

In blessed memory of Professor A.A. Pasyanskii

Unusual Formation of the Paramagnetic Complex $(\eta^4\text{-C}_4\text{Me}_4)\text{CoI}_2(\text{PhTeI})$ and Specific Features of Its Electronic, Molecular, and Crystal Structures

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Abstract—The 18-electron $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ compound noticeably decomposes upon prolonged storage and also unusually transforms into the 17-electron $(\eta^4\text{-C}_4\text{Me}_4)\text{CoI}_2(\text{PhTeI})$ complex (**I**). The X-ray diffraction study of the molecular and crystal structures of complex **I** (CIF file CCDC no. 1991837) reveals interesting analogies with the molecular structures of the known metal complexes containing organotellurium halide ligands and with the crystal packing of the related one-component crystals of $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$ and two-component crystals of $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}\cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$. An analysis of the crystal packing of molecules of complex **I** elucidates a new supramolecular synthone $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ determining the structural motifs of self-assembly in complex **I** and related compounds $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$, $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}\cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$, and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$. The EPR study of compound **I** shows that cobalt exists in the low-spin state with the total spin $S = 1/2$.

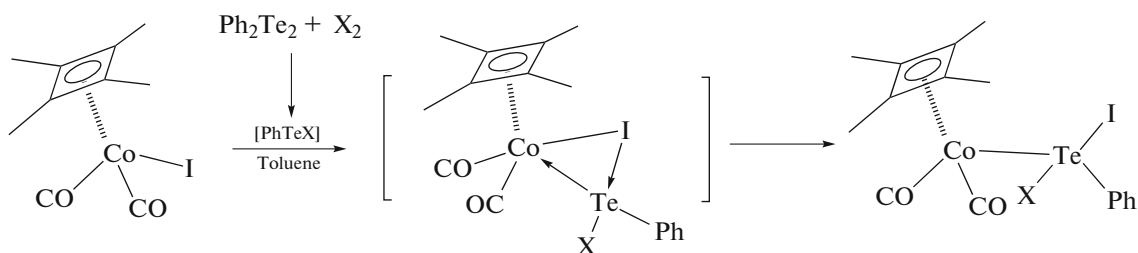
Keywords: cobalt, cyclobutadiene, metal complexes, paramagnetism, tellurium halides, EPR, X-ray diffraction analysis, halogen bond, chalcogen bond, supramolecular synthone, self-assembly

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INTRODUCTION

Catalytic and magnetic activities of the cobalt complexes evoke increasing interest in them. In particular, $\text{CpCo}(\text{CO})_2$ is successfully used as a catalyst for the trimerization of acetylenes, and $[\text{CpCo}(\text{CO})\text{I}_2]$ is applied in the functionalization of C–H bonds [1] and in C–C bond coupling [2]. The cyclobutadiene complexes $[(\text{C}_4\text{Me}_4)\text{Co}(\text{C}_6\text{H}_6)]\text{PF}_6$, $(\text{C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$, and $[(\text{C}_4\text{Me}_4)\text{Co}(\text{Bipy})(\text{MeCN})]\text{PF}_6$ catalyze the oxidation of alkanes and alcohols [3]. In 2012, we gener-

alized the results of the structural and synthetic studies of organotellurium halides as a new type of ligands in the transition metal complexes [4]. The diiodotellurophenyl cyclobutadiene-cobalt-dicarbonyl complex $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ and its heterohalide analogue $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeIBrPh}$ formed upon the formal insertion of PhTeX ($X = \text{Br}, \text{I}$) at the Co–I bond in $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$ [5] (Scheme 1) were among the most interesting representatives of this class of compounds.



Scheme 1.

After nearly 10 years, when studying the sandwich [6] and half-sandwich metal complexes with organotellurium and halide ligands in the design of crystals [7], we recrystallized a sample of $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$, which was stored at room temperature in a mechanically damaged ampule within the whole period of 10 years, and isolated the new paramagnetic complex $(\eta^4\text{-C}_4\text{Me}_4)\text{CoI}_2(\text{PhTeI})$ (**I**) for which the laboratory synthesis procedure was developed and a series of physicochemical studies was carried out.

EXPERIMENTAL

Commercial Ph_2Te_2 was recrystallized prior to use, and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ was synthesized using a described procedure [5]. Organic solvents were dehydrated using standard procedures and distilled under an argon atmosphere.

