



Dedicated to Professor Cristian Silvestru
on the occasion of his 65th anniversary

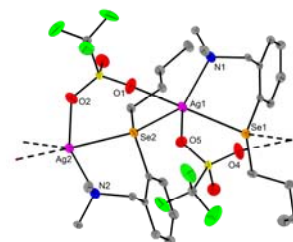
NEW SILVER(I) AND MERCURY(II) METAL COMPLEXES CONTAINING THE HETEROLEPTIC DIORGANOSELENIUM(II) LIGAND (n-Bu)[2-(Me₂NCH₂)C₆H₄]Se. SYNTHESIS AND STRUCTURAL CHARACTERIZATION

Darius DUMITRAS, Ana-Maria HODOROGEA, Dragoș MĂRGINEANU and Alexandra POP*

Supramolecular Organic and Organometallic Chemistry Centre, Chemistry Department,
Faculty of Chemistry and Chemical Engineering, Babes-Bolyai University, RO-400028 Cluj-Napoca, Roumania

Received October 31, 2019

The new ligand (n-Bu)[2-(Me₂NCH₂)C₆H₄]Se (**1**) was obtained, in very good yield and its coordination behaviour was investigated towards AgOTf (OTf = trifluoroacetate) and HgCl₂. Ligand **1** and the metal complexes [AgOTf{Se(n-Bu)[C₆H₄(CH₂NMe₂)-2}] (**2**) and [HgCl₂{Se(n-Bu)[C₆H₄(CH₂NMe₂)-2}] (**3**) were characterized in solution by multinuclear NMR spectroscopy (¹H, ¹³C, ¹⁹F, ⁷⁷Se, as appropriate) and mass spectrometry. Single crystal X-ray diffraction studies revealed the formation of a 1D coordination polymer in [AgOTf{Se(n-Bu)[C₆H₄(CH₂NMe₂)-2}]_n (**2**), when the heteroleptic diorganoselenium(II) ligand present a (N,Se)-bidentate triconnective coordination pattern.



INTRODUCTION

During last decades organoselenium chemistry became undoubtedly a well-established area of research and it was very rapidly developed, based on the valuable applications of organoselenium compounds in various fields. Organoselenium moieties can act either as nucleophiles or electrophiles and can be easily incorporated into a wide variety of substrates. Derivatives containing intramolecularly coordinating groups have been used in organic synthesis, *i.e.* asymmetric selenoxide elimination, [2,3]-sigmatropic rearrangement reactions, methoxyselenylation, oxyselenylation, asymmetric hydrosilylation, asymmetric selenocyclization, selective hydrogenation, etc.¹⁻⁵ Diorganoselenides containing organic groups with donor atoms capable for

intramolecular D→Se interaction (D = N, O) found applications in biology as enzymatic models for GPx mimics (Gpx = Glutathione peroxidase).⁶⁻⁹

Moreover, diorganoselenides bearing organic groups with *hard* donor atoms were successfully used in coordination chemistry, as neutral ligands towards transition metals, being able to behave as a *Se,D*-moiety (D = O or N) in metal complexes with promising catalytic activity.¹⁰

As part of our interest in the coordination chemistry of organoselenium(II) compounds containing organic groups with donor atoms capable to coordinate intramolecularly (*i.e.* [R₂C(OH)CH₂][2-(Me₂NCH₂)C₆H₄]Se (R = Me, Ph),¹¹ [2-(R₂NCH₂)C₆H₄][(3,5-dmpz)CH₂CH₂]Se (R = Me, Et, dmpz = dimethylpyrazole),¹² we report herein on the synthesis and structural characterization of the new

* Corresponding author: alepop@chem.ubbcluj.ro

heteroleptic diorganoselenium(II) ligand (*n*-Bu)[2-(Me₂NCH₂)C₆H₄]Se (**1**), used as building block for silver and mercury complexes.

