

Oxovanadium(V) Complexes of Salicylaldehyde and 3-Methoxysalicylaldehyde 4-phenylthiosemicarbazones. Formation of Two New Thiazoline Compounds*

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In the reaction of tris(2,4-pentandionato)vanadium(III) with salicylaldehyde 4-phenylthiosemicarbazone (H_2L^1) and 3-methoxysalicylaldehyde 4-phenylthiosemicarbazone (H_2L^2), two new mononuclear complexes of vanadium(V), $[VOL^1(OCH_3)]$ (**1**) and $[VOL^2(OCH_3)]$ (**3**), as well as thiazoline derivatives, $\{R-CHNN[CNC(C_6H_5)C(CH_3)C(O)(CH_3)S]\}$ [$R = -C_6H_4(OH)$ (L^1_{cycl} , **2**) or $-C_6H_3(OCH_3)(OH)$ (L^2_{cycl} , **4**)] have been prepared. The isolation of the thiazoline derivatives is the first example of cyclization reactions of thiosemicarbazones with appropriate β -diketone involving vanadium. In vanadium(V) complexes **1** and **3**, vanadium is coordinated by one doubly bonded oxygen atom, ONS atoms from the salicylaldehyde thiosemicarbazone ligand and by an oxygen atom from the methoxo ligand. All compounds have been characterized by means of chemical analyses and IR spectroscopy. The crystal and molecular structures of **1–4** have been determined by single-crystal X-ray diffraction.

INTRODUCTION

This investigation is a part of our research on vanadium and molybdenum complexes with thiosemicarbazones of salicylaldehyde and its derivatives, ligands with interesting ONS donor and chelating properties.^{1–3} Thiosemicarbazones are capable of stabilizing both higher and lower oxidation states of transition metal ions.^{4–7} They can act as single and double charged ions depending on pH values. The dianionic form is dominant at higher pH values whereas the monoanionic form of the ligand is favoured at lower pH values.⁸

Thiosemicarbazones can exist in several tautomeric forms, but the most interesting ones are those denoted with

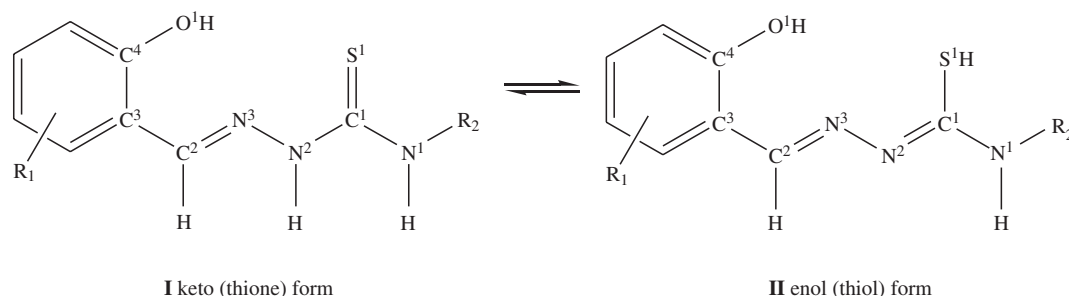
I and **II** (Scheme 1). Such molecular isomerization can result in different binding modes. The molecule can act as tridentate (involving N, S and O atoms) in form **I** or as bidentate (involving N and S atoms) ligand in form **II**.

Oxovanadium(V), oxovanadium(IV) or vanadium(III) complexes^{9–13} with thiosemicarbazones are interesting because of their significant pharmacological activities and catalytic roles in many biological systems and industrial processes.^{14–17}

Since the recent discovery of the presence of trivalent vanadium in several biological systems the interest for vanadium(III) chemistry has considerably grown.¹⁸ The trivalent state is indeed one of its unstable oxidation

* Dedicated to Professor Nikola Kallay on the occasion of his 65th birthday.

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Scheme 1.

states and can be easily oxidized to vanadium(IV) or vanadium(V) especially in solution where these reactions can be very fast. The literature survey revealed only few papers reporting the synthesis and characterization of vanadium(III) complexes with ONS-donor ligands.

Having all these facts in mind, we have been interested in preparing and characterizing vanadium(III) complexes coordinated by thiosemicarbazones, as ONS-donors. However, all our attempts resulted not in vanadium(III) but vanadium(V) thiosemicarbazonato complexes, as well as unexpected thiazoline derivatives as products of condensation (followed by ring closure) of 2,4-pentadione with thiosemicarbazones were obtained.

