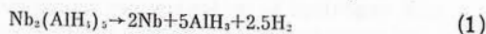


exothermic effect at 595°C, without release of gases, which corresponds to fusion of the Al-Nb eutectic<sup>4</sup>.

The thermal decomposition of Nb<sub>2</sub>(AlH<sub>4</sub>)<sub>5</sub> was studied. The precipitate filtered off at -78°C was outgassed in a Töpler pump<sup>1,2</sup> and kept for a long time at -78°, 0°, and 25°C, then decomposed with dilute (1:4) sulphuric acid. The results obtained (Table) show that at 0°C the amount of hydrogen released corresponds approximately to decomposition into metallic niobium and aluminium hydride:



Thermal decomposition of Nb<sub>2</sub>(AlH<sub>4</sub>)<sub>5</sub>.

Temperature, °C	Time kept, h	H <sub>2</sub> released, %	
		found	calculated
-78	8	0	—
0	8	11	14 (1)
25	64	32	43 (2)
H <sub>2</sub> SO <sub>4</sub>	—	57	43

However, the AlH<sub>3</sub> formed starts to decompose into the elements under the catalytic action of metallic niobium even at room temperature:



Dissolving the residue in dilute H<sub>2</sub>SO<sub>4</sub> showed that reaction (2) proceeds extremely slowly. Keeping the product at room temperature for a month leads to complete decomposition of Nb<sub>2</sub>(AlH<sub>4</sub>)<sub>5</sub> into the elements.

Nb<sub>2</sub>(AlH<sub>4</sub>)<sub>5</sub> is a dark red powder which burns in air and in the presence of moisture, does not dissolve in diethyl ether or tetrahydrofuran. The X-ray diffraction pattern recorded at liquid nitrogen temperature shows that the substance is amorphous to X-rays. On the infrared spectrum recorded in a cryostat on a UR-10 instrument there is an absorption band at 1830 cm<sup>-1</sup> which seems to belong to the Al-H stretching vibrations<sup>5</sup>.

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## Spectrophotometric Determination of the Formation Constants of Tellurium(IV) Hydroxo-complexes

V. A. Nazarenko, G. G. Shitareva, and E. N. Poluektov

The formation constants of the monomeric tellurium(IV) hydroxo-complexes in the range of ionic strengths 0.1-1.0 have been determined spectrophotometrically with 2,3,7-trihydroxy-9-pentadecyl-6-fluorone as competing ligand and the hydrolysis constants of its ions have been calculated. Interpolation equations for calculating the hydrolysis constants in this range of *I* are derived.

Tellurium(IV) oxide TeO<sub>2</sub> is amphoteric and this must be borne in mind in studying the behaviour of tellurium as a complex-forming substance. The state of tellurium(IV) in aqueous solutions has been studied repeatedly, but the halogeno-complexes or the behaviour of TeO<sub>2</sub> as an acid has been studied most.

Kasarnowsky<sup>1</sup> found the existence of the Te<sup>4+</sup> cation in acid solutions. He studied the behaviour of TeO<sub>2</sub> as a base<sup>2</sup> and determined the "apparent" cumulative basicity constant of tellurous acid. In subsequent studies of the cationic tellurium(IV) hydroxo-complexes different investigators obtained different results. They determined either individual constants or a series of constants under conditions that were not comparable (Table 1). There has been no systematic determination of the constants which characterise TeO<sub>2</sub> as a base under comparable conditions.

Table 1. Constants of the tellurium(IV) hydroxo-complexes.

Method	Values of the constants	Reference
Solubility, 18°C	$K_{\text{bas.d.}} = 1.5 \times 10^{-46}$ $K_1 \text{ bas.d.}$	2
Solubility	$K_{\text{bas.d.}} = 8.9 \times 10^{-17}$	3
Solubility	$\text{p}K_{\text{bas.}} = 10.5$	4
Distribution	$\text{p}K_{\text{a0}} = \text{p}K_{\text{4h}} = 3.16$	
<i>I</i> = 0.1		
<i>I</i> = 3.0	$\text{p}K_{\text{4h}} = 4.17$	5
Solubility		
<i>I</i> = 0.5	$\beta_4 = (2.1 \pm 0.6) \times 10^{12}$	6
Comparative dialysis		
<i>I</i> = 2.5	$\beta_{1,2} = 6.3 \times 10^{28}$	
<i>I</i> = 1.0	$\beta_3 = 1.0 \times 10^{14}$	7
<i>I</i> = 0.5	$\beta_4 = 1.7 \times 10^{12}$	