RESULTS

The heteroleptic diorganoselenide **1** was obtained based on the succession of reactions depicted in Scheme 1: the *ortho*-lithiation of *N,N*-dimethylbenzylamine, insertion of elemental selenium into the new formed carbon-lithium bond and, finally, LiBr elimination in the reaction between the lithium organoselenolate and butyl bromide.

The reaction between AgOTf and HgCl₂, respectively, and the ligand **1** in a 1:1 molar ratio, at room temperature, yielded the complexes **2** and **3**. These synthetic procedures required the use of dichloromethane (DCM) as solvent, a short reaction time and darkness in case of the silver complex. Recrystallization of **2** from a mixture of DCM and *n*-hexane yielded colourless single-crystals suitable for X-ray diffraction.

The compounds were obtained as air-stable species, a colourless oil (**1**) and crystalline solids (**2** and **3**). They are soluble in organic solvents, such as chloroform, acetone or toluene. HRMS and NMR data are consistent with the expected formulas. Characteristic ions are present in the mass spectra, which contain the molecular ion as the base peak.

DISCUSSION

Solution behaviour

The room temperature ¹H and ¹³C NMR spectra of the ligand **1** and the complexes **2** and **3** exhibit the expected resonances for the organic groups attached to selenium, both in the aliphatic and in the aromatic regions. Multiplet resonances were observed in the aliphatic region of the ¹H NMR spectra, corresponding to the butyl fragment and two sharp singlet resonances were assigned to the

methyl and the methylene protons in the [2-(Me₂NCH₂)C₆H₄] group. No evidence for the intramolecular N→E interaction in solution could be outlined from the ¹H NMR spectra. The ¹H NMR spectra of the silver(I) and mercury(II) complexes in CDCl₃ display a similar pattern, but the resonances are shifted in comparison with those corresponding to ligand **1**.

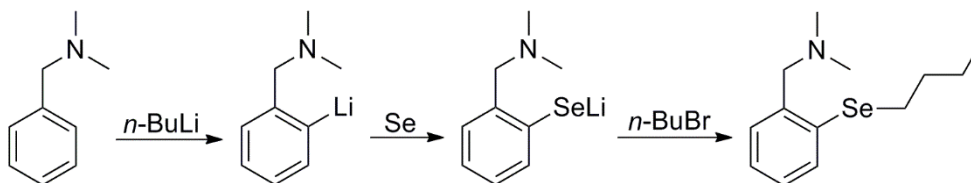
The ⁷⁷Se NMR spectra of compounds **2** and **3** display one singlet resonance (δ 188 ppm and 217 ppm, respectively), high field shifted when compared to the starting ligand **1** (δ 249 ppm) and in agreement with the values for several close related compounds described previously (*i.e.* δ 199 ppm [Me₂C(OH)CH₂][2-(Me₂NCH₂)C₆H₄]Se,¹¹ and different heteroleptic derivatives containing methyl,¹³ butyl,¹⁴ cinnamyl and naphthalene groups, for which the ⁷⁷Se resonances appear in the range 240 - 290 ppm¹⁵⁻¹⁷).

For complex **2** the ¹⁹F NMR spectrum exhibits only one singlet resonance, thus suggesting the presence of only one OTf species in solution.

The ESI+ HRMS spectra of the ligand and the metal complexes are consistent with the formation of the target compounds. The molecular peak at *m/z* 272.09125 [M+H]⁺ was observed as a base peak for the ligand **1**, while for the metal complexes were observed peaks corresponding to [M-OTf]⁺ at *m/z* 377.98897 for **2** and to [M-Cl]⁺ at *m/z* 508.02219 for **3**, respectively.

Single-crystal X-ray diffraction studies

Single crystals of **2** were obtained by slow diffusion of *n*-hexane into a DCM solution of the complex and the structure was determined by X-ray diffraction. The molecules of **2** are associated in a *zig-zag* 1D chain-like polymeric structure, formed by repeating dinuclear units. The thermal ellipsoids representation of such a dinuclear unit, with the atom numbering scheme, is depicted in Figure 1, while the 1D coordination polymer is shown in Figure 2. Relevant interatomic distances and bond angles are given in Table 1.