In this paper we report the syntheses, molecular and crystal structures of $[\text{VOL}^1(\text{OCH}_3)]$ (**1**), $[\text{VOL}^2(\text{OCH}_3)]$ (**3**), L^1_{cycl} (**2**) and L^2_{cycl} (**4**) ($L^1_{\text{cycl}} = 1\text{-}\{2\text{-}[(2\text{-hydroxybenzylidene)-hydrazono]-4\text{-methyl-3-phenyl-2,3-dihydrothiazol-5-yl}\}\text{-ethanone}$, $L^2_{\text{cycl}} = 1\text{-}\{2\text{-}[(2\text{-hydroxy-3-methoxybenzylidene)-hydrazono]-4\text{-methyl-3-phenyl-2,3-dihydrothiazol-5-yl}\}\text{-ethanone}$).

EXPERIMENTAL SECTION

Materials

Salicylaldehyde, 3-methoxysalicylaldehyde, 4-phenylthiosemicarbazide, tris(2,4-pentanedionato)vanadium(III) were of reagent grade and were used as purchased. Salicylaldehyde 4-phenylthiosemicarbazone (H_2L^1) and 3-methoxysalicylaldehyde 4-phenylthiosemicarbazone (H_2L^2) were prepared by addition of a methanolic solution containing appropriate aldehyde to a methanolic solution of 4-phenylthiosemicarbazide. The reaction mixture was then stirred at room temperature for 2–3 h. The resulting white needle-like products were collected by filtration and dried over KOH. Methanol was dried using magnesium turnings and iodine followed by distillation. All reactions were performed in argon atmosphere.

Methods

IR Spectra. – Infrared spectra were recorded as KBr pellets using a Perkin-Elmer Fourier-Transform Spectrum RX1 spectrophotometer in the 4500–450 cm^{-1} region.

Elemental Analyses. – The Analytical Services Laboratory of Ruđer Bošković Institute, provided C, H, N and S analyses. Vanadium was determined according to the previously described method.¹⁹

X-ray Analyses. – The compounds were obtained as black (**1** and **3**) and yellow (**2** and **4**) prismatic crystals. Each single-crystal of **1–4** was mounted on a glass fiber and used for measurements of unit cell parameters and intensity data collection. Diffracted intensities were collected on the Oxford Diffraction Xcalibur 3 diffractometer using graphite-monochromated $\text{MoK}\alpha$ radiation at room temperature, except for **2** for which diffracted intensities were collected at 200 K. Data reduction was performed using the CrysAlis software package.²⁰ The structures were solved by direct methods (SHELXS)²¹ and refined by the full-matrix least-squares method based on F^2 against all reflections (SHELXL-97),²² both programs operating under the WinGX²³ program package. All non-hydrogen atoms were refined anisotropically. The final Fourier maps revealed positions of almost all hydrogen atoms. Those with poor geometry were assigned calculated positions and allowed to ride on their parent atoms. The final difference Fourier maps showed no peaks of chemical significance. Crystal data, data collection and structure refinement for **1–4** are summarized in Table I.

Syntheses

Methoxo-oxo-[salicylaldehyde 4-phenylthiosemicarbazonato(2-)]vanadium(V), $[\text{VOL}^1(\text{OCH}_3)]$ (**1**) and 1- $\{2\text{-}[(2\text{-hydroxybenzylidene)-hydrazono]-4\text{-methyl-3-phenyl-2,3-dihydrothiazol-5-yl}\}\text{-ethanone}$, L^1_{cycl} (**2**). – Salicylaldehyde 4-phenylthiosemicarbazone (460 mg, 1.69 mmol) was dissolved in dry methanol (20 mL) and the solution was refluxed under argon atmosphere 15 minutes before addition of $[\text{V}(\text{acac})_3]$ (200 mg, 0.57 mmol). The reaction mixture was then refluxed for additional eight hours in argon atmosphere. During the reaction the colour of solution changed from pale yellow to dark brown. Yellow crystals of product (**2**) that separated out from the solution after 1–2 days (on cooling) were filtered off, washed with cold methanol and dried *in vacuo*. Yield: 50 mg (8 %). After two weeks black crystals of vanadium(V) complex (**1**) were obtained from the mother liquor. Yield: 110 mg (52 %).

