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THERMOCHEMISTRY OF SOME COMPLEXES OF CHROMIUM(III) WITH IMIDAZOLES

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The thermal decomposition in air of several complexes of chromium(III) with imidazole, *N*-methylimidazole and 2-methylimidazole has been studied with the aid of differential thermal analysis (DTA), thermogravimetry (TG) and derivative thermogravimetry (DTG) in the temperature range 25–600 °C. Although the final process of the decomposition gives Cr_2O_3, there are interesting differences in the complete process of decomposition. The reasons for these differences appear to be related to the trans-effect and to the presence in the imidazole complexes of hydrogen bonds. Enthalpies of the several decomposition reactions have been determined by differential thermal analysis.

In three previous papers [1–3] the synthesis and the vibrational (IR, Raman) and EPR spectra of imidazole, *N*-methylimidazole and 2-methylimidazole complexes with Cr(III) having the formulae: $(CrIm_6)Cl_3$, $(CrIm_5X)X_2$ where $X = Cl$, Br; *cis*- and *trans*-$(CrIm_4Cl_2)Cl$, *trans*-$(CrNIm_4Cl_2)Cl$ and *trans*-$(Cr2MIm_4Cl_2)Cl$ were discussed. In this work we describe the thermal decomposition of these complexes in air. No attempt was made to obtain kinetic data, our main interest being to determine the existence of new species formed during the process of decomposition. The thermal decompositions of many Cr(III) amine complexes have been thoroughly investigated. The results obtained correspond to the formation of intermediate species containing less of volatile ligand. In the case of the complexes *cis*-$[Cr(ethylene\text{-}diamine)_2Cl_2]Cl$ [4] and $Cr(pyridine)_3X_3$ ($X = Cl$, Br), [5] these species are polymeric with bringing halide. A similar behaviour is shown by the complexes studied here.

Experimental

The complexes were prepared as described elsewhere, and characterized by chemical analyses and i.r. spectroscopy [1, 2]. The thermoanalytical curves were obtained on a Mettler 181 thermoanalyser. Samples of ~ 10 mg were studied in Pt-cups supplied with lids. The runs were performed in still air. For the DTA, the sensitivity was fixed

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at 50 μV for full scale deflection while for the DTG it was 5 mg/min. The heating rate was standardized at 5 degree/min. The instrument was calibrated by use of a number of standard compounds with well-known heats of reaction. The results are listed in Table 1.

Table 1 Properties of standard compounds in thermal analysis

Compound	Type of reaction	Heat of reaction, mJ/mg	*T*, °C	Ref.	Our data	
					Tm	K calcd, mJ/°C min
KNO_3	tr	58.1	128	[13]	134	712.06
$AgNO_3$	m	69.8	212	[14]	217	791.23
Na_2SO_4	tr	52.7	241	[13]	247	836.66
$NaNO_3$	m	171.6	306	[13]	314	885.82

tr = transition; m = melting; Tm = mean reaction temperature

Heats of reaction were obtained with the relation $Q = K \int T dt = K \cdot A$ in which: Q = the heat of reaction in mJ; K = a constant for given *Tm* (the mean reaction temperature) and obtained from Table 1 (expressed in mJ/°C min); A = peak area expressed in °C · min. When K is known, the heat of reaction can be determined by measuring the peak area. Our peak areas were determined with a planimeter. The identification of the final decomposition products were obtained by means of a Philips PW 1010/30 X-ray spectrometer. It was found, in all cases, that the diffraction patterns corresponded to Cr_2O_3.

The IR spectra of some intermediate products were obtained as Nujol mulls on a Perkin–Elmer 325 spectrophotometer.

Results and discussion

The results of TG, DTG and DTA are summarized in Table 2 whereas in Figs 1 to 7 and 9 some decomposition curves are shown in detail for illustration of the typical reactions. All curves could be interpreted with one or more of the following decomposition reactions: (s = solid, l = liquid, g = gas).

