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**Aspects of the Coordination Chemistry of 1,1'-
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Aspects of the Coordination Chemistry of 1,1'-Di(benzothiazol-2-ylthio)ferrocene

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Abstract. 1,1'-Di(benzothiazol-2-ylthio)ferrocene (**1**) acts as a bridging bidentate N,N-ligand and affords coordination polymers with a range of Cu^I, Ag^I, Cu^{II}, Zn^{II} and Hg^{II} salts. The crystal structures of [Ag(CF₃SO₃)(μ-**1**)]_n, [CuCl₂(μ-**1**)]_n, [ZnCl₂(μ-**1**)]_n and [HgBr₂(μ-**1**)]_n have been determined. The silver atom exhibits a weak contact to a cyclopentadienyl π-system in addition to its trigonal-planar coordination. The copper atom is in a square-planar coordination environment, while the coordination of zinc and mercury is best described as distorted tetrahedral in the corresponding complexes.

Keywords: Coordination polymers; Copper; Mercury; Silver; Zinc

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Introduction

We recently reported the synthesis and crystal structure of 1,1'-di(benzothiazol-2-ylthio)ferrocene (**1**) [1]. This compound is a symmetrically disubstituted thioether derivative of ferrocene with pendant heterocyclic groups. It contains six potentially ligating heteroatoms, viz. two 'hard' nitrogen and four 'soft' sulfur atoms, two of which are directly connected to the ferrocene nucleus. Chelate complexes of 1,1'-dithioether-functionalised ferrocene derivatives which contain additional sulfur or nitrogen donor atoms in the periphery have been described for a wide range of 'soft' mono- and divalent metal centres, viz. Rh^{I} , Cu^{I} , Ag^{I} , Au^{I} , Pd^{II} , Pt^{II} , Cu^{II} and Hg^{II} [2]. Benzothiazole coordinates almost exclusively through its nitrogen atom. Reports concerning S-coordination are scarce [3], and no structurally characterised example is known. We have been interested in studying the binding profile of **1** towards a range of transition metal ions in order to probe the suitability of **1** as a molecular electrochemical sensor with potential selectivity for 'soft' metal ions. If in our case coordination occurs through the two thioether-type sulfur atoms, which are prone to bind preferentially to 'soft' metal centres, the effect on the redox potential of the ferrocene moiety is expected to be much larger than in the case of exclusive coordination through the nitrogen atoms of the pendant substituents. We note that one of the most efficient molecular sensors with pronounced Hg^{II} selectivity contains two thioether units for metal coordination [4].

Results and Discussion

Ligand **1** was allowed to react in dichloromethane solution in a 1:1 ratio with CuCl_2 , ZnCl_2 , HgCl_2 , HgBr_2 and also with $[\text{Cu}(\text{CH}_3\text{CN})_4][\text{PF}_6]$, $\text{Ag}(\text{CF}_3\text{SO}_3)$ and AuCl . The