(**1**) *Anal.* Calcd. for $\text{C}_{15}\text{H}_{14}\text{N}_3\text{O}_3\text{SV}$ ($M_r = 367.302$): C 49.05, H 3.84, N 11.44, S 8.73, V 13.87 %; found: C 49.10, H 3.76,

N 11.38, S 8.81, V 13.90 %. IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: (1) 3332 (N–H), 1598, 1586 (C=N), 1318, 756 (C–S), 980 (V=O)

(2) *Anal.* Calcd. for $\text{C}_{19}\text{H}_{17}\text{N}_3\text{O}_2\text{S}$ ($M_r = 351.429$): C 64.94, H 4.88, N 11.96, S 9.12 %; found: C 64.90, H 4.81, N 12.01, S 8.99 %. IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: (2) 1628, 1608, 1576, 1536 (C=N) and (C=O), 1312, 756 (C–S).

Methoxo-oxo-[3-methoxysalicylaldehyde 4-phenylthiosemicarbazono(2-)] vanadium(V), [VOL²(OCH₃)] (3) and 1-{2-[(2-Hydroxy-3-methoxy-benzylidene)-hydrazono]-4-methyl-3-phenyl-2,3-dihydro-thiazol-5-yl}-ethanone, L²_{cycl} (4).

– These products were obtained as yellow crystals (4) and black crystals (3) by adopting the procedure similar to that for 1 and 2 but using 30 mL of methanolic solution of 3-methoxysalicylaldehyde 4-phenylthiosemicarbazone (260 mg, 0.86 mmol) instead of salicylaldehyde 4-phenylthiosemicarbazone. Yield (3): 110 mg (48 %). Yield (4): 80 mg (24 %).

(3) *Anal.* Calcd. for $\text{C}_{16}\text{H}_{16}\text{N}_3\text{O}_4\text{SV}$ ($M_r = 397.328$): C 48.37, H 4.06, N 10.58, S 8.07, V 12.82 %; found: C 48.35, H 3.98, N 10.44, S 8.05, V 12.83 %. IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: (3) 3339 (N–H), 1590, 1565 (C=N), 1318, 763 (C–S), 978 (V=O).

(4) *Anal.* Calcd. for $\text{C}_{20}\text{H}_{19}\text{N}_3\text{O}_3\text{S}$ ($M_r = 382.463$): C 62.97, H 5.02, N 11.02, S 8.41 %; found: C 62.90, H 5.19, N 10.87, S 8.30 %. IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: (4) 1628, 1602, 1578, 1532 (C=N) and (C=O), 1314, 763 (C–S).

RESULTS AND DISCUSSION

Synthesis and Characterization of Vanadium(V) Complexes [VOL¹(OCH₃)] (1) and [VOL²(OCH₃)] (3) and Thiazole Derivatives L¹_{cycl} (2) and L²_{cycl} (4)

Our attempt to prepare new vanadium(III) complexes did not result with desired products, but instead we obtained two different kinds of compounds: new vanadium(V) complexes (1, 3) and thiazoline derivatives (2, 4). In both complexes (1, 3) the ligands are coordinated to vanadium as tridentate ONS-donors through phenolic-oxygen, imine-nitrogen and thiol-sulphur. The distorted tetragonal-pyramidal coordination of vanadium ion is completed by a methoxo-ligand and by the doubly bonded oxygen.

