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Original Scientific Paper

**Radiometric Determination of  $\text{AgI-TII-(}^{131}\text{I)}$ ,  $\text{AgI-HgI}_2\text{-(}^{131}\text{I)}$ ,  
and  $\text{HgI}_2\text{-TII-(}^{131}\text{I)}$  Systems***R. Despotović, Lj. A. Despotović, Z. Despotović, N. Filipović, Z. Grabarić,  
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The principle and procedure for radioanalytical determination of intermolecular interactions in a polycomponent system of »solid-liquid« type is described. As models  $\text{AgI-TII-(}^{131}\text{I)}$ ,  $\text{AgI-HgI}_2\text{-(}^{131}\text{I)}$ , and  $\text{HgI}_2\text{-TII-(}^{131}\text{I)}$  systems were used. The  $\text{AgI-TII-(}^{131}\text{I)}$  system was analysed in detail and radioanalytical results obtained were compared using X-ray analysis, conductivity and micro differential thermal analysis. Of the other two systems the results obtained were compared only with the results of X-ray analysis. As all the results obtained agree with literature data, it is concluded that the described radioanalytical method is applicable for adequate analysis of intermolecular interactions in mixed systems.

## INTRODUCTION

Many results of heterogeneous exchange of radioiodide  $^{131}\text{I}^-$  are obtained in the systems with silver iodide suspensions<sup>1</sup>. All the results obtained show a marked influence of various experimental conditions on the course of the radioiodide fixation. In the presence of thallium the precipitate of silver iodide changes its properties, and between  $\text{AgI}$  and  $\text{TII}$  intermolecular compounds were formed<sup>2,3</sup>. Since the solubility of silver iodide, thallos iodide<sup>4,5</sup>, and the compounds between  $\text{AgI}$  and  $\text{TII}$  is not equal, the equilibrium radioactivities in fixation processes could be determined only from good solubility curves for the systems with various molar fractions of  $\text{Ag}$  or  $\text{Tl}$ . In order to determine the solubility curves and for the analysis of the data obtained in mixed systems some radioanalytical results for three mixed systems  $\text{AgI-TII-(}^{131}\text{I)}$ ,  $\text{AgI-HgI}_2\text{-(}^{131}\text{I)}$  and  $\text{HgI}_2\text{-TII-(}^{131}\text{I)}$  are given in the present paper and compared with literature data<sup>2,3,9-11</sup>.

## EXPERIMENTAL

*Materials*

The standard solution of  $\text{Hg}(\text{NO}_3)_2$  was prepared by dissolution of weighed  $\text{HgO}$  (Merck, *p. a.*) in 3 M  $\text{HNO}_3$  (Merck, *p. a.*). The water used was double distilled from Duran 50 apparatus. Radioiodine  $^{131}\text{I}$  was obtained from the Nuclear Institute Vinča (Yugoslavia), as a solution of carrier free  $^{131}\text{I}^-$  of the  $\text{NaI}$  form.  $\text{NaI}$  and  $\text{KI}$  solutions were standardized by using di-iodo-di-methylfluorescein as an end point indicator. Using the chromate method the silver nitrate solution was standardized by means of standard  $\text{NaCl}$  solution. Standard  $\text{TlNO}_3$  solution was prepared by weighing (vacuum dried for 24 hours at room temperature)  $\text{TlNO}_3$ . Solutions of  $\text{La}(\text{NO}_3)_3$  and  $\text{NaNO}_3$  were prepared by weighing of *p. a.* chemicals.