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6 reactions cleanly afforded coordination compounds in all cases but two. Coordination was
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8 affected by redox processes in the case of AuCl, where formation of a gold mirror was
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10 observed, and HgCl₂, where an intractable mixture was obtained whose dark colour
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12 indicated generation of elemental mercury. The metal complexes obtained turned out to be
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14 essentially insoluble in dichloromethane and other common organic solvents, suggesting
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16 that coordination polymers, instead of mononuclear chelates, had formed. Unfortunately,
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18 this behaviour prevents the use of **1** as a molecular electrochemical sensor in solution.
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22 Structural characterisation by single crystal X-ray diffraction analysis was possible
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24 for [CuCl₂(**1**)] (Fig. 1), [ZnCl₂(**1**)] (Fig. 2), [HgBr₂(**1**)] (Fig. 3), and [Ag(CF₃SO₃)(**1**)] (Fig.
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26 4). Suitable crystals were obtained by a diffusion technique, which involved layering of a
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28 solution of **1** with pure solvent and subsequently with a solution of the corresponding metal
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30 salt. All four complexes are coordination polymers. **1** exclusively acts as a bridging
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32 bidentate N,N-ligand. S-coordination is not observed. Possibly, thioether S-coordination
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34 occurs in the case of AuCl and HgCl₂, but triggers intramolecular redox processes between
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36 the ferrocene nucleus and the metal centre coordinated in very close proximity.
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38 Uncoordinated **1** exhibits eclipsed cyclopentadienyl rings [1], while a staggered
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40 conformation with diametrically opposed substituents is observed for all four
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42 crystallographically characterised complexes. Metal coordination has no pronounced effect
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44 on intraligand bond lengths and angles.
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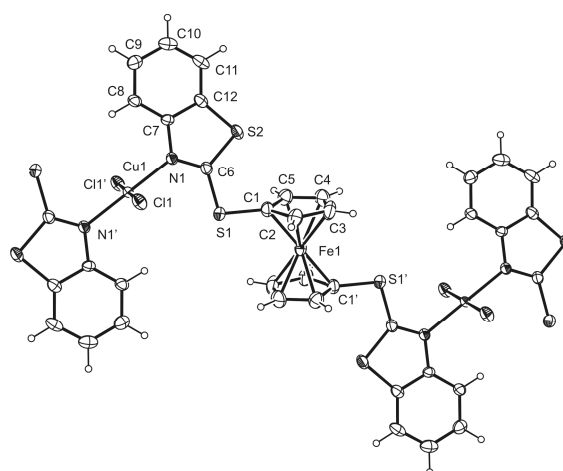


Fig.1 Short section of a polymeric chain of $[\text{CuCl}_2(\mu\text{-1})]_n$ in the crystal. Thermal displacement ellipsoids are drawn at the 30% probability level. Selected bond lengths/Å and angles/°: Cu(1)–Cl(1) 2.2423(16), Cu(1)–N(1) 2.012(4), N(1)–C(6) 1.318(6), N(1)–C(7) 1.408(5), S(1)–C(1) 1.767(5), S(1)–C(6) 1.744(5), S(2)–C(6) 1.750(5), S(2)–C(12) 1.742(5); Cl(1)–Cu(1)–N(1) 91.12(12).

The Cu atom of $[\text{CuCl}_2(\mu\text{-1})]_n$ is in a square-planar coordination environment. The ligands are in a *trans* arrangement. Bond lengths and angles are very similar to those reported for the closely related *trans*-dichlorobis(3,5-dimethylthiazole)copper(II) [5] and *trans*-dichlorodi(2-aminobenzothiazole)copper(II) [6], while the dichlorocopper(II) complex of $(\text{bzt-2})_2\text{-}o\text{-C}_6\text{H}_4$ (bzt-2 = benzothiazol-2-yl) is a *cis*-chelate which exhibits a notably distorted square-planar coordination [7]. We note that Cu^{II} complexes which contain imidazole or related heterocyclic N-ligands together with additional thioether ligands are currently attracting increased attention due to their relevance to the catalytic centre of peptidylglycine α -hydroxylating monooxygenase (PHMcc) and related oxygenases with an essentially mononuclear catalytic core [8].

