



Regio- and stereo-specific addition of organotellurium trihalides to ferrocenylacetylene: Molecular and crystal structure of (*Z*)-halovinyl organotellurium dihalides

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ABSTRACT

Organotellurium(IV) trihalides RTeX_3 ($\text{X} = \text{Br}, \text{I}$) reacts readily with ferrocenylacetylene to give (*Z*)-products of electrophilic addition to C–C triple bond: (*Z*)- $\text{FcXC} = \text{CTeX}_2\text{R}$ ($\text{R} = \text{Ph}, \text{X} = \text{Br}$ (**1**) or I (**2**); $\text{R} = \text{trans-8-ethoxy-4-cyclooctenyl}, \text{X} = \text{Br}$ (**3**)). In case of PhTeX_3 ($\text{X} = \text{Br}$ or I) the room temperature reaction is spontaneous and the structure of the product does not depend on the polarity of the solvent used; this is in contrast to the reaction of aryl-acetylenes with RTeBr_3 which were reported to afford (*E*)-bromovinyl aryltellurium dibromides in methanol and its (*Z*)-isomer in benzene. Molecular and crystal structures of new compounds and effect of bulky and electron-rich ferrocenyl substituent on the reactivity of acetylene moiety are discussed in this paper.

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1. Introduction

The electrophilic addition of tellurium tetrahalides (TeX_4 , $\text{X} = \text{Cl}, \text{Br}$) to alkynes is one of the most traditional methods to prepare vinylic tellurides. It was first developed by Campos and Petragani in the early 1960s [1] and could be used for the transformation of both terminal and disubstituted acetylenic substrates. The reactivity of the organotellurium trihalides (RTeX_3 , $\text{X} = \text{Cl}$ or Br) is similar to that of tellurium tetrahalides, but only terminal acetylenes could be used as a substrates. These reactions are highly regioselective, and its stereoselectivity can be controlled by changing the polarity of the solvent used [2]. Treatment of terminal acetylenes with RTeBr_3 in methanol, where dissociation of Te-X bond and consequent stabilization of tellurium ions can be assumed due to the more polar environment, gives (*E*)-bromovinyl aryltellurium dibromides. On the other hand, when non-polar benzene was used, the reaction proceeded through a four-membered transition state, resulting in formation of (*Z*)-isomers (Scheme 1).

For a long time there were no reports on the addition of organotellurium triiodides RTeI_3 to acetylenes. Very recently, the addition of PhTeI_3 to PhC_2H has been reported [3] to take place under 48 h reflux in benzene. The dihalides obtained can be further reduced to the corresponding vinylic tellurides (Scheme 2) [9] or used as ligands in metal–carbonyl complexes [3] in a manner previously utilized for their diphenyl analogs (Scheme 3) [4].

As a part of our interest in the coordination chemistry of organotellurium halides [5,6] and recent advances in the investigation of unusual behavior of ferrocenyl acetylene in reactions with metal–carbonyls [7,8], we studied the interaction of ferrocenyl acetylene with some organotellurium trihalides.

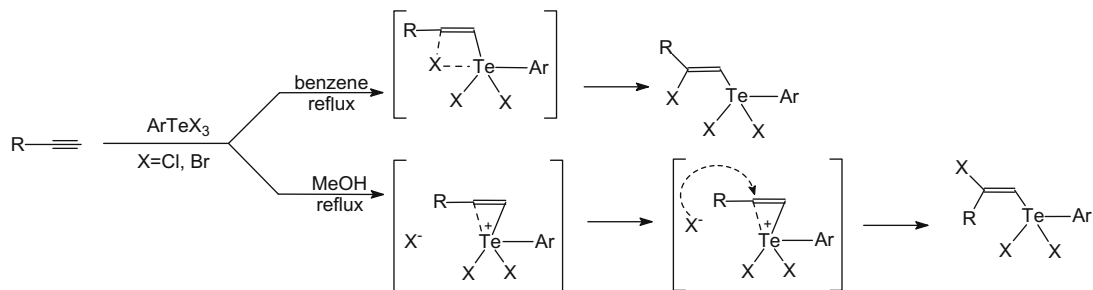
2. Results and discussion

Ferrocenylacetylene undergoes easy regio- and stereo-specific electrophilic addition of organo-tellurium trihalides (PhTeBr_3 , PhTeI_3 , *trans*-8-ethoxy-4-cyclooctenyltellurium tribromide) to give vinylic tellurium dihalides of a general formula (*Z*)- $\text{FcXC} = \text{CTeX}_2\text{R}$ ($\text{R} = \text{Ph}, \text{X} = \text{Br}$, (**1**) or I (**2**); $\text{R} = \text{trans-8-ethoxy-4-cyclooctenyl}, \text{X} = \text{Br}$ (**3**)) (Schemes 4 and 6) as air stable red crystalline substances, soluble in aliphatic hydrocarbons.