Scheme 1 – Synthesis of ligand **1**.

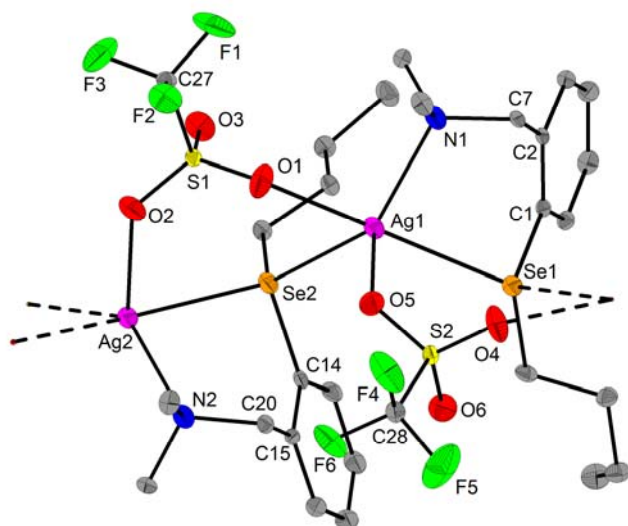


Fig. 1 – Ellipsoid representation of the repeating unit in the crystal of **2**. Ellipsoids are drawn with 30% probability. Hydrogen atoms were omitted for clarity.

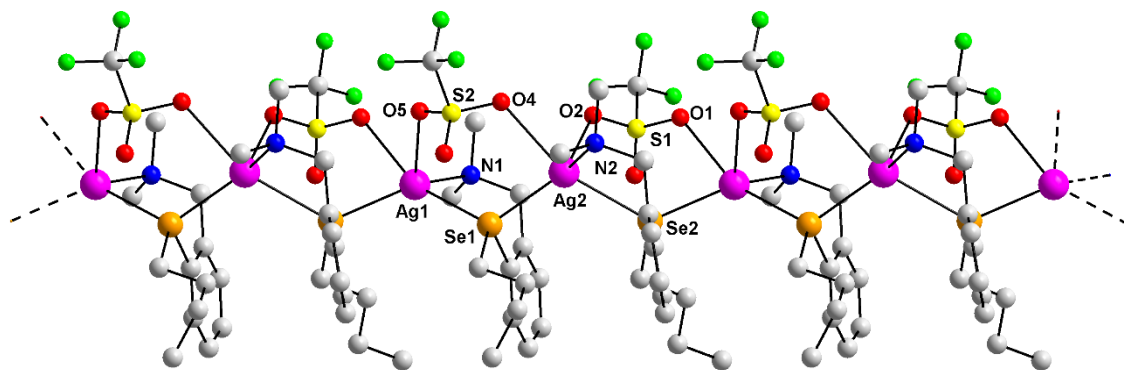


Fig. 2 – View along *c* axis of the 1D coordination polymer in crystal of **2**.

Table 1

Selected bond distances [Å] and angles [°] for compound **2**^a

Ag1–O1	2.6225(5)	Se1–Ag1–Se2	114.46(1)
Ag1–O5	2.5409(5)	Se1–Ag1–O1	158.58(1)
Ag1–Se1	2.7729(5)	Se1–Ag1–O5	87.98(1)
Ag1–Se2	2.6020(5)	Se1–Ag1–N1	86.08(1)
Ag1–N1	2.3300(5)	Se2–Ag1–O1	81.09(1)
Ag2–O2'	2.5333(5)	Se2–Ag1–O5	103.76(1)
Ag2–O4	2.6274(5)	Se2–Ag1–N1	142.24(1)
Ag2–Se1	2.6286(5)	O1–Ag1–O5	73.50(1)
Ag2–Se2'	2.7534(5)	O1–Ag1–N1	89.67(1)
Ag2–N2	2.3568(5)	O5–Ag1–N1	108.52(1)
		Se1–Ag2–O4	84.54(1)
		Se1–Ag2–Se2'	113.00(1)
H4...F2	2.4917(1)	Se1–Ag2–O2'	113.19(1)
H24A...F4	2.5364(2)	Se1–Ag2–N2'	135.63(1)
		O4–Ag2–Se2'	161.43(1)
		O4–Ag2–O2'	78.52(1)
		O4–Ag2–N2'	85.70(1)
		Se2'–Ag2–O2'	88.48(1)
		Se2'–Ag2–N2'	85.51(1)
		O2'–Ag2–N2'	106.98(1)