This paper describes an attempt to determine the formation constants of the cationic tellurium(IV) hydroxo-complexes under comparable conditions, over a wide range of the ionic strengths of the solutions, and from these to calculate the hydrolysis constants of its cations. We chose as working method spectrophotometry, which is used for determining the hydrolysis constants of the monomeric ions of other elements of high oxidation state<sup>8-10</sup>.

2,3,7-Trihydroxy-9-pentadecyl-6-fluorone (pentadecylfluorone) was used as competing ligand. The experiments were carried out under conditions which prevented the formation of tellurium complexes other than those with trihydroxyfluorone or  $\text{OH}^-$  ions.

Tellurium(IV) reacts with trihydroxyfluorones in weakly acid solutions, to form sparingly soluble compounds, which, however, remain in a colloidal state at low concentrations<sup>11,12</sup>. Stabilised gelatinous colloidal solutions of the trihydroxyfluorone complexes of tellurium remain transparent and obey Beer's law over a wide range of concentrations. It was found spectrophotometrically that tellurium forms a 1:1 complex with pentadecylfluorone at pH 2-6. The solutions of the tellurium pentadecylfluorone complex obey Beer's law in the concentration range  $2 \times 10^{-6}$ - $2 \times 10^{-5}$  g-atom Te litre<sup>-1</sup> ( $\lambda_{\text{opt}} = 510$  nm). The optical density of the solutions of the complex reaches a maximum after 4 h and then does not alter in the following 24 h.

Table 2. Initial calculated values of  $\ln S$  at  $I = 0.1$ .  $2 \times 10^{-5}$  g-ion Te litre<sup>-1</sup>,  $8 \times 10^{-5}$  M pentadecylfluorone, 10% ethanol,  $\lambda = 510$  nm,  $l = 10$  mm,  $D_{\text{max}} = 0.840$  at pH 5.85;  $pK_{\text{W}} = 13.8962$ ,  $28^\circ\text{C}$ .

pH	$10^{11}[\text{OH}^-]$	$D$	$10^5 c_c$	$-\ln S$
2.70	0.63	0.090	0.21	107.57
2.85	0.90	0.130	0.30	105.78
3.05	1.42	0.150	0.36	103.86
3.23	2.01	0.180	0.43	102.22
3.30	2.53	0.190	0.45	101.20
3.50	4.02	0.212	0.50	99.56
3.60	5.06	0.223	0.53	98.72

Since the trihydroxyfluorones in acid solutions react as monobasic acids with cations of elements<sup>8</sup> but tellurium forms a sparingly soluble compound with pentadecylfluorone, presumably the complex-forming substance in this system is the singly-charged hydroxo-cation  $\text{Te}(\text{OH})_3^+$ . Then  $S$  in the main equation of the spectrophotometric method for determining the formation constant of the hydroxo-complexes of the element:

$$\frac{d \ln S}{d[\text{OH}^-]} = \frac{\bar{n}}{[\text{OH}^-]}$$

must be calculated, by starting from the solubility product of the complex:

$$\text{SP} = [\text{Te}(\text{OH})_3^+][\text{H}_2\text{F}^-]$$

and the value of  $S$  in this instance can be expressed by the equation:

$$S = \frac{[\text{OH}^-]^2 [c_{\text{Te}} - c_c] [c_{\text{L}} - c_c] K_0 K_1}{[\text{H}^+]^2 + K_0 [\text{H}^+] + K_0 K_1}$$

where  $c_{\text{Te}}$  and  $c_{\text{L}}$  are the total concentrations of tellurium and pentadecylfluorone,  $c_c$  the concentration of the complex calculated from the equation

$$c_c = c_{\text{Te}} \cdot D / D_{\text{max}} \cdot D$$

$D$  and  $D_{\text{max}}$  are the optical densities at the given pH of the solution and at complete bonding of the tellurium in the

complex respectively;  $K_0$  and  $K_1$  the ionisation constants of pentadecylfluorone<sup>13</sup>,

$$pK_0 = 3.15 - 0.29\sqrt{I};$$

and

$$pK_1 = 6.51 - 0.28\sqrt{I}.$$

( $I = 0.1-1.0$ ).