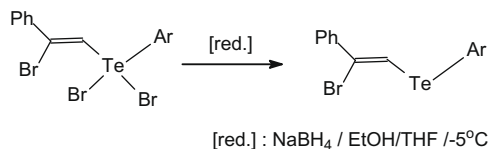
Higher solubility of ferrocenyl-substituted vinylic telluro-halides in comparison with their aryl analogs is apparently due to the lower polarity of the ferrocenyl group. Also, the relatively stronger electron-donor activity of the ferrocenyl group as compared to aryls facilitates a facile and spontaneous electrophilic RTeX_3 addition reaction to occur at room temperature; reactions of arylacetylenes, in contrast require thermolytic conditions and longer reaction times [9]. In the same work it was also demonstrated that interaction of PhTeCl_3 with arylacetylene having electron-withdrawing substituent (*p*-bromophenyleacetylene) requires 24 h reflux in benzene against 2 h for electron-rich *p*-ethoxyphenylacetylene.

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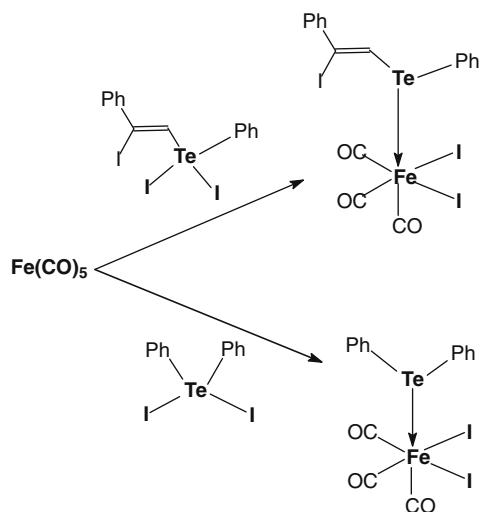
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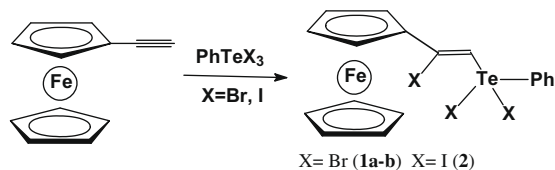
Scheme 1.



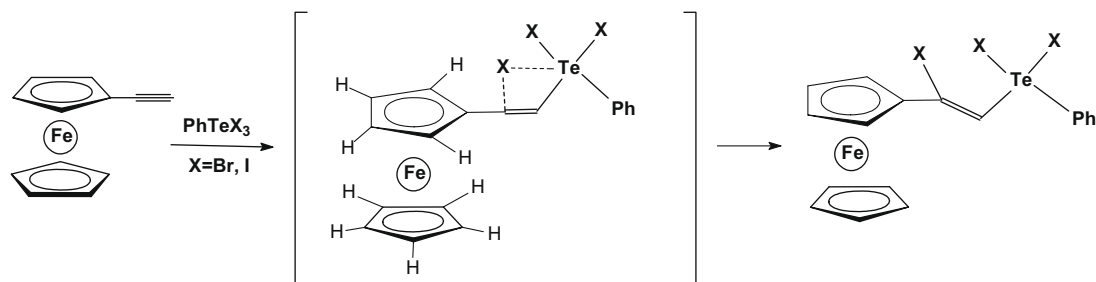
Scheme 2.



Scheme 3.



Scheme 4.



Scheme 5.

Described addition of $PhTeBr_3$ to ferrocenyl acetylene gives (Z)-vinyltellurium dibromide (**1**) as the sole product, suggesting that the nature of the solvent used does not influence the stereospecificity. This is in contrast to the reported earlier formation of (E)-bromovinyl aryltellurium dibromides in methanol and appropriate (Z)-isomers in benzene [2].

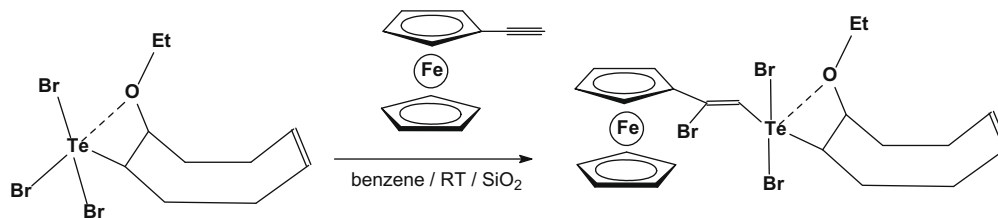
While the electronic effect of the ferrocenyl group may account for the spontaneity of the reaction of ferrocenylacetylene with $RTeX_3$, the bulkiness of the ferrocenyl group very likely contributes to the selective formation of the (Z)-intermediate (see Scheme 5 and Fig. 1).