Synthesis of $(\eta^4\text{-C}_4\text{Me}_4)\text{CoI}_2(\text{PhTeI})$ (I**).** Crystalline iodine (0.026 g, 0.1 mmol) was added by small portions to a vigorously magnetically stirred orange solution of Ph_2Te_2 (0.041 g, 0.1 mmol) in toluene (7 mL) at room temperature. The resulting dark blue mixture was additionally stirred for 20 min, and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$ (0.70 g, 0.2 mmol) was added to the mixture at on go. The obtained solution was vigorously stirred at 40°C for 12 h with a reflux condenser or was refluxed for 2 h with an effective reflex condenser open to atmosphere. The reaction course was monitored by the IR spectra. After the CO peaks of the initial $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$ disappeared, the reaction mixture was cooled to room temperature, the solvent was removed in a vacuum of a water-jet pump, and the residue was extracted with CH_2Cl_2 (10 mL). The filtered off extract was diluted with heptane (2 mL), concentrated in a vacuum of a water-jet pump to slight turbidity, and kept at -10°C for several days. The formed dark red prismatic crystals were filtered off, washed with hexane, and dried in vacuo. The yield was 0.07 g (46%).

According to the X-ray diffraction data, the crystals of complex **I** were identical to those obtained by the recrystallization of the $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$

sample, which was stored at room temperature in a damaged ampule for 10 years.

IR spectra were recorded on a Bruker Alpha FTIR spectrophotometer equipped with an ATR accessory (for solid samples) and in 0.1-mm CaF_2 cells (for solutions). EPR spectra were recorded on a Bruker ELEXSYS E-680X instrument in the X-range at 100 and 293 K.

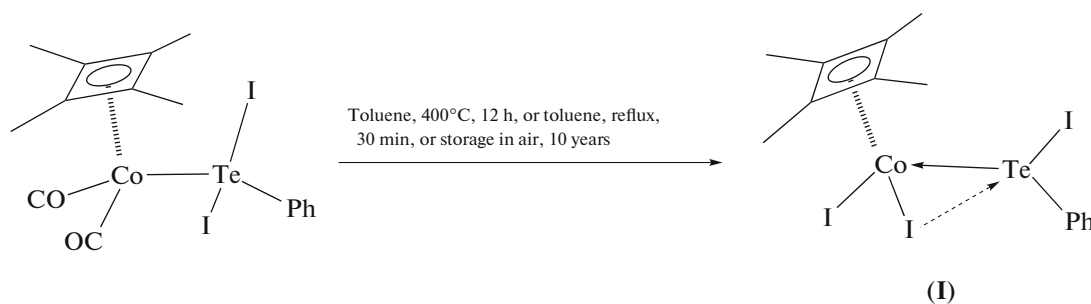
X-ray diffraction analysis (XRD) was carried out on a Bruker APEXII CCD diffractometer. An absorption correction was applied by multiple measurements of equivalent reflections using the SADABS program. The structure was determined by a direct method and refined by least squares for F^2 in the anisotropic approximation of non-hydrogen atoms using the SHELXTL and Olex2 program packages [8, 9]. The positions of hydrogen atoms were calculated geometrically. Selected crystallographic data and structure refinement parameters for complex **I** are presented in Table 1. Selected bond lengths and bond angles in the structure of compound **I** are given in the captions to Figs. 2–4.

The atomic coordinates and other structural parameters for complex **I** were deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC no. 1991837; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

Quantum-chemical calculations. The energy structures in the crystals of complex **I**, $(\eta^4\text{-C}_4\text{Me}_4)\text{-Co}(\text{CO})_2\text{I}$, $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}\cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$, and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ were constructed by the data of intermolecular energies calculated in CrystalExplorer 17.5 (TONTO, B3LYP-DGDZVP) [10] for all unique molecular pairs in the first coordination sphere of the molecule using the experimental geometry of the crystal.

RESULTS AND DISCUSSION

The routine XRD of the single crystals obtained by recrystallization showed that, within 10 years of storage under nonhermetic conditions, the initial 18-electron complex $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ decomposed noticeably and also unusually transformed into 17-electron $(\eta^4\text{-C}_4\text{Me}_4)\text{CoI}_2(\text{PhTeI})$ (**I**) (Scheme 2).



Scheme 2.