### Radioanalytical Experiments

All the systems described were prepared with an excess of iodide ions in the liquid phase using *in statu nascendi* (metal halides were precipitated by the direct mixing of the precipitation components) method and thermostated at  $20.0 \pm 0.2^\circ \text{C}$  by means of a Haake ultrathermostate. Solutions containing cationic components ( $\text{Ag}^+ + \text{Tl}^+$ ,  $\text{Hg}^{2+}$ ,  $\text{Ag}^+ + \text{Hg}^{2+}$ ,  $\text{Tl}^+ + \text{Hg}^{2+}$ ) were added by a pipet into agitated KI or NaI solutions (labelled by  $^{131}\text{I}$ ), then stirred 5 minutes and left to age for  $t_A = 1500$  minutes. In all observed systems the molar fraction of each metal was continuously changed by 5% so that each system contained 21 different samples (except in systems shown in Fig. 1.). The clear supernatant was used for determination of equilibrium radioactivity  $A_\infty$  by using EKCO Electronics well type NaI(Tl) scintillation counter and semiautomatic scaler. Systems prepared in the same way (three samples) were used for the determination of the radioactivity  $A_\infty$ . The sample of a radioactivity  $A_0$  was prepared in the same manner as above but there were no precipitated metal halogenides in the systems. The clear solution was used for determination of the standard radioactivity  $A_0$ .

### Differential Thermal Analysis

In order to obtain the phase diagram differential thermal analysis (DTA) was used. The apparatus used was commercial unit manufactured by the Gebrüder Netzsch (Selb/Bayern, Germany). The measuring head for dynamical differential calorimetry according to H. E. Schwiete<sup>6</sup> with alumina cup was used. The heating rate was  $5^\circ \text{C}/\text{min}$ . and  $10^\circ \text{C}/\text{min}$ .

### X-Ray Diffraction Analysis

The structural properties of the TlI—AgI systems *in statu nascendi* prepared under various conditions were examined by X-ray diffraction technique (RTG) using a Philips diffractometer with a scintillation counter and a single-channel pulse height analyser. The radiation was Ni-filtered  $\text{CuK}\alpha$ . X-ray diffraction patterns were taken from the wet samples with their original supernatant liquor. A narrow range of Bragg angles from  $2\Theta = 20^\circ$  to  $2\Theta = 50^\circ$  was selected. The recording of patterns was done one day after preparation ( $t_A = 1500$  minutes). Several independent experiments were run with each sample and it was found that the preferred orientation of the precipitate particles could be neglected<sup>7</sup>. The relative intensities of the most prominent diffraction lines of TlI, AgI and coprecipitates for various systems, are shown in Fig. 4. In order to obtain diffractograms for AgI— $\text{HgI}_2$  systems Bragg angles from  $2\Theta = 20^\circ$  to  $2\Theta = 50^\circ$  were selected and for the TlI— $\text{HgI}_2$  systems Bragg angles from  $2\Theta = 20^\circ$  to  $2\Theta = 50^\circ$  were recorded. The results obtained are shown in Figs. 7 and 8.

### Conductivity Experiments

The high frequency Cambridge conductivity bridge was used for the determination of the conductivity of the supernatants in the TlI—AgI systems. Measured conductivities shown in Fig. 3 were obtained as conductivities of the equilibrated and saturated liquors obtained after washing TlI—AgI precipitates.

## RESULTS AND DISCUSSION

As our principal aim is to describe the radiometric principles on which this radioanalytical procedure is based we will make an attempt to explain the distribution of the radioactivity in such a system.

Compounds AX and BX are sparingly soluble substances. If they are in contact they give the sparingly soluble compounds  $\text{ABX}_2$ . In the system the well soluble compounds CX were present. Anion X is the same for compounds AX, CX, BX and  $\text{ABX}_2$  and it is labelled by its radioactive isotope. Total amount of X in liquid phase is  $n_R^L$ , in solid phase  $n_R^S$  and the ratio is  $\alpha_R = n_R^S / n_R^L$ .

