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SYNTHESIS, ^{19}F, ^{125}Te NMR SPECTRA AND CRYSTAL STRUCTURE OF NBu_4TeF_5 AND NH_4TeF_5 $(NH_4F)_{0.25}$ ('$(NH_4)_2TeF_6$')

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SUMMARY

^{19}F and ^{125}Te NMR in different organic solvents and X-Ray investigations of NBu_4TeF_5 (I) and '$(NH_4)_2TeF_6$' (II) show that TeF_5^- anion in (I) both solution and the solid state has a square pyramidal structure. No octahedral arrangement of fluorine was found in (II). This compound consists of NH_4TeF_5 and NH_4F molecules, the NH_4F being in channels between the layers of NH_4TeF_5.

INTRODUCTION

The reaction of TeO_2 with MF (where M = K^+, Cs^+, NH_4^+) in hydrofluoric acid leads to the formation of complex tellurium (IV) fluorides with not more than 5 fluorine atoms in the inner coordination sphere [1]. The pentafluorotellurite anion has a distorted pseudooctahedral configuration with a stereoactive lone pair (LP) of electrons occupying one of the coordination positions.

$[TeX_6]^{2-}$ anions (where X = Cl,Br) which have a regular octahedral configuration, are rather stable [2-5]. In such complexes the lone pair is apparently delocalized on the central atom and, evidently, is stereochemically inactive. We synthesized mixed pentacoordinated fluorotellurites, containing the ions Cl^-, NO_3^-,

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SO_4^{2-}. X-Ray investigations showed that neither of these anions can compete with fluorine for entering the first coordination sphere and localizes on the lone pair side of the central atom with distances of Te...L 2.7 - 3.4 Å ($L = Cl^-$, NO_3^-, SO_4^{2-}) [6,7]. Such contacts are specific for the second coordination sphere of the Te atom.

It is not surprising, that information [8] about $(NH_4)_2TeF_6$ synthesis caused great interest. The authors obtained the compound by several methods and proposed the existence of an octahedral anion TeF_6^{2-} on the basis of Raman spectroscopy and elemental analysis [9]. Both $(NH_4)_2TeCl_6$ and TeO_2 were used for preparation.

Later W.Abriel and co-workers [10] showed by the X-ray investigation that the phase obtained by interaction of $(NH_4)_2TeCl_6$ and HF was a pentafluorotellurite (IV) $NH_4TeF_5 \cdot (HCl)_{0.25} \cdot (H_2O)_{0.25}$. However, in a more recent paper [11] the authors confirm that the product described earlier as $(NH_4)_2TeF_6$ actually contains Te(IV) atoms octahedrally coordinated by six fluorine atoms. It is shown that the product is a solid solution of $nNH_4F \cdot mTeF_4$ with n/m close to 2 and containing small amounts of HF. Since the lone pair is stereoactive in all previously studied tellurium (IV) complexes, the existence of TeF_6^{2-} anion seemed to us to be unlikely.

The aim of the present work was the synthesis and study of NBu_4TeF_5 and '$(NH_4)_2TeF_6$' by X-ray structural analysis, ^{19}F and ^{125}Te NMR and IR-spectroscopy. We assumed that the presence of a cation such as NBu_4^+ in NBu_4TeF_5 will increase the solubility of this compound in organic solvents which will allow study of the structure of this complex in solution by ^{125}Te NMR. On the other hand it will lead to the decrease of distortion of the Te atom arrangement geometry due to weakening of mutual influence of neighbouring ions $[TeF_5]^-$ in the crystals of the compound .

EXPERIMENTAL

Synthesis of NBu_4TeF_5 (I) was carried out as follows. TeO_2 was first dissolved in 40% of HF. A stoichiometric quantity of NBu_4Br was added with stirring into the solution.The mixture was kept for crystallization.(Analysis: Required for NBu_4TeF_5 Te=27.46%, F=20.44%, Found Te=27.3%, F=20.8%). Solutions of NBu_4TeF_5 in acetonitrile, acetone and dimethylformamide were used for NMR investigations. To obtain '$(NH_4)_2TeF_6$' (II) the procedure suggested in [8] was used. The compound has the same values of chemical analysis as one in [8].

^{19}F and ^{125}Te NMR spectra were recorded by VARIAN XL-100 and BRUKER WP-80 spectrometers with internal stabilization on the deuterium. Chemical shifts are given relative to CCl_3F and $Te(OH)_6$ (40% solution in D_2O) for ^{19}F and ^{125}Te respectively.