Table 3. Values of  $-\ln S$  at  $I = 0.3-1.0$ .

pH	$10^{11}[\text{OH}^-]$	$I = 0.3$	$I = 0.5$	$I = 0.7$	$I = 1.0$
2.75	0.71	106.56	106.22	106.15	105.86
2.85	0.90	105.55	105.18	105.14	104.92
3.00	1.27	104.41	103.72	103.52	103.46
3.10	1.60	103.41	102.73	102.65	102.46
3.20	2.01	102.40	101.76	101.66	101.48
3.30	2.53	101.42	100.78	100.68	100.51
3.55	4.50	98.72	98.84	98.73	98.58

The quantity  $n/[\text{OH}^-]$  is determined graphically as the slope of the tangent at the given point on the curve of  $\ln S$  against  $[\text{OH}^-]$ . In calculating the formation constants of the tellurium complexes the auxiliary functions  $G_1$ ,  $G_2$ ,  $G_3$ , and  $G_4$  were obtained by Fomin and Maiorova's method<sup>14</sup>. In every instance the  $G_i([\text{OH}^-])$  functions were extrapolated to zero  $\text{OH}^-$  concentration by the linear equation

$$1/G_i = a_i + b_i[\text{OH}^-]$$

by the least squares method.

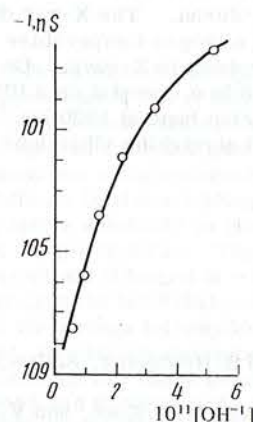


Figure 1. Variation of  $-\ln S$  with  $[\text{OH}^-]$ ;  $I = 0.1$ .

#### EXPERIMENTAL

The initial  $1 \times 10^{-2}$  M  $\text{TeO}_2$  solution was made up by dissolving a weighed portion of special purity metallic tellurium in  $\text{HNO}_3$ . The solution was evaporated to dryness, then twice evaporated with water to dryness and the

dry residue dissolved in 1 N  $\text{HClO}_4$ . The working solutions were prepared when required by diluting the initial one.  $2 \times 10^{-3}$  M pentadecylfluorone solution was prepared by dissolving a weighed portion of the reagent in 96% ethanol with addition of 0.5 ml 6 N  $\text{HCl}$  to a volume of 100 ml. The solutions examined on the photometer contained  $2 \times 10^{-5}$  g-ion Te litre<sup>-1</sup>,  $8 \times 10^{-5}$  M pentadecylfluorone, 0.04% gelatin, and 10% ethanol. The ionic strength of the solution was maintained by adding potassium nitrate. The necessary concentration of hydrogen ions was maintained by a glycol buffer. The hydrogen ion concentration was measured with a glass electrode on an LPU-01 pH-meter. The optical density of the solutions was measured with an SF-4A spectrophotometer. The temperature was held at  $(28 \pm 0.5)^\circ\text{C}$ .