TABLE I. Crystal data, data collection and refinement

Compound	1	2	3	4
Chemical formula	$\text{C}_{15}\text{H}_{14}\text{N}_3\text{O}_3\text{SV}$	$\text{C}_{19}\text{H}_{17}\text{N}_3\text{O}_2\text{S}$	$\text{C}_{16}\text{H}_{16}\text{N}_3\text{O}_4\text{SV}$	$\text{C}_{20}\text{H}_{20}\text{N}_3\text{O}_3\text{S}$
Molecular weight	367.302	351.429	397.328	382.463
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$	$P\bar{1}$
$a / \text{\AA}$	10.3774(9)	11.8394(13)	10.4120(12)	7.8143(17)
$b / \text{\AA}$	9.9754(7)	10.1465(15)	10.0030(12)	9.895(2)
$c / \text{\AA}$	15.3836(13)	14.9689(18)	16.2612(19)	12.068(2)
$\alpha / ^\circ$	90.00	90.00	90.00	86.928(15)
$\beta / ^\circ$	100.183(7)	96.590(9)	96.665(9)	90.395(15)
$\gamma / ^\circ$	90.00	90.00	90.00	87.592(17)
$V / \text{\AA}^3$	1567.4(2)	1786.3(4)	1682.2(3)	930.9(3)
Z	4	4	4	2
Temperature / K	293	293	293	293
$\rho(\text{calcd.}) / \text{g cm}^{-3}$	1.556	1.307	1.569	1.364
μ / mm^{-1}	0.783	0.198	0.741	0.200
$F(000)$	752	736	816	402
Crystal size / mm^3	0.67×0.29×0.21	0.76×0.29×0.25	0.85×0.39×0.28	0.53×0.30×0.26
Crystal colour	Black	Yellow	Black	Yellow
θ range / $^\circ$	4.40–27.49	4.10–26.00	4.27–26.00	4.25–24.99
Reflections collected	14559	10236	14978	8033
Independent reflections	3557	3496	3288	3250
Observed reflections	3351	2425	2756	2115
Absorption correction	Analytical	Analytical	Analytical	Analytical
$R^{(a)}$, $wR^{(b)}$ [$I \geq 2\sigma(I)$]	0.0339, 0.0837	0.0554, 0.1531	0.0490, 0.1317	0.0576, 0.1566
Goodness-of-fit, $S^{(c)}$	1.063	1.118	1.103	1.031
Maximum/minimum	0.371/–0.326	0.357/–0.183	0.606/–0.415	0.436/–0.508
$\Delta\rho$ (e \AA^{-3})				

^(a) $R = \sum ||F_o| - |F_c|| / \sum F_o$

^(b) $w = 1 / [\sigma^2(F_o^2) + (g_1P)^2 + g_2P]$ where $P = (F_o^2 + 2F_c^2) / 3$

^(c) $S = \sum [w(F_o^2 - F_c^2)^2 / (N_{\text{obs}} - N_{\text{param}})]^{1/2}$

The complexes **1** and **3** are stable in air and highly soluble in DMF, DMSO, acetone, γ -picoline and pyridine but moderately soluble in MeOH, EtOH, CH₃CN, CHCl₃ and CH₂Cl₂. The results of elemental analyses are in good agreement with the assigned formulations. In the IR spectra of complexes the single strong absorption bands found at 980 cm⁻¹ (**1**) and 978 cm⁻¹ (**3**) are assigned to V=O stretching. The bands around 1600 cm⁻¹ are assigned to the stretching of C–N and C–O(phenolic) bonds of the thiosemicarbazones. The absorption bands located at about 1318 (**1**) and 1314 (**3**) cm⁻¹ are attributed to the coordinated C–S(thiol) stretching. The absence of $\nu(\text{O–H})$ bands, found in the spectra of free ligands at about 3100 cm⁻¹, confirms that the coordination occurs *via* phenolic oxygen. These data and the remaining frequencies in the IR spectra are in accordance to the literature data for the same type of complexes.¹³

The thiazoline compounds, 1-{2-[(2-hydroxy-benzylidene)-hydrazono]-4-methyl-3-phenyl-2,3-dihydro-thiazol-5-yl}-ethanone (L¹_{cycl}, **2**) and 1-{2-[(2-hydroxy-3-methoxy-benzylidene)-hydrazono]-4-methyl-3-phenyl-2,3-dihydro-thiazol-5-yl}-ethanone (L²_{cycl}, **4**) were isolated as the first products of the previously described reactions. The thiazole/thiazoline ring can be prepared using

several different methods, although the most widely used approach relies on Hantzsch's synthesis, which involves reaction between haloketones and thioamides.²⁵ Recently, several articles were published reporting the synthesis of thiazolines from thiosemicarbazones.^{26,27} Various cyclization reactions of thiosemicarbazones and related molecules induced by metal ions are well described in the literature.²⁸ Therefore, it seems reasonable to conclude that compounds **2** and **4** in our case are the result of the condensation between thiosemicarbazones and 2,4-pentandione (from [VO(acac)₃]) promoted by the presence of vanadium. The role of vanadium is not precisely known at this time, but what is known and confirmed by several control experiments is that presence of a base (triethylamine for example) is an obligatory step in generation of the thiazoline ring. Finally, as far as we are aware, this is the first example of cyclization reactions of thiosemicarbazones with appropriate β -diketone involving vanadium.