^a Symmetry equivalent position $-1+x, y, z$ is given by "prime".

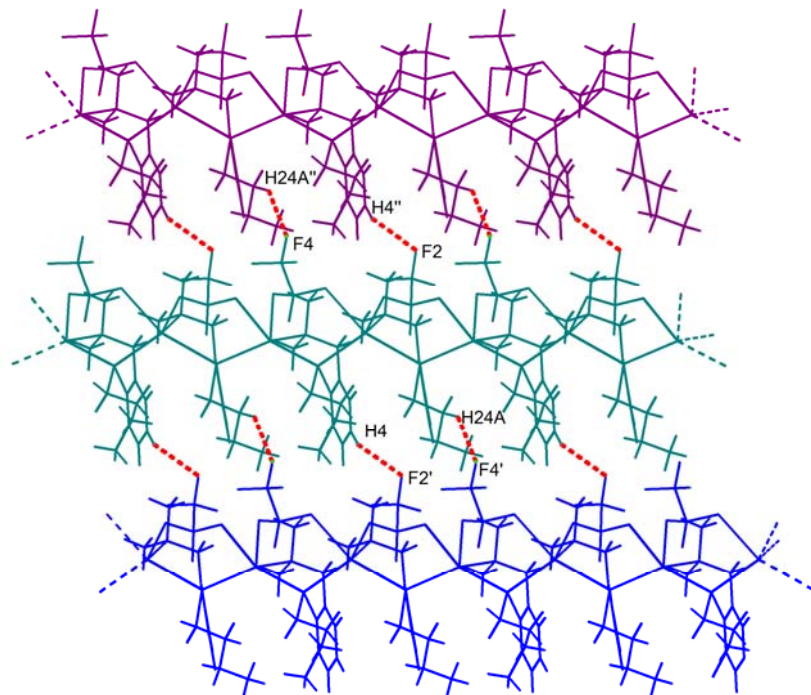


Fig. 3 – View along *c* axis of a supramolecular layer based on F \cdots H contacts in the crystal **2** [symmetry equivalent atoms (*x*, $-1+y$, *z*), ($-1+x$, $1+y$, *z*) are given by “prime” and “double prime”, respectively].

The Ag–Se bond distances (Se1–Ag1 2.7729(5) and Se2–Ag1 2.6020(5) Å, cf. $\Sigma_{\text{vdw}}(\text{Se}, \text{Ag})$ 3.7 Å¹⁸) are in the range found for other silver(I) complexes, *i.e.* [Ag(TFA)(*t*-Bu₂Se)₂], [Ag₄I₄(*t*-Bu₂Se)₄],¹⁹ and [Ag₂{ μ -*o*-C₆H₄(SeMe)₂}{*o*-C₆H₄(SeMe)₂}]₂²⁺.²⁰ The nitrogen atoms from the pendant arms are strongly coordinated to silver (N1–Ag1 2.3300(3) and N2–Ag1 2.3568(5) Å, cf. $\Sigma_{\text{vdw}}(\text{N}, \text{Ag})$ 3.25 Å¹⁸), at interatomic distances close to that one found in the related species [AgOTf{S[CH₂C(OH)Me₂][C₆H₄(CH₂NMe₂)-2]}] (N–Ag 2.251(3) Å)¹¹ and slightly stronger than those found in [AgOTf{P[C₆H₄(CH₂NMe₂)-2]}₃] (N–Ag 2.387(5) and 2.398(4) Å).²¹ The strong N→Ag intramolecular coordination results in the formation of a non-planar six-membered AgNC₃Se chelate ring, folded about the imaginary Se1 and C7 axis, adopting a distorted boat conformations. While the heteroleptic diorganoselenium(II) ligand displays a (*N*,*Se*)-bidentate triconnective coordination pattern, with the selenium atom bridging two neighboring silver atoms, the trifluoroacetato group has a *O*,*O*-bidentate, biconnective, bridging coordination behaviour, thus resulting in a distorted trigonal bipyramidal environment around each silver atom, with O(1) and Se(1) in apical positions [O(1)–Ag(1)–Se(1) 158.58(6)°]. The Ag–O distances (O1–Ag1 2.6225(5) and O2–Ag2 2.5333(2) Å; cf.