In the system with AX and CX  $n_R^S = n_{AX}^S$ ,  $n_R^L = n_{CX}^L$  and if the compounds AX are measurably soluble, then  $n_X^L = n_{CX}^L + n_{AX}^L$  and  $\alpha_\Sigma = n_{AX}^S / (n_{CX}^L + n_{AX}^L)$ . In the case of AX + ABX<sub>2</sub> + CX in the system,  $n_{\Sigma X}^S = n_{AX}^S + n_{ABX_2}^S$  and  $n_{\Sigma X}^L = n_{CX}^L + n_{AX}^L + n_{ABX_2}^L$  or  $\alpha_\Sigma = n_X^S / n_X^L = (n_{AX}^S + n_{ABX_2}^S) / (n_{CX}^L + n_{AX}^L + n_{ABX_2}^L)$ . In the system with ABX<sub>2</sub> + BX + CX,  $n_{\Sigma X}^S = n_{ABX_2}^S + n_{BX}^S$ ,  $n_{\Sigma X}^L = n_{CX}^L + n_{ABX_2}^L + n_{BX}^L$  and  $\alpha_\Sigma = (n_{ABX_2}^S + n_{BX}^S) / (n_{CX}^L + n_{ABX_2}^L + n_{BX}^L)$ . Assuming that  $n_{AX}^L$ ,  $n_{BX}^L$  and  $n_{ABX_2}^L$  are equal to zero, only sparingly soluble compounds will be present in the measured system in form of the precipitates and of well soluble compounds CX. Then  $\alpha_\Sigma$  will be equal to  $\alpha_R$ . On the basis of the amounts of the precipitating components,  $\alpha_R$  values were obtained if the AX, BX, and ABX<sub>2</sub> are insoluble compounds. The values of  $\alpha_\Sigma$  were determined by means of the radiometric technique. By measuring the radioactivities,  $A_\infty$  and  $A_0$ ,  $\alpha_\Sigma$  can be determined as  $\alpha_\Sigma = (A_0 - A_\infty) / A_\infty$ . If the determined  $\alpha_\Sigma$  is smaller than  $\alpha_R$ ,  $n_{\Sigma X}^S$  is smaller than  $n_R^S$ . Then  $n_{\Sigma X}^L$  must be larger than  $n_R^L$  and the solubility of the present solid phases increases. If the  $\alpha_\Sigma$  is plotted against the molar fraction of  $f_{AX}$  or  $f_{BX}$ , inflection points indicate the formation of compounds ABX<sub>2</sub> at  $f_{AX} = 0.5$  and  $f_{BX} = 0.5$ . In our case the metals were A or B and iodide labelled with <sup>131</sup>I<sup>-</sup> was the common anion X. In the presentation of the results,  $n_R^L$  is presented as  $pI_R = -\log n_R^L$ . There are certainly many other combinations depending on the solubilities of components formed in the system and also on the total quantity of substances in the system. In the graphical scheme  $\alpha_\Sigma - f_{AX}, f_{BX}$  several maxima, plateaus or minima will be formed in the case of a poly-component systems. For illustration three systems are shown in the present paper.

Silver iodide and thallos iodide are sparingly soluble in pure water. The solubility of sparingly soluble iodides increases, as a rule, with the increase of the concentration of iodide ions in a solution. Solubilities of AgI and TlI are also dependent on the presence of various nonhalide compounds<sup>5</sup>. From  $pI = 1$  up to  $pI = 5$  the solubility of AgI is approx. constant if  $\alpha_R = 1$  (Fig. 1). Between  $pI = 5$  up to  $pI = 8$   $\alpha_\Sigma$  decreases if  $\alpha_R = 1$  because at  $pI = 8$  the silver iodide is completely dissolved. These radiometric results are in very good agreement with the above mentioned results. The solubility of TlI is constant within very narrow limits between  $pI = 1$  and  $pI = 2.2$  and then increases so fast, that at  $pI = 3.3$  all present TlI is dissolved if  $\alpha_R = 1$  (Fig. 1). This is also in very good agreement with the cited results. In the mixture of AgI—TlI the measured  $\alpha_\Sigma$  decreases as the  $pI$  value and the part of molar fraction of Tl increase (Fig. 1). These changes are caused by different solubility of AgI, TlI and also by intermolecular compounds AgI · TlI and AgI · 2 TlI as will be seen in the next consideration.

Analysis of the AgI—TlI systems show that in all cases, irrespective of the differences in initial conditions of the preparation of the systems, distribution curves which have inflections at the same molar fractions of silver (or thallium) were obtained (Fig. 2). High frequency conductivity measurements show a minimum conductivity in systems containing AgI only (point A in Fig. 3); then an increase up to  $0.1 f_{TlI}$ , and up to  $0.5 f_{TlI}$  constant conductivity, then a positive inflection, a second positive inflection after  $0.6 f_{TlI}$  and after