Diffraction data were collected on an automated Syntex $P2_1$ diffractometer (graphite-monochromatized λMoK_α-radiation, $\theta/2\theta$ scanning, $2\theta_{max}=63^\circ$, 4108 unique observed reflections (for compound I), $2\theta_{max}=65^\circ$, 758 reflections (for compound II).

The crystals of NBu_4TeF_5 (I) are monoclinic; a=12.556(2), b=13.032(1), c=12.635(2)$\mathring{A}$, β=96.73(1)°, V=2053.3(4) $\mathring{A}^3$, Z=4, space group is $P2_1$.

The crystals of '$(NH_4)_2TeF_6$' (II) are tetragonal; a=13.754(2), c=5.9258(6)$\mathring{A}$, V=1120.9(5)$\mathring{A}^3$, Z=8, space group is $P\bar{4}2_1c$. Both structures were solved by Patterson and Fourier methods and refined by full-matrix LS technique. Non-H atoms were refined with anisotropic thermal factors. The final R values are 0.043 (compound I) and R=0.066 (compound II). H atoms were located from difference Fourier-synthesis (compound I), we could not locate H atoms in structure II. All calculations were done on an IBM PC/XT computer using a PC-SHELX-76 package. Atomic coordinates and equivalent isotropic thermal parameters are given in Tables 1 and 2, bond lengths and angles in Tables 3 - 5 , projections of structures are in Figs.1 and 2.

For synthesis of compound II interaction of NH_4F or NH_4HF_2 with TeO_2 in the concentrated (50%) solution of HF has been proposed [8]. We have made a preliminary study in the system $NH_4F(NH_4HF_2)$-TeO_2-$HF_{conc.}$ in the range of ratio $NH_4F(NH_4HF_2) : TeO_2$ from 0.1:1 to 5:1 with the identification of the obtained compounds by chemical and IR-spectroscopic analyses. It was found that in the range of ratio from 0.1:1 to 1:1 the compound NH_4TeF_5 was formed. In the presence of excess of NH_4F (ratio of NH_4F:TeO_2 2.5:5.1) the compound, in the shape of well faced crystals, was precipitated from the solution on crystallization. The salt obtained was recrystallized from HF. The needle-shaped crystals obtained were used for X-ray structural investigations.

Comparing the IR spectra of the synthesized compound with that of NH_4TeF_5 give preliminary data of its structure. In the region of frequencies of Te-F stretching both spectra are much alike indicating the presence of $[TeF_5]^-$anions in both compounds. The band is split into two components (635 and 620 cm^{-1}) in the IR spectrum obtained for NH_4TeF_5, which is in agreement with the presence of two crystallographically independent $[TeF_5]^-$ anions in the structure [12] .

The presence of only one unsplit band at 625 cm^{-1} in the IR spectra of '$(NH_4)_2TeF_6$' indicates, that only one type of anion $[TeF_5]^-$ is present. There are two additional bands of average intensity at 1200 cm^{-1} with several bends and a wide band at (1300-1800 cm^{-1}) with a maximum near 1500 cm^{-1} as compared with spectrum of NH_4TeF_5.