The important IR bands of **2** and **4** are in accordance with the literature data for the same type of compounds.²⁵ The spectra of compounds **2** and **4** exhibit four bands in the 1628–1536 cm⁻¹ region, corresponding to C=N and C=O stretching, respectively. The absorption bands lo-

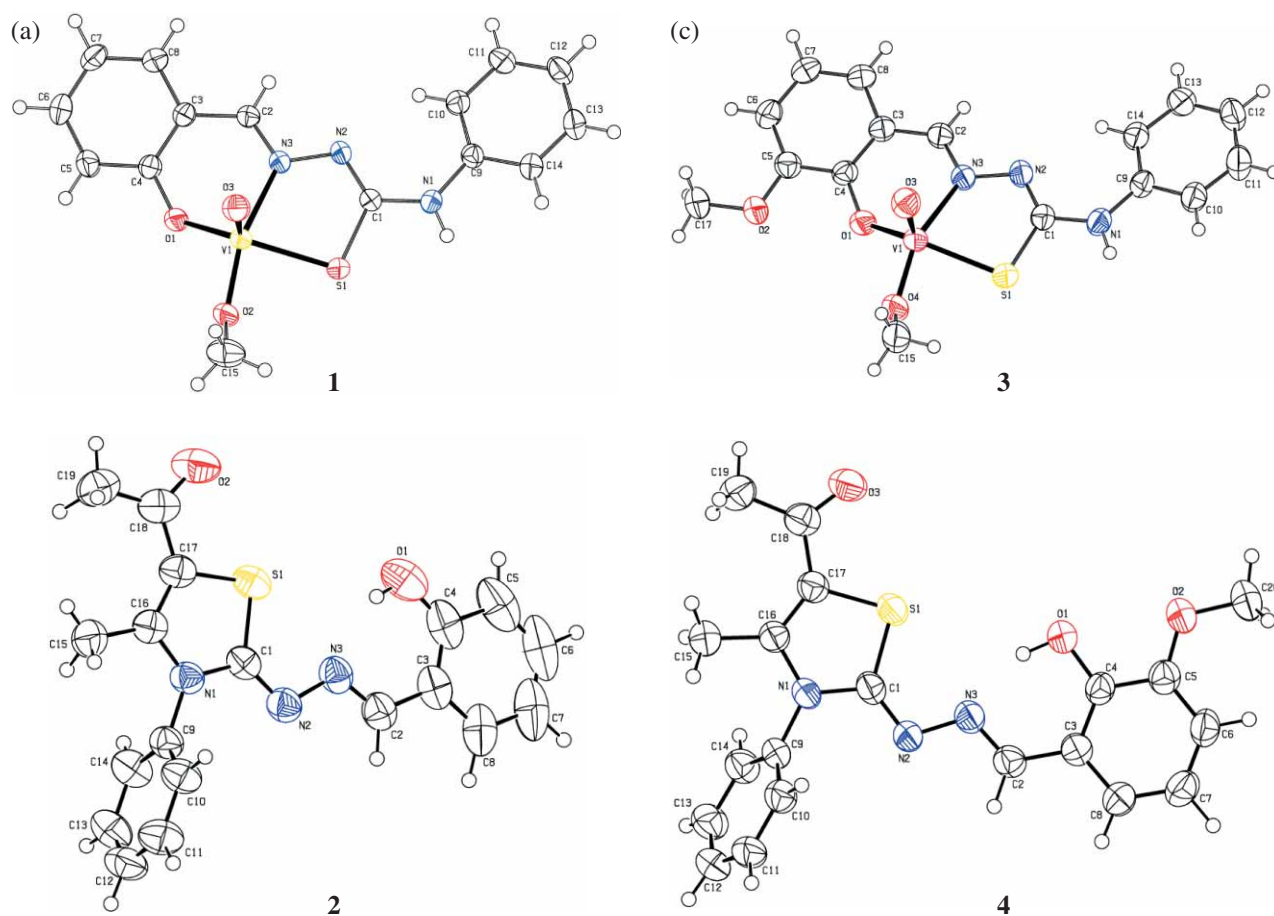


Figure 1. ORTEP²³ drawings of the compounds **1–4** with atom labelling schemes. Ellipsoids are drawn at the 50 % probability level. Hydrogen atoms are presented as spheres of arbitrary small radii.