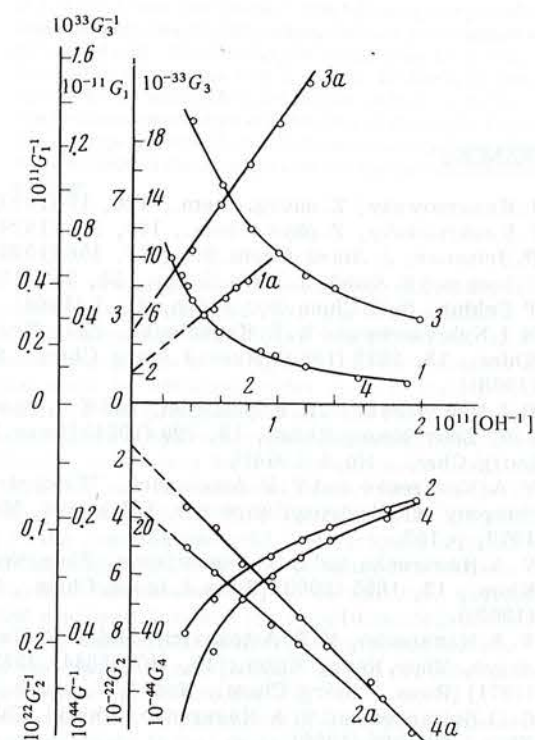


Figure 2. Determination of the formation constants  $\beta_i$  of the tellurium(IV) hydroxo-complexes,  $I = 0.1$ . 1) and 1a)  $\beta_1$ ; 2) and 2a)  $\beta_2$ ; 3) and 3a)  $\beta_3$ ; 4) and 4a)  $\beta_4$ .

Table 2 gives the initial experimental results for calculating  $\ln S$  and Table 3 the  $\ln S$  at various  $I$ . Fig. 1 shows the variation of  $-\ln S$  with  $[\text{OH}^-]$  and Fig. 2 the variation of the auxiliary functions  $G_1$ ,  $G_2$ ,  $G_3$ , and  $G_4$  at ionic strength 0.1.

The extrapolation of these auxiliary functions to zero  $\text{OH}^-$  concentration was carried out by the equations:

$$I=0.1$$

$$1/G_1 = 0.11 \cdot 10^{-14} + 0.15 [\text{OH}^-] \quad ([\text{OH}^-] = 1.0 \cdot 10^{-11} - 2.0 \cdot 10^{-11})$$

$$1/G_2 = -6.44 \cdot 10^{-24} - 12.95 \cdot 10^{-13} [\text{OH}^-] \quad ([\text{OH}^-] = 0.4 \cdot 10^{-11} - 1.2 \cdot 10^{-11})$$

$$1/G_3 = 2.44 \cdot 10^{-35} + 7.30 \cdot 10^{-24} [\text{OH}^-] \quad ([\text{OH}^-] = 0.4 \cdot 10^{-11} - 1.2 \cdot 10^{-11})$$

$$1/G_4 = -5.34 \cdot 10^{-17} - 2.65 \cdot 10^{-35} [\text{OH}^-] \quad ([\text{OH}^-] = 0.6 \cdot 10^{-11} - 2.0 \cdot 10^{-11})$$

$$I=0.3$$

$$1/G_1 = 0.81 \cdot 10^{-12} + 1.88 \cdot 10^{-1} [\text{OH}^-] \quad ([\text{OH}^-] = 0.8 \cdot 10^{-11} - 2.0 \cdot 10^{-11})$$

$$1/G_2 = -3.62 \cdot 10^{-24} - 7.82 \cdot 10^{-13} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.2 \cdot 10^{-11})$$

$$1/G_3 = 1.72 \cdot 10^{-35} + 3.55 \cdot 10^{-24} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.2 \cdot 10^{-11})$$

$$1/G_4 = -0.79 \cdot 10^{-14} - 1.76 \cdot 10^{-35} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.2 \cdot 10^{-11})$$

$$I=0.5$$

$$1/G_1 = 5.98 \cdot 10^{-12} + 22.17 \cdot 10^{-2} [\text{OH}^-] \quad ([\text{OH}^-] = 1.2 \cdot 10^{-11} - 3.0 \cdot 10^{-11})$$

$$1/G_2 = -1.53 \cdot 10^{-24} - 5.91 \cdot 10^{-13} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.2 \cdot 10^{-11})$$

$$1/G_3 = 3.62 \cdot 10^{-36} + 1.55 \cdot 10^{-23} [\text{OH}^-] \quad ([\text{OH}^-] = 0.4 \cdot 10^{-11} - 1.2 \cdot 10^{-11})$$

$$1/G_4 = -8.41 \cdot 10^{-18} - 3.57 \cdot 10^{-36} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.2 \cdot 10^{-11})$$

$$I=0.7$$

$$1/G_1 = 4.85 \cdot 10^{-12} + 18.95 \cdot 10^{-2} [\text{OH}^-] \quad ([\text{OH}^-] = 0.6 \cdot 10^{-11} - 1.6 \cdot 10^{-11})$$