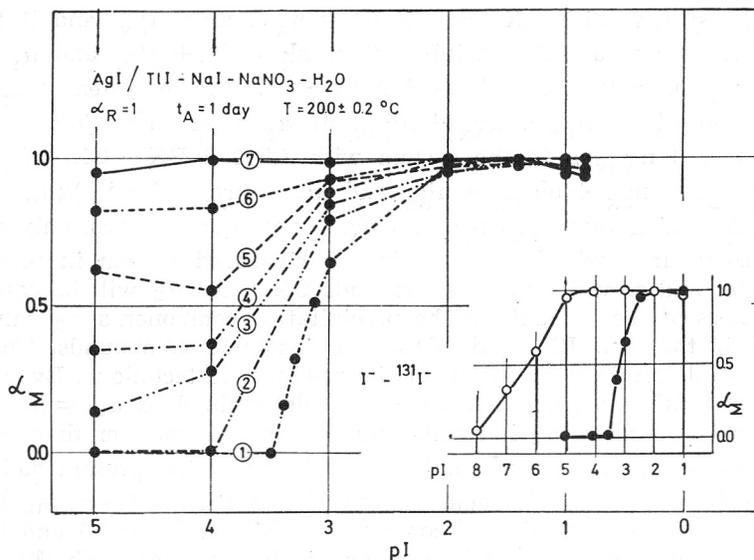


Fig. 1. Ratio  $\alpha_{\Sigma}$  plotted against  $pI$  for various AgI + TlI mixtures in  $\text{NaNO}_3 + \text{NaI}$  ( $^{131}\text{I}$ ) solutions. The molar fractions of TlI were  $f_{\text{TlI}} = 1.0$  (curve 1), 0.9 (curve 2), 0.7 (curve 3), 0.5 (curve 4), 0.3 (curve 5), 0.1 (curve 6) and  $f_{\text{TlI}} = 0$  i. e.  $f_{\text{AgI}} = 1.0$  (curve 7). In the small figure the ratio  $\alpha_{\Sigma}$  is plotted against  $pI$  for AgI in  $\text{NaNO}_3 + \text{NaI}$  ( $^{131}\text{I}$ ) solutions.

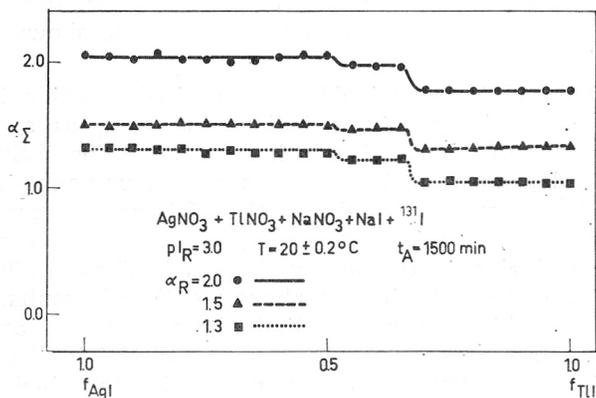


Fig. 2. Ratio  $\alpha_{\Sigma}$  plotted against molar fractions of silver iodide,  $f_{\text{AgI}}$  and thallium iodide,  $f_{\text{TlI}}$  at different  $\alpha_{\text{R}}$ .

0.7  $f_{\text{TlI}}$  up to 1.0  $f_{\text{TlI}}$  constant conductivity. The inflections on the conductivity curve (points B and C) lie at the same molar compositions of the systems as the inflections in curves ( $\alpha_{\Sigma}$ ,  $f$ ) in Fig. 2. In Fig. 4 the relative intensities of the most prominent diffraction lines of TlI, AgI, and co-precipitates, for various systems, are shown. It can be seen that the pure hexagonal-cubic (mixed) AgI and the pure orthorhombic TlI exist only at 100% Ag ions and at 100% Tl ions, respectively (Fig. 4). Besides, one can see two groups of reflections, corresponding to two coprecipitates. When the molar ratio Tl/Ag

