

# Poster Presentations

## [MS25-P07] Polymorphism and isostructurality in the products of reaction of imidazolidine-2-thione with iodine.

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Polyiodides have very different structural and physical properties. Reactions of diiodine with heterocycles such as thiones are well known to produce various types of iodine compounds, including charge transfer complexes, “spoke structures” (D-I<sub>2</sub>; D as a donor), “extended spoke structures” (D-I<sub>2</sub>-I<sub>2</sub>), iodonium salts, monocationic or dicationic disulfides<sup>[1]</sup>. We decided to check how a small change in reaction conditions affects the resulting products.

In the course of our studies we synthesized and crystallographically characterized the ionic complexes of nine novel compounds. We have noticed, for instance, that in the course of one reaction there can be as many as four different salts obtained.

The reactions of imidazoline-2-thione (tHIMT) with HBr (in molar ratio 1:3) and HCl (in molar ratio 1:6) in methanol solutions resulted in formation of two isostructural salts: **(1)** [C<sub>3</sub>H<sub>7</sub>N<sub>2</sub>S<sup>+</sup> Br<sup>-</sup>] and **(2)** [C<sub>3</sub>H<sub>7</sub>N<sub>2</sub>S<sup>+</sup> Cl<sup>-</sup>]. Direct reaction of tHIMT with diiodine and HCl in a molar ratio 1:1:1 in methanol/dichloromethane solution resulted in two complexes of formulae: **(3)** [C<sub>3</sub>H<sub>6</sub>N<sub>2</sub>S]I<sub>2</sub>·I<sub>2</sub> (extended spoke structure) and **(4)** [C<sub>6</sub>H<sub>9</sub>N<sub>2</sub>S]·I<sub>3</sub>. We have found out that **(3)** is isostructural with the previously characterized structure with thiazolidinethione as a ligand<sup>[2]</sup>. Moreover, this compound **(3)** is highly preferred (received six times in different synthesis), which makes it even more interesting. On the other hand compound **(4)** is a polymorph of the structure reported in 2003<sup>[3]</sup>. This certain reaction appeared to be very sensitive for the presence of acetone.

Even traces of acetones cause the formation of **(4)** or its polymorph.

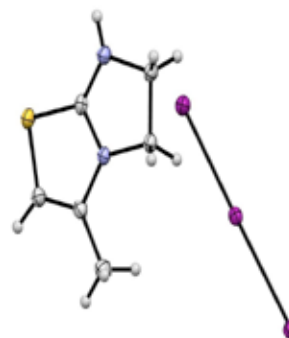


Fig 1. ORTEP diagram of **4**, thermal ellipsoids drawn at the 50% probability level.

Equally sensitive and interesting result of a reaction was a co-crystallization of three various crystals, obtained after leaving crystal **(2)** in the very low concentrated vapor of the iodine. This resulted in formation of: -

**(5)** [(C<sub>3</sub>H<sub>6</sub>N<sub>2</sub>S)<sub>2</sub>I<sup>+</sup>]Cl<sup>-</sup>·H<sub>2</sub>O, **(6)** [(C<sub>3</sub>H<sub>6</sub>N<sub>2</sub>S)<sub>2</sub>I<sup>+</sup>]I<sup>-</sup>[I<sub>3</sub>] and **(7)** [(C<sub>3</sub>H<sub>6</sub>N<sub>2</sub>S)<sub>2</sub>I<sup>+</sup>]<sub>2</sub>I<sup>-</sup>·2(H<sub>2</sub>O). Crystals **(5)** appeared to be isostructural with also newly synthesized crystal **(8)** [(C<sub>3</sub>H<sub>6</sub>N<sub>2</sub>S)<sub>2</sub>I<sup>+</sup>]Br<sup>-</sup>·H<sub>2</sub>O].

Our studies has confirmed that the amount of iodine, molar ratio and many different factors used in the reaction significantly affect the resulting products.

### References:

- [1] P.H. Svensson, L.Kloo. *Chemical Reviews*, 2003: 1649–1684.
- [2] I-E. Parigoridi, G.J. Corban, S.K. Hadjikakou, N. Hadjiliadis, N. Kourkoumelis, G. Kostakis, V. Psycharis, C.P. Raptopoulou and M. Kubicki. *Dalton Trans.*, 2008, 5159–5165
- [3] Y. Thanyasirikul, C. Pakawatchai, M.L. Cole, P.C. Junk, B.W. Skelton and A.H. White. *Org. Biomol Chem.* 2003 Sep 21;1(18):3217-22