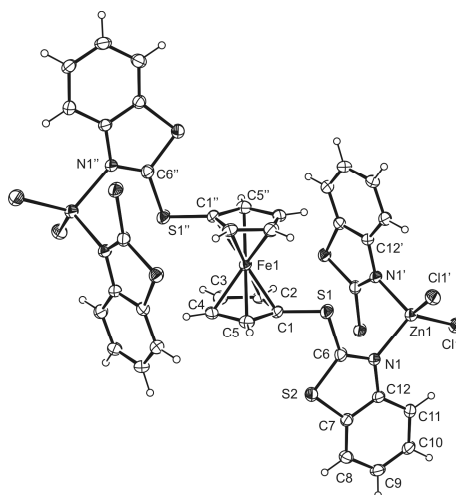


Fig. 2 Short section of a polymeric chain of $[\text{ZnCl}_2(\mu\text{-1})]_n$ in the crystal. Thermal displacement ellipsoids are drawn at the 30% probability level. Selected bond lengths/Å and angles/°: Zn(1)–Cl(1) 2.2343(13), Zn(1)–N(1) 2.106(3), N(1)–C(6) 1.318(6), N(1)–C(12) 1.414(5), S(1)–C(1) 1.771(4), S(1)–C(6) 1.755(4); N(1)–Zn(1)–Cl(1) 116.62(10), N(1)–Zn(1)–Cl(1') 103.72(11).

The Zn atom of $[\text{ZnCl}_2(\mu\text{-1})]_n$ is in a distorted tetrahedral coordination environment. The N–Zn–N and Cl–Zn–Cl angles are $101.8(2)^\circ$ and $114.19(7)^\circ$, respectively. The corresponding angles are ca. 103.3° and 116.8° for the closely related $[\text{ZnCl}_2(\text{bzt-7-NH}_2)]$ (bzt-7 = benzothiazol-7-yl) [9] and ca. 116.3° and 112.5° for the bulkier thio-functionalised $[\text{ZnCl}_2\{\text{bzt-2-N}=\text{C}(\text{SMe})_2\}]$ [10].

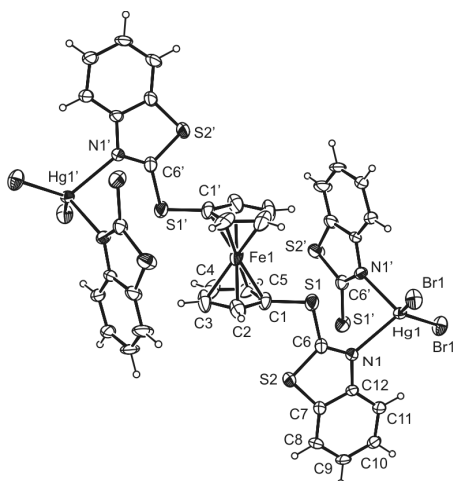


Fig. 3 Short section of a polymeric chain of $[\text{HgBr}_2(\mu\text{-1})]_n$ in the crystal. Thermal displacement ellipsoids are drawn at the 30% probability level. Selected bond lengths/Å and angles/°: Hg(1)–N(1) 2.409(5), Hg(1)–Br(1) 2.4973(8), N(1)–C(6) 1.301(8), N(1)–C(12) 1.427(7), S(1)–C(1) 1.769(6), S(1)–C(6) 1.762(7); N(1)–Hg(1)–Br(1) 98.84(13), N(1)–Hg(1)–Br(1') 116.09(12).

The Hg atom of $[\text{HgBr}_2(\mu\text{-1})]_n$ is in a very distorted tetrahedral coordination environment. The N–Hg–N and Br–Hg–Br angles are $90.1(2)^\circ$ and $130.32(4)^\circ$, respectively, which is similar to the corresponding values of ca. 89.9° and 127.4° reported for the closely related $[\text{HgI}_2(\text{bzt-2-CH=CHPh})]$ [11].