TABLE II. Selected interatomic distances / Å for **1** and **3**

	1	3
V1-O (methoxo)	1.7593(12)	1.7655(19)
V1-N3	2.1482(14)	2.149(2)
V1-S1	2.3168(5)	2.3295(8)
V1-O1	1.8318(12)	1.8198(18)
V1-O3 (apical)	1.5925(13)	1.589(2)
C1-S1	1.7525(16)	1.744(3)
C4-O1	1.340(2)	1.341(3)
C2-N3	1.288(2)	1.292(3)
N2-N3	1.3946(18)	1.399(3)
C1-N2	1.288(2)	1.286(4)
C1-N1	1.367(2)	1.376(3)
C2-C3	1.446(2)	1.435(4)
C3-C4	1.408(2)	1.400(4)

cated at 1312 and 756 cm^{-1} (**2**) and at 1282 and 760 cm^{-1} (**4**) are attributed to the C–S bond vibrations.

Crystal and Molecular Structures

Crystal data, data collection and structure refinement for **1–4** are summarized in Table I.

The relevant bond lengths and angles for **1–4** are given in Tables II, III and IV. ORTEP drawings of the complexes with the atom numbering schemes are given in Figure 1.

The vanadium atom in complexes **1** and **3** shows the distorted tetragonal-pyramidal coordination. The tridentate thiosemicarbazone ligand is bonded to the V=O group through the phenolic-oxygen, imine-nitrogen and thiolato-sulphur atoms forming six- and five-membered chelate rings. The fifth coordination site is occupied by a methoxo-ligand. Selected interatomic distances are reported in Table II.

Pentacoordinate vanadium(V) complexes may adopt either trigonal bipyramidal or tetragonal pyramidal geometry. However, perfect geometries are seldom encoun-

tered and distortion from either of these geometries is best shown by the τ -value proposed by Addison *et al.*²⁹ In accordance to this criterion, τ -values of 0.28 and 0.29 for **1** and **3**, respectively, would suggest the tetragonal pyramidal environment around the vanadium ion (Table III.). The coordination is 28 % and 29 % along the pathway of distortion from tetragonal pyramidal to trigonal bipyramidal. Almost the same degree of distortion is found in complex [VO (ONS) (OEt)] {ONS = (R)-salicylaldehyde[benzylmercaptothio-carbonylhydrazone(2-)], R = H or 3-OMe}.⁵

Like in many uncomplexed and unprotonated thiosemicarbazones,³⁰ the *trans* configuration of the S1 atoms with respect to N3 is observed (and with N1 in the *cis* configuration with respect to N3) while in chelation with vanadium the relative positions of above-mentioned atoms change.

The vanadium ion is shifted for about 0.55 Å above the basal plane (base of the tetragonal pyramid), which is defined with O1, N1, S1 and the oxygen atom from the methoxo ligand, so that the six and five-membered chelate rings formed by chelation the vanadium(V) ion are not planar. As a consequence, delocalisation of the π -electrons within the mentioned rings is slightly interrupted as can be seen easily from the bond length distribution.

The salicylaldiminic part of the molecule is almost planar. Shortening of C5–C6 [1.3763(29) in **1** and 1.3807(40) Å in **3**] is consistent with the quinoid effect.³¹