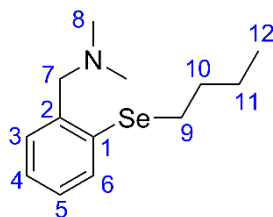
$\Sigma_{\text{vdw}}(\text{O}, \text{Ag})$ 3.10 Å¹⁸) are in the range found for other complexes with coordinated trifluoroacetato ligands.^{22–23}

In the crystal of **2** each chain is further interconnected with two other neighbouring chains, by very weak H \cdots F interactions (H4 \cdots F2 2.4927(1) and H24A \cdots F4 2.5364(2) Å, cf. $\Sigma_{\text{vdw}}(\text{H}, \text{F})$ 2.55 Å²⁴), thus resulting in supramolecular 2D layers (Figure 3).

EXPERIMENTAL

General experimental information

The starting materials were commercial reagents (*n*-BuLi, Se powder, AgOTf, HgCl₂) purchased from Sigma Aldrich and used without supplementary purification. All the solvents were dried and distilled under argon prior to use, by following standard techniques. Reactions involving air sensitive compounds were performed under argon atmosphere. Melting points were measured on an Electrothermal 9200 apparatus. HRMS ESI+ mass spectra were recorded using a Thermo Scientific LTQ-OrbitrapXL instrument equipped with a standard ESI/APCI source. Multinuclear NMR spectra were recorded at room temperature in dry CDCl₃, on a BRUKER AVANCE 400 instrument operating at 400.13, 100.61, 376.49 and 76.31 MHz for ¹H, ¹³C, ¹⁹F and ⁷⁷Se, respectively. The ¹H and ¹³C chemical shifts are reported in ppm units relative to the residual peak of the solvent (ref. CHCl₃: ¹H 7.26, ¹³C 77.00 ppm). ¹H and ¹³C resonances were assigned using 2D NMR experiments (COSY, HMQC and HMBC) and are given according to the numbering illustrated in Scheme 2. The NMR data were processed using the MestReNova software.²⁶



Scheme 2

Synthesis of (n-Bu)[2-(Me₂NCH₂)C₆H₄]Se (1)