$$(CrIm_6)X_3(s) \rightarrow (CrIm_5X)X_2(s) + Im(g) \quad (1)$$

$$(CrIm_6)X_3(s) \rightarrow (CrIm_3X_3)(s) + 3\ Im(g) \quad (2)$$

$$(CrIm_5X)X_2(s) \rightarrow (CrIm_4X_2)X(s) + Im(g) \quad (3)$$

$$(CrIm_5X)X_2(s) \rightarrow (CrIm_3X_3)(s) + 2\ Im(g) \quad (4)$$

$$(CrIm_4X_2)X(s) \rightarrow (CrIm_3X_3)(s) + Im(g) \quad (5)$$

$$2\ (CrIm_4X_2)X(s) \rightarrow Cr_2(X)Im_5X_5(s) + 3\ Im(g) \quad (6)$$

$$2\ (CrIm_4X_2)X(s) \rightarrow Cr_2(X)_2Im_4X_4(s) + 4\ Im(g) \quad (7)$$

$$2\ (CrIm_3X_3)(s) \rightarrow Cr_2(X)Im_5X_5(s) + Im(g) \quad (8)$$

$$2\ (CrIm_3X_3)(s) \rightarrow Cr_2(X)_2Im_4X_4(s) + 2\ Im(g) \quad (9)$$

$$2\ (CrIm_4X_2)X(s) \rightarrow [Cr_2(X)_2Im_6X_2]X_2(s) + 2\ Im(g) \quad (10)$$

$$2\ [Cr_2(X)_2Im_6X_2]X_2(s) \rightarrow [Cr_4(X)_6Im_{10}X_2]X_4(s) + 2\ Im(g) \quad (11)$$

$$[Cr_4(X)_6Im_{10}X_2]X_4(s) \rightarrow Cr_4(X)_6Im_6X_6(s) + 4\ Im(g) \quad (12)$$

$$4\ CrIm_3X_3(s) \rightarrow Cr_4(X)_6Im_6X_6(s) + 6\ Im(g) \quad (13)$$

$$Cr_2(X)Im_5X_5(s) \rightarrow Cr_2(X)_2Im_4X_4(s) + Im(g) \quad (14)$$

$$2\ Cr_2(X)Im_5X_5(s) \rightarrow Cr_4(X)_6Im_6X_6(s) + 4\ Im(g) \quad (15)$$

$$(CrIm_5X)X_2 \cdot (H_2O)_m(s) \rightarrow (CrIm_5X)X_2(s) + mH_2O(g) \quad (16)$$

$$(CrIm_4X_2)X \cdot (H_2O)_m(s) \rightarrow (CrIm_4X_2)X(s) + mH_2O(g) \quad (17)$$

The complexes $(CrIm_5Cl)Cl_2$, *cis*-$(CrIm_4Cl_2)Cl$, $Cr(NIm_5Cl)Cl_2$ are very hygroscopic and hydrated products are formed if special precautions are not taken. In this case, one of the dehydration reactions (16), (17) ($m = 2$), occurred first. After that, they show the same behaviour as the anhydrous products.

Some reactions were found, for which it was not possible to determine accurate ΔH values, because of overlap with other reactions.

In all cases the number of imidazole molecules evolved was determined from the TG and DTG measurements.

The value of ΔH_l, Table 2, gives an indication for the stability of Cr-ligand binding. Nevertheless, since most reactions occur above 250° the sublimation energy of imidazole and evaporation energy of *N*-methylimidazole are included in these values. The experimentally obtained heats of sublimation and evaporation of imidazole and *N*-methylimidazole via reactions (18) and (19) respectively are also listed in Table 2.

$$Im(s) \rightarrow Im(g) \quad (18)$$

$$N\text{-}Im(l) \rightarrow N\text{-}Im(g) \quad (19)$$

It is impossible to obtain a realistic measure of the metal-ligand bond because data for the heats of sublimation are lacking for the compounds $(CrIm_{6-p}X_p)X_{3-p}$. However, we can attempt to estimate the strength of metal-ligand bond from reactions of the general type

$$(CrIm_{6-p}X_p)X_{3-p}(s) \rightarrow (CrIm_{6-p-n}X_{p+n})X_{3-p-n}(s) + nIm(s)$$