Table 1. Selected crystallographic data and structure refinement parameters for complex **I**

Parameter	Value
Empirical formula	C ₁₄ H ₁₇ I ₃ CoTe
<i>FW</i>	752.51
Radiation (λ , Å)	MoK α (0.71073)
<i>T</i> , K	150
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>
<i>Z</i>	4
<i>a</i> , Å	17.9930(13)
<i>b</i> , Å	7.1900(5)
<i>c</i> , Å	14.8942(10)
β , deg	93.8320(10)
<i>V</i> , Å ³	3965.85(13)
ρ_{calc} , g/cm ³	2.600
μ , mm ⁻¹	7.185
<i>F</i> (000)	1356.0
Range of data collection over θ , deg	5.482–57.998
Number of measured reflections	13590
Number of independent reflections	4947
<i>R</i> _{int} , <i>R</i> _{sigma}	0.0264, 0.0340
Number of reflections with <i>I</i> > 2 σ (<i>I</i>)	5420
Number of refined parameters	173
<i>R</i> ₁ , <i>wR</i> ₂ (<i>I</i> > 2 σ (<i>I</i>))	0.0376, 0.0939
<i>R</i> ₁ , <i>wR</i> ₂ (for all data)	0.0441, 0.0976
GOOF	1.053
Residual electron density (max/min), e/Å ³	2.92/–1.46

As can be seen from Scheme 2, the coordination of the PhTeI ligand in complex **I** resembles the assumed structure of the transient complex in the insertion of PhTeI at the Co–I bond in (η^4 -C₄Me₄)-Co(CO)₂TeI₂Ph (Scheme 1), which can be considered as an indirect proof for the possibility of this transition state to exist.

In order to prepare complex **I** in amounts sufficient for studying its physicochemical properties, we elucidated that the complex can purposefully be obtained from a solution of (η^4 -C₄Me₄)-Co(CO)₂TeI₂Ph in toluene with reflux (without isolation from the outer atmosphere) for 30 min or with vigorous stirring under the same conditions at 40°C for 12 h.

The molecular structure of monomeric complex **I** can be presented as the tetramethyl-cyclobutadiene-cobalt fragment coordinating two one-electron iodides and one two-electron PhTeI ligands stabilized due to the hypervalent binding of Te \cdots I with one of the

terminal iodide ligands with the short intramolecular contacts Te \cdots I (3.171 Å, Fig. 1). The counting of electrons according to this description represents compound **I** as a 17-electron complex.

Compared to the initial compound (η^4 -C₄Me₄)-Co(CO)₂TeI₂Ph, in complex **I** the Co–Te distance slightly elongates (from 2.547 to 2.569 Å, respectively) and the Te–I distance noticeably shortens (from ~2.95 to 2.777 Å, respectively). The crystal structure of complex **I** is characterized by the absence of appreciable intermolecular Te \cdots I chalcogen bonds. The intermolecular chalcogen bonds characteristic of the crystals of uncoordinated organotellurium halides are strongly weakened upon their coordination with transition metals. This occurs due to the competition with the intramolecular halogen binding Te \cdots X (X = Br, I) and a general decrease in the electrophilicity of tellurium upon the coordination with the metal enhanced by the backward donation M \rightarrow Te) [4].

Interestingly, there are common motifs in the crystal packings of the molecules of complex **I** and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$. The crystal structures of the latter and its cocrystal 1,4- $\text{C}_6\text{H}_4\text{I}_2$ contain columns of the molecules associated due to the intermolecular interactions $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ [5] (Figs. 2b, 2c). Similar columns are formed due to the $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ and $\text{Te}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ in the crystal of complex **I** (Fig. 2a), whereas in the crystal of $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ they stabilize the zigzag chain (Fig. 2d), which allows one to speak about the supramolecular synthon $[\text{I}\cdots(\eta\text{-}\pi\text{-ligand})\text{-M}]$ determining the long-range order of the packing [11].