A single crystal X-ray analysis of compound **1** was carried out. When crystals of **1** were grown from benzene/heptane solvent, crystals (**1a**) incorporating one solvate benzene molecule per molecule of **1** were obtained, while the crystals obtained from methanol and recrystallized from dichloromethane/heptane solvent mixture (**1b**) contain no solvent but have 2 independent molecules of **1** (**1b₁** and **1b₂**, respectively) in the unit cell.

In the solid state **1a** exhibits short inermolecular $Te-Br$ contacts (3.484 Å versus sum of Te/Br van der Waals radii 3.91 Å) which assembles the molecules of **1a** into the zig-zag chains along the b -axis of the unit cell (see Fig. 2).

Apart from this, the molecules of compounds **1a** and **1b** are generally identical (see Table 2) and all the subtle differences in the inter-atomic distances and angles are the result of crystal packing effects and mainly Te -halogen secondary interactions, which fit well with the sum of Te and Br covalent radii [10] as well as usually observed $Te-Br$ distances in previously reported diorganotellurium dibromides.

Although the intermolecular contacts $Te \cdots Br$ in the crystal of **1b** (3.668 and 3.800 Å, respectively) sometime may appear to be shorter than the sum of Te and Br Van der Waals radii they do not affect the geometry of the molecule **1b₁** and **1b₂**. However, they are responsible for the formation of two types of chains (Fig. 3) which give rise to formation of two crystallographically independent molecules in the crystal of **1b**. Although the difference in the bond distances is negligible, the tendency for the elongation of intramolecular $Te-Br$ distance ($Te(1A)-Br(1A)$ 2.6616(17) Å,



Scheme 6.

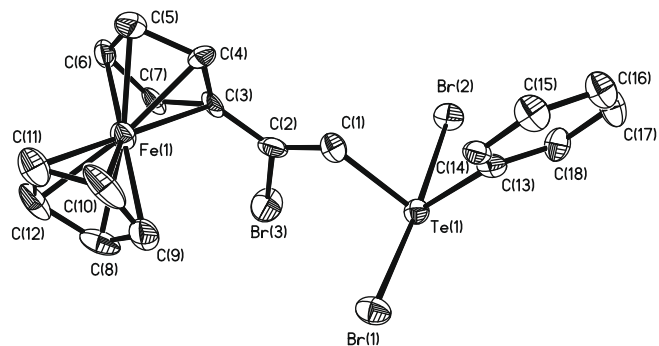


Fig. 1. ORTEP diagram of (*Z*)-(2-bromo-2-ferrocenyl-vinyl)phenyltellurium dibromide (**1a**) with 50% probability ellipsoids. Selected bond distances (Å) and bond angles (°): Te(1)–Br(1) 2.6470(10), Te(1)–Br(2) 2.7198(10), C(1)–C(2) 1.351(10), C(2)–C(1)–Te(1) 124.3(6), Br(1)–Te(1)–Br(2) 176.11(3).

Te(1A)–Br(2A) 2.7207(17) Å, Te(1A)–Br(3A) 3.423 Å) in the “dimeric” **1b₂**, containing intermolecular Te–Br interaction (3.529 Å, which is 0.37 Å shorter than the sum of Te and Br Van der Waals radii) in comparison with “single” molecule **1b₁**, Te(1)–Br(2) 2.6718(17) Å, Te(1)–Br(1) 2.7094(17) Å, Te(1)–Br(3) 3.412 Å, which forms only weak intermolecular Te–Br (~4 Å, what is 0.1 Å longer than appropriate sum of Van der Waals radii) contacts is quite clear (see Table 2 and Fig. 3).

Addition of PhTeI₃ to FcC₂H follows the same pattern as for PhTeBr₃ and at room temperature readily forms (*Z*)-(2-iodo-2-ferro-

ocenyl-vinyl)phenyltellurium diiodide (**2**) which if crystallized from dichloromethane/heptane mixture gives crystals of **2**, which are isostructural with **1b** (see Table 1). Also, we observe the same set of two independent molecules (see Table 4) with the same peculiarities of crystal packing as **1b** with a short intermolecular Te–I distance 3.718 Å in dimeric associate and 4.05 Å between the “single” molecules (see Figs. 4 and 5).

It is noteworthy that under the same conditions as those employed in the reaction of phenyltellurium tribromide, the reaction of ferrocenylacetylene with *trans*-8-ethoxy-4-cyclooctenyltellurium tribromide gives the (*Z*)-vinylic dibromide, **3**. The reaction, is however much slower than in the case of phenyltellurium tribromide, but it accelerates significantly after addition of a small amount of silica gel.

