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Synthesis, X-ray structure analysis and computational studies of novel bis(thiocarbamoyl) disulfides with non-covalent S···N and S···S interactions

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Abstract

In this Letter, we describe the synthesis of several bis(thiocarbamoyl) disulfides that present interesting intramolecular S···N and S···S interactions. In one case, crystals suitable for X-ray characterization have been obtained. The non-covalent interactions have been studied analyzing the crystal structure and by means of high level density functional theory (DFT) calculations (RI-PB86/TZVP) using both 'atoms-in-molecules' (AIM) and natural bond orbital (NBO) analyses.

1. Introduction

It is well known that bis(thiocarbamoyl) disulfides such as antabus (tetraethylthiuram disulfide) act as antioxidants and inhibitors of aldehyde dehydrogenase [1]. In addition, compounds of this type are found to possess antiviral activity [2] and they are potential drug formulations for the prevention and treatment of cataracts [3]. On the other hand, the non-bonding S···N, S···S and S···O interactions play an important role in mechanism of action of anticancer [4,5] and antibacterial [6] agents as well as antagonists of angiotensine II (AT_1) receptors [7–9].

In this Letter, we describe a convenient synthetic methodology for the preparation of a novel class of bis(thiocarbamoyl) disulfides which exhibit both the S···N and S···S short contacts. Nature of these interactions was investigated by means of X-ray structure analysis and quantum chemical calculations. The non-covalent interactions have been studied using the Bader's theory of 'atoms-in-molecules' [10], which has been used successfully to understand a great variety of non-covalent interactions. Additionally, the interactions have been rationalized using the natural bond orbital (NBO) method [11], taking advantage of the perturbative analysis of NBO donor–acceptor interactions.

2. Experimental and theoretical methods

2.1. Synthesis

Melting points were measured on a Boetuis apparatus and are not corrected. IR spectra were taken in KBr pellets on a Perkin–Elmer FTIR 1600 spectrometer. NMR spectra were recorded on a Varian Unity 500 apparatus using TMS as the internal standard. Elemental analyses of C, H, N were within ±0.4% of the theoretical values. Starting *N*-{1-[(3-thioxo-5,6-dihydroimidazo[2,1-*c*][1,2,4]thiadiazol-7-lythio)-thiocarbonyl]2-imidazolidene}-arylsulfonamides (**1**–**5**) were prepared according to the described procedure [12].

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*2.1.1. Preparation of bis{[2-(arylsulfonylimino)imidazolidin-1-yl]thiocarbonyl}disulfides (**6–10**)*

2.1.1.1. General procedure. *N*-{1-[(3-thioxo-5,6-dihydroimidazo[2,1-*c*][1,2,4]thiadiazol-7-lythio)-thiocarbonyl]2-imidazolidene}arylsulfonamide **1–5** (0.4 mmol) was dissolved in wet DMF (15 mL) and the reaction mixture was heated under reflux for 10 min. The product that precipitated was isolated according to the procedure described below and purified by crystallization from suitable solvent.

*2.1.2. Bis{[2-(phenylsulfonylimino)imidazolidin-1-yl]thiocarbonyl}disulfide (**6**)*

The reaction mixture was concentrated to a volume of 5 mL under reduced pressure. Then chloroform (10 mL) was added. After 12 h the product **6** that precipitated was filtered off and washed with chloroform. Yield: 0.060 g (50%); m.p., 212–213 °C (DMSO/methanol);^{1}H NMR (DMSO-d_6): δ 3.63 (t, 4H, CH_2, $J = 7.9$ Hz), 4.23 (m, 4H, CH_2, $J = 7.9$ Hz), 7.55–7.67 (m, 6H, CH), 7.94–7.98 (m, 4 H, CH), 9.12 (s 2H, NH); ^{13}C NMR (DMSO-d_6) δ 40.9, 51.8, 126.4, 129.3, 132.8, 142.2, 151.2, 193.5; IR: 3392, 1636, 1410, 1382, 1341, 1307, 1283 cm^{-1}.

The filtrate obtained after isolation of compound **6** was concentrated under reduced pressure to a volume of 2 mL. After 6 h the compound **11** that deposited was separated by suction and washed with methanol. Yield 30%; m.p., 190–192 °C (Ref. [13]: 190–192 °C).

