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Synthesis and Structure Determination of Selenium(IV) Cyanides

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The reaction of SeF_4 with Me_3SiCN did not result in the preparation of the designated $Se(CN)_4$ but $Se(CN)F_3$ and $Se(CN)_2F_2$ were obtained as first known selenium(IV) cyanide compounds and characterized by their NMR spectra. $Se(CN)_2F_2$ was crystallized as 1,2-dimethoxyethane solvate as well as the corresponding tellurium compound $Te(CN)_2F_2$ with very similar structures. NMR spectroscopic data of some more miscellaneous tellurium cyanides and the crystal structures of solvates of $Se(CN)_2$ and oxygen-bridged $TeO(CN)_2$ are presented.

Introduction

The chemistry of selenium cyanides is limited to $Se(CN)_2$,[1] $Se_2(CN)_2$[2] and $Se_3(CN)_2$.[3] Very recently, Klapötke et al. reported an attempted preparation of $Se(CN)_4$ that failed.[4] No selenium cyanide with selenium in the oxidation state IV has been characterized so far. The chemistry of sulfur(IV) cyanides consists out of sulfur cyanide trifluoride and a poorly characterized, unstable sulfur dicyanide difluoride.[5] Very recently, tellurium tetracyanide solvates were synthesized and structurally characterized[6] whereas the unsolvated $Te(CN)_4$ has been described as very unstable and even explosive.[7] In continuation of our work on tellurium and selenium cyanides we report on the synthesis and structural characterization of selenium and tellurium dicyanide difluoride and the formal hydrolysis product tellurium dicyanide oxide which crystallizes as a dimer.

Results and Discussion

The compounds were synthesized as outlined in Scheme 1 using trimethylsilyl cyanide as cyanide transfer agent. The reactions were performed in FEP or MFA reaction vessels in DME (1,2-dimethoxyethane) or DME/THF mixtures as solvent. ^{13}C-enriched trimethylsilyl cyanide was used for the low temperature NMR experiments to detect reaction intermediates and structures in solution. The use of a glass flask for the reaction results in the formation of tellurium dicyanide oxide which crystallizes from the reaction mixture as DME solvate.

Selenium cyanide trifluoride and selenium dicyanide difluoride can be detected in the ^{19}F, ^{13}C and ^{77}Se NMR spectra (Table 1, Figure 1). No resonances of $Se(CN)_3F$ or $Se(CN)_4$ could be detected even if an excess of Me_3SiCN is used. An excess of Me_3SiCN together with a long reaction time and higher temperature results in the formation of $(CN)_2$ and $Se(CN)_2$ which can be detected by NMR spectroscopy and crystallized as DME solvate.

$$SeF_4 + 2\,Me_3SiCN \xrightarrow{DME} Se(CN)_2F_2dme_2 + 2\,Me_3SiF$$
$$TeF_4 + 2\,Me_3SiCN \xrightarrow{DME} Te(CN)_2F_2dme_2 + 2\,Me_3SiF$$
$$TeF_4 + 2\,Me_3SiCN \xrightarrow[glass]{DME} (Te(CN)_2Odme_2)_2 + 2\,Me_3SiF$$

Scheme 1.

Table 1. NMR spectroscopic data of SeF_4, $Se(CN)F_3$, $Se(CN)_2F_2$ and $Se(CN)_2$ in a DME/[D_8]THF mixture at –80 °C (δ in [ppm], J in [Hz]).