Probable mechanism of such catalytic effect could be rationalized in term of competition between the partly hydrated silica gel surface and ethoxy-group oxygen atom for the coordination site in the Te center and resulting in the activation of Te atom.

Red crystals of **3** (see Table 4) (as well as **1a** and **2**) demonstrates intramolecular Te–O secondary bond (2.6636(6) Å), but no considerable intermolecular Te–Br interactions, probably due to the curtaining effect of the bulky halo-vinylferrocenyl group as well as additional interaction of Te center with vinylic halogen atom which occurs in the same direction as the Te lone electron pair at a distance (3.3856(6) Å) that is shorter than Te/Br van der Waals radii sum (3.90 Å), so that tellurium atom acquires typical pseudo-octahedral geometry by means of intramolecular secondary interactions only (see Fig. 6).

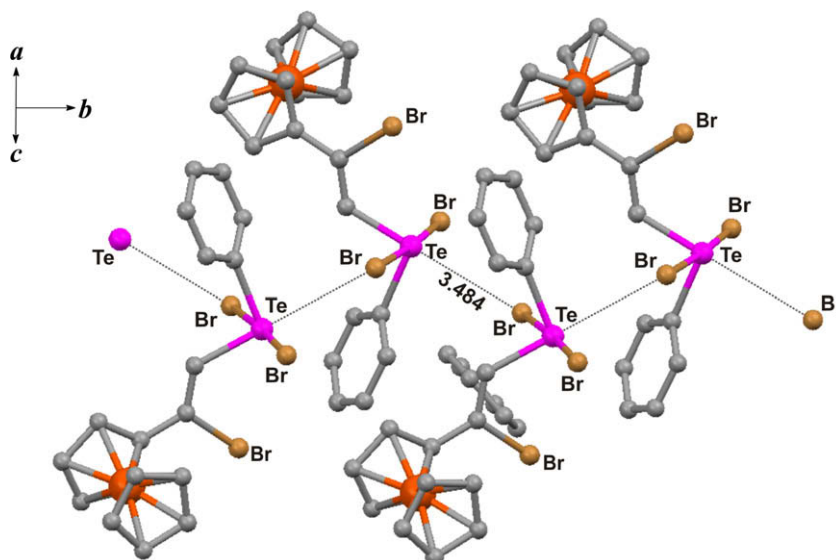
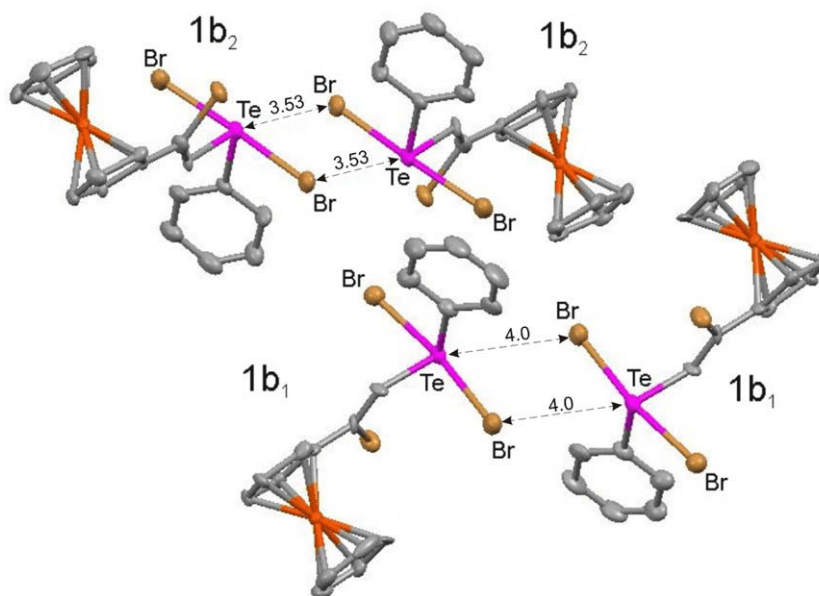


Fig. 2. Chain assembly of **1a** molecules in the solid state.

Table 1
Crystal data and structure refinement for **1–3**.