A solution of *n*-BuLi in hexane (6.02 mL, 1.6 M, 9.62 mmol) was added dropwise to a stirred solution of *N,N*-dimethylbenzylamine (1.3 g, 9.62 mmol) in 30 mL anhydrous *n*-hexane, at r.t., under argon. The reaction mixture was refluxed for 3 h and then allowed to cool to room temperature. Evaporation of the solvent gave the lithium derivative as a colourless compound. The solid was dissolved in 40 mL anhydrous THF, elemental selenium (0.76 g, 9.62 mmol) was added and the reaction mixture was stirred for 4 h at room temperature, under argon. To the resulting yellow solution *n*-butyl bromide (1.30 g, 9.62 mmol) was added and the reaction mixture was stirred at room temperature for 24 h. The solvent was removed in vacuum, dichloromethane was added and LiBr was removed by filtration. Evaporation of the solvent gave the title compound as a yellow oil. Yield 1.7 g, 65 % ¹H NMR, δ (ppm) 7.47 (d, ²J_{HH} = 7.51 Hz, 1H, H-6), 7.28 – 7.30 (m, 1H, H-3), 7.15 – 7.23 (m, 2H, H-4, H-5), 3.50 (s, 2H, H-7), 2.91 (t, ²J_{HH} = 7.56 Hz, 2H, H-9), 2.26 (s, 6H, H-8), 1.74 (qv, ²J_{HH} = 7.4 Hz, 2H, H-10), 1.49 (sext, ²J_{HH} = 7.57 Hz, 2H, H-11), 0.96 (t, ²J_{HH} = 7.3 Hz, 3H, H-12). ¹³C NMR, δ (ppm) 139.86 (C-2), 133.91 (C-1), 130.32 (C-6), 129.79 (C-3), 127.64 (C-5), 125.55 (C-4), 64.40 (C-7), 45.06 (C-8), 31.93 (C-10), 26.44 (C-9), 23.29 (C-11), 13.72 (C-12). ⁷⁷Se NMR, δ (ppm) 249. HRMS (ESI+), m/z (%): 272.09125 (100), [M+H]⁺ calcd. for C₁₃H₂₂NSe: m/z = 272.09120.

Synthesis of [AgOTf{Se(n-Bu)[C₆H₄(CH₂NMe₂)-2}] (2)

AgOTf (0.199g, 0.77 mmol) was added to a solution of 1-(2-(butylselenanyl)phenyl)-*N,N*-dimethylmethanamine (1) (0.210 g, 0.77 mmol) in acetone (10 mL), at room temperature, under stirring for 1 h. From the clear solution the solvent was removed at reduced pressure and the remained yellowish solid was washed with cold Et₂O. Yield: 0.287 g, 70 %. M.p. 108.6°C. ¹H NMR, δ (ppm) 7.57 (d, ²J_{HH} = 7.51 Hz, 1H, H-6), 7.37 – 7.28 (m, 3H, H-3, H-4, H-5), 3.72 (s, 2H, H-7), 3.19 (t, ²J_{HH} = 7.75 Hz, 2H, H-9), 2.59 (s, 6H, H-8), 1.59 (qv, ²J_{HH} = 7.5 Hz, 2H, H-10), 1.35 (sext, ²J_{HH} = 7.54 Hz, 2H, H-11), 0.85 (t, ²J_{HH} = 7.27 Hz, 3H, H-12). ¹³C NMR, δ (ppm) 136.83 (C-1), 133.95 (C-2), 133.60 (C-6), 130.50 (C-3), 129.15 (C-5), 128.66 (C-4), 120.45 (CF₃, ¹J_{HF} = 321 Hz), 65.87 (C-7), 48.54 (C-8), 33.97 (C-10), 31.85 (C-9), 22.75 (C-11), 13.41 (C-12). ⁷⁷Se NMR, δ (ppm) 188. ¹⁹F NMR, δ (ppm) -77. HRMS (ESI+), m/z (%): 377.98897 (100), [M-OTf]⁺ calcd. for C₁₃H₂₂NSeAg: m/z = 377.98847; m/z (%): 270.07590 (60), [(n-Bu)(2-Me₂NCH₂C₆H₄)Se]⁺.

Synthesis of [HgCl₂{Se(n-Bu)[C₆H₄(CH₂NMe₂)-2}] (3)

HgCl₂ (0.2 g, 0.74 mmol) was added to a solution of 1 (0.2 g, 0.74 mmol) and DCM. The mixture was stirred for 30 minutes and the solvent was removed at reduced pressure.

