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## Direct synthesis and crystal structure of a novel tetranuclear $\text{Co}_2^{\text{III}}\text{Fe}_2^{\text{III}}$ Schiff base complex

Eduard Nikolaevich Chygorin <sup>1</sup>, Vladimir Nikolayevich Kokozay <sup>2</sup>,  
 Iryna Vasylyivna Omelchenko <sup>3</sup> and Julia Anatoliyivna Rusanova <sup>4,\*</sup>

<sup>1</sup> Department of Chemistry, Taras Shevchenko National University of Kyiv, 64/13, Volodymyrska str., Kyiv, 01601, Ukraine  
[chygorin.eduard@gmail.com](mailto:chygorin.eduard@gmail.com) (E.N.Ch.)

<sup>2</sup> Department of Chemistry, Taras Shevchenko National University of Kyiv, 64/13, Volodymyrska str., Kyiv, 01601, Ukraine  
[kokozay@univ.kiev.ua](mailto:kokozay@univ.kiev.ua) (V.N.K.)

<sup>3</sup> Scientific and Technological Center, Institute for Single Crystals, National Academy of Sciences of Ukraine, 60 Nauky ave., Kharkiv, 61072, Ukraine  
[irina@xray.isc.kharkov.com](mailto:irina@xray.isc.kharkov.com) (I.V.O.)

<sup>4</sup> Department of Chemistry, Taras Shevchenko National University of Kyiv, 64/13, Volodymyrska str., Kyiv, 01601, Ukraine  
[rusanova\\_j@yahoo.com](mailto:rusanova_j@yahoo.com) (J.A.R.)

\* Corresponding author at: Department of Chemistry, Taras Shevchenko National University of Kyiv, 64/13, Volodymyrska str., Kyiv, 01601, Ukraine.  
 e-mail: [usanova\\_j@chem.knu.ua](mailto:usanova_j@chem.knu.ua) (J.A. Rusanova).

### RESEARCH ARTICLE



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### ABSTRACT

The title compound, *tetra*( $\mu$ -2-3-(2-oxybenzylideneamino)-1-hydroxypropan-2-olato)-4-nitrophenolatedi-cobalt(III)-di-iron(III) dimethylsulfoxidehexasolvate, crystallizes in the monoclinic space group  $P2_1/c$  and represent the first example of heterometallic  $\text{Co}^{\text{III}}\text{-Fe}^{\text{III}}$  complex with 3-((5-nitro-2-hydroxybenzylidene)amino)propane-1,2-diol/2-(((2,3-dihydroxypropyl)iminio)methyl)-4-nitrophenolate) - a hydroxyl rich Schiff base ligand which was obtained *in situ*. Crystal data for  $\text{C}_{52}\text{H}_{74}\text{Cl}_2\text{Co}_2\text{Fe}_2\text{N}_8\text{O}_{26}\text{S}_6$  ( $M = 1720.01$  g/mol): monoclinic, space group  $P2_1/c$  (no. 14),  $a = 16.353(3)$  Å,  $b = 15.234(2)$  Å,  $c = 15.201(3)$  Å,  $\beta = 113.99(2)^\circ$ ,  $V = 3460.0(12)$  Å<sup>3</sup>,  $Z = 2$ ,  $T = 173(2)$  K,  $\mu(\text{MoK}\alpha) = 1.225$  mm<sup>-1</sup>,  $D_{\text{calc}} = 1.651$  g/cm<sup>3</sup>, 14130 reflections measured ( $5.7^\circ \leq 2\theta \leq 57.266^\circ$ ), 7748 unique ( $R_{\text{int}} = 0.1051$ ,  $R_{\text{sigma}} = 0.2148$ ) which were used in all calculations. The final  $R_1$  was 0.0914 ( $I > 2\sigma(I)$ ) and  $wR_2$  was 0.2279 (all data). The metal ions have distorted octahedral coordination geometry and are joined in a tetranuclear  $\{\text{Co}_2\text{Fe}_2(\mu\text{-O})_6\}$  core by O-bridging atoms from the ligand. There are numerous intermolecular interactions occurring between the components of the crystal:  $\pi$ -hole interaction between  $\text{NO}_2 \cdots \text{NO}_2$  groups of the ligands, short  $\text{S} \cdots \text{S}$ ,  $\text{O} \cdots \text{O}$  and  $\text{C} \cdots \text{C}$  interactions and weak and strong hydrogen bonds.