Identification code	1a	1b	2	3
Formula	C ₂₁ H ₁₈ Br ₃ FeTe	C ₁₈ H ₁₅ Br ₃ FeTe	C ₁₈ H ₁₅ FeI ₃ Te	C ₂₂ H ₂₆ Br ₃ FeOTe
Formula weight	693.53	654.48	795.45	729.61
T (K)	150(2)	296(2)	296(2)	296(2)
λ (Å)	0.71073			
Crystal system	monoclinic			
Space group	C2/c	P21/c	P21/c	P21/n
a (Å)	24.441(3)	14.398(5) E	14.585(3)	14.500(4)
b (Å)	7.8452(10)	10.421(4) E	10.682(2)	11.912(3) Å
c (Å)	24.740(3)	25.714(9) E	26.603(5)	14.901(4)
α (°)	90			
β (°)	116.479(2)	98.303(5)	97.129(3)	106.441(4)
γ (°)	90			
V (Å ³)	4246.1(9)	3818(2)	4112.9(14)	2468.5(10)
Z	8	8	8	4
D _{calc} (mg/m ³)	2.170	2.277	2.569	1.963
Absorption coefficient (mm ⁻¹)	7.706	8.564	6.624	6.636
F(0 0 0)	2616	2448	2880	1396
θ (°)	1.86–29.00	1.99–28.00	1.95–29.00	2.23–29.00
Reflections collected	22 464	37 765	40 406	20 083
Independent reflections (R _{int})	5659 (0.1345)	9225 (0.1851)	10 894 (0.0672)	6508 (0.0742)
Maximum and minimum transmission	0.699 and 0.148	0.783 and 0.170	0.557 and 0.050	0.503 and 0.211
Data/restraints/parameters	5659/0/235	9225/0/415	10 894/0/415	6508/0/254
Goodness-of-fit (GOF) on F ²	0.937	0.963	0.991	0.964
Final R indices [I > 2σ(I)]	R ₁ = 0.0515, wR ₂ = 0.0855	R ₁ = 0.0644, wR ₂ = 0.1265	R ₁ = 0.0482, wR ₂ = 0.0924	R ₁ = 0.0512, wR ₂ = 0.1032
R indices (all data)	R ₁ = 0.1366, wR ₂ = 0.1105	R ₁ = 0.1526, wR ₂ = 0.1661	R ₁ = 0.0901, wR ₂ = 0.1090	R ₁ = 0.1180, wR ₂ = 0.1299
Largest difference peak and hole (e Å ⁻³)	0.871 and -0.893	1.939 and -1.943	1.235 and -0.937	1.305 and -0.593

**Fig. 3.** Two independent molecules (**1b₁** and **1b₂**) in the dimeric assemblies in the solid state.

3. Experimental

3.1. General procedures

All reactions were performed using standard Schlenk techniques under an atmosphere of dry argon. Solvents were purified, dried and distilled under an argon atmosphere. Infrared spectra were recorded on a Specord 75IR spectrometer in THF, diethyl ether or heptane solutions and in KBr pellets. Elemental analyses were performed on a Carlo-Erba automatic analyzer. FcC₂H [11], cyclo-C₈H₁₂(OEt)TeBr₃ [12], and PhTeBr₃ [13], were prepared using reported methods.

3.2. Synthesis of compounds **1a–b**

3.2.1. Synthesis of (Z)-(2-bromo-2-ferrocenyl-vinyl)phenyltellurium (**1a**) dibromide in benzene

Yellow powder of PhTeBr₃ (0.9 g 0.2 mmol) was added in one portion to the orange solution of FcC₂H (0.042 g 0.2 mmol) in benzene (10 mL) at the ambient temperature. Reaction mixture turned red quickly and was stirred for 30 min. Resulting clear burgundy-red solution was concentrated with heptane (10 ml) to half of the initial volume and kept at -10 °C for 12 h. Long red prisms precipitated; these were separated and washed with hexanes, dried in vacuum and used for single crystal X-ray investigation. Further

Table 2
Selected bond lengths (Å) and bond angles (°) for **1a–b**.

1a		1b₁		1b₂	
Te(1)–C(1)	2.081(7)	Te(1)–C(1)	2.072(11)	Te(1A)–C(1A)	2.083(10)
Te(1)–C(13)	2.116(7)	Te(1)–C(13)	2.132(11)	Te(1A)–C(13A)	2.142(11)
Te(1)–Br(1)	2.6470(10)	Te(1)–Br(1)	2.7094(17)	Te(1A)–Br(1A)	2.6616(17)
Te(1)–Br(2)	2.7198(10)	Te(1)–Br(2)	2.6718(17)	Te(1A)–Br(2A)	2.7207(17)
C(1)–C(2)	1.351(10)	C(1)–C(2)	1.326(14)	C(1A)–C(2A)	1.340(15)
C(2)–Br(3)	1.909(8)	C(2)–Br(3)	1.913(10)	C(2A)–Br(3A)	1.908(12)
C(1)–C(2)–C(3)	126.4(7)	C(1)–C(2)–C(3)	126.2(10)	C(1A)–C(2A)–C(3A)	124.6(11)
C(1)–C(2)–Br(3)	117.6(6)	C(1)–C(2)–Br(3)	117.7(8)	C(1A)–C(2A)–Br(3A)	118.4(9)
C(2)–C(1)–Te(1)	124.3(6)	C(2)–C(1)–Te(1)	125.2(8)	C(2A)–C(1A)–Te(1A)	124.3(8)
Br(1)–Te(1)–Br(2)	176.11(3)	Br(1)–Te(1)–Br(2)	171.89(5)	Br(1A)–Te(1A)–Br(2A)	171.83(5)
C(1)–Te(1)–Br(1)	88.30(19)	C(1)–Te(1)–Br(1)	85.9(3)	C(1A)–Te(1A)–Br(1A)	85.6(3)
C(1)–Te(1)–Br(2)	87.82(19)	C(1)–Te(1)–Br(2)	86.6(3)	C(1A)–Te(1A)–Br(2A)	87.4(3)
C(13)–Te(1)–Br(1)	91.61(18)	C(13)–Te(1)–Br(1)	91.2(3)	C(13A)–Te(1A)–Br(1A)	93.1(3)
C(13)–Te(1)–Br(2)	88.92(18)	C(13)–Te(1)–Br(2)	92.4(3)	C(13A)–Te(1A)–Br(2A)	91.3(3)