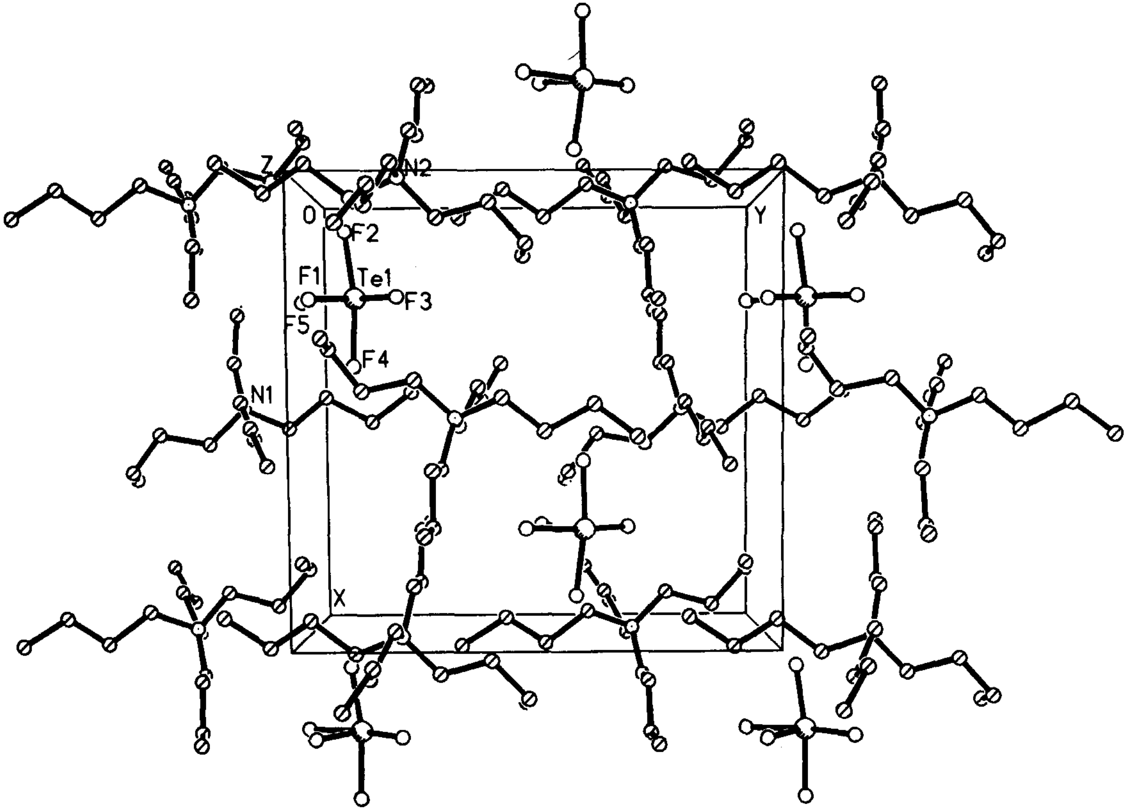

Fig. 1. The crystal structure of NBu_4TeF_5 viewed along Z-axis

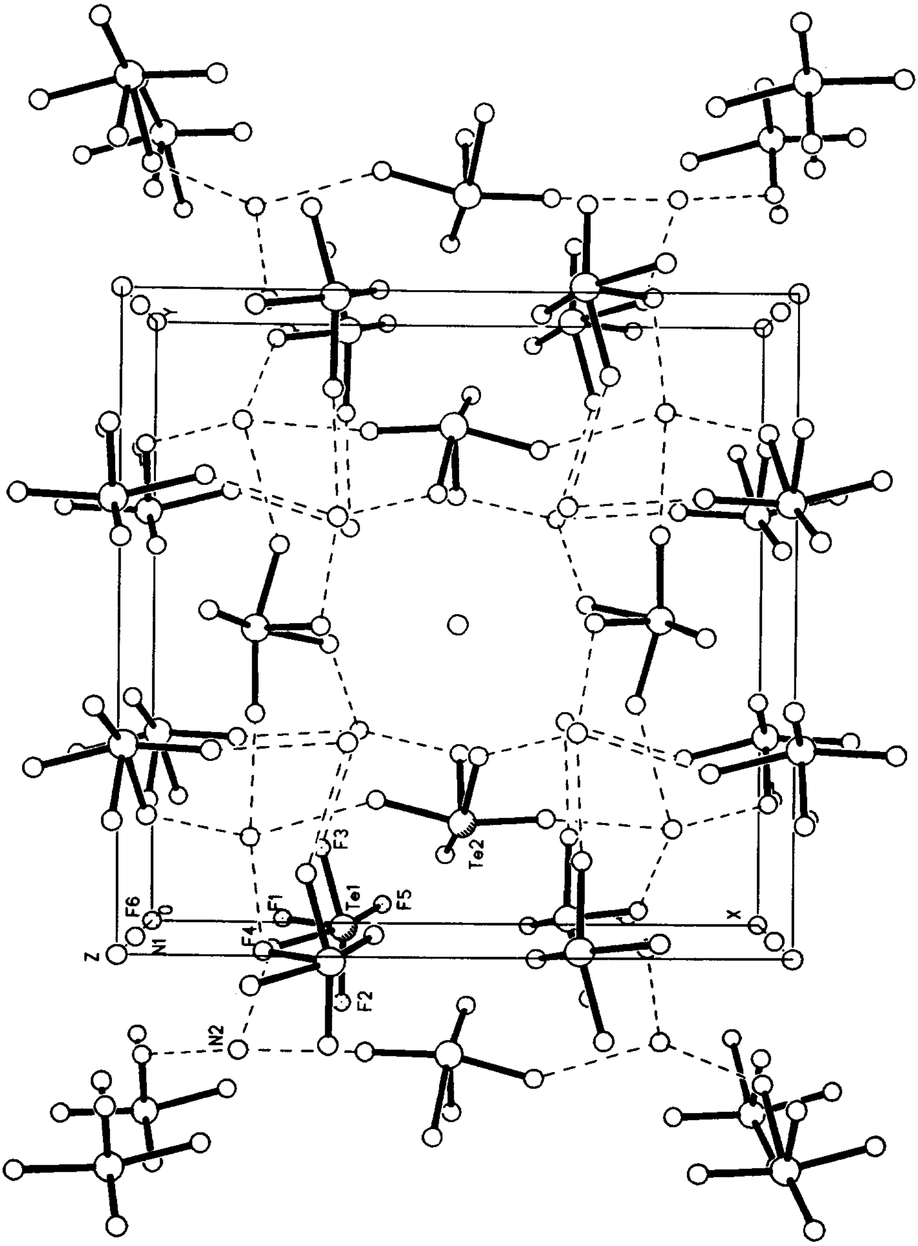

Fig. 2. The crystal structure of $NH_4TeF_5(NH_4F)_{0.25}$ viewed along Z-axis.

TABLE 1

Fractional atomic coordinates and

isotropic thermal parameters $U_{(eq)}$ of compound I

Atom	X/A	Y/B	Z/C	$U_{(eq)}$
TE(1)	0.2378(1)	0.1005(1)	0.4311(1)	0.0224
TE(2)	0.2608(1)	0.4338(1)	0.0678(1)	0.0253
F(1)	0.2442(6)	0.0070(6)	0.5441(6)	0.037
F(2)	0.0869(5)	0.0809(5)	0.4375(5)	0.041
F(3)	0.2388(7)	0.1989(7)	0.5470(6)	0.049
F(4)	0.3929(5)	0.0991(8)	0.4586(5)	0.053
F(5)	0.2435(5)	-0.0260(5)	0.3462(4)	0.033
F(6)	0.2430(7)	0.5299(7)	-0.0367(7)	0.052
F(7)	0.4117(6)	0.4613(7)	0.0569(6)	0.063
F(8)	0.2656(7)	0.3423(6)	-0.0535(5)	0.036
F(9)	0.1048(6)	0.4419(8)	0.0372(6)	0.053
F(10)	0.2598(6)	0.5578(6)	0.1533(6)	0.045
N(1)	0.4892(6)	-0.1711(6)	0.2403(6)	0.019
N(2)	0.9905(7)	0.2128(7)	0.7349(7)	0.025
C(1)	0.5346(8)	-0.0724(9)	0.2008(7)	0.025
C(2)	0.4609(9)	0.0159(9)	0.204(1)	0.028
C(3)	0.520(1)	0.1196(9)	0.1812(8)	0.036
C(4)	0.449(1)	0.2106(8)	0.176(1)	0.046
C(5)	0.5718(7)	-0.2538(7)	0.2203(5)	0.022