	SeF_4	$Se(CN)F_3$	$Se(CN)_2F_2$	$Se(CN)_2$
δ (^{77}Se)	1075	933	575	286
δ (^{19}F)	24.2 (F_{ax}) 10.4 (F_{eq})	12.5 (F_{ax}) –37.2 (F_{eq})	–27.4	–
δ (^{13}C)	–	121	111	99.9
^1J (^{77}Se-^{19}F)	158 (F_{ax}) 1170 (F_{eq})	144 (F_{ax}) 782 (F_{eq})	406	–
^1J (^{77}Se-^{13}C)	–	274	242	231
^2J (^{13}C-^{19}F)	–	16 (F_{ax}) 21 (F_{eq})	18	–
^2J (^{19}F-^{19}F)	26[8]	140	–	

Quite in contrast the reaction of tellurium tetrafluoride with $Me_3Si^{13}CN$ results in the formation of $Te(CN)_2F_2$, $Te(CN)_3F$ and $Te(CN)_4$, respectively as can be seen by the analysis of the ^{19}F, ^{13}C and ^{125}Te NMR spectra besides small amounts of $Te(CN)_2FCl$ (**8**), $Te(CN)_2Cl_2$ and $Te(CN)_3Cl$ from an Me_3SiCl impurity (Figure 2). The NMR spectroscopic data are summarized in Tables 2 and 3. The NMR spectroscopic data of all of these compounds are consistent with the principal structure shown in Scheme 2. The sub-

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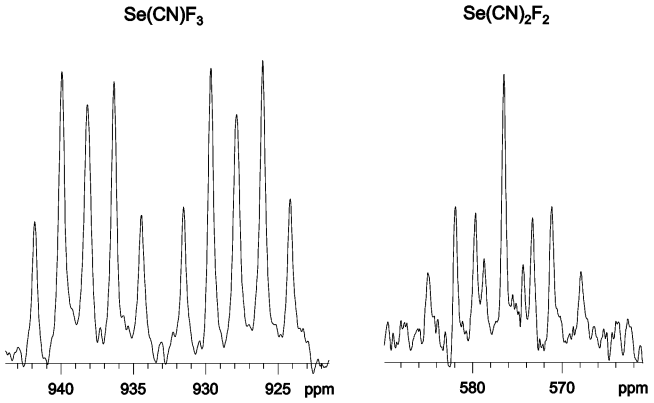


Figure 1. ^{77}Se NMR spectra of ^{13}C labelled $Se(CN)F_3$ (signal multiplicity: ddt) and $Se(CN)_2F_2$ (signal multiplicity: tt) in [D_8]THF/DME mixture at –80 °C, Me_2Se as reference standard.

stituents occupy two axial and two equatorial positions of a ψ-trigonal bipyramid with the lone pair in the equatorial position. In solution the coordination sphere is presumably completed by solvent molecules like DME or THF. According to the VSEPR model the more electronegative substituents (fluorine) occupy the axial position and the cyanide substituent prefers the equatorial position. This is reflected in the smaller ^{77}Se-^{19}F coupling constants for the axial fluorine substituent in comparison to that of the equatorial fluorine substituents of SeF_4 and $Se(CN)F_3$ as expected due to the smaller s character for the axial bond. The differences in $^1J(^{77}Se\text{-}^{19}F)$ for SeF_4 in comparison to literature data is due to the different solvent, DME/THF and CH_3F.[8] The same observation is made for the $^1J(^{125}Te\text{-}^{13}C)$ couplings to axial and equatorial cyanide substituents, respectively.

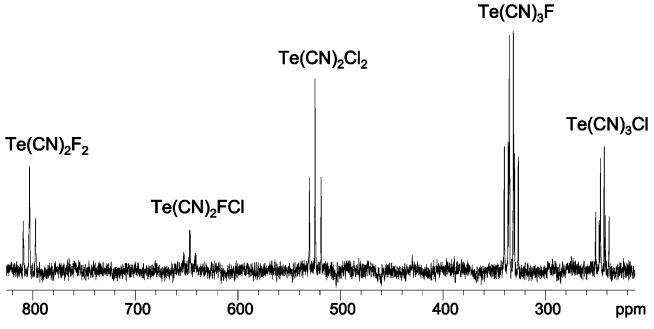


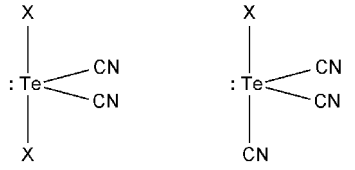
Figure 2. ^{125}Te NMR spectra of ^{13}C-labelled $Te(CN)_2F_2$, $Te(CN)_2FCl$, $Te(CN)_2Cl_2$, $Te(CN)_3F$ and $Te(CN)_3Cl$ in [D_8]THF/DME mixture at –70 °C, Me_2Te as reference standard.