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### 1. Introduction

During the past several decades, polynuclear complexes have attracted considerable interest due to their potential for a wide range of physicochemical properties such as magnetic [1], catalytic [2], and useful light- and/or redox-induced functions [3]. High-nuclearity solids may exist in a wide range of shapes and different arrangements of the metal atoms [4,5]. Iron is an important bioelement and is a part of heteronuclear cores in metalloproteins such as in purple acid phosphatase (FeZn) [6], humancalcineurin (FeZn) [7] and human protein phosphatase 1 (MnFe) [8]. Fe(III) oxo- and hydroxo-bridged complexes are promising materials in the field of bioinorganic chemistry owing to their relevance as models of the protein active sites [9,10] and their activity in important biological processes [11,12].

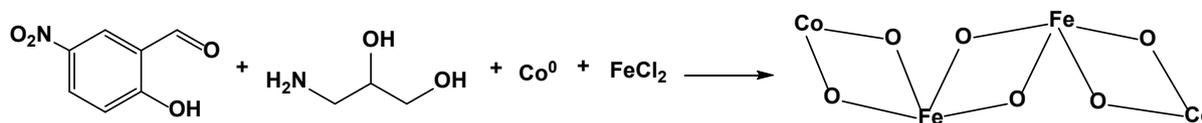
In recent years, one of the most widely utilized classes of ligands in metal coordination chemistry are the Schiff bases.

Due to their synthetic flexibility, these compounds demonstrate exceptionally rich coordination chemistry and diverse properties in the areas of magnetism, luminescence, chirality, catalysis, cytotoxicity, and ferroelectricity [13-15]. Using of these multipurpose ligands in a synthetic approach named direct synthesis of coordination compounds (which based on spontaneous self-assembly of free metal ions with commonly simple and flexible ligands without significant geometrical restrictions) appears to be an extremely powerful tool for the construction of novel hetero-polynuclear complexes [16].

In this study, we have continued our investigations in the field of direct synthesis and present the synthesis and crystal structure of a novel tetranuclear heterometallic (Fe and Co) complex with a mixed N,O,O-donor hydroxyl rich Schiff base ligand derived from the condensation of 5-nitrosalicylaldehyde with 3-amino-1,2-propanediol and formed *in situ*.

**Table 1.** Crystal data and details of the structure refinement for title compound.

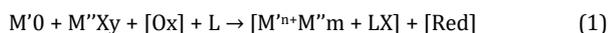
Parameters	Title compound
Empirical formula	C <sub>52</sub> H <sub>74</sub> Cl <sub>2</sub> Co <sub>2</sub> Fe <sub>2</sub> N <sub>8</sub> O <sub>26</sub> S <sub>6</sub>
Formula weight	1720.01
Temperature (K)	173(2)
Crystal system	Monoclinic
Space group	P2 <sub>1</sub> /c
a, (Å)	16.353(3)
b, (Å)	15.234(2)
c, (Å)	15.201(3)
β, (°)	113.99(2)
Volume (Å <sup>3</sup> )	3460.0(12)
Z	2
ρ <sub>calc</sub> (g/cm <sup>3</sup> )	1.651
μ (mm <sup>-1</sup> )	1.225
F(000)	1772.0
Crystal size (mm <sup>3</sup> )	0.100 × 0.100 × 0.050
Radiation	MoK <sub>α</sub> (λ = 0.71073 Å)
2θ range for data collection (°)	2.850 to 28.633
Index ranges	-21 ≤ h ≤ 16, -17 ≤ k ≤ 20, -17 ≤ l ≤ 20
Reflections collected	14130
Independent reflections	7748 [R <sub>int</sub> = 0.1051, R <sub>sigma</sub> = 0.2148]
Data/restraints/parameters	7748/0/442
Goodness-of-fit on F <sup>2</sup>	0.989
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0914, wR <sub>2</sub> = 0.1694
Final R indexes [all data]	R <sub>1</sub> = 0.2083, wR <sub>2</sub> = 0.2279
Largest diff. peak/hole (e Å <sup>-3</sup> )	0.98/-0.63

**Scheme 1.** Reaction scheme for the synthesis of the title compound.

## 2. Experimental

### 2.1. Materials and methods

All reagents were of commercial quality and were used as received. Solvents were dried and purified using standard procedures [17]. The synthetic method, direct synthesis, has been intensively used and reported in literature [16,18-20]. Its basis lies in the oxidative dissolution of metal(s) during the synthesis. In our case of the synthesis of a heterometallic complex we use of one of the metals in a zero-valent state (Co<sup>0</sup>), with the other one in the form of a salt (FeCl<sub>2</sub>) (Scheme 1), which, in general, can be described by Reaction (1)



where ligand L is the product of the condensation of 5-nitrosalicylaldehyde with 3-amino-1,2-propanediol, formed *in situ*.