C(6)	0.549(1)	-0.365(1)	0.244(1)	0.037
C(7)	0.6390(9)	-0.4318(8)	0.2212(8)	0.029
C(8)	0.6591(9)	-0.439(1)	0.108(1)	0.046
C(9)	0.3794(9)	-0.199(1)	0.1786(8)	0.025
C(10)	0.3784(8)	-0.2110(8)	0.0561(7)	0.031
C(11)	0.2597(9)	-0.210(1)	0.008(1)	0.052
C(12)	0.247(1)	-0.221(1)	-0.111(1)	0.056
C(13)	0.4733(7)	-0.1670(7)	0.3538(7)	0.019
C(14)	0.559(1)	-0.1275(9)	0.4317(9)	0.028
C(15)	0.533(1)	-0.125(1)	0.5450(8)	0.031
C(16)	0.615(1)	-0.081(1)	0.6267(8)	0.055
C(17)	1.0318(8)	0.110(1)	0.7003(8)	0.028
C(18)	0.957(1)	0.014(1)	0.705(1)	0.033
C(19)	1.0188(9)	-0.0757(9)	0.6820(9)	0.038
C(20)	0.953(1)	-0.175(1)	0.680(1)	0.058
C(21)	0.8840(9)	0.2356(9)	0.6742(9)	0.023
C(22)	0.8808(9)	0.244(1)	0.5553(8)	0.029
C(23)	0.7654(8)	0.2469(7)	0.5051(7)	0.020
C(24)	0.7523(9)	0.2610(9)	0.3854(6)	0.035
C(25)	0.9687(9)	0.2035(9)	0.8557(8)	0.033
C(26)	1.0639(9)	0.154(1)	0.9296(8)	0.033
C(27)	1.031(1)	0.168(1)	1.0428(9)	0.043
C(28)	1.115(1)	0.110(2)	1.122(1)	0.052
C(29)	1.0761(9)	0.2905(8)	0.7215(8)	0.032
C(30)	1.0435(8)	0.4002(8)	0.7408(9)	0.027
C(31)	1.132(1)	0.477(1)	0.715(1)	0.042
C(32)	1.152(1)	0.4701(9)	0.5992(8)	0.046