Table 2. NMR spectroscopic data of $Te(CN)_2F_2$, $Te(CN)_2FCl$ and $Te(CN)_2Cl_2$ in a DME/[D_8]THF mixture at –80 °C (δ in [ppm], J in [Hz]).

	$Te(CN)_2F_2$	$Te(CN)_2FCl$	$Te(CN)_2Cl_2$
δ (^{125}Te)	804	647	525
δ (^{19}F)	–77.6	–59.7	–
δ (^{13}C)	122	117	111
^1J (^{125}Te-^{19}F)	48	85	–
^1J (^{125}Te-^{13}C)	744	722	723
^2J (^{13}C-^{19}F)	31	32	–

Table 3. NMR spectroscopic data of $Te(CN)_3F$ and $Te(CN)_3Cl$ in a DME/[D_8]THF mixture at –80 °C (δ in [ppm], J in [Hz]).

	$Te(CN)_3F$	$Te(CN)_3Cl$
δ (^{125}Te)	333	245
δ (^{19}F)	–23.5	–
δ (^{13}C)	125 (C_{ax})	121 (C_{ax})
	113 (C_{eq})	109 (C_{eq})
^1J (^{125}Te-^{19}F)	31	–
^1J (^{125}Te-^{13}C)	477 (C_{ax})	459 (C_{ax})
	613 (C_{eq})	603 (C_{eq})
^2J (^{13}C-^{19}F)	149 (C_{ax})	–
	39 (C_{eq})	
^2J (^{13}C-^{13}C)	8	5



Scheme 2. Principal structures of ψ-trigonal bipyramidal di- and trisubstituted tellurium(IV) cyanides with X = F, Cl.

Crystals of solvates of $TeO(CN)_2$, $Se(CN)_2F_2$, $Te(CN)_2F_2$, and $Se(CN)_2$ were obtained from DME solutions by cooling the reaction mixture to –80, –35, –35 and –80 °C, respectively. Quite unexpected $Se(CN)_2F_2$·2DME and $Te(CN)_2F_2$·2DME crystallize isomorphous in the monoclinic space group $C2/c$ with very similar lattice constants whereas $TeCl_4$·2DME and $SeCl_4$·DME have completely different structures.[9] As can be seen from Figure 3 half a molecule forms the asymmetric unit of the C_2-symmetric molecule. Two DME molecules are coordinated to the sele-