### 2.2. Measurements

IR spectra were recorded on a Perkin-Elmer Spectrum BX spectrometer (KBr tablet). Elemental analyses for metals were performed with an ICP spectrometer (Fisons Instruments, ARL Model 3410+) and with a Perkin-Elmer 2400 analyzer for C, H, N and Cl. Quantitative determinations of the metals were performed by atomic absorption spectroscopy.

### 2.3. X-ray crystallography

For the crystal structure determination, the single-crystal of the title compound was used for data collection on an Xcalibur Sapphire 3 diffractometer equipped with graphite monochromatic MoK<sub>α</sub> radiation (λ = 0.71073 Å). The crystal data, data collection and structure refinement details are summarized in Table 1. The structure was solved by direct methods and refined by the full-matrix least-squares technique in the anisotropic

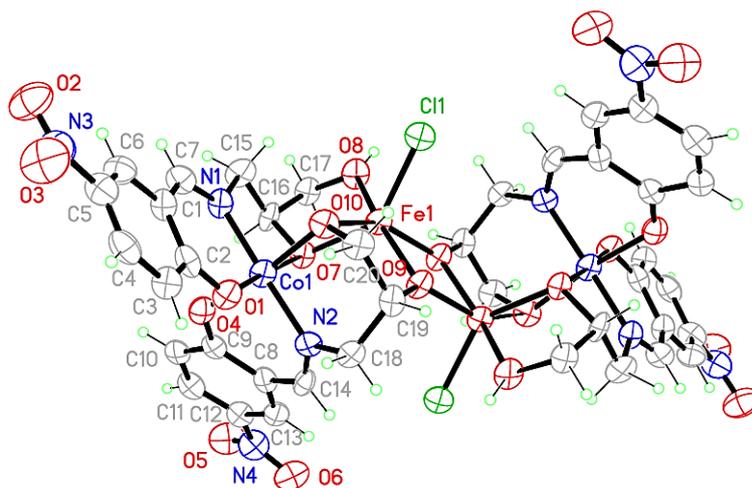
approximation for non-hydrogen atoms. The hydrogen atoms were added geometrically and refined as riding, with  $U_{iso} = 1.2U_{eq}(C)$  for carbon atoms of the ligand and  $U_{iso} = 1.5U_{eq}(C)$  for methyl atoms of DMSO solvate molecules. For data collection: CrysAlisPro, Agilent Technologies, 2014 [21]; cell refinement: CrysAlisPro, Agilent Technologies, 2014 [21]; data reduction: CrysAlisPro, Agilent Technologies, 2014 [21]; program(s) used to solve structure: SHELXT [22]; program(s) used to refine structure: SHELXL [23]; molecular graphics: SHELXTL [24].