TABLE 2

Fractional atomic coordinates and isotropic thermal parameters $U_{(eq)}$ of compound II

Atom	X/A	Y/B	Z/C	$U_{(eq)}$
Te(1)	0.3153(1)	-0.0090(1)	-0.0222(3)	0.0444
N(1)	0.0000(0)	0.0000(0)	-0.5000(0)	0.19
N(2)	0.161(1)	-0.178(1)	0.499(7)	0.06
F(1)	0.209(1)	-0.004(2)	-0.212(2)	0.09
F(2)	0.311(1)	-0.1422(8)	-0.126(3)	0.08
F(3)	0.279(1)	0.1255(8)	-0.007(4)	0.09
F(4)	0.204(1)	-0.033(1)	0.185(3)	0.10
F(5)	0.376(2)	0.018(2)	-0.296(4)	0.17
F(6)	0.000(0)	0.000(0)	0.000(0)	0.18

TABLE 3

Main interatomic distances d (Å) of compound I

Bond	d	Bond	d
TE(1)-F(1)	1.872(8)	TE(1)-F(2)	1.923(6)
TE(1)-F(3)	1.945(8)	TE(1)-F(4)	1.938(6)
TE(1)-F(5)	1.973(6)	TE(2)-F(6)	1.815(9)
TE(2)-F(7)	1.949(8)	TE(2)-F(8)	1.948(7)
TE(2)-F(9)	1.954(8)	TE(2)-F(10)	1.945(8)
N(1)-C(1)	1.52(1)	N(1)-C(5)	1.54(1)
N(1)-C(9)	1.54(1)	N(1)-C(13)	1.47(1)
N(2)-C(17)	1.52(2)	N(2)-C(21)	1.49(1)
N(2)-C(25)	1.59(1)	N(2)-C(29)	1.50(1)
C(1)-C(2)	1.48(2)	C(2)-C(3)	1.58(2)
C(3)-C(4)	1.49(2)	C(5)-C(6)	1.51(2)
C(6)-C(7)	1.49(2)	C(7)-C(8)	1.48(2)
C(9)-C(10)	1.55(1)	C(10)-C(11)	1.54(2)
C(11)-C(12)	1.49(2)	C(13)-C(14)	1.47(2)
C(14)-C(15)	1.51(2)	C(15)-C(16)	1.49(2)
C(17)-C(18)	1.56(2)	C(18)-C(19)	1.45(2)
C(19)-C(20)	1.53(2)	C(21)-C(22)	1.50(2)
C(22)-C(23)	1.51(2)	C(23)-C(24)	1.51(1)
C(25)-C(26)	1.57(2)	C(26)-C(27)	1.55(2)
C(27)-C(28)	1.56(2)	C(29)-C(30)	1.51(2)
C(30)-C(31)	1.56(2)	C(31)-C(32)	1.52(2)