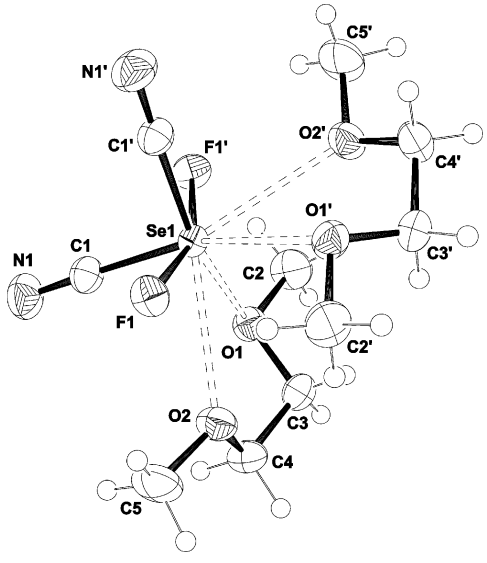


Figure 3. Molecular structure (ORTEP[10]) of $Se(CN)_2F_2$·2DME. Atoms labelled with prime (′) are generated by the symmetry operation 1 –x, y, 0.5-z. Selected bond lengths and angles: Se1/Te1–C1 1.8934(12)/2.1076(11), Se1/Te1–F1 1.8332(7)/1.9653(6), Se1/Te1–O1 2.9414(10)/2.7882(9), Se1/Te1–O2 2.8558(9)/2.8243(8) Å, F1–Se1–F1 157.11(5)/F1–Te1–F1 146.82(4), C1–Se1–C1 98.40(7), C1–Te1–C1 96.10(6), F1–Se1–C1 82.62(4) and F1–Te1–C1 78.96(4)°.

nium and tellurium centre with large element oxygen distances. The four ligands form a ψ-trigonal bipyramid with the fluorine substituents in axial and the cyanide groups and the lone pair in equatorial position as expected from the VSEPR model and in agreement with NMR spectra. Se–F bonds (1.833 Å) are 0.06 Å shorter than Se–C bonds and the angle between the axial fluorine ligands is significant smaller than 180°. The cyanide groups are almost ideal with a typical C–N triple bond length (1.137 Å) and linear orientation at the carbon atom. Tellurium dicyanide oxide forms a centrosymmetric oxygen-bridged dimer with Te–O distances of 1.899 and 2.096 Å. Again the coordination sphere of the tellurium is completed by two DME molecules with large Te–O distances (Figure 4). $Se(CN)_2$·DME (Figure 5) forms a cyclic dimer with two DME molecules bridging two $Se(CN)_2$ units. Therein the two cyanide groups are almost identical in bond length and angle whereas the unsolvated structure reported by Linke and Lemmer[1b] of $Se(CN)_2$ contains two different cyanide groups. In both structures the selenium centre is square-planar coordinated, in unsolvated $Se(CN)_2$ via secondary Se–N contacts and in $Se(CN)_2$·DME via coordination of two DME molecules.

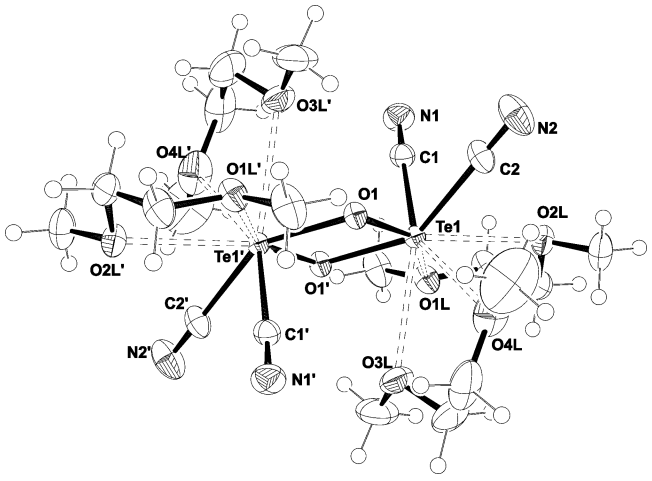


Figure 4. Molecular structure (ORTEP[10]) of $TeO(CN)_2$·2DME. Atoms labelled with prime (′) are generated by the symmetry operation 2 –*x*, –y, 1 – *z*. Selected bond lengths and angles: Te1–C1 2.1367(13), Te1–C2 2.2447(14), Te1–O1 1.8991(9), Te1–O1′ 2.0960(9), Te1–OL 2.8509(12)–2.9143(11) Å, O1–Te1–O1′ 76.47(4), C1–Te1–C2 79.02(6), Te1–O1–Te1′ 103.53(4)°.