When analyzing the crystal packing, it is fruitful to remind the remarks by C.B. Aakeroy (editor of *CrystEngComm* from 2010 to 2019) about a subjective nature of the visible but not confirmed by calculations motifs of the packing [12, 13]. Another member of the editorial board of *CrystEngComm* A. Gavezzetti (from 2005 to 2008) was more categorical and insisted on that “no sensible journal should any more accept sentences like ‘the crystal structure consists of pairs/chains/layers held together by such and such interactions...’ and similar assertions, when they are not supported by reliable energy numbers” [14]. Since the appearance of the Crystal Explorer program package [15] equipped with the Energy Framework function (energy structure) [16], which makes it possible to efficiently calculate and clearly represent the energy crystal structure, it became possible to find the energetically substantiated motifs of the packing in the crystal and also visualize supramolecular synthones [17] and supramolecular reactions between them [18]. Our calculations of the energy of pair intermolecular interactions (TONTO/CE-B3LYP DGDZVP) in the crystals of complex **I**, $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$, $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}\cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$, and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ and the representation of the strongest interactions as energy structures (Figs. 3a–3d) showed the complete correspondence with the $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ -associated columns and zigzags proposed above (Fig. 2).

In the crystal of complex **I**, the binding energy in the $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ -associated chains (−9.3 kcal/mol) turned out to be close to the binding energy between the chains (−10.3 kcal/mol), but the chains $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ in $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$ and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}\cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$ and zigzags $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ turned out to be most strongly associated by molecular aggregates appreciably exceeding others. Interestingly, in the cocrystal $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}\cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$, the energy of the $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ binding (−8 kcal/mol) even exceeded the energy of the intermolecular interaction between the halogen-bonded ($\text{I}\cdots\text{I}$) $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$ and $1,4\cdots\text{C}_6\text{H}_4\text{I}_2$ (−5.7 kcal/mol). The dispersion and

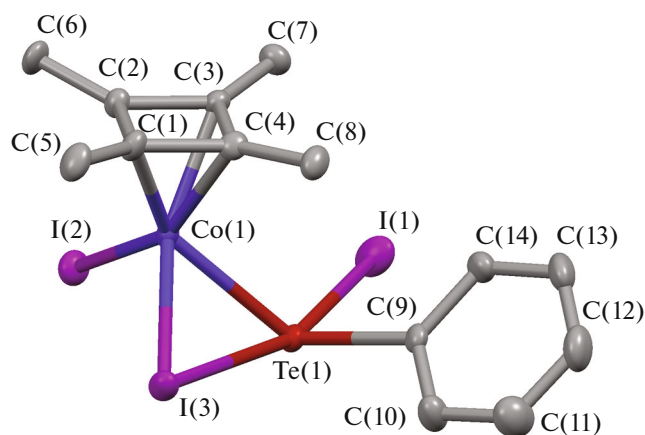


Fig. 1. Molecular structure of complex **I**. Bond lengths and bond angles: $\text{Co}(1)\text{—Te}(1)$ 2.5688(6), $\text{Co}(1)\text{—I}(3)$ 2.6260(7), $\text{Co}(1)\text{—I}(2)$ 2.5664(7), $\text{Te}(1)\text{—I}(1)$ 2.7769(4); $\text{Te}(1)\text{—I}(3)$ 3.1713 Å and $\text{I}(3)\text{Te}(1)\text{I}(1)$ $164.79(1)^\circ$, $\text{Co}(1)\text{Te}(1)\text{C}(9)$ $111.8(1)^\circ$. Hydrogen atoms are omitted.

electrostatic forces introduce approximately equal contributions to the total energy of the intermolecular $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ binding in the chains in complex **I**, $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$, and $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}\cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$ and zigzags in $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$.

The same zigzag chains as in $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ (Fig. 2d) are observed in the isostructural heterohalide compound $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeIBrPh}$. This observation suggests that the predominant formation of contacts $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ rather than $\text{Br}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ should be observed in $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeIBrPh}$, which is characterized by the disordering of the positions of Br and I [5], due to a noticeably higher tendency of iodine to form halogen bonds. This means, in turn, that the positions of the iodine and bromine atoms should be refined with the “free population” rather than 50/50 as in the original article [5]. Indeed, the repeated structure refinement for $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeIBrPh}$ in this regime made it possible to decrease R_1 from 3.36 to 3.01% and showed that the position of the halogen contacting with $(\eta^4\text{-C}_4\text{Me}_4)$ is populated with iodine by 80% and the remained position (having no these contacts) is populated with bromine by 80% (Fig. 4). This is one more important example demonstrating how an analysis of halogen bonds can be useful in the resolution of the positional disorder in crystals [7, 19].