TABLE 4

Main bond angles ω (°) of compound I

Angle	ω	Angle	ω
F(1)-TE(1)-F(2)	80.6(3)	F(1)-TE(1)-F(3)	81.9(4)
F(1)-TE(1)-F(4)	84.5(3)	F(1)-TE(1)-F(5)	82.5(3)
F(2)-TE(1)-F(3)	88.6(3)	F(2)-TE(1)-F(4)	164.9(3)
F(2)-TE(1)-F(5)	90.6(3)	F(3)-TE(1)-F(4)	87.3(4)
F(3)-TE(1)-F(5)	164.3(3)	F(4)-TE(1)-F(5)	89.4(3)
F(6)-TE(2)-F(7)	81.9(4)	F(6)-TE(2)-F(8)	82.1(4)
F(6)-TE(2)-F(9)	77.5(4)	F(6)-TE(2)-F(10)	79.8(4)
F(7)-TE(2)-F(8)	86.4(3)	F(7)-TE(2)-F(9)	159.4(4)
F(7)-TE(2)-F(10)	87.5(3)	F(8)-TE(2)-F(9)	90.0(3)
F(8)-TE(2)-F(10)	161.5(3)	F(9)-TE(2)-F(10)	89.6(3)
C(1)-N(1)-C(5)	104.7(7)	C(1)-N(1)-C(9)	112.3(8)
C(1)-N(1)-C(13)	112.9(7)	C(5)-N(1)-C(9)	109.5(7)
C(5)-N(1)-C(13)	110.9(7)	C(9)-N(1)-C(13)	106.6(7)
C(17)-N(2)-C(21)	110.1(8)	C(17)-N(2)-C(25)	108.1(8)
C(17)-N(2)-C(29)	106.9(8)	C(21)-N(2)-C(25)	105.3(8)
C(21)-N(2)-C(29)	114.7(8)	C(25)-N(2)-C(29)	111.5(8)
N(1)-C(1)-C(2)	113.0(9)	C(1)-C(2)-C(3)	110.5(9)
C(2)-C(3)-C(4)	113(1)	N(1)-C(5)-C(6)	119.3(8)
C(5)-C(6)-C(7)	111(1)	C(6)-C(7)-C(8)	116(1)
N(1)-C(9)-C(10)	115.4(9)	C(9)-C(10)-C(11)	106.8(9)
C(10)-C(11)-C(12)	112(1)	N(1)-C(13)-C(14)	119.6(8)
C(13)-C(14)-C(15)	115(1)	C(14)-C(15)-C(16)	117(1)
N(2)-C(17)-C(18)	118(1)	C(17)-C(18)-C(19)	107(1)
C(18)-C(19)-C(20)	113(1)	N(2)-C(21)-C(22)	116.5(9)
C(21)-C(22)-C(23)	109.5(9)	C(22)-C(23)-C(24)	114.1(9)
N(2)-C(25)-C(26)	113.5(9)	C(25)-C(26)-C(27)	103.6(9)
C(26)-C(27)-C(28)	108(1)	N(2)-C(29)-C(30)	114.1(9)
C(29)-C(30)-C(31)	111.0(9)	C(30)-C(31)-C(32)	111(1)

TABLE 5

Main interatomic distances d (Å) and bond angles ω (°) of compound II

Bond	d	Angle	ω
Te(1) - F(1)	1.85(2)	F(1)--Te(1)-F(2)	79.8(7)
Te(1) - F(2)	1.93(1)	F(1)-Te(1)-F(3)	77.3(8)
Te(1) - F(3)	1.92(1)	F(1)-Te(1)-F(4)	76.9(8)
Te(1) - F(4)	1.99(2)	F(1)-Te(1)-F(5)	79.3(9)
Te(1) - F(5)	1.86(2)	F(2)-Te(1)-F(3)	157.0(7)
Te(1)....F(2)	3.45(2)	F(2)-Te(1)-F(4)	90.9(7)
Te(1)....F(5)	3.47(2)	F(2)-Te(1)-F(5)	85.4(9)
Te(1)....F(5)	3.23(2)	F(3)-Te(1)-F(4)	86.1(7)
		F(3)-Te(1)-F(5)	88.1(9)
		F(4)-Te(1)-F(5)	156.2(9)