A white solid was obtained after washing the resulting product with hexane (3 x 10 mL). Yield 0.383 g, 96% M.p. 104.6 °C. ¹H NMR, δ (ppm) 7.44 (d, ²J_{HH} = 7.73 Hz, 1H, H-6), 7.4 (t, ²J_{HH} = 7.4 Hz, 1H, H-4), 7.35 (t, ²J_{HH} = 7.37 Hz, 1H, H-5), 7.24 (d, ²J_{HH} = 7.37 Hz, 1H, H-3), 3.77 (s, 2H, H-7), 3.37 (t, ²J_{HH} = 7.6 Hz, 2H, H-9), 2.47 (s, 6H, H-8), 1.88 (qv, ²J_{HH} = 7.5 Hz, 2H, H-10), 1.52 (sext, ²J_{HH} = 7.54 Hz, 2H, H-11), 0.98 (t, ²J_{HH} = 7.4 Hz, 3H, H-12). ¹³C NMR, δ (ppm) 134.54 (C-1), 134.07 (C-3), 131.37 (C-6), 130.51 (C-4), 129.43 (C-2), 128.67 (C-5), 65.03 (C-7), 47.75 (C-8), 31.73 (C-9), 31.40 (C-10), 23.20 (C-11), 13.64 (C-12). ⁷⁷Se NMR, δ (ppm) 217. HRMS (ESI+), m/z (%): 508.02219 (100), [M-Cl]⁺ calcd. for C₁₃H₂₂NSeHgCl: m/z = 508.02285; m/z (%): 270.07584 (30), [(n-Bu)(2-Me₂NCH₂C₆H₄)Se]⁺.

Crystal structure determination

The details of the crystal structure determination and refinement for compound 2 are given in Table 2. The crystal was mounted on MiTeGen microMounts cryoloops and data were collected on a Bruker D8 VENTURE diffractometer using Mo-Kα radiation (λ = 0.71073 Å) from a IμS 3.0 microfocus source with multilayer optics, at low temperature (100 K). The structure was refined with anisotropic thermal parameters for non-H atoms. Hydrogen atoms were placed in fixed, idealized positions and refined with a riding model and a mutual isotropic thermal parameter. For structure solving and refinement the Bruker APEX3 Software Package was used.²⁶ The drawings were created using the Diamond program.²⁸

CONCLUSIONS

Multinuclear NMR spectroscopy and mass spectrometry confirmed the formation of the reported compounds. No evidence for intramolecular N→Se interaction in solution could be outlined from the ¹H NMR spectra, as only one set of sharp singlet resonances were observed in the aliphatic region for the CH₂N and the NCH₃ protons in each case. The single-crystal X-ray diffraction studies revealed the formation of a 1D coordination polymer in case of compound 2, with (Se,*N*)-chelating building blocks, and strongly coordinated *O,O*-bridging trifluoroacetato groups.

SUPPLEMENTARY MATERIAL

CCDC 1962256 contain the supplementary crystallographic data for compound 2. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgments. Financial support from Babeş-Bolyai University (Advanced Fellowship STAR-UBB) is greatly acknowledged.

Table 2

Crystal data and structure refinement for compound **2**

Empirical formula	C ₁₄ H ₂₁ AgF ₃ NO ₃ SSe	
Formula weight	527.21	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P 1	
Unit cell dimensions	a = 8.9964(17) Å	α = 77.789(6)°
	b = 10.121(2) Å	β = 80.750(5)°
	c = 10.570(2) Å	γ = 84.577(6)°
Volume	926.6(4) Å ³	
Z	2	
Density (calculated)	1.890 Mg/m ³	
Absorption coefficient	3.205 mm ⁻¹	
F(000)	520	
Crystal size	0.171 x 0.100 x 0.028 mm ³	
Theta range for data collection	2.298 to 28.301°	
Index ranges	-11 ≤ h ≤ 11, -13 ≤ k ≤ 13, -14 ≤ l ≤ 14	
Reflections collected	43573	
Independent reflections	9163 [R(int) = 0.0334]	
Completeness to theta = 25.242°	99.9 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	9163 / 3 / 1658	
Goodness-of-fit on F ²	3.476	
Final R indices [I > 2σ(I)]	R1 = 0.0317, wR2 = 0.1092	
R indices (all data)	R1 = 0.0347, wR2 = 0.1095	
Absolute structure parameter	0.024(3)	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.692 and -0.929 e.Å ⁻³	

