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THERMAL DECOMPOSITION OF THE SOLVENT-EXTRACTED CHLORO COMPLEXES OF TRIVALENT GALLIUM, INDIUM AND THALLIUM WITH TRIOCTYLAMINE AND TRIOCTYLMETHYLAMMONIUM CHLORIDE*

T. Sato and *T. Takahashi*

DEPARTMENT OF APPLIED CHEMISTRY, FACULTY OF ENGINEERING, SHIZUOKA UNIVERSITY, HAMAMATSU, JAPAN

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The complexes of trivalent gallium, indium and thallium with trioctylamine (TOA, R_3N) and trioctylmethylammonium chloride (TOMAC, $R_3R'NCl$) were prepared by drying in vacuo the organic extracts from aqueous solutions containing the chlorides of their metals with TOA and TOMAC in benzene: $R_3NHGaCl_4$, $(R_3NH)_2InCl_5$, $R_3NHTlCl_4$, $R_3R'NGaCl_4$, $R_3R'NInCl_4$ and $R_3R'TlCl_4$. The prepared complexes were examined by TG and DTA under an atmosphere of nitrogen, and the products of their thermal decompositions, such as volatile matter and residues, were examined by gas chromatography, X-ay diffraction study and infrared spectrophotometry. It was found that the complexes of these trivalent metals with TOA and TOMAC decompose thermally to the chlorides ($GaCl_3$, $InCl_3$ and TlCl) through the cracking of alkyl groups.

In order to obtain further information concerning the compositions of the complexes isolated from the organic solutions in the solvent extraction of metals with long-chain aliphatic amines, studies have been made on the thermal decomposition of such complexes of the chloro complexes of copper(II) [1], zirconium(IV) [2], vanadium(IV) [3] and uranium(VI) [4] in air, and of divalent manganese, cobalt, copper and zinc [5] and vanadium [6] under an atmosphere of nitrogen. The present paper extends this work to an examination of the thermal behaviour of chloro complexes of trivalent gallium, indium and thallium with trioctylamine (TOA, R_3N) and trioctylmethylammonium chloride (TOMAC, $R_3R'NCl$) under an atmosphere of nitrogen.

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Experimental

Chemicals

TOA and TOMAC of high purity (both >99%, Koei Chemical Co. Ltd., Osaka) were used without further purification, diluted with benzene. Aqueous solutions of indium(III) and thallium(III) were prepared by dissolving trivalent indium and thallium chlorides, $InCl_3$ and $TlCl_3$, while that of gallium(III) was prepared by dissolving gallium metal (99.9999%, Swiss Aluminium Ltd., Switzerland) in hydrochloric acid solution. All chemicals used were of analytical reagent grade.

Preparation of complexes

On the basis of the distribution results [7], the organic solutions saturated with trivalent metals were prepared as follows: 0.05 mol dm^{-3} TOA (or 0.05 mol dm^{-3} TOMAC) in benzene was shaken for 15 min with an aqueous solution containing 0.085 mol dm^{-3} $GaCl_3$ in 5 mol dm^{-3} HCl, 0.226 mol dm^{-3} $InCl_3$ in 6 mol dm^{-3} HCl (or 5 mol dm^{-3} HCl for TOMAC) or 0.048 mol dm^{-3} $TlCl_3$ in 0.1 mol dm^{-3} HCl (or 0.01 mol dm^{-3} HCl for TOMAC) at 20°; the organic phase was centrifuged and dried in vacuo. The organic extracts so obtained contained the extractant/chloride/metal in the molar ratios, indicating the stoichiometries to be $R_3NHGaCl_4$, $(R_3NH)_2InCl_5$, $R_3NHTlCl_4$, $R_3R'NGaCl_4$, $R_3R'NInCl_4$ and $R_3R'NTlCl_4$ (Table 1).