### 2.4. Synthesis of title compound

3-Amino-1,2-propanediol (0.194 g, 2.5 mmol), 5-nitrosalicylaldehyde (0.418 g, 2.5 mmol), and triethylamine (0.35 mL, 2.5 mmol) were dissolved in dimethylsulfoxide (DMSO, 25 mL) and magnetically stirred at 323-333 K (10 min). To the resulting hot yellow solution of the ligand, cobalt powder (0.074 g, 1.25 mmol) and FeCl<sub>2</sub>·4H<sub>2</sub>O (0.248 g, 1.25 mmol) were added and the mixture was stirred magnetically until the cobalt was fully dissolved (6 h). Brown crystals suitable for X-ray analysis were formed from the resulting dark colored solution after one month. The crystals were filtered off, washed with dry isopropyl alcohol, and finally dried at room temperature. Yield: 0.3 g, 54% (per iron). Anal. calcd. for C<sub>52</sub>H<sub>74</sub>N<sub>8</sub>O<sub>26</sub>S<sub>6</sub>Cl<sub>2</sub>Co<sub>2</sub>Fe<sub>2</sub>: C, 36.31; H, 4.34; N, 6.51. Found: C, 36.33; H, 4.41; N, 6.49%. FT-IR (KBr, ν, cm<sup>-1</sup>): 3100 br, 2998 w, 2863 w, 1640 m, 1595 vs, 1544 m, 1481 m, 1439 w, 1390 w, 1311 vs, 1250 m, 1200 w, 1125 w, 1100 m, 1015 m, 945 m, 920 w, 841 w, 794 w, 755 w, 731 w, 688 w, 662 w, 595 w, 544 w, 492 w, 442 w. In the high-frequency region, broad medium-intensity bands in the 3100-2998 cm<sup>-1</sup> range can be attributed to ν(CH) due to aromatic C-H stretching, strong bands appearing at 1595 cm<sup>-1</sup> were assigned to ν(C=N) stretching vibrations, a very strong absorption at 1311 cm<sup>-1</sup> was assigned to ν(NO<sub>2</sub>). The presence of the solvate molecules (DMSO) is shown by the medium-intensity bands at 1015 and 945 cm<sup>-1</sup>. The compound is sparingly soluble in DMF and DMSO and insoluble in water.

**Table 2.** Selected bond lengths and angles \*.

Bond	Length (Å)	Bond	Angles (°)
Fe1-O9 <sup>i</sup>	1.961 (5)	O9 <sup>i</sup> -Fe1-O9	71.2 (2)
Fe1-O9	2.037 (5)	O9-Fe1-O7	107.3 (2)
Fe1-O7	2.041 (5)	O7-Fe1-O10	74.9 (2)
Fe1-O10	2.059 (5)	O9 <sup>i</sup> -Fe1-Cl1	104.85 (17)
Fe1-O8	2.139 (5)	O9-Fe1-Cl1	102.99 (17)
Fe1-Cl1	2.303 (3)	O7-Fe1-Cl1	141.69 (16)
S1S-O1S	1.508 (7)	O10-Fe1-Cl1	89.11 (17)
S1S-C1S	1.753 (10)	O8-Fe1-Cl1	85.14 (17)
S1S-C2S	1.764 (10)	O1-Co1-N1	96.2 (3)
Co1-O1	1.863 (5)	O1-Co1-O4	91.2 (2)
Co1-N1	1.881 (7)	O4-Co1-O7	92.5 (2)
Co1-O4	1.891 (6)	N1-Co1-N2	176.9 (3)
Co1-O7	1.892 (5)	O4-Co1-N2	92.8 (3)
Co1-N2	1.915 (6)	O7-Co1-N2	89.7 (2)
Co1-O10	1.916 (5)	O1-Co1-O10	95.0 (2)
O1-C2	1.283 (9)	N1-Co1-O10	85.3 (2)
C1-C6	1.395 (11)	O4-Co1-O10	170.8 (2)

\* Symmetry code: (i)  $-x+1, -y+1, -z+1$ .**Figure 1.** The molecular structure of the title compound, showing the atom numbering scheme, showing 50% probability displacement ellipsoids.

### 3. Results and discussion

The X-ray investigation of the title complex showed the molecular structure as determined in the crystalline phase. The crystal and the refinement data are shown in Table 1. It crystallizes in the monoclinic space group  $P2_1/c$ . The centrosymmetric unit consists of a complex molecule  $\{[\text{Co}_2\text{Fe}_2(\text{HL})_2(\text{L})_2\text{Cl}_2] \cdot 6\text{DMSO}\}$  in which metal atoms are joined by O bridging atoms from the deprotonated Schiff base ligands in the tetranuclear core  $\{\text{Co}_2\text{Fe}_2(\mu\text{-O})_6\}$  forming a nonlinear  $\text{Co}^{\text{III}} \cdots \text{Fe}^{\text{III}} \cdots \text{Fe}^{\text{III}} \cdots \text{Co}^{\text{III}}$  chain-like arrangement. The structure also contains six DMSO solvent molecules of crystallization (Figure 1). The inversion center is situated at the mid-point of the  $\text{Fe}_2\text{O}_2$  unit. The Schiff base ligands, which contains a tetradentate  $\{\text{NOOO}\}$  donor set with three hydroxyl groups that provide their chelating and bridging capabilities, compensate for the charges on the metal ions by existing in two forms, doubly deprotonated (dianionic) and triply deprotonated (trianionic).