Table 2 The decomposition of chromium(III) imidazole complexes

Compound	Reaction number *	Temperature region, °C	Weight loss, %		ΔH_l, ** kJ · mol^{-1}	ΔH_L, *** kJ · mol^{-1}
			calc.	obs.		
$(CrIm_6)Cl_3$	(2)	170–295	36.0	35.4	79.0 (5)	18.9
	(8)	310–335	6.0	6.6	96.1 (5)	36.0
	(14)	360–405	6.0	6.0	–	–
$(CrIm_5Cl)Cl_2$	(4)	175–280	27.3	25.9	77.3 (4)	17.2
	(8)	290–330	6.8	6.0	97.8 (9)	37.7
	(14)	340–395	6.8	6.7	–	–
$(CrIm_5Br)Br_2$	(3)	160–290	10.8	11.3	89.4 (8)	29.3
	(7)	320–440	21.5	21.2	–	–
cis-$(CrIm_4Cl_2)Cl$	(5)	240–280	15.8	14.9	77.7 (10)	17.6
	(8)	290–350	7.9	7.8	97.3 (1)	37.2
	(14)	360–400	7.9	7.6	–	–
trans-$(CrIm_4Cl_2)Cl$	(10)	230–320	15.8	15.6	94.4 (15)	34.3
	(11)	330–390	7.9	7.8	–	–
	(12)	400–450	15.8	15.1	–	–
$(CrNIm_5Cl)Cl_2$	(3)	130–175	14.4	13.2	109.9 (5)	70.7
	(7)	175–280	28.8	28.2	94.8 (3)	55.6
trans-$(CrNIm_4Cl_2)Cl$	(5)	200–250	16.8	17.9	100.3 (4)	61.1
	(9)	270–350	16.8	14.7	100.3 (9)	61.1
trans-$(Cr_2MIm_4Cl_2)Cl$	(6)	155–215	25.3	24.7	58.5 (2)	–
Imidazole (s)	(18)	60–250	100.0	100.0	60.1 (2)	–
N-methylimidazole (l)	(19)	50–165	100.0	100.0	39.2 (6)	–

*The reaction number corresponds to the overall process which area peak is possible to measure. **Uncertainties in the last digit are indicated in parentheses. ***Calculated from reaction (20), uncertainties are 4.18 kJ · mol^{-1}

The heat of this reaction can be calculated as: $\Delta H_L = \Delta H_l - \Delta H_s$, where ΔH_s is either the experimentally obtained heat of sublimation or vaporization of the ligand. Although variations in *Cp* are usually smaller than 0.83 kJ · mol^{-1} · 100°, comparison of heats of a particular decomposition reaction for several similar compound seems to be allowed.

In the thermoanalytical curves (DTA, TG and DTG) of the several compounds studied it can be seen that the complete process of decomposition in air, in the range of temperatures employed, 25–600°, includes several thermal effects in all cases. In Fig. 1 the curves corresponding to $(CrIm_6)Cl_3$ are plotted. It is clear that there are five endothermic peaks with their maxima at the temperatures of 205, 250, 275, 330 and 410°. There is also a very marked exothermic effect with its maximum at 485°.