Table 1 Molar ratios of $[R_3N]$ or $[R_3R'N]/[Cl]/[M]/[H_2O]$ in trivalent metal-saturated organic solutions from solvent extraction with TOA and TOMAC

Metal	Molar ratio	
	$[R_3N]/[Cl]/[M]/[H_2O]$	$[R_3R'N]/[Cl]/[M]/[H_2O]$
Ga(III)	1.1/4.2/1.0/0.1	1.2/4.1/1.0/0.2
In(III)	2.1/4.9/1.0/0.1	1.0/4.0/1.0/0.1
Tl(III)	1.0/4.2/1.0/—	1.0/4.1/1.0/—

Analysis

The complexes were examined by thermogravimetry (TG) and differential thermal analysis (DTA), and the products of their thermal decompositions such as volatile matter and residues, were checked by gas chromatography, X-ray diffration study and infrared spectroscopy [6]. The TG and DTA were performed under an atmosphere of nitrogen at a heating rate of 5 deg min^{-1} and a flow rate of nitrogen of 50 ml min^{-1} with alumel-chromel thermocouples, using 40 mg of the sample and α-alumina as reference material for the measurement of differential thermal electromotive force. The residues were derived from the complexes by heating to the required temperatures at a rate of 5 deg min^{-1} under the same atmosphere. Infrared spectra were recorded on Japan Spectroscopic Co. Ltd. Model IRA–1 (4000–650 cm^{-1}) and IR–F (700–200 cm^{-1}) grating spectrometers. X-ray powder diffraction diagrams were obtained on a Geigerflex recording X-ray diffractometer with filtered copper radiation. The volatile matter generated during the DTA was collected with a 2 ml syringe at the gas outlet of the DTA apparatus and analysed with a Shimazu Model GC–6AMPTF gas chromatograph. Each component detected was identified by gas chromatography-mass spectrometry (GC–MS). Hydrogen chloride in the volatile matter was trapped in 0.05 mol dm^{-3} silver nitrate solution in an interval of 10 degrees at a temperature between 200 and 350°, by passing it through the solution for 3 min. For chemical analysis, the resulting materials were dissolved in benzene, and the chloride concentration and water content in proportions of the solution were determined by Volhard and Karl Fischer titrations. Additionally, the benzene solutions were washed with 1 mol dm^{-3} nitric acid (1 mol dm^{-3} sodium carbonate in the extraction of thallium(III)), and the metals in the acidic layers were then titrated with EDTA with xylenol orange as indicator [8].

Results and discussion

The TG and DTA curves of the trivalent gallium, indium and thallium complexes with TOA and TOMAC are shown in Figs 1–2. For the complexes with TOA, the