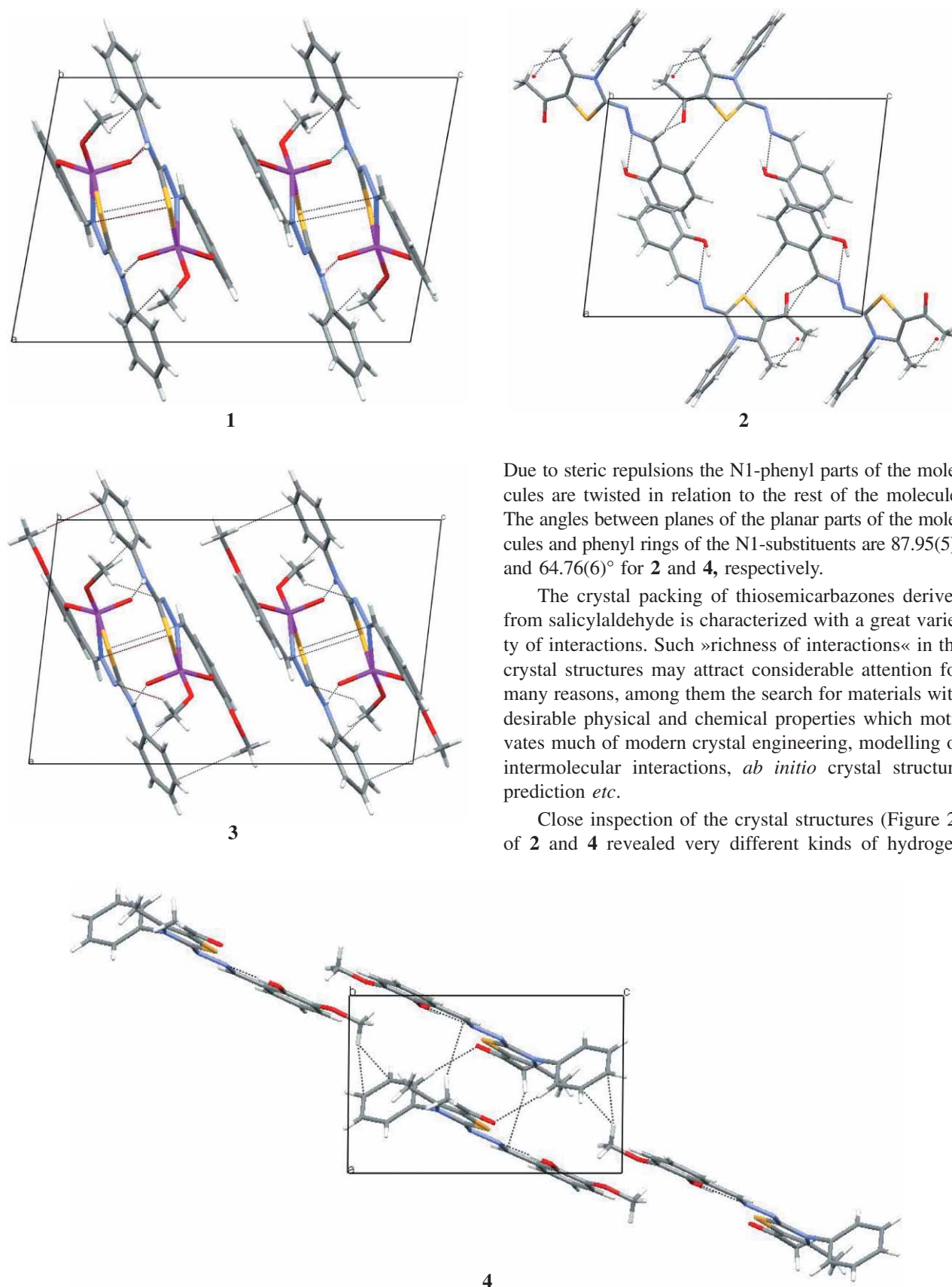
Salicylaldiminic and thiosemicarbazone parts of molecules **2** and **4** are planar. Delocalization of π -electron accounts for the bond lengths in the above mentioned parts of molecules being intermediate between single and double bonds. Other geometric parameters (Table IV) are comparable with those in the Cambridge Structural Database.³²

TABLE III. Selected interatomic angles / ° for **1** and **3**

	1	3
O3-V1-O (methoxo)	106.36(7)	105.86(10)
O1-V1-O (methoxo)	99.02(6)	100.79(9)
N3-V1-O (methoxo)	157.46(6)	156.27(9)
S1-V1-O (methoxo)	85.75(5)	84.53(7)
N3-V1-S1	77.41(4)	77.32(6)
O1-V1-S1	137.25(5)	139.06(7)
O3-V1-O1	109.93(7)	109.15(10)
O1-V1-N3	83.85(5)	83.70(8)
O3-V1-S1	109.25(5)	108.18(8)
O3-V1-N3	93.41(6)	94.35(9)

TABLE IV. Selected interatomic distances / Å for **2** and **4**

	2	4
C1-N2	1.304(3)	1.299(3)
C1-N1	1.376(3)	1.388(3)
C1-S1	1.731(2)	1.741(3)
C2-N3	1.276(3)	1.282(3)
C2-C3	1.446(3)	1.457(4)
C3-C4	1.381(4)	1.398(4)
C4-O1	1.361(4)	1.368(3)
N2-N3	1.398(3)	1.387(3)
C15-C16 (C16-C17)	1.478(3)	1.495(4)
C16-C17 (C17-C18)	1.362(3)	1.354(4)
C16-N1 (C17-N1)	1.393(3)	1.392(3)
C17-C18 (C18-C19)	1.453(3)	1.461(4)
C17-S1 (C18-S1)	1.758(3)	1.765(3)
C18-C19 (C19-C20)	1.484(4)	1.499(4)



Due to steric repulsions the N1-phenyl parts of the molecules are twisted in relation to the rest of the molecule. The angles between planes of the planar parts of the molecules and phenyl rings of the N1-substituents are $87.95(5)^\circ$ and $64.76(6)^\circ$ for **2** and **4**, respectively.