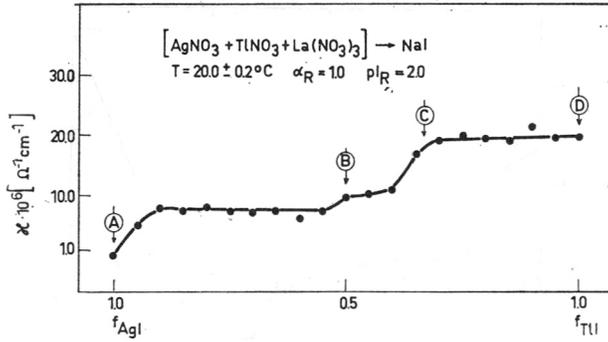


Fig. 3. The conductivity,  $\kappa \cdot 10^6$  ( $\Omega^{-1} \text{cm}^{-1}$ ), of the equilibrated supernatant liquor plotted against the molar fractions  $f_{\text{AgI}}$  and  $f_{\text{TlI}}$ . Arrows mark the points on the curve indicating in the projections on the abscissa the molar compositions of the insoluble compounds.

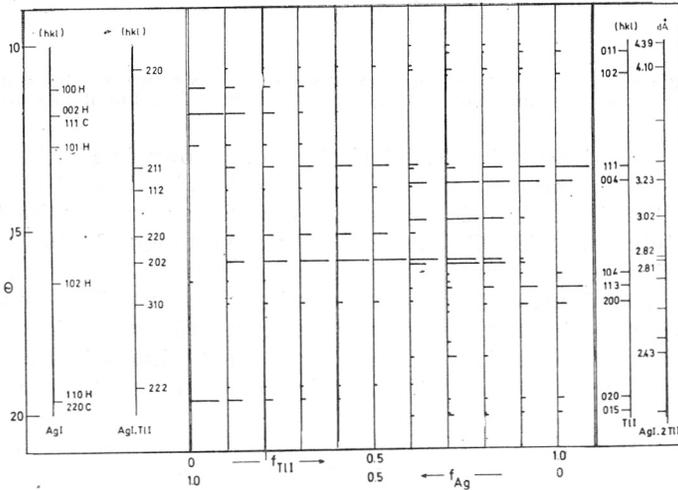


Fig. 4. Relative intensities of the most prominent diffraction lines of TlI, AgI and co-precipitates. Ordinate: Bragg angles and selected reflection for AgI, AgI · TlI, TlI and AgI · 2TlI. Abscissa: the molar fractions of AgI,  $f_{\text{AgI}}$ , TlI,  $f_{\text{TlI}}$ , respectively, and belonging relative intensities (represented by length of mark).

is 1:1 a deep yellow precipitate of the salt AgITlI crystallizes. At the molar ratio  $\text{Tl}/\text{Ag} = 2:1$  a white precipitate of the salt AgI.2TlI is obtained. X-ray diffraction analysis shows that the former salt is the equilibrium phase in presence of an excess of AgI, and the latter salt is the equilibrium phase in an excess of TlI. Intermediate compositions give rise to mixtures of both modifications, as has also been observed before.<sup>7</sup> The compound AgI.TlI is tetragonal, with  $a = 8.34 \text{ \AA}$ ,  $c = 7.67 \text{ \AA}$ , this being in good agreement with the cited results.<sup>3</sup> The sample with 100% Ag ions contains approx. 50% cubic and 50% hexagonal modifications of AgI.<sup>7</sup> By decreasing the percent of Ag ions to 60%, the hexagonal modification increases to approx. 75–80%. RTG results obtained show the presence of the definite compounds at the same molar fractions of  $f_{\text{AgI}}$  and  $f_{\text{TlI}}$  as does the radioanalysis. The results of the

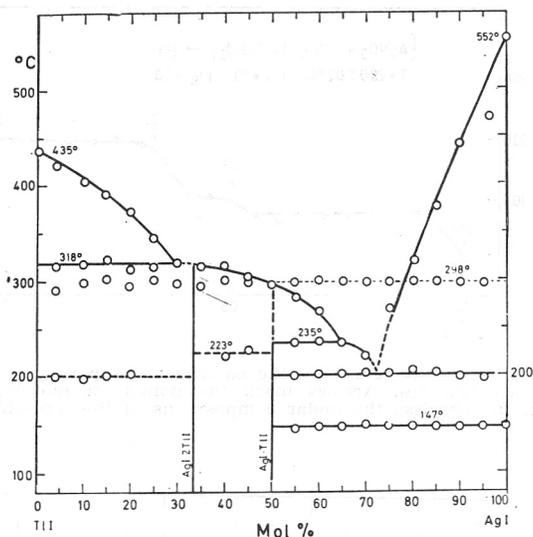


Fig. 5. Phase diagram of the TlI—AgI system. Ordinate: temperature of the system. Abscissa: the molar percentage of AgI, TlI, respectively, in the initial mixture.