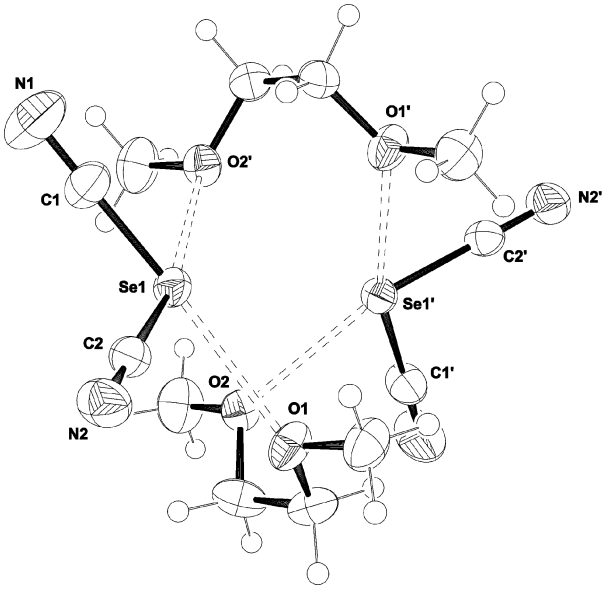


Figure 5. Molecular structure (ORTEP[10]) of $Se(CN)_2$·DME. Atoms labelled with prime (′) are generated by the symmetry operation 1 – *x*, y, 0.5 – *z*. Selected bond lengths and angles: Se1–C1 1.8584(19), Se1–C2 1.8673(18), Se1–O1 2.728(1), Se1–O2′ 2.703(1) Å, C1–Se1–C2 90.22(8), O1–Se1–O2′ 118.19(8)°.

Conclusions

As already observed for tellurium tetracyanide the solvent molecule has a strong influence on the structure and stability of the compounds. Attempts to isolate solvent-free tellurium tetracyanide resulted in an extremely sensitive material which could not be further characterized. We expect that selenium tetracyanide is less stable and a suitable solvent needs to be found to prevent the decomposition into selenium dicyanide and cyanogen which is exothermic by 300 kJ mol^{-1} on the basis of MP2 calculations.[4]

Experimental Section

General: All reactions were carried out under vacuum in carefully dried FEP or MFA tubes by condensing all volatile reagents into the reaction vessel using a glass vacuum line system. SeF_4 and TeF_4 were prepared as described in the literature[11] and handled in an automatic drybox. 1,2-dimethoxyethane was carefully dried and distilled onto molecular sieves. $(CH_3)_3SiCN$ (from Acros) was distilled before use.

In a typical NMR experiment 0.5 mmol of EF_4 (E = Se, Te) was dissolved in a DME/[D_8]THF mixture (5:1) in a MFA NMR tube. $(CH_3)_3Si^{13}CN$ (0.1 g, 1 mmol) was condensed to this solution and the tube was flame sealed. NMR spectra were taken after reaction at –60 °C (selenium compounds) or 20 °C (tellurium compounds).

$Se(CN)F_3$: ^{19}F NMR (DME/[D_8]THF, –80 °C): δ = 12.5 (dd, $^2J_{FF}$ = 140, $^2J_{FC}$ = 16 Hz, 2 F_{ax}), –37.2 (dt, $^2J_{FF}$ = 140, $^2J_{FC}$ = 21 Hz, 1 F_{eq}) ppm. ^{13}C NMR (DME/[D_8]THF, –80 °C): δ = 121 (dt, $^2J_{CF(eq)}$ = 21, $^2J_{CF(ax)}$ = 16 Hz, 1 C) ppm. ^{77}Se NMR (DME/[D_8]-THF, –80 °C): δ = 933 (ddt, $^1J_{SeF(eq)}$ = 782, $^1J_{SeC}$ = 274, $^1J_{SeF(ax)}$ = 144 Hz) ppm.

$Se(CN)_2F_2$: ^{19}F NMR (DME/[D_8]THF, –80 °C): δ = –27.4 (t, $^2J_{FC}$ = 18 Hz, 2 F_{ax}) ppm. ^{13}C NMR (DME/[D_8]THF, –80 °C): δ = 111 (t, $^2J_{CF}$ = 17.8 Hz, 2 C) ppm. ^{77}Se NMR (DME/[D_8]THF, –80 °C): δ = 575 (tt, $^1J_{SeF}$ = 406, $^1J_{SeC}$ = 242 Hz) ppm.

