Fig. 4. In the infrared spectra of **1** and **2**, well-defined absorptions at 3325 and 3408 cm^{-1} are assigned to the N_2—H stretching modes. Quantum chemical calculations for the most stable conformers of the title compounds isolated in a vacuum compute that the $\nu(N_2—H)$ stretching modes appear as a very intense absorption shifted to lower frequencies as compared with the $\nu(N_1—H)$ fundamental. However, broad and not well-resolved bands are observed near 3100 cm^{-1} in the infrared spectra of the studied compounds, making difficult an unambiguous assignment of these features. This is especially conformed in the infrared spectrum of **2** for which superposition with C—H stretching modes is anticipated. However, the very low intense signal at 3124 cm^{-1} in the Raman spectrum of **2** can be associated with the $\nu(N_2—H)$ stretching. Weiqun et al. [30] reported a low ν(N—H) frequency value (3086 cm^{-1}) for the related N-(4-chloro)benzoyl-N′(4-tolyl)thiourea species. In the present case, however, the bands observed clearly in the Raman spectra at 3073 and 3099 cm^{-1} for compounds **1** and **2**, respectively, are better assigned as ν(C—H) stretching fundamentals from the halophenyl group, in agreement with the comprehensive vibrational study by Estévez–Hernández and coworkers [31] on 3-monosubstituted 1-furoylthioureas.

Strong IR absorptions at 1675 and 1680 cm^{-1} with clear-defined counterparts at 1679 and 1681 cm^{-1} Raman bands are observed (see Fig. 5), which were assigned to the ν(C=O) modes. Such a low value for the carbonyl double bonds is also a signature for the presence of a intramolecular N_2—H···O=C interaction in the —C(O)NHC(S)NH— moiety [32,33].

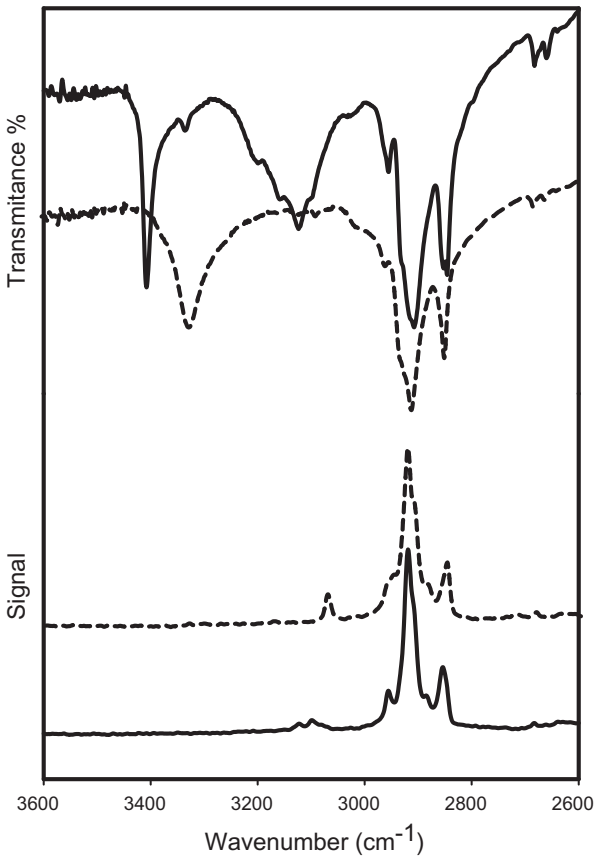


Fig. 4. FT-Raman and FTIR spectra for compounds **1** (dashed line) and **2** (solid line) in the 3600–2600 cm^{-1} region.