Each ligand spanning the cobalt atom meridionally; thus, the coordination geometry around the  $\text{Co}^{\text{III}}$  ion is slightly distorted octahedral with an  $\text{N}_2\text{O}_4$  coordination sphere: two oxygen atoms and one nitrogen atom of the dianionic ligand and one nitrogen atom of the trianionic ligand are in the equatorial plane, apical positions are filled by two oxygen atoms of the trianionic ligand. The chelating fragments coordinated to the  $\text{Co}^{\text{III}}$  ion are twisted, as defined by the dihedral angles of  $70.24(2)^\circ$  between the mean planes of atoms O4/N2/C9/C8/C14 and O1/N1/C7/C1/C2. The Co-O and Co-N bond

lengths are within the ranges 1.863(5) - 1.916(5) Å and 1.881(7) - 1.915(6) Å (Table 2), respectively, which are comparable with those for previously reported related compounds (CSD Refcodes: JIJGUH and PENVIQ) [25,26] ( $\text{Co}^{\text{III}}\text{-O/N}$  distances which fall in the range 1.880(4) - 1.995(4) Å).

The coordination of the  $\text{Fe}^{\text{III}}$  ion is a highly distorted octahedral  $\text{O}_5\text{Cl}$  geometry. Three of the four coordination sites in the equatorial plane are occupied by oxygen atoms of the trianionic Schiff base ligand, one of which is symmetry related (O10, O9, O9  $(-x+1, -y+1, -z+1)$ ), and the fourth site is filled by an oxygen atom of the dianionic ligand (O8); octahedral environment being completed with chlorine atom and one oxygen atom of the dianionic ligand in apical positions with an O7Fe1Cl1 angle of  $141.69(16)^\circ$ . (Figure 2) The Fe-O bond lengths are in the range 1.961(5) - 2.139(5) Å, and the Fe-Cl distance is 2.3033(25) Å. These data are in good agreement with analogous data for previously reported related structures (CSD Refcodes: CINSUQ, QIMVEN, and TOJDED) [27-29].

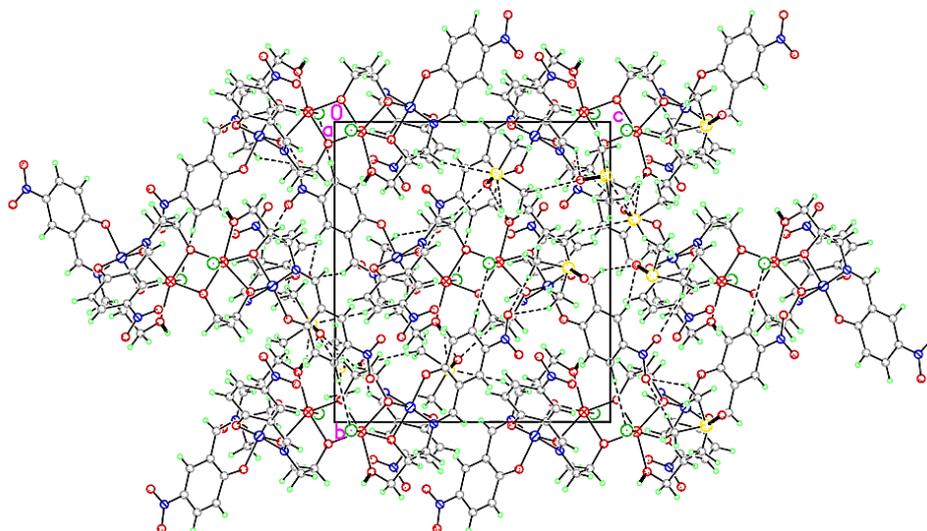
The separation between the  $\text{Co}^{\text{III}}$  and  $\text{Fe}^{\text{III}}$  ions within the binuclear fragment is 3.0321(16) Å and between two symmetry-related  $\text{Fe}^{\text{III}}$  ions is 3.2513(24) Å. Those distances as well as all bonding parameters and the dimensions of the angles in the title complex are in good agreement with those encountered in related complexes [30,31].

As it has been noted [29], one of the forces that make supramolecular system binding blocks stick to one another is  $\pi$ -hole contacts. The shortest reported contact of that type is 2.80 Å [33].