*2.1.3. Bis{[2-((4-methylphenyl)sulfonylimino)imidazolidin-1-yl]thiocarbonyl}disulfide (**7**)*

Chloroform (10 mL) was added to the reaction mixture. After 12 h compound **7** thus obtained was filtered off and washed with chloroform. Yield: 0.055 g (40%); m.p., 218–219 °C (DMF/chloroform); ^{1}H NMR (DMSO-d_6) δ 2.36 (s, 3H, CH_3), 3.59 (t, 4H, CH_2, $J = 8.2$ Hz), 4.19 (t, 4H, CH_2, $J = 8.2$ Hz), 7.36 (d, 4H, CH, $J = 8.1$ Hz), 7.81 (d, 4H, CH, $J = 8.1$ Hz), 9.02 (s 2H, NH); ^{13}C NMR (DMSO-d_6) δ 21.2, 41.5, 51.8, 126.1, 130.0, 139.9, 143.7, 151.4, 193.5; IR: 3400, 1632, 1415, 1331, 1297, 1146, 1082 cm^{-1}.

*2.1.4. Bis{[2-((4-methoxyphenyl)sulfonylimino)imidazolidin-1-yl]thiocarbonyl}disulfide (**8**)*

The reaction mixture was concentrated under reduced pressure to a volume of 5 mL. Then chloroform (10 mL) was added. After 12 h the product **8** was filtered off and washed with chloroform. Yield: 0.028 g (21%); m.p., 203–204 °C (DMF/chloroform); ^{1}H NMR (DMSO-d_6) δ 3.57–3.64 (m, 4H, CH_2), 4.16–4.24 (m, 4H, CH_2), 7.08 (d, 4H, CH, $J = 8.9$ Hz), 7.86 (d, 4H, CH, $J = 8.9$ Hz), 9.02 (s 2H, NH); IR: 3408, 1631, 1592, 1497, 1412, 1332, 1266 cm^{-1}.

*2.1.5. Bis{[2-((4-chlorophenyl)sulfonylimino)imidazolidin-1-yl]thiocarbonyl}disulfide (**9**)*

Chloroform (10 mL) was added to the reaction mixture. After 12 h compound **9** was filtered off and washed with chloroform. Yield: 0.073 g (40%); m.p., 224–225 °C (DMF/chloroform); ^{1}H NMR (DMSO-d_6) δ 3.60 (t, 4 H, CH_2, $J = 8.3$ Hz), 4.19 (t, 4H, CH_2, $J = 8.3$ Hz), 7.64 (d, 4H, CH, $J = 8.8$ Hz), 7.91 (d, 4H, CH, $J = 8.8$ Hz), 9.15 (s 2H, NH); IR: 3396, 1629, 1473, 1417, 1330, 1297, 1147 cm^{-1}.

Crystallization of the amorphous compound **9** from DMF in the presence of equimolar amount of $CuCl_2$ yielded crystals of **9** × 2 DMF suitable for X-ray structure analysis.

*2.1.6. Bis{[2-((4-trifluoromethylphenyl)sulfonylimino)imidazolidin-1-yl] thiocarbonyl} disulfide (**10**)*

The reaction mixture was evaporated to dryness under reduced pressure. The solid residue was treated with hot methanol (20 mL). After 12 h product **10** that precipitated was collected by filtration and washed with methanol. Yield: 0.095 g (64%); m.p. 227–229 °C (DMF);^{1}H NMR (DMSO-d_6) δ 3.58–3.66 (m, 4H, CH_2), 4.16–4.24 (m, 4H, CH_2), 7.96 (d, 4H, CH, $J =$ 8.4 Hz), 8.14 (d, 4H, CH, $J = 8.4$ Hz), 9.24 (s 2H, NH); IR: 3404, 1662, 1477, 1420, 1328, 1300, 1155 cm^{-1}.

2.2. X-ray structure analysis

The diffraction data were collected at 130 K with a KumaCCD diffractometer using graphite monochromated Mo Kα radiation. The intensity data were collected and processed using Oxford Diffraction CrysAlis Software [14]. The structure was solved by direct methods with the program SHELXS-97 [15] and refined by full-matrix least-squares method on F^2 with SHELXL-97 [16].

Crystal data for **9**: $C_{20}H_{18}Cl_2N_6O_4S_6 \cdot 2(C_3H_7NO)$, monoclinic, space group $P2_1/n$, $a = 7.2427(4)$ Å, $b = 11.5538(5)$ Å, $c = 20.7267(9)$ Å, $\beta = 93.078(4)°$, $V = 1731.92(14)$ Å^3, $Z = 2$, $d_x = 1.564$ g cm^{-3}, $T = 130$ K. Final R indices for 2486 reflections with $I > 2\sigma(I)$ and 257 refined parameters are: $R_1 = 0.0329$, $wR_2 = 0.0822$ ($R_1 = 0.0430$, $wR_2 = 0.0863$ for all 3042 data). The solvent molecule is disordered over two overlapping positions with the occupation ratio of 2:1.