The existence of the 17-electron cobalt atom ($S = 7/2$) in the environment of iodine atoms ($S = 5/2$) in complex **I** allows one to expect the unusual hyperfine splitting of the singlet in the EPR spectrum. Nevertheless, the measurements in the solution and in the solid sample showed no resolved hyperfine structure.

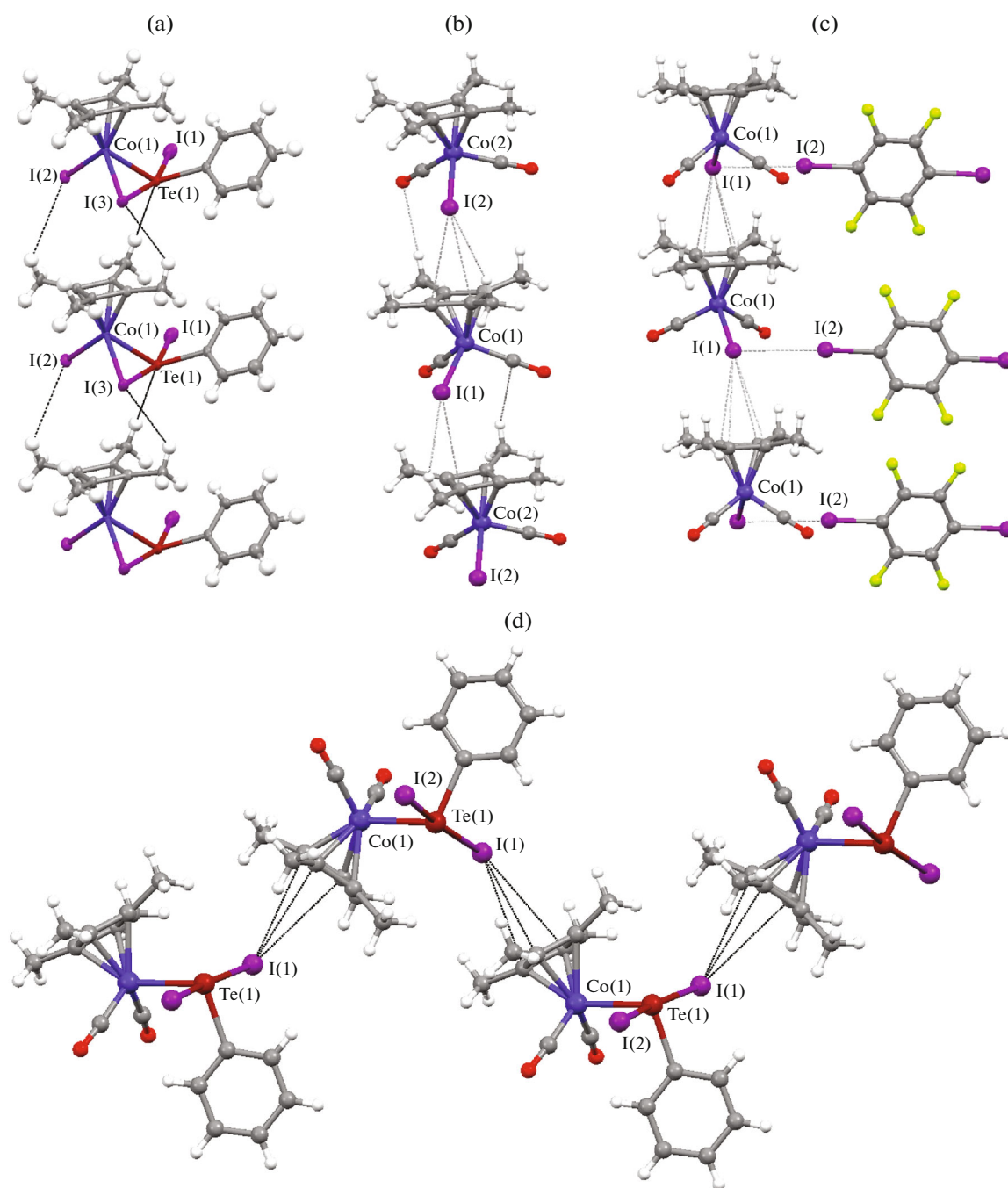


Fig. 2. Chains of molecules of (a) complex **I**, (b) $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$, (c) $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I} \cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$, and (d) $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ associated due to the $\text{I} \cdots (\eta^4\text{-C}_4\text{Me}_4)$ interactions.