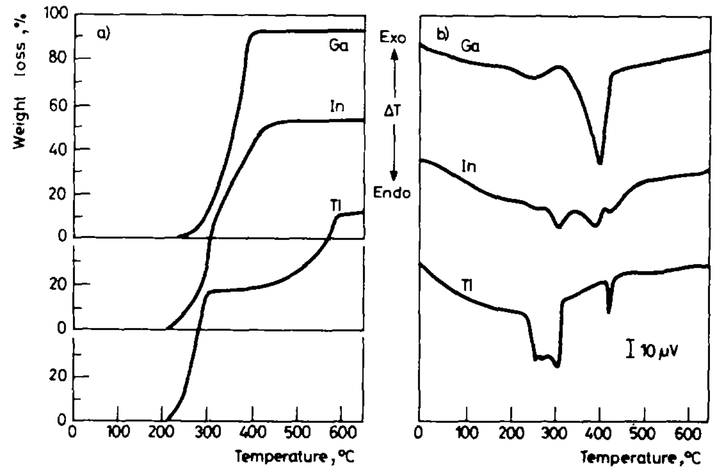


Fig. 1 TG (a) and DTA (b) curves of trivalent metal complexes with TOA

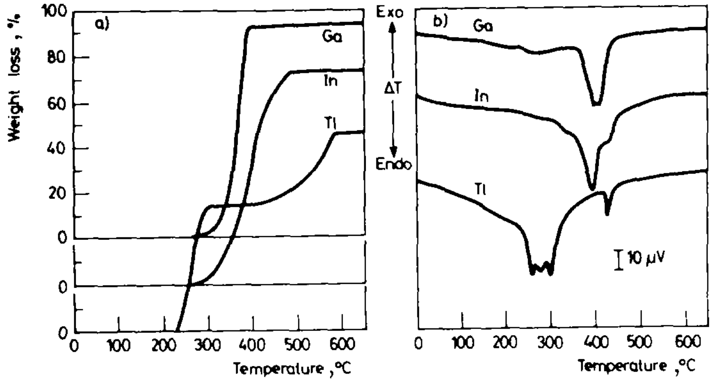


Fig. 2 TG (a) and DTA (b) curves of trivalent metal complexes with TOMAC

TG curves exhibit weight losses of 92.7% at 410° for gallium, 89.0% at 440° for indium, and 58.4% at 300–440° and 92.2% at 585° for thallium, while the DTA curves of the complexes reveal endothermic reactions at 270 and 400° for gallium, 270, 310, 400 and 440° for indium and 255, 270, 310 and 425° for thallium. The complexes with TOMAC exhibit losses of 93.2% at 400° for gallium, 92.0% at 470° for indium, and 54.3% at 300–430° and 86.4% at 580° for thallium in the TG curves, and endotherms at 270, 400 and 410° for gallium, 290, 350 and 440° for indium and 255, 265, 300 and 425° for thallium in the DTA curves. In Figs 1–2 it is observed that the endothermic reactions in the DTA curves occur at points near the change of shape in the TG curve. Additionally, it is presumed that the thermal stabilities of the complexes with TOA resemble those of the complexes with TOMAC.

The volatile matter generated in the thermal decomposition of the trivalent gallium, indium and thallium complexes with TOA and TOMAC contains aliphatic compounds such as allene, ethylene, ethane, propane, 1-butene, 2-butene and methyl chloride, identified by GC–MS, and hydrogen chloride, trapped in silver nitrate solution. Some representative gas chromatographic data are illustrated in Tables 2–7, where the relative peak heights in the gas chromatogram for some of the components are indicated as a function of the heating temperature. The variation of the molar ratios, [Cl]/[M], where M = Ga, In and Tl, in the residues derived from the complexes with TOA and TOMAC by heating to the stated temperatures, is given in Table 8. The molar ratios are 3 : 1 for gallium and indium, and 1 : 1 for thallium at 350°. It is postulated, therefore, that the studied metal complexes with TOA and TOMAC decompose into the metal chloride, MCl_3, except that the thallium complexes decompose to MCl. These facts are supported by the X-ray diffraction results shown in Table 9 and Figs 3–4, where the diagrams for the complexes with TOA are indicated as representative data, since their patterns are

Table 2 Relative peak heights of some components in the gas chromatogram of the chloro complex of gallium(III) with TOA as a function of heating temperature

Temp., °C	Relative peak height*, mm						
	C_3H_4	C_2H_4	C_2H_6	C_3H_6	CH_3Cl	$1-C_4H_8$**	$2-C_4H_8$**
200	4						
225							
250	10		2		1		
275	8		2				
300	8		3		4		
325	15	10	4	3	8	11	23
350	14	6	9	9	17	6	5
375	12	12	4	60	64	42	20
400	102	352	210	1540	62	950	540
425	26	46	54	142	12	73	50
450	15	33	30	54	5	21	5
475	20	71	45	62	6	25	4
500	24	100	54	75		33	5

* Sensitivity range 4×10^3 μV

** $1-$ or $2-C_4H_8$ denotes 1- or 2-butene.

Table 3 Relative peak heights of some components in the gas chromatogram of the chloro complex of indium(III) with TOA as a function of heating temperature

Temp., °C	Relative peak height*, mm						
	C_3H_4	C_2H_4	C_2H_6	C_3H_6	CH_3Cl	$1-C_4H_8$**	$2-C_4H_8$**
200							
225					2		
250	1			2	5		
275	7			5	4		
300	3	2		4	16	7	2
325	38	1		11	14	5	2
350	53	5	2	22	20	2	5
375	37	16	3	45	35	3	
400	170	23	5	81	34	13	23
425		105	56	180	27	40	28
450	10	20	3	69	5	22	7
475	17	8	2	22		16	13
500	15	5	1	7		10	7

* Sensitivity range 4×10^3 μV

** $1-$ or $2-C_4H_8$ denotes 1- or 2-butene.

Table 4 Relative peak heights of some components in the gas chromatogram of the chloro complex of thallium(III) with TOA as a function of heating temperature