Table 3
Selected bond lengths (Å) and bond angles (°) for **2**.

2₁		2₂	
Te(1)–C(1)	2.084(7)	Te(1A)–C(1A)	2.091(7)
Te(1)–C(13)	2.141(7)	Te(1A)–C(13A)	2.145(7)
Te(1)–I(1)	2.9540(9)	Te(1A)–I(1A)	2.8914(9)
Te(1)–I(2)	2.9191(9)	Te(1A)–I(2A)	2.9746(9)
I(3)–C(2)	2.089(6)	I(3A)–C(2A)	2.095(7)
C(2)–C(1)	1.332(9)	C(1A)–C(2A)	1.536(5)
C(1)–Te(1)–I(1)	86.79(18)	C(1A)–Te(1A)–I(1A)	86.66(19)
C(1)–Te(1)–I(2)	86.30(18)	C(1A)–Te(1A)–I(2A)	87.50(19)
C(13)–Te(1)–I(1)	93.20(19)	C(13A)–Te(1A)–I(1A)	94.6(2)
C(13)–Te(1)–I(2)	93.9(2)	C(13A)–Te(1A)–I(2A)	91.8(2)
I(2)–Te(1)–I(1)	170.38(2)	I(2A)–Te(1A)–I(1A)	171.63(2)
C(1)–C(2)–C(3)	123.6(6)	C(1A)–C(2A)–C(3A)	123.5(6)
C(1)–C(2)–I(3)	118.8(5)	C(1A)–C(2A)–I(3A)	118.7(5)
C(2)–C(1)–Te(1)	124.1(5)	C(2A)–C(1A)–Te(1A)	124.7(5)

concentration and cooling of the mother-liquor allowed additional quantity of red crystalline precipitate of **1a**. Yield 0.12 g (85%).

3.2.2. Synthesis of (Z)-(2-bromo-2-ferrocenyl-vinyl)phenyltellurium diiodide (**1b**) in methanol

Yellow powder of PhTeBr₃ (0.045 g 0.1 mmol) was added in one portion to the orange solution FcC₂H (0.021 g 0.1 mmol) in methanol (2 mL) at the ambient temperature. Reaction mixture quickly turned dark-red and was stirred for 30 min. The solvent was removed in vacuum. Dark-red residue was extracted with CH₂Cl₂ (20 mL) and filtered through the thin layer of silica gel. Resulting clear burgundy-red solution was concentrated with heptane (10 mL) to half of the initial volume and kept at –10 °C for 12 h.

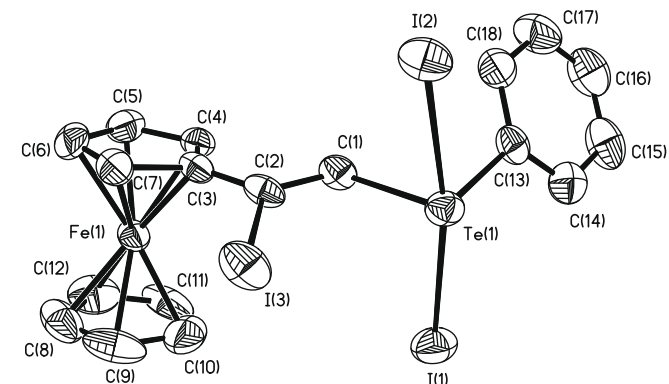


Fig. 4. ORTEP diagram of (Z)-(2-bromo-2-ferrocenyl-vinyl)phenyltellurium diiodide (**2**) with 50% probability ellipsoids. Selected bond distances (Å) and bond angles (°): Te(1)–I(1) 2.9540(9), Te(1)–I(2) 2.9191(9), C(2)–C(1) 1.332(9), I(2)–Te(1)–I(1) 170.38(2), C(2)–C(1)–Te(1) 124.1(5).