$Te(CN)_2F_2$: ^{19}F NMR (DME/[D_8]THF, –60 °C): δ = –77.6 (t, $^2J_{FC}$ = 31 Hz, 2 F_{ax}) ppm. ^{13}C NMR (DME/[D_8]THF, –70 °C): δ = 122 (t, $^2J_{CF}$ = 31 Hz, 2 C) ppm. ^{125}Te NMR (DME/[D_8]THF, –70 °C): δ = 804 (tt, $^1J_{TeF}$ = 48, $^1J_{TeC}$ = 744 Hz) ppm.

$Se(CN)_2F_2$·2DME: To 0.5 g (0.3 mmol) of selenium tetrafluoride in a MFA tube 1 mL of DME was condensed and the SeF_4 allowed to dissolve at ambient temperature. The solution was cooled to –196 °C and 0.6 g (0.6 mmol) of $(CH_3)_3SiCN$ was condensed into the tube. The tube was flame-sealed and warmed to –10 °C to dissolve solid $(CH_3)_3SiCN$. Then the reaction mixture was kept for

two days at –60 °C and then three days at –70 °C. After 10 days at –80 °C, $Se(CN)_2F_2(dme)_2$ was obtained as colourless and very temperature-sensitive crystals. Crystal structure: $C_{10}F_2H_{20}N_2O_4Se$, M = 349.24, monoclinic, $C2/c$, a = 15.141(3), b = 8.0531(13), c = 14.257(2) Å, β = 113.310(4)°, V = 1596.4(5) Å^3, Z = 4, $\rho_{calcd.}$ = 1.453 g· cm^{-1}, T = 173 K, structure solution by direct methods and full-matrix least-squares refinement,[12] 8380 measured reflections, 2425 crystallographic unique reflections (R_{int} = 0.018) and 2281 reflections with $F_o > 4\sigma(F_o)$, Mo-K_α, λ = 0.71073 Å, $2\theta_{max}$ = 61.01°, R_1 = 0.0188, wR_2 = 0.0480, 89 parameters, anisotropic displacement parameters, H-atoms isotropic, riding model.

$Se(CN)_2$·DME: The reaction was carried out similar to the formation of $Se(CN)_2F_2$·2DME but with 0.2 g (1.3 mmol) of SeF_4 and 0.5 g (5 mmol) of $(CH_3)_3SiCN$. Solid $(CH_3)_3SiCN$ was dissolved at –50 °C and the MFA tube was stored at –80 °C for 4 weeks, then at –30 °C for 20 h and again 4 weeks at –80 °C and colourless crystals were obtained. Raman (–90 °C): $\tilde{\nu}$ = 117, 134, 314, 350, 365, 374, 455, 499, 512, 842, 852, 1021, 1028, 1079, 1095, 1130, 1167, 1246, 1283, 1415, 1437, 1445, 1470, 1479, 2169, 2175, 2728, 2794, 2817, 2834, 2854, 2876, 2905, 2919, 2947, 2962, 2996 cm^{-1}. Crystal structure: $C_{12}H_{20}N_4O_4Se_2$, M = 442.2, monoclinic, $C2/c$, a = 17.138(3), b = 8.6309(15), c = 14.247(3) Å, β = 114.518(3)°, V = 1917.3(6) Å^3, Z = 4, $\rho_{calcd.}$ = 1.532 g cm^{-1}, T = 173 K, structure solution by direct methods and full-matrix least-squares refinement,[12] 15358 measured reflections, 2940 crystallographic unique reflections (R_{int} = 0.027) and 2342 reflections with $F_o > 4\sigma(F_o)$, Mo-K_α, λ = 0.71073 Å, $2\theta_{max}$ = 61.10°, R_1 = 0.0242, wR_2 = 0.0560, 102 parameters, anisotropic displacement parameters, H-atoms isotropic, riding model.