The crystal packing of thiosemicarbazones derived from salicylaldehyde is characterized with a great variety of interactions. Such »richness of interactions« in the crystal structures may attract considerable attention for many reasons, among them the search for materials with desirable physical and chemical properties which motivates much of modern crystal engineering, modelling of intermolecular interactions, *ab initio* crystal structure prediction *etc.*

Close inspection of the crystal structures (Figure 2) of **2** and **4** revealed very different kinds of hydrogen

Figure 2. Crystal packing of the compounds **1-4** in the unit cell viewed down *b*-axis. The hydrogen bonds, C-H \cdots π and $\pi\cdots\pi$ interactions are represented as black dashed lines.

bonds, T-shaped C–H... π interactions between the molecules and the intramolecular O1–H1O...N3 (x , y , z) hydrogen bond. Molecules in **1** and **3** are mutually bonded in a similar way (Figure 2). C–H... π and π ... π interactions dominate in the crystal structure of **1**, while sheets of molecules are formed by the C–H... π and S... π interactions in **3**.

CONCLUSIONS

Four new compounds, methoxo-oxo-[salicylaldehyde 4-phenylthiosemicarbazonato(2-)]vanadium(V) (**1**), 1-[2-[(2-hydroxy-benzylidene)-hydrazono]-4-methyl-3-phenyl-2,3-dihydro-thiazol-5-yl]-ethanone (**2**), methoxo-oxo-[3-methoxysalicylaldehyde 4-phenylthiosemicarbazonato(2-)]vanadium(V) (**3**) and 1-[2-[(2-hydroxy-3-methoxy-benzylidene)-hydrazono]-4-methyl-3-phenyl-2,3-dihydro-thiazol-5-yl]-ethanone (**4**) were isolated and characterized. The vanadium(V) complexes (**1**, **3**) and thiazoline derivatives (**2**, **4**) were obtained by the reaction of [V(acac)₃] with 4-phenylthiosemicarbazone ligands derived from salicylaldehyde or 3-methoxy salicylaldehyde. The oxidation of vanadium(III) to vanadium(V) was caused by traces of oxygen present in the methanolic solution. Thiazoline derivatives (**2** and **4**) are result of the reaction of the appropriate thiosemicarbazone and 2,4-pentandione.

Vanadium centres in complexes [VOL¹(OCH₃)] (**1**) and [VOL²(OCH₃)] (**3**) have pentacoordinated environments completed by oxo-oxygen, tridentate thiosemicarbazonato (ONS) and methoxo ligands.

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Supplementary Materials. – Supplementary crystallographic data sets for the structures of **1–4** are available through the Cambridge Structural Data base with deposition numbers 626931 (for **1**), 626932 (for **2**), 626933 (for **3**) and 626934 (for **4**). Copies of this information may be obtained free of charge from the director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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SAŽETAK

Kompleksi oksovanadija(V) sa salicilaldehid- i 3-metoksisalicilaldehid 4-feniltiosemikarbazonom. Nastanak dva nova tiazolinska spoja.

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Novi mononuklearni kompleksi vanadija(V) (**1** i **3**) i derivati tiazolina (**2** i **4**) priređeni su reakcijom *tris*(2,4-pentandionato)vanadija(III) sa salicilaldehid- i 3-metoksisalicilaldehid 4-feniltiosemikarbazonom. Izolacija tiazolinskih derivata prvi je primjer reakcije ciklizacije tiosemikarbazona i β -diketona u prisutnosti vanadija. U kompleksima vanadija(V), atom vanadija koordiniran je okso-kisikovim atomom, ONS atomima tiosemikarbazonskog liganda te kisikovim atomom metokso skupine. Svi priređeni spojevi identificirani su na osnovi podataka elementne analize i infracrvene spektroskopije, te su im određene molekulska i kristalna struktura difrakcijom rentgenskih zraka na monokristalu.