The spectrum of powdered compound **I** at room temperature represents a broad unresolved line with $g = 2.25$. The spectrum of a solution of compound **I** in toluene at room temperature represents a broad unresolved line with $g = 2.228$. The spectrum of a solution of complex **I** in toluene at the low temperature (Fig. 5) is described by the orthorhombic spin-Hamiltonian

$$\hat{H} = g_z \beta H_z S_z + g_x \beta H_x S_x + g_y \beta H_y S_y,$$

where g_z , g_x , and g_y are the z , x , and y components of the g tensor; S_z , S_x , and S_y are the projections of the spin operator on the coordinate axes; and $S = 1/2$.

The parameters of the complex were determined by the best fit algorithm between the experimental and theoretical spectra constructed by the method described earlier [20]. The sum of the Lorentzian and Gaussian functions was used as a function of the line

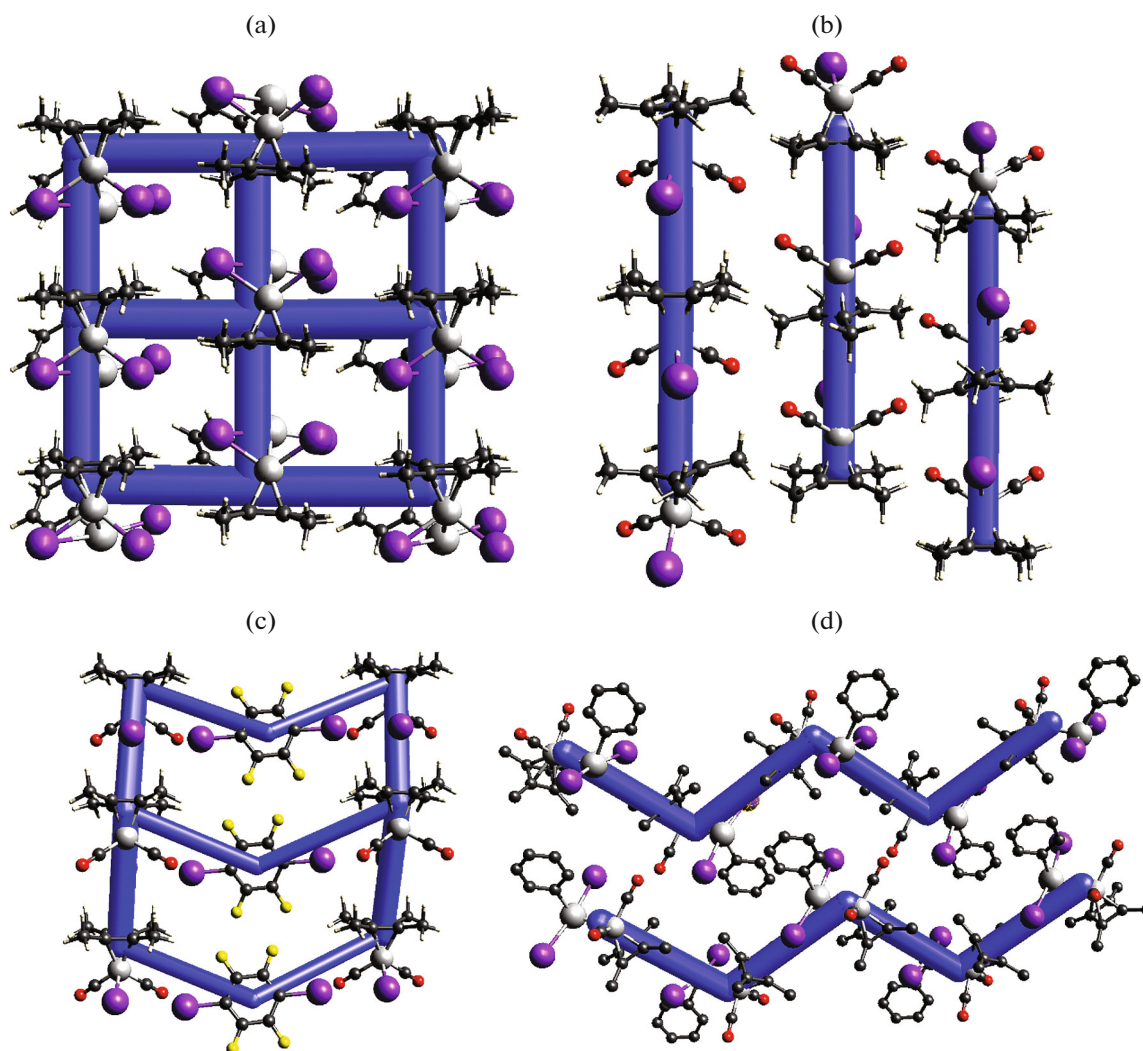


Fig. 3. Energy structures in the crystals of (a) complex I, (b) $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}$, (c) $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{I}\cdots 1,4\text{-C}_6\text{H}_4\text{I}_2$, and (d) $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$ constructed on the basis of the strongest intermolecular packing interactions. The energy structure is built of cylinders, whose diameter and direction reflect the magnitude of the molecular packing energy in different directions.