phase analysis carried out by DTA technique are shown in Fig. 5. The diagram is very similar to the one of L. G. Berg and I. N. Lepeshkov<sup>2</sup>, with the exception of the line at 298° C. On the DTA diagrams for all unheated samples we find a minimum at 298° C. When DTA over the range 50—100% mol of AgI was repeated with a sample which was preheated 300° C or higher, the minimum at 298° C was found to be missing. It is possible to suppose the minimum corresponding to the secondary reaction or disproportion. Accordingly, we must draw the conclusion that the phase compositions in the precipitated samples (heated up to 298° C or higher with constant component ratio) are not equal.

The analysis of the ratio  $\alpha_{\Sigma}$  in the  $\text{HgI}_2$ —KI— $^{131}\text{I}$  systems shows (Fig. 6) an increase of the  $\alpha_{\Sigma}$  values with the increase of  $\alpha_{\text{R}}$  and the decrease of the  $p\text{I}_{\text{R}}$  values (increase of the initial KI concentrations). The radioanalytic determination of the  $\alpha_{\Sigma}$  values in the various mixtures of the  $\text{AgI} + \text{HgI}_2$  precipitates (Fig. 7 DEF curve) shows at composition  $\text{HgI}_2 \cdot 2\text{HgI}$  (point E in Fig. 7) an inflection of the curve which corresponds to the maximum of the RTG diffraction at  $2\Theta = 24.32^\circ$  (point B in Fig. 7). The points A and D in Fig. 7 correspond to the pure AgI and the points C and F to the pure  $\text{HgI}_2$ . All these results are in very good agreement with literature data<sup>9,10</sup>. The results obtained on the radioanalytic determination of the  $\alpha_{\Sigma}$  in various precipitated mixtures of TlI +  $\text{HgI}_2$  show that there are two inflections on the distribution curve of the investigated system, one at point E, the second at point F in Fig. 8. The inflection at point E corresponds to point B of the diffractograms (maximum of the RTG reflections at the  $2\Theta = 28.56^\circ$ ) in the same figure. These inflections B and E correspond to the  $\text{HgI}_2 \cdot 4\text{TlI}$  compound. There is no RTG maximum which could correspond to the distribution of point F and we must draw the conclusion that the new compound is not registered in the solid phase under these conditions.

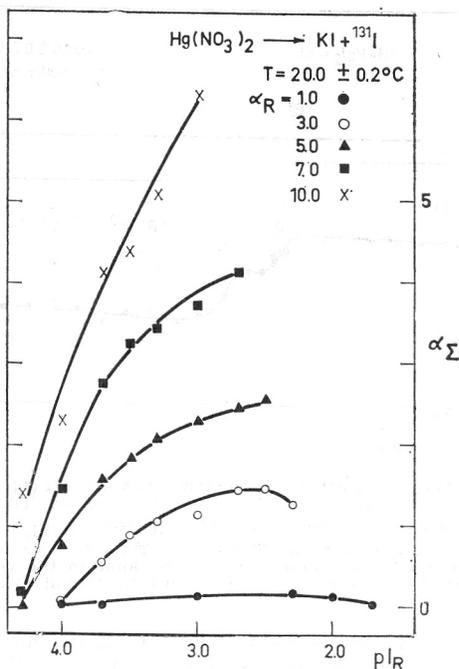


Fig. 6. Solubility of  $\text{HgI}_2$  solutions of KI of various concentrations. Ordinate: the ratio  $\alpha_\Sigma$ . Abscissa: initial concentration of KI expressed as  $pI_R$ .