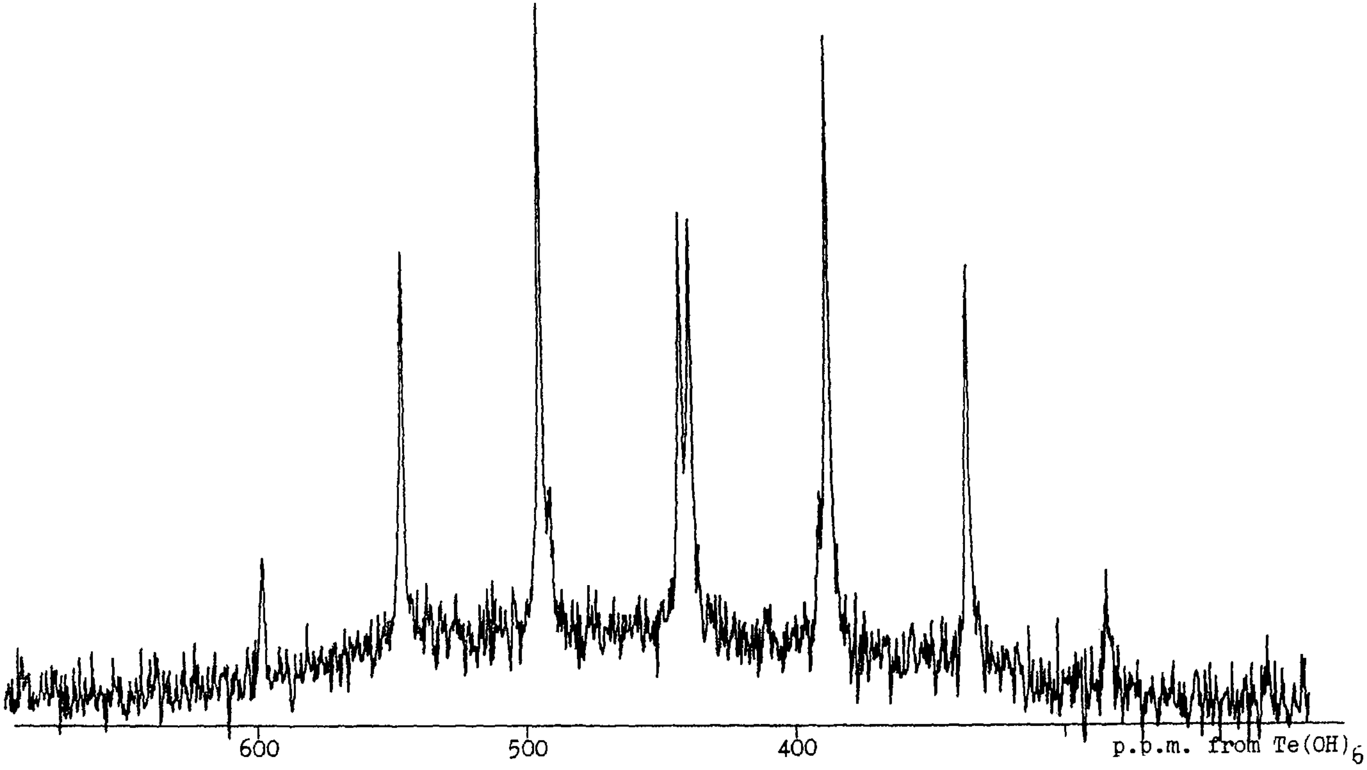

Fig. 3. ^{125}Te NMR spectrum of NBu_4TeF_5 in acetonitrile.

The ^{19}F NMR spectrum of compound I in solution consists of a doublet and quintet; the ^{125}Te NMR spectrum is a doublet of quintets. The patterns of ^{19}F and ^{125}Te NMR spectra is caused by pseudooctahedral configuration of the $[TeF_5]^-$ anion (four fluorine atoms lying in a equatorial plane, equivalent to each other and unequivalent to axial). The spin-spin interaction for axial (J_{Te-F_a}= 2911 Hz) and equatorial bonds (J_{Te-F_e}= 1380 Hz) differ considerably, which allows us to assume that axial and equatorial bonds of particle $[TeF_5]^-$ are unequivalent (Fig. 3).

The ^{19}F NMR spectrum of a solution of compound II in DMFA also consists of a doublet and quintet with a ratio of integral intensities 4:1. In addition a broad signal (low intensity) in the range of F^- chemical shifts is observed . The pattern of ^{19}F NMR spectra indicates that only $[TeF_5]^-$ anion and F^- ion in insignificant concentration are present in the solution of DMFA. No other signals (including those, which could correspond to complex anion $[TeF_6]^{2-}$) were found in the spectrum of compound II. No evidence for $[TeF_6]^{2-}$ in the solution of compound II was obtained.