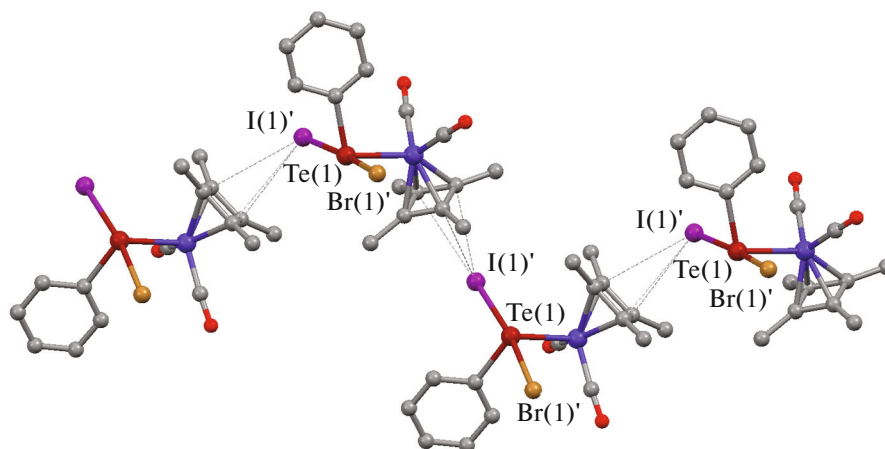


Fig. 4. Chains of the $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeIBrPh}$ molecules predominantly associated due to the $\text{I}\cdots(\eta^4\text{-C}_4\text{Me}_4)$ (not $\text{Br}\cdots(\eta^4\text{-C}_4\text{Me}_4)$!) interactions. Hydrogen atoms are omitted for clarity, and halogen atoms are presented with the maximum population.

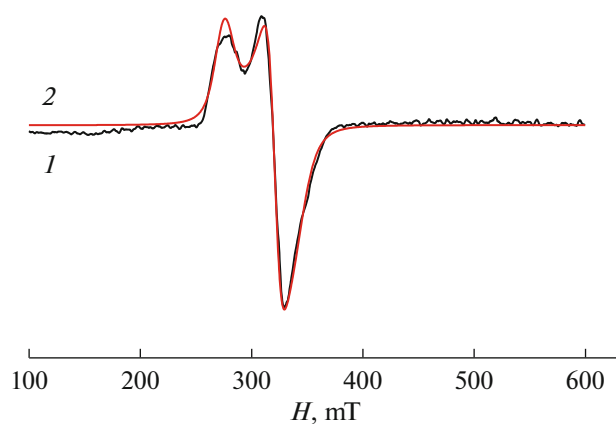


Fig. 5. Spectrum of a solution of complex **I** in toluene at $T = 100$ K: (1) experimental spectrum and (2) simulation with the spin-Hamiltonian parameters ($g_z = 2.490$, $g_x = 2.132$, $g_y = 2.030$).

shape [21]. The g factors, linewidths, and line shapes were varied in the course of minimization. The best spin-Hamiltonian parameters are given in the caption to Fig. 5.

Thus, we showed that the 17-electron cyclobutadienylcobalt complex **I** can be synthesized by the decomposition of $(\eta^4\text{-C}_4\text{Me}_4)\text{Co}(\text{CO})_2\text{TeI}_2\text{Ph}$. The parameters of the spin-Hamiltonian of complex **I** were determined by the best-fit algorithm between the experimental and theoretical spectra. The binding energies of the associated chains in the crystal were determined. Since the 17-electron cobalt complexes play an important role in catalysis, we are planning to continue the studies in this trend.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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