$$1/G_2 = -10.88 \cdot 10^{-24} - 4.99 \cdot 10^{-13} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.4 \cdot 10^{-11})$$

$$1/G_3 = 2.26 \cdot 10^{-36} + 0.11 \cdot 10^{-23} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.4 \cdot 10^{-11})$$

$$1/G_4 = -6.57 \cdot 10^{-18} - 1.99 \cdot 10^{-36} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.4 \cdot 10^{-11})$$

$$I=1.0$$

$$1/G_1 = 3.29 \cdot 10^{-12} + 23.78 \cdot 10^{-2} [\text{OH}^-] \quad ([\text{OH}^-] = 1.2 \cdot 10^{-11} - 2.2 \cdot 10^{-11})$$

$$1/G_2 = -4.45 \cdot 10^{-24} - 32.99 \cdot 10^{-13} [\text{OH}^-] \quad ([\text{OH}^-] = 0.4 \cdot 10^{-11} - 1.4 \cdot 10^{-11})$$

$$1/G_3 = 3.10 \cdot 10^{-37} + 0.27 \cdot 10^{-24} [\text{OH}^-] \quad ([\text{OH}^-] = 0.4 \cdot 10^{-11} - 1.4 \cdot 10^{-11})$$

$$1/G_4 = -1.82 \cdot 10^{-17} - 0.33 \cdot 10^{-36} [\text{OH}^-] \quad ([\text{OH}^-] = 0.2 \cdot 10^{-11} - 1.4 \cdot 10^{-11})$$

The auxiliary functions obtained were used to calculate the formation constants of the hydroxo-complexes and the hydrolysis constants of the tellurium(IV) ions. Table 4 gives the results of these calculations. The least squares method was used to calculate the equations of the linear relations between  $pK_{hi}$  and  $I$  (Fig. 3):

$$pK_{h1} = 1.980 - 0.577 I, \quad I = 0.1-1.0,$$

$$pK_{h2} = 2.357 - 0.513 I, \quad I = 0.1-1.0,$$

$$pK_{h3} = 2.632 - 0.473 I, \quad I = 0.1-0.7,$$

$$pK_{h4} = 2.925 - 0.450 I, \quad I = 0.1-0.5.$$

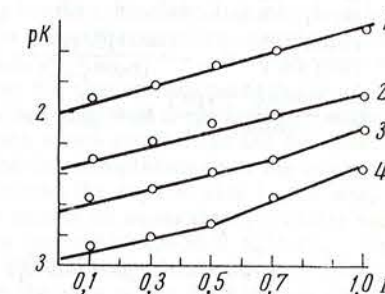


Figure 3. Variation of  $pK_{hi}$  of the tellurium ions with  $I$ . 1)  $pK_{h1}$ ; 2)  $pK_{h2}$ ; 3)  $pK_{h3}$ ; 4)  $pK_{h4}$ .

#### DISCUSSION

The hydrolysis constants of the tellurium(IV) ions we have calculated depend linearly on the ionic strength of the solutions for  $pK_1$  and  $pK_2$  in the range 0.1-1.0; for  $pK_3$ ,  $I = 0.1-0.7$ ; and for  $pK_4$  only for  $I = 0.1-0.5$ . Similar behaviour had been observed for the germanium hydroxo-complexes<sup>9</sup>. On assuming that the linear variation of  $pK_1$  will also be observed at  $I < 0.1$ , then at  $I = 0$  the hydrolysis constants of the tellurium ions will be:

$$K_{h1}^0 = 1.05 \cdot 10^{-2} \quad K_{h2}^0 = 2.33 \cdot 10^{-2},$$

$$K_{h3}^0 = 4.40 \cdot 10^{-3} \quad K_{h4}^0 = 1.19 \cdot 10^{-2}.$$

Fig. 4 shows the distribution of the various tellurium hydroxo-complexes as a function of the concentration of  $\text{H}^+$  ions.