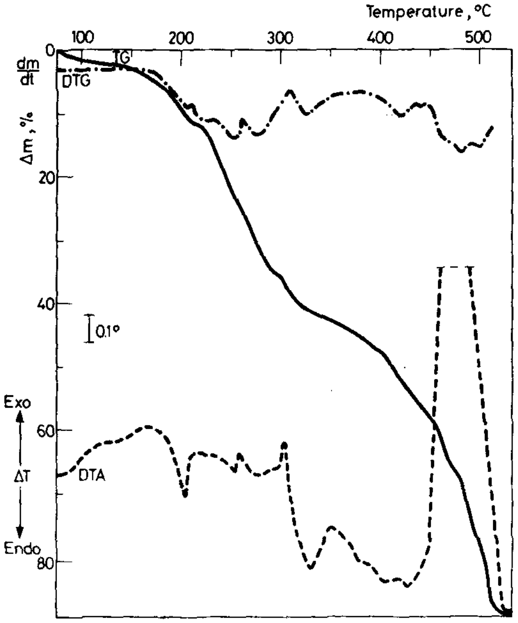


Fig. 1 Thermoanalytical curves for $(CrIm)_6Cl_3$

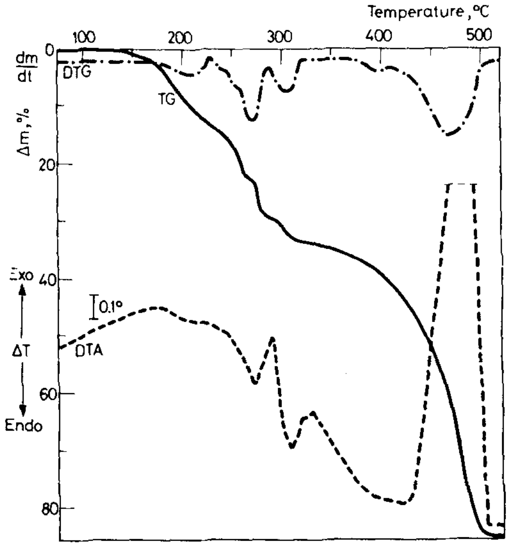


Fig. 2 Thermoanalytical curves for $(CrIm_5Cl)Cl_2$

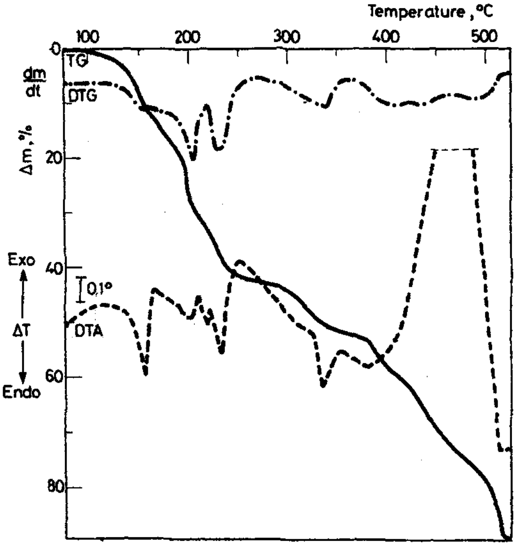


Fig. 3 Thermoanalytical curves for $(CrNIm_5Cl)Cl_2$

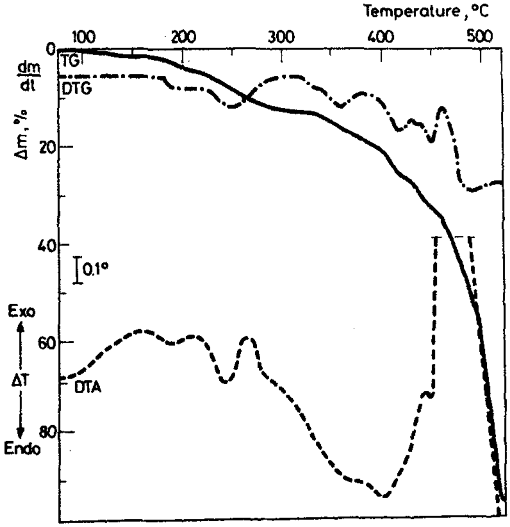


Fig. 4 Thermoanalytical curves for $(CrIm_5Br)Br_2$

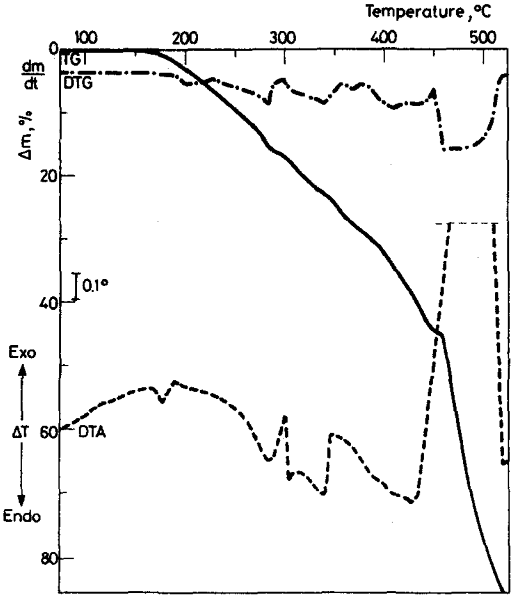


Fig. 5 Thermoanalytical curves for *cis*-(CrIm$_4$Cl$_2$)Cl

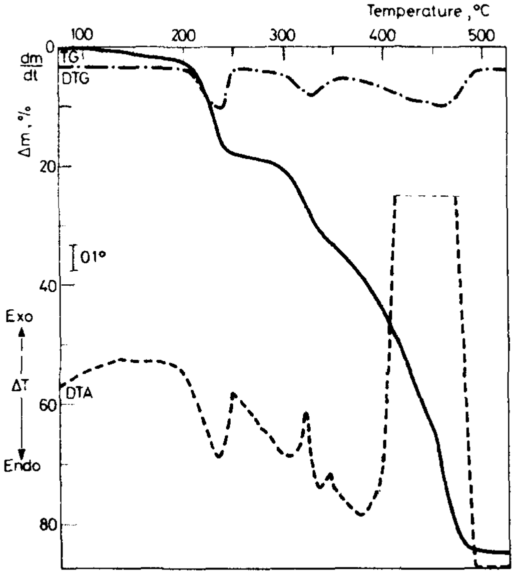


Fig. 6 Thermoanalytical curves for *trans*-(CrNIm$_4$Cl$_2$)Cl

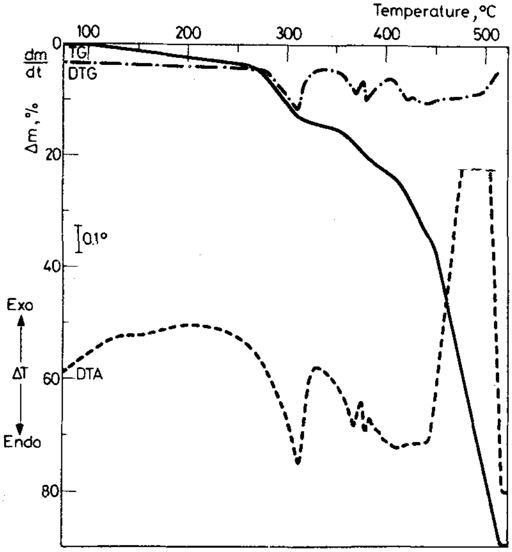


Fig. 7 Thermoanalytical curves for *trans*-(CrIm$_4$Cl$_2$)Cl

From TG plot it appears that the decomposition starts at about 160° and although there are no clear steps in this curve, the information contained on the DTG curve can help in deciding the stoichiometry of the process occurring. It seems then that under this dynamic condition the maximum decomposition rate is attained near 200°. However, before this process of decomposition ends another increase on the rate of weight loss is observed and it is accompanied by two small endotherms on the DTA curve. The weight loss recorded up to the end of these peaks is, respectively, 12.19% and 23.21% and can be fitted to the reactions (1) and (4), for which the calculated weight losses are 12.0 and 24.0%.

This behaviour is not different from that shown by the chromium-hexa-amine complexes [Cr(NH$_3$)$_6$]Cl$_3$ which, according to Wendlandt and Chow [6], evolve initially one mole of ammonia and other two moles in a second step.