Temp., °C	Relative peak height*, mm						
	C_3H_4	C_2H_4	C_2H_6	C_3H_6	CH_3Cl	$1-C_4H_8$**	$2-C_4H_8$**
200	13	2	2	4	2	1	1
225	18	10	9	14	1	47	7
250	26	5	5	10	3	4	4
275	31	12	35	47	6	17	18
300	18	4	12	13		9	13
325	35	3	3	1	1	2	2
350	6	3	4	2		2	3
375			3	1		1	3
400	15		3	1		1	6
425	28	2	4	6		4	
450	10	1					1
475	13	1				1	1
500	7	1					

* Sensitivity range 4×10^3 μV

** $1-$ or $2-C_4H_8$ denotes 1- or 2-butene.

Table 5 Relative peak heights of some components in the gas chromatogram of the chloro complex of gallium(III) with TOMAC as a function of heating temperature

Temp., °C	Relative peak height*, mm						
	C_3H_4	C_2H_4	C_2H_6	C_3H_6	CH_3Cl	$1-C_4H_8$**	$2-C_4H_8$**
200							
225		3			1		
250		3		5	2		
275	8	1	1		4		
300	16	3	3	6	32		
325	15	6	5	3	114		
350	10	5	4	2	95		
375	9	9	5	13	124	6	6
400	11	20	9	134	79	97	36
425	152	400	400	1100	280	512	290
450	28	56	46	113	82	27	57
475	5	9	7	21	9	8	1
500	4	3	3	8	7		

* Sensitivity range 4×10^3 μV

** $1-$ or $2-C_4H_8$ denotes 1- or 2-butene.

Table 6 Relative peak heights of some components in the gas chromatogram of the chloro complex of indium(III) with TOMAC as a function of heating temperature

Temp., °C	Relative peak height*, mm						
	C_3H_4	C_2H_4	C_2H_6	C_3H_6	CH_3Cl	$1-C_4H_8$**	$2-C_4H_8$**
200							
225							
250					7		
275	7	3			11	1	
300	6	2			7		
325	12	7	5	2	220		
350	66	59	25	50	220	16	19
375	136	36	54	75	95	50	30
400	173	98	79	152	8	51	55
425	35	22	18	14	2	15	22
450	25	44	21	19	1	13	6
475	22	17	11	29		4	7
500	21	23	7	14		1	6

* Sensitivity range 4×10^3 μV

** $1-$ or $2-C_4H_8$ denotes 1- or 2-butene.

Table 7 Relative peak heights of some components in the gas chromatogram of the chloro complex of thallium(III) with TOMAC as a function of heating temperature

Temp., °C	Relative peak height*, mm						
	C_3H_4	C_2H_4	C_2H_6	C_3H_6	CH_3Cl	$1-C_4H_8$**	$2-C_4H_8$**
200					5		
225					14		
250	7	3	2		9800	2	2
275		1	1	1	4480		
300	20	1	1	1	35		
325	15	2	1		2	3	6
350	11	1				1	1
375	13	4	5	5	3	4	4
400	13	2	2			2	2
425	16	6	7	5		3	2
450	25	15	30	37		11	11
475	15	4	6	9		9	5
500	14	3	4	6		3	3

* Sensitivity range 4×10^3 μV

** $1-$ or $2-C_4H_8$ denotes 1- or 2-butene.

Table 8 Variation of molar ratios, [Cl]/[M]*, in the residues derived from trivalent metal complexes with TOA and TOMAC by heating to the stated temperature

Temp., °C	Molar ratio [Cl]/[M]					
	Ga(III)		In(III)		Tl(III)	
	TOA	TOMAC	TOA	TOMAC	TOA	TOMAC
100	4.4	4.1	4.7	3.9	4.1	4.3
200	—	—	4.6	3.8	4.1	4.2
250	3.7	3.9	3.9	3.8	2.2	2.8
300	—	—	3.3	3.5	1.4	1.2
350	3.2	2.9	3.1	3.2	1.1	1.2
400	—	—	2.8	2.9	1.1	1.0

* M = Ga, In or Tl.