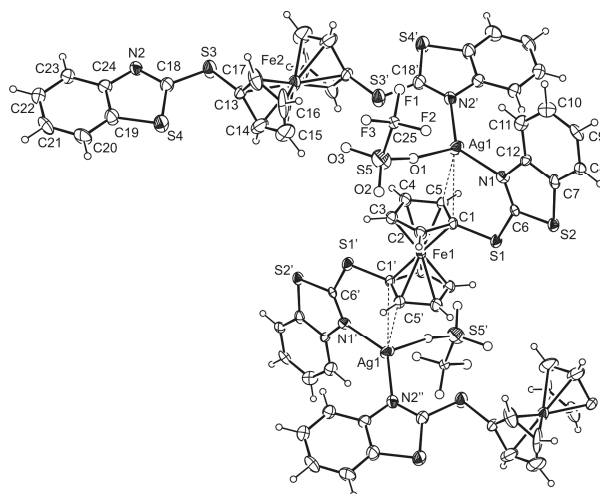


Fig. 4 Short section of a polymeric chain of $[\text{Ag}(\text{CF}_3\text{SO}_3)(\mu\text{-1})]_n$ in the crystal. Thermal displacement ellipsoids are drawn at the 30% probability level. O and F atoms are shown as circles of arbitrary size for clarity. Selected bond lengths/ \AA : Ag(1)–N(1) 2.215(7), Ag(1)–N(2') 2.177(8), Ag(1)–O(1) 2.354(8), N(1)–C(6) 1.325(9), N(1)–C(12) 1.427(10), S(1)–C(1) 1.768(8), S(1)–C(6) 1.757(8).

The Ag atom of $[\text{Ag}(\text{CF}_3\text{SO}_3)(\mu\text{-1})]_n$ is surrounded by two benzothiazole nitrogen atoms and one triflate oxygen atom in a distorted trigonal-planar arrangement. The N–Ag–N angle has a value of $135.5(3)^\circ$, while the two N–Ag–O angles are $117.7(4)^\circ$ and $96.3(4)^\circ$. The sum of angles is ca. 349.5° , which deviates considerably from the ideal value of 360° and is indicative of a weak additional interaction. Indeed, the Ag atom forms a contact with one of the cyclopentadienyl ring. It is positioned almost perpendicularly with respect to one of the edges of this ring. The corresponding Ag–C distances of ca. 2.88 and 2.94 \AA (indicated by dotted lines in Fig. 4) are well below the sum of the estimated *van der Waals* radii of ca. 3.4 \AA . A similar interaction between a ferrocene-type π -system and an Ag^{I} centre has been reported by Laguna and coworkers for $[\text{Ag}\{\text{fc}(\text{S}_2\text{CNEt}_2)_2\}]_n[\text{ClO}_4]_n$ (fc

= 1,1'-ferrocenediyl), where the corresponding Ag–C distances are close to 3.0 Å [12].

Coordinative interactions between Ag^I and aromatic π-systems have been firmly established [13] ever since the seminal crystal structure determination of the benzene complex [Ag(μ-η²: η²-C₆H₆)_n][ClO₄]_n [14].

Crystals of the product obtained from the reaction of **1** with Cu[PF₆] proved to be unsuitable for a single-crystal X-ray structure analysis. The insolubility of this compound clearly indicates the polymeric nature of this complex, which therefore can be formulated as [Cu(μ-**1**)_n][PF₆]_n. However, details of the Cu^I coordination environment remain unclear.

Experimental Part

1,1'-Di(benzothiazol-2-ylthio)ferrocene (**1**) was prepared as described previously [1]. All other materials were procured from standard commercial sources and used as received. Elemental analyses were performed by the microanalytical laboratories of the University of Kassel and the University of Halle.

General procedure for the preparation of the metal complexes

In the case of [Cu(CH₃CN)₄][PF₆] and Ag(CF₃SO₃) as starting material a solution of the metal salt (0.10 mmol) in dichloromethane (5 mL) was added rapidly to a stirred solution of **1** (52 mg, 0.10 mmol) in dichloromethane (5 mL). In the case CuCl₂, ZnCl₂ and HgBr₂ as starting material a solution of the metal salt in acetone (5 mL) was used. The reaction mixture was allowed to stand at room temperature in the dark for 14 h. The precipitate was isolated by filtration, washed with dichloromethane (2 × 0.5 mL) and dried in vacuo.