At higher temperature there is another loss of weight, accompanied by an endotherm (peak at 330°), which is followed by two more decomposition steps, also reflected on the DTG curve. The first of these processes, for which the weight loss recorded is 6.60%, corresponds to the elimination of one imidazole molecule for two chromium atoms (weight loss calculated 6.00%), and under these conditions the formation of a halogen bridged dimer, reaction (8), can be postulated. The next step, weight loss 6.02%, is responsible for the elimination of another imidazole molecule giving either *cis* or *trans* dichloro-tetrachloro-tetraimidazole dichromium(III) reaction (14). A further process seems to produce a tetramer, but this cannot be clearly differentiated from the oxidation of the chromium complex to form Cr_2O_3, which will be responsible for a very marked exotherm centred at 485°. The thermoanalytical curves of $(CrIm_5Cl)Cl_2$, Fig. 2, were analogous to those first discussed for $(CrIm_6)Cl_3$, with one only difference that is caused by the expected absence of the first decomposition step.

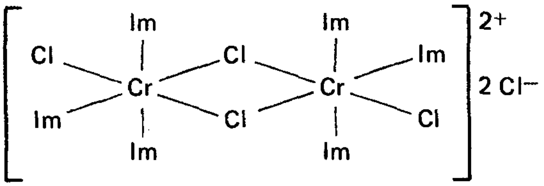
The thermal decomposition of $(CrNIm_5Cl)Cl_2$ is shown in Fig. 3. There are no clear steps in the TG curve, however the DTG curve exhibits four peaks accompanied by five endotherms on the DTA curve. The first of these processes can be attributed to a reaction type (3) giving either *cis*-or *trans*-$(CrNIm_4Cl_2)Cl$. The second process, peak centred at 200°, corresponds to the elimination of another imidazole molecule for each chromium atom, weight loss recorded 15.03%. This process will then produce $CrNIm_3Cl_3$, reaction (5). On the DTA curve the peak area of this step appears to overlap the one corresponding to the following process. In this the amount decomposed is equivalent to a molecule of imidazole for every chromium atom. At first sight it appears that this behaviour could be different from that shown by the $(CrIm_5Cl)Cl_2$.