Crystallographic data for compound **9** have been deposited with Cambridge Crystallographic Data Centre (CCDC Deposition Number CCDC 294718). Copies of the data can be obtained upon request from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, quoting the deposition numbers.

2.3. Computational methods

The geometry of compound **9** was fully optimized using the density functional BP86 method with the resolution of the identity (RI) approximation and the TZVP (triple-ζ with polarization) basis set. The RI method [17,18] uses an auxiliary fitting basis to avoid treating the complete set of two-electron repulsion integrals. The calculations at the RI-BP86/TZVP level of theory were performed using the program TURBOMOLE version 5.7 [19]. The AIM analysis was performed by means of the AIMPAC program [20]

using the BP86/TZVP//RI-BP86/TZVP wavefunction. The NBO analysis was performed using the NBO 3.0 program implemented in the GAUSSIAN-03 package [21] at the BP86/TZVP//RI-BP86/TZVP level of theory.

3. Results and discussion

3.1. Synthesis

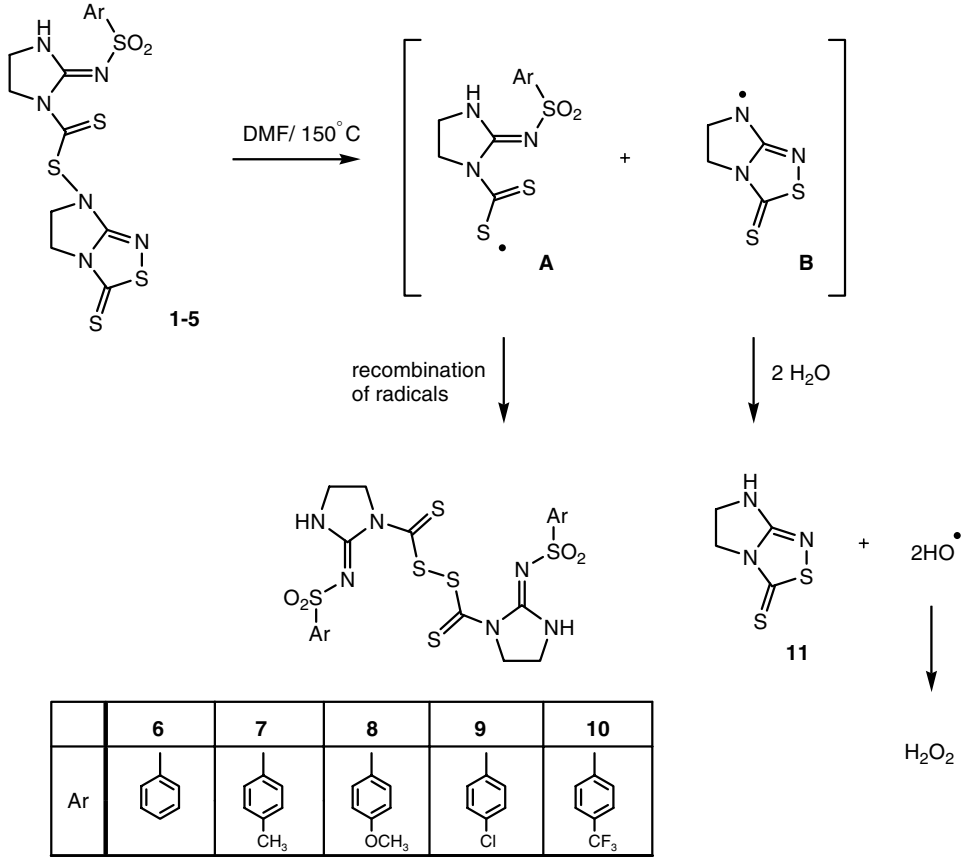
The target bis{[2-(arylsulfonylimino)imidazolidin-1-yl]thiocarbonyl}disulfides **6**–**10** were obtained by refluxing a solution of corresponding *N*-{1-[(3-thioxo-5,6-dihydroimidazo[2,1-*c*][1,2,4]thiadiazol-7-lythio)-thiocarbonyl]2-imidazolidene}arylsulfon-amide **1**–**5** in wet dimethylformamide (DMF) for 10 min. A proposed mechanism of this reaction is presented in Scheme 1.