Long red prisms precipitated; these were separated and washed with hexanes, dried in vacuum and used for single crystal X-ray investigation. Further concentration and cooling of the mother-liquor allowed additional quantity of red crystalline precipitate of **1b**. Yield 0.05 g (76%). Anal. Calc. for C₁₈H₁₅Br₃FeTe (M = 654): C, 33.03; H, 2.31. Found: C, 34.03; H, 2.85%.

3.3. Synthesis of (Z)-(2-iodo-2-ferrocenyl-vinyl)phenyltellurium diiodide (**2**)

Dark-red powder of PhTeI₃ (0.117 g 0.2 mmol) was added in one portion to the orange solution FcC₂H (0.042 g 0.2 mmol) in CH₂Cl₂ (20 mL) at the ambient temperature. Reaction mixture slowly turned darker and was stirred for 2 h. Resulting clear dark-red solution was concentrated with heptane (5 mL) to half of the initial volume and kept at –10 °C for 3 h. Elongated dark-red prisms precipitated; these were separated and washed with hexanes, dried in vacuum and used for single crystal X-ray investigation. Further concentration and cooling of the mother-liquor allowed additional quantity of red crystalline precipitate of **2**. Yield 0.11 g (69%). Anal. Calc. for C₁₈H₁₅I₃FeTe (M = 795.5): C, 27.18; H, 1.90. Found: C, 27.95; H, 2.14%.

3.4. Synthesis of (Z)-(2-bromo-2-ferrocenyl-vinyl)(trans-8-ethoxy-4-cyclooctenyl)-tellurium dibromide (**3**)

Yellow crystalline cyclo-C₈H₁₂(OEt)TeBr₃ (0.1 g 0.2 mmol) was added in one portion to the orange solution of FcC₂H (0.042 g 0.2 mmol) in benzene (10 mL) at the ambient temperature. The color of the reaction mixture did not change after stirring during 30 min, but turned red being groped at the TLC (Silufol) plate. Three grams of silica gel was added to the stirred reaction solution and its color has started to turn darker. Reaction mixture slowly turned darker and was stirred for 1 h. Resulting red mixture was dried in vacuum and extracted with CH₂Cl₂ (20 mL) on a glass filter. Burgundy-red filtrate was concentrated with heptane (10 mL) to half of the initial volume and kept at –10 °C for 3 h. Elongated dark-red prisms precipitated; these were separated and washed with hexanes, dried in vacuum and used for single crystal X-ray investigation. Further concentration and cooling of the mother-liquor allowed additional quantity of red crystalline precipitate of **3**. Yield 0.11 g (75%). Anal. Calc. for C₂₂H₂₇Br₃FeOTe (M = 730.62): C, 36.17; H, 3.72. Found: C, 36.04; H, 3.701%.

3.5. X-ray crystallography

Suitable X-ray quality crystals of **3–5** were obtained directly during preparation (see synthetic part for details). A Bruker APEX II CCD area detector diffractometer equipped with a low-temperature

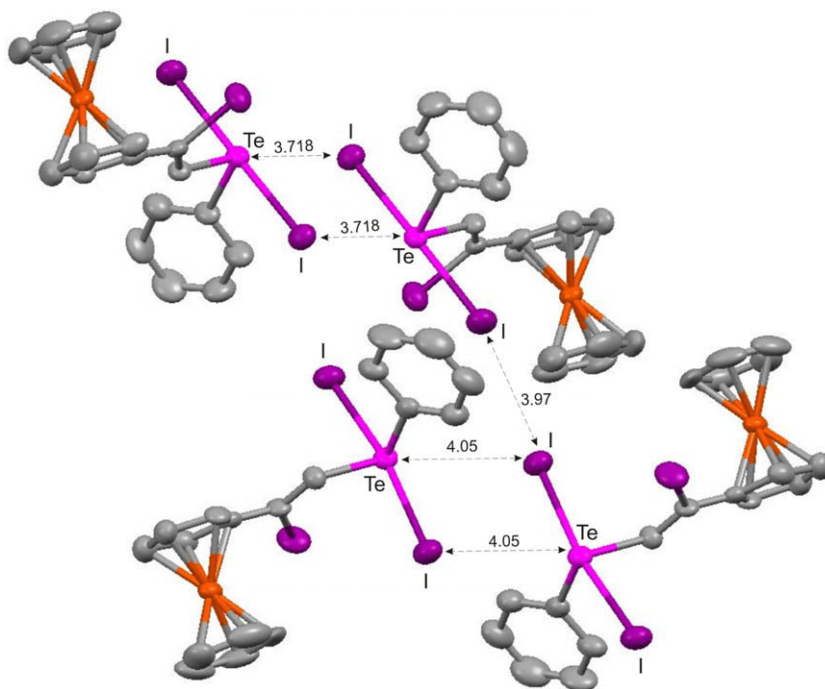


Fig. 5. Two independent molecules of **2** in the dimeric assemblies in the solid state.