REFERENCES

1. A. J. Mukherjee, S. S. Zade, H. B. Singh and R. B. Sunoj, *Chem. Rev.*, **2010**, *110*, 4357.
2. M. Tiecco, L. Testaferri, C. Santi, C. Tomassini, F. Marini, L. Bagnoli and A. Temperini, *Tetrahedron Asymmetry*, **2000**, *11*, 4645.
3. Y. Nishibayashi and S. Uemura, *Top. Curr. Chem.*, **2000**, *208*, 201.
4. Y. Miyake, M. Oda, A. Oyamada, H. Takada, K. Ohe and S. Uemura, *J. Organomet. Chem.*, **2000**, *611*, 475.
5. L. Uehlin, G. Fragale and T. Wirth, *Chem. Eur. J.*, **2002**, *8*, 1125.
6. B. Mishra, K. I. Priyadarsini and H. Mohan, G. Mugesh, *Bioinorg. Med. Chem. Lett.*, **2006**, *16*, 5334.
7. K. P. Bhabak and G. Mugesh, *Chem. Eur. J.*, **2009**, *15*, 9846.
8. G. Mugesh and H. B. Singh, *Chem. Soc. Rev.*, **2000**, *29*, 347.
9. K. P. Bhabak and G. Mugesh, *Acc. Chem. Res.*, **2010**, *43*, 1408.
10. A. Kumar, G. K. Rao, F. Saleem and A. K. Singh, *Dalton Trans.*, **2012**, *41*, 11949.
11. A. Pop, R. Mitea and A. Silvestru, *J. Organomet. Chem.*, **2014**, *768*, 121.
12. A. Pop, D. Rosca, R. Mitea and A. Silvestru, *Inorg. Chim. Acta*, **2013**, *405*, 235.
13. M. Iwaoka and S. Tomoda, *J. Am. Chem. Soc.*, **1996**, *118*, 8077.
14. E. Fragoso, R. Azpiroz, P. Sharma, G. Espinosa-Perez, F. Lara-Ochoa, A. Toscano, R. Gutierrez and O. Portillo, *J. Mol. Struct.*, **2018**, *1155*, 711.
15. T. C. Bourland, R. G. Carter and A. F. T. Yokochi, *Org. Biomol. Chem.*, **2004**, *2*, 1315.
16. K. Fujita, M. Kanakubo, H. Ushijima, A. Oishi, Y. Ikeda and Y. Taguchi, *SYNLETT.*, **1998**, 987.
17. S. Kumar, H. B. Singh and G. Wolmershauser, *Organometallics*, **2006**, *25*, 382.
18. J. Emsley, *Die Elemente*, Walter de Gruyter, Berlin, **1994**.
19. S. Mishra, D. Du, E. Jeanneau, F. Dappozze, C. Guillard, J. Zhang and S. Daniele, *Chem. Asian J.*, **2016**, *11*, 1658.
20. J. R. Black, N. R. Champness, W. Levason and G. Reid, *Inorg. Chem.*, **1996**, *35*, 1820.
21. M. Leschke, G. Rheinwald, and H. Lang, *Z. Anorg. Allg. Chem.*, **2002**, *628*, 2470.
22. M. Wen, M. Munakata, Y.-Z. Li, Y. Suenaga, T. Kuroda-Sowa, M. Maekawa and M. Anahata, *Polyhedron*, **2007**, *26*, 2455.
23. C. Rim, H. Zhang and D. Y. Son, *Inorg. Chem.*, **2008**, *47*, 11993.
24. J. D. Dunitz and R. Taylor, *Chem. Eur. J.*, **1997**, *3*, 89.
25. MestReC and MestReNova, Mestrelab Research S.L., A Coruña 15706, Santiago de Compostela.
26. G. M. Sheldrick, *Acta Crystallogr., Sect. C: Struct. Chem.*, **2015**, *C71*, 3.
27. DIAMOND – Visual Crystal Structure Information System, CRYSTAL IMPACT, Postfach 1251, 53002 Bonn, Germany, 2001.