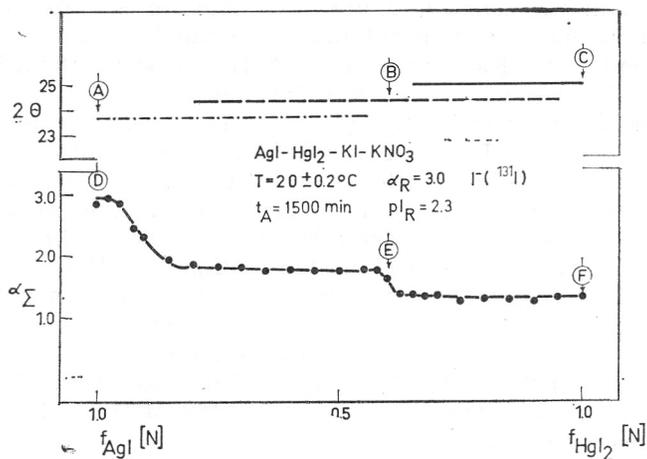


Fig. 7. Relative intensities of selected diffraction lines of  $\text{AgI}$  ( $2\theta = 23.72^\circ$ ),  $\text{HgI}_2 \cdot 2\text{AgI}$  ( $2\theta = 24.32^\circ$ ) and  $\text{HgI}_2$  ( $2\theta = 24.94^\circ$ ). A, B, C lines and the  $\alpha_\Sigma$  of  $\text{AgI}$ ,  $\text{HgI}_2$  and co-precipitates (curve DEF) plotted against the normal fraction of  $\text{AgI}$ ,  $f_{\text{AgI}}$ ,  $\text{HgI}_2$   $f_{\text{HgI}_2}$  respectively, in the initial mixtures of the system at  $T = 20.0 \pm 0.02^\circ\text{C}$ ,  $\alpha_R = 3.0$ ,  $pI_R = 2.3$ . Arrows A, and C mark RTG maxima which correspond to the compounds of  $\text{AgI}$  and  $\text{HgI}_2$  (see the intersections of the projections of arrows D, E and F to the abscissa).

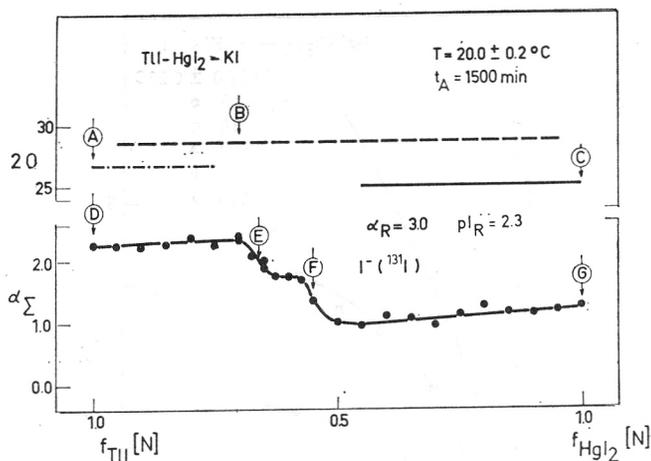


Fig. 8. Relative intensities of selected diffraction lines of TlI ( $2\theta = 26.72^\circ$ ),  $\text{HgI}_2 \cdot 4\text{TlI}$  ( $2\theta = 28.56^\circ$ ) and  $\text{HgI}_2$  ( $2\theta = 24.94^\circ$ ) A, B, C lines and the  $\alpha_\Sigma$  of TlI,  $\text{HgI}_2$  and co-precipitates (curve DEFG) plotted against the fraction of TlI,  $f_{\text{TlI}}$ ,  $\text{HgI}_2$ ,  $f_{\text{HgI}_2}$ , respectively, in the initial mixtures of the system at  $T = 20.0 \pm 0.2^\circ\text{C}$ ,  $\alpha_R = 3.0$  and  $pI_R = 2.3$ . Arrows A, B and C mark RTG maxima which correspond to the compounds of TlI and  $\text{HgI}_2$  (see the projection for the adequate arrows D, E and G to the abscissa).