The crystal structure of NBu_4TeF_5

The X-ray investigation revealed that structure I is built up of isolated complex ions of $[TeF_5E]^-$ (E= lone pair) and NBu_4^+ cations (Fig. 1). It was found that unit cell contains two crystallographically independent ions of each type. In the pseudooctahedral Te(1) and Te(2) complexes the axial bonds (1.872(8) and 1.815(9)Å) are shorter than the equatorial ones (1.923(6) - 1.973(6)Å).The Te atoms are shifted from the equatorial plane of pseudooctahedra by 0.26(1) and 0.34(2) Å towards the lone pair.

Angles F_a-Te-F_e (77.7(4) - 84.5(3)°) are significantly less than ideal 90°. The Te atoms have weak intermolecular (Van der Waals) contacts only with the carbon atoms of NBu_4^+ ions. The geometry of the $[TeF_5E]^-$ ions is rather similar to that found in the structure of NH_4TeF_5 [12]. The small difference in bond lengths Te-F_e from 1.910(8) to 2.055(8)Å in $[TeF_5E]^-$ can be explained by the participation of F atoms in an interaction with the NH_4^+ions. It leads to the considerable deflection of angles H-N-H from tetrahedral (78.5 - 121.3°). The geometry of the N atoms in structure I is as expected close to ideal tetrahedral: C-N-C angles lie between 104.7(7) and 114.7(9)°. C-N and C-C distances in NBu_4^+ ions are in the range of 1.45(2) - 1.59(2)Å, the mean C-N and C-C distances 1.51(2) - 1.53(2) Å are equal within the experimental error.

The crystal structure of $NH_4TeF_5(NH_4F)_{0.25}$

It was finally found that the composition of crystals of compound II is $NH_4TeF_5(NH_4F)_{0.25}$ and the hypothetical anion $[TeF_6]^{2-}$ is not formed. The tellurium complex has a pseudooctahedral configuration, analogous to those found in the structures NH_4TeF_5 [12] and $KTeF_5$ [13]. The axial Te-F bond is shorter than the equatorial (Table 3). The Te atom is shifted (0.40(4) Å) from the equatorial plane. The $N(2)H_4^+$ cation is surrounded by eight F atoms with the average distance N(2) - F 3.0 Å. Four shorter contacts N(2)-F have values 2.79(3) - 2.87(3) Å and can be interpreted as hydrogen bonds N-H-F. The $[TeF_5]^-$ anions are grouped around centers in the direction 1/2,0,Z (0,1/2,Z). The lone pair of Te atoms are directed inside thus forming channels (Fig. 2). The larger channels in the directions 0,0,Z (1/2,1/2,Z respectively) contain ions of $N(1)H_4^+$ and F^- (analogous channels occupied by neutral molecules of HCl and H_2O were found in the structure of $NH_4TeF_5 \cdot (HCl)_{0.25} \cdot (H_2O)_{0.25}$ [10]).The 'Channel' NH_4^+ cation is surrounded by ten 'neighbours'. Among the N(1)-F contacts two shortest distances (2.98(3)Å x 2) correspond to the bonds with the 'channel' F^- ion. The unusual shortening of the Te-F(5) bond 1.86(2)Å should be noted (analogous shortening of the bond with one of the equatorial ligands was noted in the work [10]). The possible cause is that F(5) atom has no contacts with NH_4^+ cations.

CONCLUSION

The **existence** of $(NH_4)_2TeF_6$ under the conditions described is not confirmed and the crystals obtained by us and in [8] (the same group of symmetry) are $NH_4TeF_5(NH_4F)_{0.25}$. It was shown that in both structures as well as in $MTeF_5$ compounds (M= NH_4^+, K^+, Rb^+, Tl^+) [14] pseudooctahedral configuration of Te-complexes occurs. In all cases in the Te-complexes axial bonds are shortened in comparison with equatorial and the angles between valent bonds at the Te atom are decreased in comparison with the same in ideal configuration. In mixed compounds $K_2TeF_5 \cdot L$ (L= NO_3^-,Cl^-) [14] pseudooctahedral configuration of Te-complexes is preserved and L- ligands are situated on the lone pair side at long distances within the second coordination sphere of Te atom.

In all known structures of tellurium (IV) fluorides the lone pair of electrons is highly stereoactive. Evidently, due to this reason the formation of octahedral $[TeF_6]^{2-}$ ion similar to the known octahedral complexes of $[TeCl_6]^{2-}$ and $[TeBr_6]^{2-}$ is hardly probable [14].

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