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6 [Cu(μ -**1**)]_n[PF₆]_n: yellow, microcrystalline solid (64 mg, 88%). Elemental analysis:

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8 C₂₄H₁₆N₂S₄CuF₆FeP (725.02): C 38.81 (calcd. 39.76), H 2.55 (2.24), N 3.83 (3.86), S
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10 17.11 (17.69).
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15 [Ag(CF₃SO₃)(μ -**1**)]_n: yellow, microcrystalline solid (66 mg, 85%). Elemental analysis:

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17 C₂₅H₁₆N₂S₅AgF₃FeO₃ (773.42): C 38.70 (calcd. 38.82), H 2.54 (2.09), N 3.77 (3.62), S
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19 17.01 (16.58).
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25 [CuCl₂(μ -**1**)]_n: brown, microcrystalline solid (54 mg, 83%). Elemental analysis:

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27 C₂₄H₁₆N₂S₄Cl₂CuFe (650.97): C 43.56 (calcd. 44.28), H 2.54 (2.48), N 4.24 (4.30), S 18.85
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29 (19.70).
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35 [ZnCl₂(μ -**1**)]_n: yellow, microcrystalline solid (56 mg, 86%). Elemental analysis:

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37 C₂₄H₁₆N₂S₄Cl₂FeZn (652.75): C 43.65 (calcd. 44.16), H 2.94 (2.47), N 4.30 (4.29), S 19.35
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39 (19.65).
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44 [HgBr₂(μ -**1**)]_n: dark yellow, microcrystalline solid (74 mg, 84%). Elemental analysis:

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46 C₂₄H₁₆N₂S₄Br₂FeHg (876.89): C 33.44 (calcd. 32.87), H 2.35 (1.84), N 3.52 (3.19), S
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48 14.66 (14.63).
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52 53 *X-ray crystallography*

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55 Single crystals were obtained by a diffusion technique which involved layering of a
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57 dichloromethane solution of **1** with pure dichloromethane and subsequently with a solution
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6 of the corresponding metal salt in acetone or dichloromethane. For each data collection a
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8 single crystal was mounted on a glass fibre and all geometric and intensity data were taken
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10 from this sample. Data collection using MoK α radiation ($\lambda = 0.71073 \text{ \AA}$) was made on a
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12 Stoe IPDS2 diffractometer equipped with a 2-circle goniometer and an area detector.
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14 Absorption correction was done by integration using X-red [15]. The data sets were
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16 corrected for Lorentz and polarisation effects. The structures were solved by direct
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18 methods (SHELXS97) and refined using alternating cycles of least squares refinements
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20 against F^2 (SHELXL97) [16]. All non H atoms were found in difference Fourier maps and
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22 were refined with anisotropic displacement parameters, except C(25) in the silver complex
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24 [Ag(CF₃SO₃)(μ -1)] which was refined isotropically. H atoms were placed in constrained
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26 positions according to the riding model with the 1.2 fold isotropic displacement
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28 parameters. Pertinent crystallographic data are collected in Table 1. Graphical
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30 representations were made using ORTEP-3 win [17].
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39 Supplementary Material. Crystallographic data (excluding structure factors) for the
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41 structures reported in this paper have been deposited with the Cambridge Crystallographic
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43 Data Centre as supplemental publication CCDC-713654 ([CuCl₂(1)]·CH₂Cl₂), CCDC-
44
45 713655 ([ZnCl₂(1)]), CCDC-713656 ([HgBr₂(1)]) and CCDC-713657·([Ag(CF₃SO₃)(1)]).
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48 Copies of the data can be obtained free of charge on application to The Director, CCDC,
49
50 12 Union Road, Cambridge CB2 1 EZ, UK (Fax: int.code+(1223)336-033; e-mail for
51
52 enquiry: fileserv@ccdc.cam.ac.uk).
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Table 1 Crystal data and structure refinement for the coordination polymers [CuCl₂(**1**)], [ZnCl₂(**1**)], [HgBr₂(**1**)] and [Ag(CF₃SO₃)(**1**)]