In comparison with the results of Huart and Durif<sup>11</sup> our RTG analysis indicate only  $\text{HgI}_2 \cdot 4\text{TlI}$  in the systems with free TlI or  $\text{HgI}_2$ . Then the inflections E and F are probably caused by the change in composition of the solution. This change may be caused by the change of sediment compositions without the formation of new compounds or by complex dissolution of present components as in the  $\text{AgI}-\text{PbI}_2$  systems.<sup>8</sup>

$\alpha_\Sigma$  values calculated for the conditions at  $0.4 f_{\text{Ag}}$  and  $0.6 f_{\text{Hg}}$  [when we take into account the complex solubility of  $\text{HgI}_2$  in KI (Fig. 6)] correspond to the values obtained by radioanalytical experiments (Fig. 7) and are in very good agreement with Katelaaras results.<sup>9</sup> He always obtained  $\text{HgI}_2 \cdot 2\text{AgI}$  compounds. A decrease of  $\alpha_\Sigma$  values in the region of  $0.85$  to  $0.95 f_{\text{Ag}}$  is caused by an increase in the quantity of  $\text{AgI}$  and by total complex dissolution of the present  $\text{HgI}_2$  (ratio  $\text{Hg}:\text{I}$  is  $1:4$  at  $0.9 f_{\text{Ag}}$ ). In the region of  $0.95 f_{\text{Ag}}$  and  $0.45 f_{\text{Ag}}$   $\alpha_\Sigma$  is constant because  $(\text{HgI}_2)_{\text{solid}} \leftrightarrow (\text{HgI}_2)_{\text{solution}}$  equilibria are obtained (in the systems there is enough  $\text{HgI}_2$  for the formation of a  $\text{HgI}_2 \cdot 2\text{AgI}$  precipitate). The region of the decrease of  $\alpha_\Sigma$  values where  $\text{HgI}_2 \cdot 2\text{AgI}$  is formed corresponds to the change of the  $(\text{HgI}_2)_{\text{solid}} \leftrightarrow (\text{HgI}_2)_{\text{solution}}$  equilibria, because  $\text{HgI}_2$  is precipitated in precisely predetermined amounts equivalent to present  $\text{AgI}$ , while the rest of  $\text{HgI}_2$  is complexly dissolved. After that equilibrium is again reached in the system because one part of mercury is in the form of  $\text{HgI}_2$ , another as  $\text{HgI}_2 \cdot 2\text{AgI}$  and the rest is complexly dissolved as  $\text{HgI}_4^{2-}$  (see RTG data: the diffraction intensities of  $\text{HgI}_2 \cdot 2\text{AgI}$  decrease as well intensities for  $\text{HgI}_2$ ).

Comparing the cited results with those obtained by physical and chemical methods applied it can be concluded that on the basis of the results the radioanalytical method can be used for the determination of interaction between precipitation components in similar systems. By careful interpretation of radiochemically obtained  $\alpha_\Sigma - f_{\text{AX}}, f_{\text{BX}}$  curves it is possible in such cases to

estimate the interaction in the liquid or solid phases of poly-component systems. The experimental error depends on the standard radiometric errors caused by the statistic nature of the radioactive decay. It seems that the error is from 3 to 5 percent.

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## IZVOD

**Radiometrijsko određivanje AgI—TII—<sup>(131)</sup>I, AgI—HgI<sub>2</sub>—<sup>(131)</sup>I  
i HgI<sub>2</sub>—TII—<sup>(131)</sup>I sistema**

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Opisano je radioanalitičko određivanje međumolekularnih interakcija u polikomponentnim sistemima tipa »kruto-tekuće«. Kao modeli primijenjeni su sistemi AgI—TII—<sup>(131)</sup>I, AgI—HgI<sub>2</sub>—<sup>(131)</sup>I i HgI<sub>2</sub>—TII—<sup>(131)</sup>I. Detaljno je analiziran sistem AgI—TII—<sup>(131)</sup>I tako da su radioanalitički rezultati uspoređivani s rezultatima rendgenske difrakcije, električne vodljivosti i mikrodiferencijalno termičke analize na identičnim uzorcima. Na druga dva sistema rezultati radioanalitičkog određivanja uspoređivani su samo s rezultatima rendgenske difrakcije. Budući da se svi dobiveni rezultati slažu s literaturnim podacima, zaključuje se da je opisana radioanalitička metoda primjenljiva za opisane analize intermolekularnih interakcija u miješanim sistemima.

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