First step of the reaction sequence is the homolytic cleavage of the sulfonamide S–N bond at elevated temperature leading to the radicals **A** and **B**. Such a thermal decomposition of sulfenamides is well documented in the chemical literature [22,23]. Then, recombination of sulfur radicals **A** gives rise to the formation of disulfides **6**–**10**. Simultaneously, the radical **B** reacts with water molecule to give 6,7-dihydro-5*H*-imidazo[2,1-c][1,2,4]thiadiazol-3-thione (**11**) and hydroxyl radical which subsequently recombinates to hydrogen peroxide. Analytical and spectroscopic data for **11** were identical with those described previously for authentic sample [13].

It should be pointed out that the above reaction did not proceed in dry DMF, which suggests that an alternative mechanism involving the hydrolytic cleavage of S–N bond might be operative. If it is the case, the sulfenic acid-type species with unstable R–S–OH moiety should be formed. One reagent that has been reported to be useful for trapping the unstable sulfenic acid is methyl acetylenecarboxylate [24]. However, upon heating of **1**–**5** in the presence of methyl acetylenecarboxylate, adducts with alkenyl structure were not detected (NMR evidence).

3.2. X-ray structure analysis

Molecular structure adopted by **9** in the solid state is shown in Fig. 1. This molecule is centrosymmetric with the inversion center located in the middle of the S–S bond that leads to the *trans* conformation at the disulfide S–S bond. This is a seldom case for the disulfide bridges and our survey of the Cambridge Structural Database (CSD) [25] shows that among 401 molecules containing the acyclic C–S–S–C fragment as much as 384 have the C–S–S–C torsion angle in the range 60–120° and only in 8 cases the torsion angle around the S–S bond points to the *trans* conformation. Interestingly, the change in the conforma-



Scheme 1. Proposed mechanism for the formation of disulfides **6**–**10**.

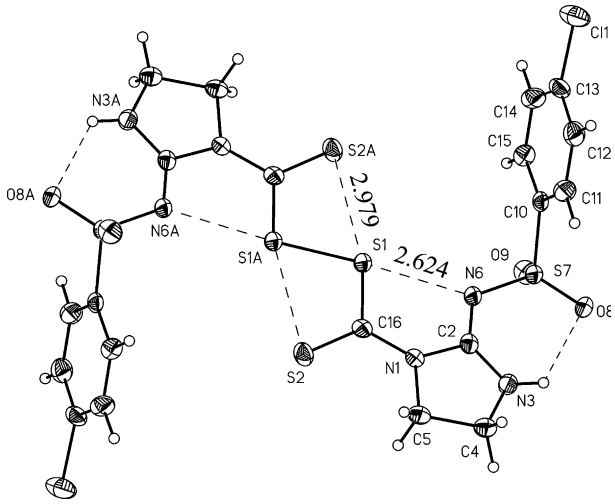


Fig. 1. Ortep drawing of **9** showing atom labelling and short intramolecular S···S and S···N contacts in Å.

tion is accompanied by a significant change in the S–S bond length: for the 384 skewed fragments and for the eight fragments in the *trans* conformation calculated mean values of the S–S bond lengths are 2.04(2) Å and 2.12(3) Å, respectively. The S–S bond length of 2.124(1) Å in **9** is close to the mean value for the trans conformation. Tetraisopropylthiuram disulfide (**12**) [26] is among the eight *trans* disulfides found in the CSD and it is the only one among 14 bis(thiocarbamoyl) disulfides found in the database which adopts the *trans* conformation. Molecular structure of **9**, which can also be considered a thiuram disulfide analogue, closely resembles that of **12**. There are two short intramolecular S···S contacts in both structures (2.979 Å and 2.989 Å in **9** and **12**, respectively) and by adopting the trans conformation the C–S···S fragments of the molecule become nearly linear, i.e. allow for the involvement of the nucleophilic S2 atom in the interaction with the C–S1 σ^* orbital. Whereas S···S contacts shorter than the sum of van der Waals radii are also observed in compounds with the nearly perpendicular thiocarbamoyl moieties, there the C–S···S angles are close to 90° and the S···S contacts are significantly longer with the mean value of 3.25(3) Å. Another geometrical parameter influenced by the conformational change and the S···S interaction in the thiuram disulfide moiety is the C–S–S angle, which is much smaller for *trans* conformers (95.0° in **9** and 97.8° in **2**) than for the skewed forms [mean value of 104.2(9)°].