$Te(CN)_2F_2$·2DME: In a MFA tube 0.1 g (0.5 mmol) of TeF_4 was dissolved in 0.7 mL of DME and 0.1 g (1.0 mmol) of $(CH_3)_3SiCN$ was condensed into this solution. The flame-sealed tube was shaken at room temperature and placed in a refrigerator (–35 °C); colourless platelets were obtained overnight. Raman (–80 °C): $\tilde{\nu}$ = 164, 181, 216, 349, 371, 410, 418, 523, 540, 838, 858, 1013, 1024, 1069, 1094, 1121, 1161, 1240, 1276, 1410, 1440, 1452, 1471, 2081, 2186, 2718, 2791, 2807, 2827, 2847, 2868, 2891, 2909, 2930, 2947, 2962, 3023, 3036 cm^{-1}. Crystal structure: $C_{10}F_2H_{20}N_2O_4Te$, M = 397.9, monoclinic, $C2/c$, a = 15.132(2), b = 7.9293(11), c = 14.201(2) Å, β = 111.911(2)°, V = 1580.9(4) Å^3, Z = 4, $\rho_{calcd.}$ = 1.972 g cm^{-1}, T = 133 K, structure solution by direct methods and full-matrix least-squares refinement,[12] 9490 measured reflections, 2412 crystallographic unique reflections (R_{int} = 0.015) and 2344 reflections with $F_o > 4\sigma(F_o)$, Mo-K_α, λ = 0.71073 Å, $2\theta_{max}$ = 61.06°, R_1 = 0.0129, wR_2 = 0.0309, 89 parameters, anisotropic displacement parameters, H-atoms isotropic, riding model.

$TeO(CN)_2$·2DME: In a Schlenk flask 0.1 g (0.5 mmol) of TeF_4 was dissolved in 2.5 mL of DME. In another Schlenk flask 0.2 g (2 mmol) of $(CH_3)_3SiCN$ was dissolved in 2.5 mL DME. Both solutions were cooled to –50 °C and the TeF_4 solution was passed with Argon pressure over Teflon tubing into the $(CH_3)_3SiCN$ solution. The reaction mixture was warmed up to –18 °C and colourless crystals precipitate. These were redissolved and at –35 °C colourless platelets were obtained overnight. Raman (–80 °C): $\tilde{\nu}$ = 194, 223, 262, 292, 311, 333, 368, 386, 400, 521, 678, 846, 859, 1020, 1078, 1129, 1293, 1443, 1457, 1477, 2149, 2166, 2731, 2814, 2827, 2849, 2891, 2921, 2947, 3010 cm^{-1}. Crystal structure: $C_{10}H_{20}N_2O_5Te$, M = 375.9, monoclinic, $P2_1/n$, a = 9.2504(10), b = 14.988(2), c = 11.1541(14) Å, β = 92.191(8)°, V = 1545.3(3) Å^3, Z = 4, $\rho_{calcd.}$ = 1.616 g cm^{-1}, T = 143 K, structure solution by direct methods and full-matrix least-squares refinement,[12] 25247 measured reflections, 4695 crystallographic unique reflections (R_{int} = 0.014) and 4331 reflections with $F_o > 4\sigma(F_o)$, Mo-K_α, λ = 0.71073 Å, $2\theta_{max}$ = 61.08°, R_1 = 0.0149, wR_2 = 0.0362, 167 parameters, anisotropic displacement parameters, H-atoms isotropic, riding model.

CCDC-695320 [for $Se(CN)_2F_2$·2DME], -695321 [for $Se(CN)_2$·DME], CCDC-695322 [for $Te(CN)_2F_2$·2DME], and CCDC-695323 [for $TeO(CN)_2$·2DME] contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.ac.uk/data_request/cif.

Acknowledgments

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