Table 9 X-ray diffraction results for the residues derived from trivalent metal complexes with TOA and TOMAC by heating to the stated temperature

Temp., °C	Phase detected					
	Ga(III)		In(III)		Tl(III)	
	TOA	TOMAC	TOA	TOMAC	TOA	TOMAC
200	—	—	Am	Am	TlCl	TlCl
250	Am	Am	Am	Am	TlCl	TlCl
300	—	—	Am	Am	TlCl	TlCl
350	Am	Am	Am	Am	TlCl	TlCl
400	—	—	Am	Am	Am	Am
450	—	—	Am	Am	Am	Am

* Am represents amorphous type.

analogous to those for the complexes with TOMAC. The X-ray results indicate that the residues produced by heating the complexes of gallium and indium with TOA and TOMAC are amorphous, independently of the heating temperature, while those of thallium exhibit the pattern arising from reduction to thallium(I) chloride at 200°, although this pattern disappears on heating at 400°. Thus, it is seen that the amount of components detected above 250° increases on increase of the temperature, in accordance with the weight loss in the TG curve, and the maximum amounts of their volatile matter correspond to the endotherms in the DTA curves. On the other hand, it is found that hydrogen chloride evolved from the complexes with TOA is detected above 200° (before detection of the organic components) and its maximum amount is obtained at about 250°, as in the thermal decomposition of

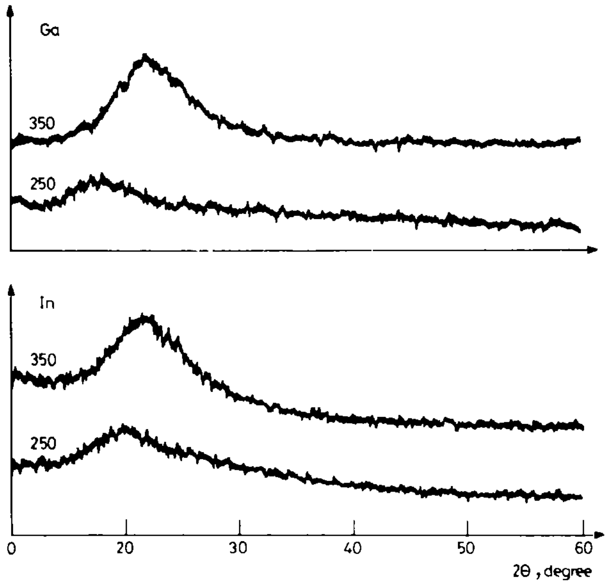


Fig. 3 X-ray diffraction diagrams of the residues derived from trivalent gallium and indium complexes with TOA by heating to the stated temperatures (numbers on curves are heating temperatures, °C)

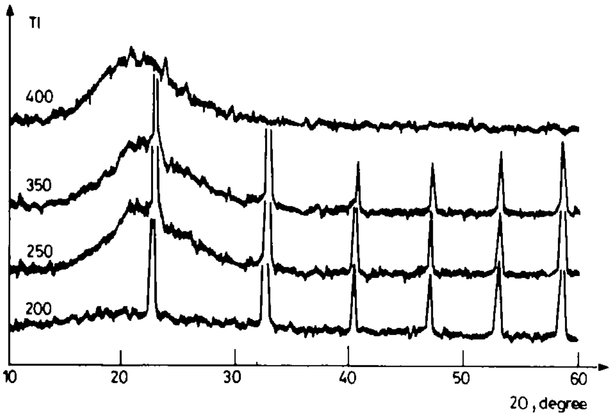


Fig. 4 X-ray diffraction diagrams of the residues derived from thallium(III) complex with TOA by heating to the stated temperatures (numbers on curves are heating temperatures, °C)

divalent metal complexes [5]. It is therefore inferred that the thermal decomposition is initiated by the release of hydrogen chloride, followed by the decomposition of TOA. This implies that in the DTA curves, the endotherms at 270, 270 and 255–270° for the trivalent gallium, indium and thallium complexes, respectively, are to be assigned to the release of hydrogen chloride accompanied by the decomposition of TOA. The TG curves reveal weight losses corresponding to the theoretical values for the thermal reactions of the complexes of trivalent metals with TOA:

$$(R_3NH)_nMCl_{3+n} \rightarrow [nR_3N\ldots MCl_3]+nHCl$$

where $n=1$ and 2 for gallium and indium, while for the thallium complex:

$$R_3NHTlCl_4 \rightarrow [R_3N\ldots TlCl]+HCl+3/2Cl_2$$

The volatile matter from the complexes with TOMAC contains the same organic components as that from the complexes with TOA, although the former complexes contain much more methyl chloride than the latter complexes. In contrast, the thermal decomposition of vanadyl complexes with TOA and TOMAC indicates the release of methyl alcohol and methyl chloride, ascribed to the presence of hydrolysed species [6]. In Tables 5–7, it is apparent that the distribution of methyl chloride is different from that of hydrocarbons: The maximum amount of methyl chloride is detected at 325–375° (250–275° for the thallium complex), and that of hydrocarbons at 400–425°. These components are generated largely at the beginning of the thermal decomposition, implying that the decomposition of the complexes with TOMAC releases methyl chloride at first. From this it is deduced that the endotherms at lower temperatures in the DTA curves for the complexes correspond to the distribution of methyl chloride and hydrocarbons. The endotherms at 270 and 380° for gallium, 290 and 350° for indium and 255 and 265° for thallium are assigned to dechlorination, due to the evolution of methyl chloride and/or cracking (Table 8).

The infrared spectra of the complexes of the trivalent metals with TOA (Figs 5–6) show the following absorptions: C—H stretching bands at 2920 and 2860 cm^{-1}; CH_3 degenerate (and CH_2 scissoring) and symmetrical bonding modes at 1465 and 1380 cm^{-1}, attributed to the presence of TOA; the NH^+ stretching vibration at 2350 cm^{-1} in TOA hydrochloride shifts to broad bands centered around 3110, 2600 and 2800 cm^{-1} for gallium, indium and thallium, respectively; and the M—Cl stretching vibations appear at 380, 330 and 290 cm^{-1} for gallium, indium and thallium, respectively, confirming the formation of the chloro complex. When the gallium complex is heated at 350°, the spectrum retains a pattern similar to that of the starting material, although the Ga—Cl stretching band becomes slightly broadened. At 400°, however, there is no indication of characteristic absorptions, because the gallium compound is thermally dissociated. For the indium complex, the NH^+ stretching band shifts to higher frequency, at 3040 cm^{-1}, as a shoulder, on heating at 250°, and disappears at 350°. On heating of the thallium complex, the NH^+ stretching band shifts to higher frequency, at 3225 cm^{-1}, at 200°, and disappears at 250°. Simultaneously, the bands due to alkyl groups and the Tl—Cl stretching band decrease in intensity. In contrast, the complexes with TOMAC

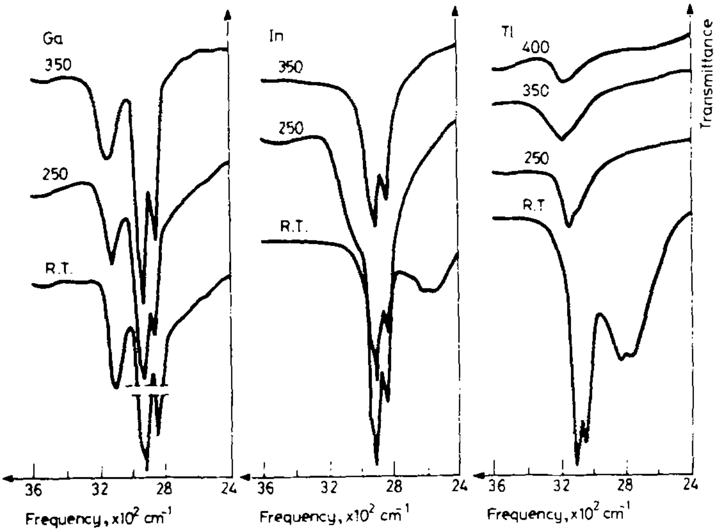


Fig. 5 Infrared spectra of the residues derived from trivalent metal complexes with TOA by heating to the stated temperatures (numbers on curves are heating temperatures, °C)