Compound	[CuCl ₂ (1)]·CH ₂ Cl ₂	[ZnCl ₂ (1)]	[HgBr ₂ (1)]	[Ag(CF ₃ SO ₃)(1)]
Empirical formula	C ₂₅ H ₁₈ Cl ₄ CuFeN ₂ S ₄	C ₂₄ H ₁₆ Cl ₂ FeN ₂ S ₄ Zn	C ₂₄ H ₁₆ Br ₂ FeHgN ₂ S ₄	C ₂₅ H ₁₆ AgF ₃ FeN ₂ O ₃ S ₅
Formula weight	735.89	652.75	876.89	773.42
Temperature / K	153(2)	153(2)	173(2)	213(2)
Crystal system	monoclinic	monoclinic	monoclinic	triclinic
Space group	<i>C2/c</i>	<i>C2/c</i>	<i>C2/c</i>	<i>P-1</i>
Lattice parameters length / Å	<i>a</i> = 24.871(9) <i>b</i> = 9.205(5) <i>c</i> = 16.612(4)	<i>a</i> = 14.271(2) <i>b</i> = 12.9492(14) <i>c</i> = 14.910(2)	<i>a</i> = 15.4795(15) <i>b</i> = 12.3068(12) <i>c</i> = 15.3354(15)	<i>a</i> = 8.9260(18) <i>b</i> = 10.208(2) <i>c</i> = 15.643(3)
angle / °	β = 130.469(17)	β = 116.271(11)	β = 118.252(7)	α = 100.485(15) β = 98.867(15) γ = 95.070(16)
Cell volume / Å ³	2893(2)	2470.9(6)	2573.4(5)	1374.9(5)
<i>Z</i>	4	4	4	2
<i>d</i> _{calc.} / g cm ⁻³	1.689	1.755	2.263	1.868
μ / mm ⁻¹	1.915	2.130	9.970	1.673
<i>F</i> (000)	1476	1312	1656	768
θ range / °	2.15 to 25.0	2.24 to 25.0	2.23 to 25.18	1.34 to 25.0
Index ranges	-29 → <i>h</i> → 29 -10 → <i>k</i> → 10 -19 → <i>l</i> → 19	-16 → <i>h</i> → 16 -11 → <i>k</i> → 15 -17 → <i>l</i> → 17	-18 → <i>h</i> → 18 -14 → <i>k</i> → 14 -18 → <i>l</i> → 18	-10 → <i>h</i> → 10 -12 → <i>k</i> → 12 -18 → <i>l</i> → 17
Refls. collected	9083	6474	8238	8998
Independent refls.	2542 [<i>R</i> _{int} = 0.0915]	2177 [<i>R</i> _{int} = 0.0874]	2250 [<i>R</i> _{int} = 0.0695]	4568 [<i>R</i> _{int} = 0.0879]
Refls. observed	1348	1627	1934	1686
<i>T</i> _{min} / <i>T</i> _{max}	0.7899 and 0.9090	0.7866 and 0.8586	0.2135 and 0.2977	0.8340 and 0.9682
Data / restraints / parameters	2542 / 0 / 171	2177 / 0 / 156	2250 / 0 / 156	4568 / 3 / 359
GOF on <i>F</i> ²	0.723	0.943	1.035	0.647
Final <i>R</i> indices	<i>R</i> 1 = 0.0391	<i>R</i> 1 = 0.0434	<i>R</i> 1 = 0.0328	<i>R</i> 1 = 0.0501
[<i>I</i> > 2σ(<i>I</i>)]	<i>wR</i> 2 = 0.0657	<i>wR</i> 2 = 0.1074	<i>wR</i> 2 = 0.0767	<i>wR</i> 2 = 0.0820
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0391 <i>wR</i> 2 = 0.0961	<i>R</i> 1 = 0.0594 <i>wR</i> 2 = 0.1134	<i>R</i> 1 = 0.0393 <i>wR</i> 2 = 0.0787	<i>R</i> 1 = 0.1395 <i>wR</i> 2 = 0.0951
Largest diff. peak and hole / eÅ ⁻³	0.356 and -0.458	0.555 and -0.649	0.731 and -1.454	1.643 and -0.553

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