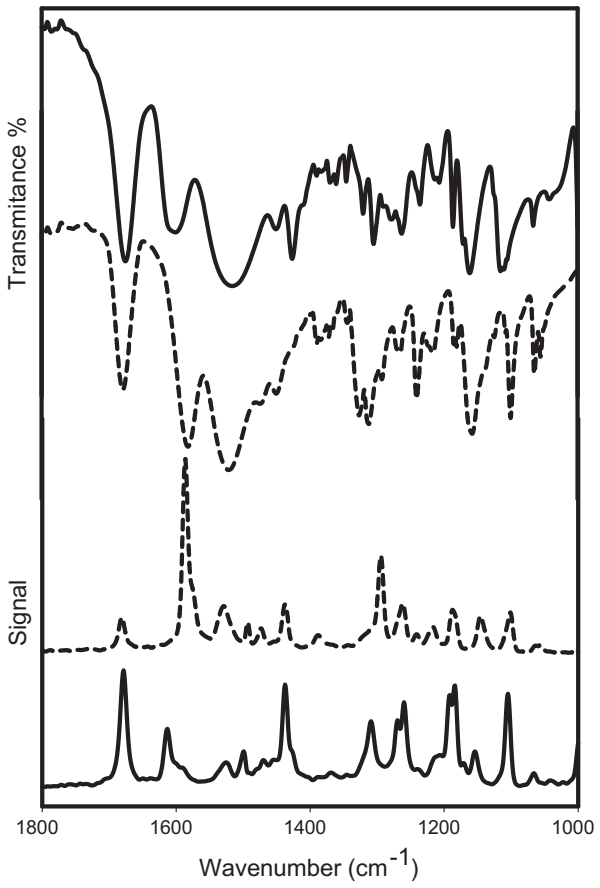


Fig. 5. FT-Raman and FTIR spectra for compounds **1** (dashed line) and **2** (solid line) in the 1800–1000 cm^{-1} region.

The fingerprint region of amide and thioamide groups present a characteristic band in the 1500–1600 cm^{-1} range of the IR spectrum, originated by the N—H deformation mode [δ(N—H)]. B3YLP/6-311+G* computations predict strong bands at 1564 and 1524 cm^{-1} due to the $\delta(N_2—H)$ and $\delta(N_1—H)$ normal mode on **2**, whereas for **1** the corresponding values are expected at 1576 and 1540 cm^{-1}, respectively. As showed in Fig. 5, the infrared spectrum of the former species shows an intense and broad band centered at 1517 cm^{-1}, whereas in the Raman spectrum two signals are defined at 1525 and 1499 cm^{-1}, which are tentatively assigned as $\delta(N_2—H)$ and $\delta(N_1—H)$, respectively. Similarly, an intense absorption is observed at 1523 cm^{-1} with a shoulder at lower wave numbers (1476 cm^{-1}), with counterparts at 1530 and 1474 cm^{-1} in the Raman spectrum, are assigned as the $\delta(N_2—H)$ and $\delta(N_1—H)$ normal mode for compound **1**, respectively.

The adamantyl group is responsible for the very strong band observed in the Raman spectrum of **2** at 774 cm^{-1} and the broad signal at 783 cm^{-1} for **1**, bands which can be assigned with confidence to the "breathing mode", in agreement with the Raman spectra observed for adamantane isotopomers [34]. In this region, also a medium intensity IR absorptions observed at around 756 cm^{-1} (761 and 755 cm^{-1} Raman) are tentatively assigned to the ν(C=S) mode for **1** and **2**, respectively. As already pointed out [35], this assignment is in agreement with previously studied thiourea derivatives [32,36]. However, it should be mentioned that for the parent the thiourea molecule, this mode appeared in the 1094 cm^{-1} in the infrared spectrum (1105 cm^{-1} Raman) [37], and higher values – up to 1325 cm^{-1} – have been also reported [23,30]. The formation of C=S···H—X intermolecular hydrogen bonds seems to strongly effect the frequency of the ν(C=S) mode [20].

Conclusion

Two novel adamantane-1-carbonylthiourea derivatives (**1, 2**) were prepared by treating adamantane-1-carbonyl isothiocyanate (produced *in situ*) with anilines in good yields. Conformational and structural properties were determined by using experimental techniques which include vibrational spectroscopies (infrared and Raman) and single crystal X-ray diffraction analysis. The X-ray molecular structure is similar for both studied compounds, with the central —C(O)NHC(S)NH— moiety adopting a typical six membered ring structure, which is favored by strong C=O···H—N intramolecular hydrogen bond. Centro-symmetric dimeric units dominate the crystal packing, however, different intermolecular interactions are observed for the title compounds. Thus, whereas compound **1** forms dimers mainly held by a N—H···S=C hydrogen bond, bifurcate N—H···O=C hydrogen bonds are present in compound **2**.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2012.10.043.

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