The cumulative constant for the basicity of  $\text{TeO}_2$  which we obtained,  $1.4 \times 10^{-46}$  ( $I = 0.1$ ) agrees very well with the constant found by Kasarnowsky<sup>2</sup>,  $1.5 \times 10^{-46}$ . The distribution of the various tellurium hydroxo-complexes as a function of  $[\text{H}^+]$  reported by Sekine<sup>5</sup> does not agree with that we found (Fig. 4), which is quite understandable, since Sekine<sup>5</sup> did not take into account all the formation constants of the tellurium hydroxo-complexes and the first ionisation constant of tellurous acid was too high. On calculating the concentration of  $\text{HTeO}_3^-$  ions in the solution at various concentrations of  $\text{H}^+$  ions, using our<sup>15</sup> ionisation constant of  $\text{H}_2\text{TeO}_3$  ( $K_1 = 1.2 \times 10^{-7}$ ) and also the constant found by other investigators<sup>16</sup>, it seems that  $[\text{HTeO}_3^-]$  in the solution is 0.01% at pH 4, 0.37% at pH 4.5, and 1.27% at pH 5. This corresponds to the diagram in Fig. 4 and agrees with Issa and Awad's observation<sup>4</sup> of an isoelectronic point for  $\text{TeO}_2$  solutions at pH  $\approx 4$ .

Table 4. Characteristics of the tellurium hydroxo-complexes at 28°C,  $pK_w = 13.8962$ .

Constant	Ionic strength of the solution				
	0.1	0.3	0.5	0.7	1.0
$10^{-12}K_{h1}$	0.909	1.235	1.695	2.062	3.040
$10^{-24}K_{h2}$	0.336	0.625	1.110	1.666	3.497
$10^{-35}K_{h3}$	0.683	1.627	3.497	6.652	23.410
$10^{-46}K_{h4}$	0.709	2.206	5.420	15.630	89.260
$10^2K_{bas.1}$	1.15	1.51	2.15	2.62	3.86
$10^2K_{bas.2}$	0.47	0.64	0.83	1.03	1.38
$10^3K_{bas.3}$	2.58	3.30	4.00	5.06	8.49
$10^3K_{bas.4}$	1.30	1.71	1.96	2.97	4.83
$10^{12}K_{bas.1}$	9.62	7.38	6.45	4.25	2.62
$10^{12}K_{bas.2}$	4.91	3.84	3.17	2.55	1.49
$10^{12}K_{bas.3}$	2.70	1.98	1.52	1.23	0.86
$10^{12}K_{bas.4}$	1.10	0.81	0.59	0.48	0.32

$$\beta_i = \frac{[\text{Te}(\text{OH})_i^{(4-i)+}]}{[\text{Te}^{4+}][\text{OH}^-]^i}; K_{bas. i} = \frac{[\text{Te}(\text{OH})_{4-i}^{(4-i)+}][\text{OH}^-]}{[\text{Te}(\text{OH})_i^{(4-i)+}]};$$

$$K_{hi} = \frac{[\text{Te}(\text{OH})_i^{(4-i)+}][\text{H}^+]}{[\text{Te}(\text{OH})_{i-1}^{(3-i)+}]}; K_{hi} = \frac{\beta_i}{\beta_{i-1}} K_w$$

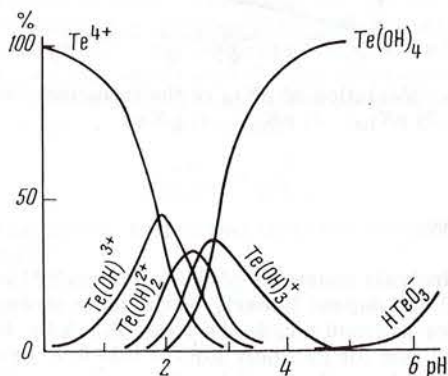


Figure 4. Distribution of the hydroxo-complexes as a function of  $[\text{H}^+]$ ;  $I = 0.1$ .

On allowing for the hydrolysis constants of the tellurium ions we calculated the mechanism of interaction of tellurium with pentadecylfluorone by a published method (p.43 of Ref. 8). This showed the complex-forming species to be

the cation  $\text{Te}(\text{OH})_3^+$ . Hence the reverse calculation confirmed the correctness of the reaction mechanism based on the calculated formation constants of the tellurium hydroxo-complexes. The solubility product calculated from our observed hydrolysis constants of the tellurium(IV) ions and the ionisation constants of pentadecylfluorone is  $(16.98 \pm 0.87) \times 10^{-21}$ .

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