In compound **9**, the disulfide S atom is additionally involved in the interaction with another nucleophilic atom as evidenced by short S1···N6 contact to the imide nitrogen atom [N6···S1 2.624 Å, <S7–N6···S1 165.4°]. There are four structures [27–30] in the CSD where the disulfide S atom is involved in the S···N interaction closing a five-membered ring, however, in all cases the N···S contacts are much longer and fall within the range 2.724–2.815 Å. The above might be indicative of a cooperative effect of the S···S and S···N interactions in **9**.

3.3. Computational studies

Starting from the X-ray structure of **9**, we have optimized its geometry at the RI-BP86/TZVP level of theory. In Fig. 2, we compare the experimental and theoretical geometries in order to verify the reliability of the theoretical level. It is worth mentioning that the distances and angles of the atoms involved in the S···S and S···N non-covalent interactions are very similar. The main difference is located on the conformation of the aromatic ring, where the theoretical and experimental N6–S7–C10–C11 (see labeling of Fig. 1) dihedral angles are 68.0 and 99.4, respectively. This difference is probably due to packing forces that act in the solid state, however it does not affect the non-covalent interactions focus of this study.

Once verified the reliability of the level of theory, we have used the AIM theory to study the N···S and S···S non-covalent interactions analyzing the topology of the charge density $\rho(r)$ distribution and properties of critical points (CP), which provides an unambiguous definition of chemical bonding [31]. The exploration of the CPs (see

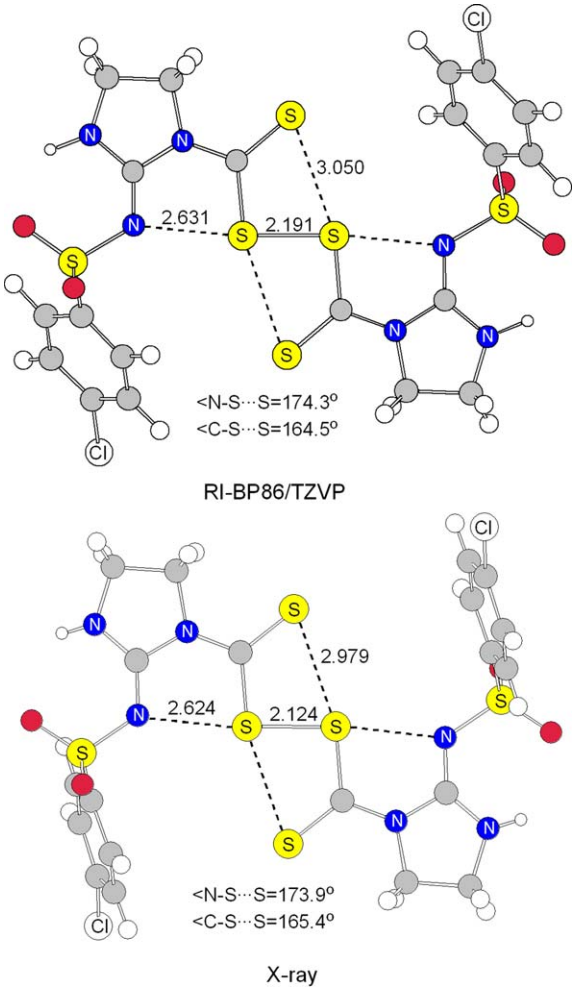


Fig. 2. Theoretical RI-PB86/TZVP optimized structure (top) and experimental X-ray structure (bottom). Distances in Å and angles in (°).

Fig. 3) revealed the presence of one (3, −1) and (3, +1) CPs for each non-bonded interaction. The bond CPs (represented by white circles) connect the S1 and S1A atoms with the N6/S2A and NA6/S2 pair of atoms, respectively. The presence of the ring CPs (black circles) indicate the formation of a four- and a five-membered rings as a consequence of the interactions. The Laplacian of the charge density at the (3, −1) CPs is positive which indicates a depletion of the electron density, as is common in closed-shell interactions [10]. The values of $\rho(r)$ and $\nabla^2\rho(r)$ at the CPs are summarized in Table 1. The value of the charge density at the bond CP of the N···S interaction ($\rho(r) = 2.889 \times 10^{-2}$ a.u.) is higher than the value obtained for the S···S interaction ($\rho(r) = 1.966 \times 10^{-2}$ a.u.) indicating that the N···S interaction is stronger. For comparison purposes we have also included in Fig. 3 and Table 1 the AIM analysis and properties of CPs of the intramolecular hydrogen bond that is formed between the oxygen atom labeled O8 and the H–N3 bond (see Fig. 1). The value of $\rho(r)$ at the bond CP is 2.707×10^{-2} a.u., which is comparable to the value obtained for the N···S bond CP indicating that the strength of the N···S non-covalent interaction is comparable to the N–H···O hydrogen bond. This result stresses the importance of these interactions which have a significant effect on the final geometry of the compound.