**Table 3.** Hydrogen geometric parameters (Å, °) of the title compound\*.

D-H...A	D-H	H...A	D...A	∠D-H...A
O8-H8-O3S <sup>i</sup>	0.82	11.74	22.562(8)	1173.9
C4-H4-Cl1 <sup>ii</sup>	0.95	2.89	3.762(9)	153.1
C6-H6-O1S <sup>iii</sup>	0.95	2.40	3.283(12)	154.5
C7-H7-O1S <sup>iii</sup>	0.95	2.46	3.326(11)	152.0
C14-H14-O2S <sup>iv</sup>	0.95	2.49	3.274(11)	139.7
C15-H15B-S3S <sup>v</sup>	0.99	2.85	3.758(9)	152.7
C18-H18A-O3S <sup>vi</sup>	0.99	2.48	3.279(9)	138.0
C1S-H1SB-O3	0.98	2.42	3.289(12)	147.0
C2S-H2SC-O3	0.98	2.20	3.101(12)	152.4
C4S-H4SC-O2 <sup>vii</sup>	0.98	2.39	3.357(13)	168.0 2
C5S-H5SA-O2 <sup>viii</sup>	0.98	2.45	3.192(11)	132.5

\* Symmetry codes: (i)  $-x+1, y+1/2, -z+1/2$ ; (ii)  $x, -y+1/2, z-1/2$ ; (iii)  $-x, y+1/2, -z-1/2$ ; (iv)  $-x+1, -y+1, -z+1$ ; (v)  $-x+1, -y+1, -z$ ; (vi)  $x, -y+1/2, z+1/2$ ; (vii)  $-x, y+1/2, -z+1/2$ ; (viii)  $x+1, -y+1/2, z+1/2$ .



**Figure 2.** The crystal packing of the title compound (along *a* axis). Several C-H...O hydrogen bonds and C-H...Cl, C-H...S, C-H...N contacts that link the components in the crystal are shown as dashed lines.

In the title complex  $\pi$ -hole interaction occur between NO<sub>2</sub> groups of the ligands forming O...N and O...C contacts with, respectively distances for [O5-N3] and [O5-C5] distances of 3.0226(108) Å and 3.0894(110) Å, respectively. These are in a good agreement with previously reported for analogous contacts [32].

In addition, in the crystal, all units are connected by a number of short connections and hydrogen bonds (Table 3, Figure 2). Thus, the solvent DMSO molecules connected with each other by short S...S ( $1-x, 0.5+y, 0.5-z$ ) contact [S2S...S3S = 3.4878(38) Å] which is essentially shorter than the sum of the Van der Waals radii for the atoms involved. There are also connections with the complex units through strong O-H...O [O8...O3S ( $1-x, 0.5+y, 0.5-z$ ) = 2.562(8) Å], O-H...S [O8...S3S ( $1-x, 0.5+y, 0.5-z$ ) = 3.596(6) Å] and a number of weak C-H...O, C-H...N, C-H...Cl and C-H...C hydrogen bonds (Table 3). These data are in good agreement with previously published data [33-36].

#### 4. Conclusion

The direct synthesis method for preparation of coordination compounds was successfully used for the open-air preparation of the novel tetranuclear heterometallic complex with polydentate Schiff base ligands formed *in situ* as a product of condensation between 5-nitrosalicylaldehyde and 3-amino-1,2-propanediol. The molecular structure of the newly tetranuclear heterometallic (Fe and Co) complex with a mixed N, O, O-donor hydroxyl rich Schiff base ligand was examined by spectroscopic (IR) and X-ray crystallography techniques.

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#### Supporting information

CCDC-2019437 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>, or by e-mailing [data\\_request@ccdc.cam.ac.uk](mailto:data_request@ccdc.cam.ac.uk), or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

#### Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.

Author contributions: All authors contributed equally to this work.

Ethical approval: All ethical guidelines have been adhered.

Sample availability: Samples of the compounds are available from the author.

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ORCID 

Eduard Nikolaevich Chygorin

 <http://orcid.org/0000-0001-5648-0451>

Vladimir Nikolayevich Kokozay

 <http://orcid.org/0000-0003-1834-3020>

Iryna Vasylyvna Omelchenko

 <http://orcid.org/0000-0002-6882-6832>

Julia Anatoliyivna Rusanova

 <http://orcid.org/0000-0002-0801-1690>

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