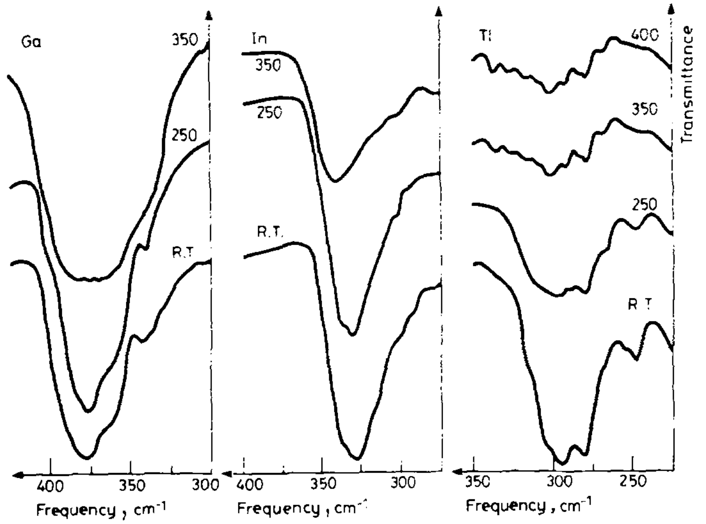


Fig. 6 Infrared spectra in the region of lower frequency of the residues derived from trivalent metal complexes with TOA by heating to the stated temperature (numbers on curves are heating temperatures, °C)

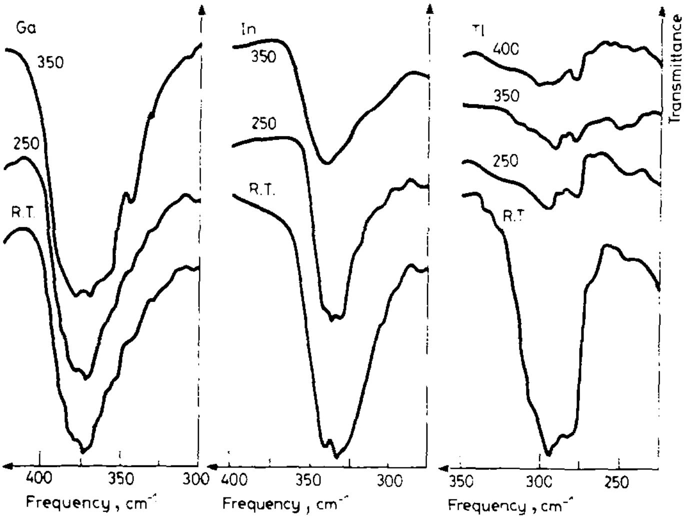


Fig. 7 Infrared spectra in the region of lower frequency of the residues derived from trivalent metal complexes with TOMAC by heating to the stated temperatures (numbers on curves are heating temperatures, °C)

(Fig. 7) give infrared spectra similar to those of the complexes with TOA, except for the absence of the NH^+ stretching bands. However, since the shapes of the M—Cl stretching bands are more symmetric than those for the TOA complexes, it is inferred that the symmetry of the latter complexes is lower than that of the former. The changes in the infrared spectra of the residues derived from the thermally decomposed TOMAC complexes are almost the same as those in the case of the

Table 10 Probable assignment of the endothermic reactions in the DTA curves of trivalent metal complexes with TOA and TOMAC

Temp., °C						Probable assignment
Ga(III)		In(III)		Tl(III)		
TOA	TOMAC	TOA	TOMAC	TOA	TOMAC	
				255		release of HCl
270		270		270		
	270		290		255	release of CH_3Cl and cracking
			350		265	
400*	400* 410*	310		310	300	cracking
		400	400			
		440	440	425	425	sublimation

*The reaction in cracking overlaps in thermal dissociation.

TOA complexes. Thus, it is considered that the thermal stabilities of these complexes follow the sequence Ga > In > Tl.