We have also analyzed the non-covalent interaction from another point of view. We have used the natural bond orbital (NBO) method to understand which orbitals are involved in the interaction and the energetic contribution to the stabilization of the molecule in terms of donor–acceptor interactions. The results obtained from the second order perturbation theory analysis of NBO donor–acceptor interactions that involve the atoms that participate in the non-covalent interactions present in compound **9** indicate that there is an interaction between the electron lone pair of nitrogen atoms N6 and N6A and the S1–S1A sigma antibond orbital (σ^*_{S-S}). The second-order energy ($\Delta E^{(2)}$) lowering computed for each $LP_N \rightarrow \sigma^*_{S-S}$ interaction is $\Delta E^{(2)} = 6.87$ kcal/mol. The NBO analysis also indicates an interaction between a lone pair of sulfur atoms S1 and S1A and the C16A–S2A and C16–S2 sigma antibond orbitals, respectively. The $\Delta E^{(2)}$ lowering computed for each $LP_S \rightarrow \sigma^*_{C-S}$ interaction is $\Delta E^{(2)} = 5.32$ kcal/mol. These results of energy lowering are in agreement with the values of charge density computed at the bond CPs obtained from the AIM analysis which indicate that in **9** the strength of the N···S interactions is higher than the strength of the S···S interactions.

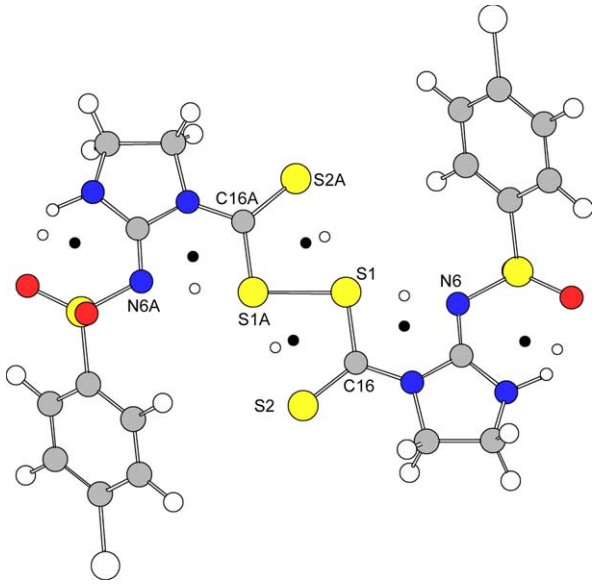


Fig. 3. Representation of the location of the (3, −1) CPs (white circles) and (3, +1) CPs (black circles) originated from the N···S and S···S interactions.

Table 1
The electron density $\rho(r)$ and its Laplacian $\nabla^2\rho(r)$ in atomics units at the critical points originated from the non-covalent interactions of compound **9** computed at the BP86/TZVP//RI-BP86/TZVP level of theory

Non-covalent interaction	CP	$10^2\rho(r)$	$10^2\nabla^2\rho(r)$
N···S–S	(3, +1)	2.889	7.806
N···S–S	(3, −1)	1.671	8.922
S···S–C	(3, +1)	1.966	5.492
S···S–C	(3, −1)	1.785	7.211
O···H–N	(3, +1)	2.707	9.865
O···H–N	(3, −1)	1.427	8.115

4. Conclusion

In summary, we have synthesized six new bis(thiocarbamoyl) disulfides derivatives. In one case, crystals suitable for X-ray spectroscopy analysis have been obtained. Several non-covalent N···S and S···S interactions have been observed in the solid state and the physical nature of these interactions has been analyzed using DFT calculations and AIM and NBO analyses. The AIM analysis confirms the formation of these interactions through the presence of bond and ring CPs. The computed values of charge density at the bond CPs give emphasis to the importance and strength of such interactions. From the NBO analysis, we learn that the interaction can be explained by means of electron donation from lone pairs of N and S atoms to sigma antibond S–S and S–C orbitals, respectively.

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