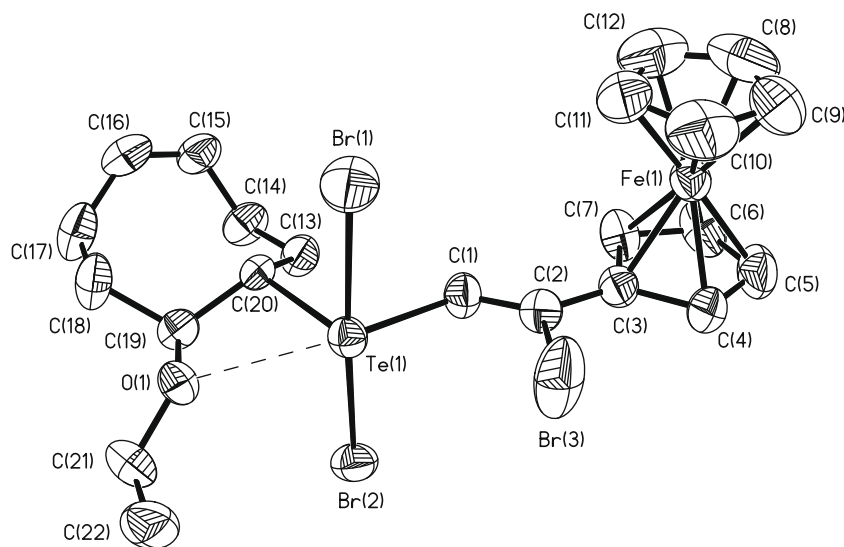


Fig. 6. ORTEP (*Z*)-(2-bromo-2-ferrocenyl-vinyl)(*S,S*-*trans*-8-ethoxy-4-cyclooctenyl)-tellurium dibromide (**3**) with 50% probability ellipsoids. Selected bond distances (Å) and bond angles (°): Te(1)–Br(2) 2.6645(10), Te(1)–Br(1) 2.6946(10), C(1)–C(2) 1.315(8), Te(1)–O(1) 2.6636(6), Br(2)–Te(1)–Br(1) 173.93(3), C(1)–C(2)–C(3) 125.5(6).

attachment was used for the cell determination and intensity data collection for compounds **1–3**. Structures **1–3** were solved by direct methods and refined by means of least squares method for F^2 in anisotropic (isotropic for H atoms) approximation in SHELXTL package [14]. Positions of H atoms were calculated geometrically. Appropriate empirical absorption corrections using the programs SADABS. Relevant crystallographic data and structure refinement details are listed in Table 1. Selected distances and angles for structures **1–3** are given in Tables 1–4.

4. Conclusions

Ferrocenylacetylene was shown to interact with organo-tellurium trihalides to give vinylic tellurium dihalides, but a significant

contrast with known aryl- and alkyl-acetylenes examples is seen in the almost instant interaction between FcC_2H with corresponding RTeX_3 in mild conditions with the formation of only the *cis*-addition product, what results from relatively higher electron-donating properties of ferrocenyl ligand whereas its bulkiness favors the *cis*-addition. Single crystal X-ray investigation revealed noticeable intermolecular Te–halogen interactions, which however are responsible only for the crystal packing effects.

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Table 4
Selected bond lengths (Å) and bond angles (°) for **3**.

Te(1)–C(1)	2.099(6)
Te(1)–C(20)	2.192(6)
Te(1)–Br(2)	2.6645(10)
Te(1)–Br(1)	2.6946(10)
Br(3)–C(2)	1.898(6)
C(1)–C(2)	1.315(8)
Te(1)–O(1)	2.6636(6)
C(1)–Te(1)–C(20)	99.9(2)
C(1)–Te(1)–Br(2)	86.32(18)
C(20)–Te(1)–Br(2)	89.74(16)
C(1)–Te(1)–Br(1)	88.40(18)
C(20)–Te(1)–Br(1)	88.22(15)
Br(2)–Te(1)–Br(1)	173.93(3)
C(1)–C(2)–C(3)	125.5(6)
C(1)–C(2)–Br(3)	119.3(5)
C(3)–C(2)–Br(3)	115.2(5)

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Appendix A. Supplementary material

CCDC 760043 (**1a**), CCDC 760042 (**1b**), CCDC 760044 (**2**), CCDC 760041 (**3**) contains the supplementary crystallographic data for

this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.jorganchem.2010.02.017](https://doi.org/10.1016/j.jorganchem.2010.02.017).

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