Hence, the endothermic reactions observed in the DTA curves are interpreted as given in Table 10. It is concluded that the thermal decompositions of the complexes of trivalent metals with TOA and TOMAC proceed in the following sequences: for the complexes with TOA:

$$R_3NHGaCl_4 \xrightarrow[-HCl]{270^\circ} GaCl_3 + A' \xrightarrow[\text{cracking}]{300\text{–}400^\circ}$$

$GaCl_3$ (thermal dissociation at $\sim 400^\circ$)

$$(R_3NH)_2InCl_5 \xrightarrow{270^\circ} InCl_3 + A' \xrightarrow[\text{cracking}]{300\text{–}400^\circ}$$

$InCl_3$ (sublimation at $> 440^\circ$)

$$R_3NHTlCl_4 \xrightarrow[\text{reduction and } -HCl]{255\text{–}270^\circ} TlCl + A' \xrightarrow[\text{cracking}]{\sim 300^\circ}$$

$TlCl$ (sublimation at 425°)

where A′ denotes the product of thermal decomposition; for the complexes with TOMAC:

$$R_3R'NGaCl_4 \xrightarrow[-CH_3Cl \text{ and cracking}]{270^\circ} GaCl_3 + A' \xrightarrow[\text{cracking}]{300\text{–}400^\circ}$$

$GaCl_3$ (thermal dissociation at $\sim 400^\circ$)

$$R_3R'NInCl_4 \xrightarrow[-CH_3Cl \text{ and cracking}]{290\text{–}350^\circ} InCl_3 + A' \xrightarrow[\text{cracking}]{300\text{–}400^\circ}$$

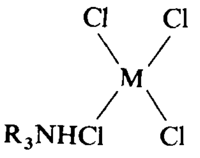
$InCl_3$ (sublimation at $> 440^\circ$)

$$R_3R'NTlCl_4 \xrightarrow[-CH_3Cl \text{ and cracking}]{255\text{–}265^\circ} TlCl + A' \xrightarrow[\text{cracking}]{\sim 300^\circ}$$

and/or reduction

$TlCl$ (sublimation at 425°),

supporting the proposed tetrahedral structures, [I] and [II], for the complexes with TOA and TOMAC:



[I]

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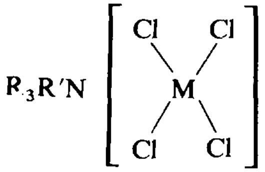

[II]

An exception is the indium complex with TOA, which has trigonal bipyramidal or square pyramidal structure.

* * *

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Zusammenfassung — Komplexe von dreiwertigem Gallium, Indium und Thallium mit Trioctylamin (TOA, R_3N) und Trioctylmethylammonium-Hydrochlorid (TOMAC, $R_3R'NCl$) wurden durch Trocknung der benzolischen Extrakte der die Metallchloride mit TOA bzw. TOMAC enthaltenden wässrigen Lösungen im Vakuum dargestellt: $R_3NHGaCl_4$, $(R_3NH)_2InCl_5$, $R_3NHTlCl_4$, $R_3R'NGaCl_4$, $R_3R'NInCl_4$ und $R_3R'TlCl_4$. Die dargestellten Komplexe wurden durch TG und DTA in Stickstoffatmosphäre untersucht. Die Produkte der thermischen Zersetzung, flüchtige Substanzen und Rückstände, wurden gas-chromatographisch, röntgendiffraktometrisch und IR-spektrophotometrisch untersucht. Es wurde festgestellt, daß die Komplexe dieser dreiwertigen Metalle sich thermisch durch Krackung der Alkylgruppen zu den Chloriden ($GaCl_3$, $InCl_3$ und $TlCl_3$) zersetzen.

Резюме — Комплексы хлоридов трехвалентных галлия, индия и таллия с триоктиламином (TOA, R_3N) и триоктилметиламмоний хлоридом (TOMAC, $R_3R'NCl$) состава $R_3NHGaCl_4$, $(R_3NH)_2InCl_5$, $R_3NHTlCl_4$, $R_3R'NGaCl_4$, $R_3R'NInCl_4$ и $R_3R'NTlCl_4$ были получены высушиванием в вакууме органических экстрактов, полученных при совместном экстрагировании хлоридов металлов с TOA и TOMAC в бензоле. Полученные комплексы были изучены методами ТГ и ДТА в атмосфере азота, а продукты термического разложения анализировались газовой хроматографией, рентгено-фазовым анализом и ИК спектрометрией. Установлено, что эти комплексы разлагаются до хлоридов металлов ($GaCl_3$, $InCl_3$ и $TlCl$) с расщеплением алкильных групп.