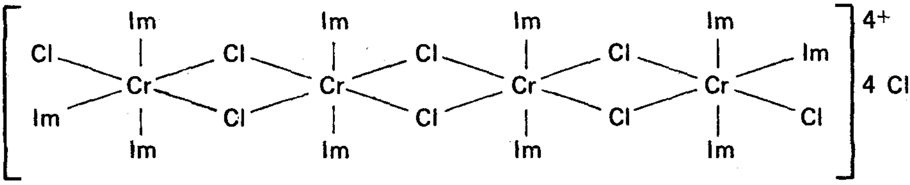
However, since the DTG curve shows one peak with a shoulder accompanied by two endothermic effects clearly differentiated, the first of these process could be due to the elimination of one imidazole molecule for two chromium atoms giving a monobridged dimer reaction (8), the second process will then produce the doubly chlorine bridged dimer, reaction (14). At higher temperatures $Cr_2(X)_2NIm_4X_4$ decomposes according to the usual pattern.

In the case of $(CrIm_5Br)Br_2$, Fig. 4, the thermal decomposition in air starts at 160°. The first step of the decomposition is clearly defined on the TG curve from which the experimental weight loss corresponds to the reaction (3). The second step, also endothermic, seems to be due to the elimination of the two imidazole for every chromium atom. If we assume for this complex a similar behaviour to the *N*-methylimidazole and chlorine analogues, this process probably means the formation of a bromine bridged dimer, reaction (7), in which the evolution of imidazole occurs in only one step.

The thermoanalytical curves corresponding to the next two complexes studied: *cis*-$(CrIm_4Cl_2)Cl$ and *trans*-$(CrNIm_4Cl_2)Cl$ are given in Figs 5 and 6 respectively. At lower temperatures, there are only three thermal effects in the both complexes. It can be appreciated that the curve of the first one shows a weight loss corresponding to the elimination of one imidazole molecule per chromium atom and this process can be attributed to reaction (5). At higher temperatures there are two not clearly differentiated endothermic peaks on the DTA curves, centred at 310° and 345°, and also reflected by one peak with a shoulder on the DTG curve. In this step one imidazole molecule is lost for two chromium atoms, giving the chlorine-bridged dimer, reaction (8). Then there is a third step, where the weight loss is the same; this last process will produce the doubly chlorine-bridged dimer (reaction (14)). This behaviour is consistent with those first discussed for $(CrIm_6)Cl_3$ and $(CrIm_5Cl)Cl_2$.

In the thermal decomposition of *trans*-(CrNIm$_4$Cl$_2$)Cl one can appreciate a similar pattern with regard to *cis*-(CrIm$_4$Cl$_2$)Cl. Nevertheless, the presence of only one peak in the second step of the process and the overlapping of its peak area with that corresponding to the third step can be observed. At higher temperatures both complexes decompose in the usual way giving the final product Cr$_2$O$_3$. In the case of the *trans*-(CrIm$_4$Cl$_2$)Cl, Fig. 7, according to the TG, DTG and DTA curves the first decomposition step seems to end near 320° and the weight loss recorded is equivalent to the loss of one imidazole molecule for each chromium atom. This process is followed by two weight losses, also reflected by two peaks on the DTG curve and two endotherms on the DTA curve. In each one the loss of weight corresponds to the elimination of one molecule for four chromium atoms. This last process suggests that a tetramer is produced in two steps, which supposes the previous formation of a dimer. This means the formation successively of:





reactions (10) and (11), the formation of tetramers are not uncommon in chromium chemistry [7]. Subsequently the loss of one imidazole molecule for one chromium atom will give a neutral tetramer, reaction (12). The formation of the above mentioned dimer is supported by infrared spectroscopic data. In Fig. 8 IR spectra of *trans*-(CrIm$_4$Cl$_2$)Cl and the corresponding products obtained when this is heated up to 320° and 370° are shown.

Group theory predicts for the dimer the appearance of one terminal metal-chloride stretching modes and two bridge metal-chloride stretching mode. The complex *trans*-(CrIm$_4$Cl$_2$) shows in the IR region two bands at 379 and 360 cm^{-1}, while in the dimer it can be observed three strong bands at 390, 370 and 348 cm^{-1}, that can be assigned to a terminal ν(Cr–Cl). Moreover, a band of medium intensity occurs at 280 cm^{-1} which has been assigned to bridge ν(Cr–Cl). The proportion $\nu(\text{Cr–Cl})_{\text{bridge}}/\nu(\text{Cr–Cl})_{\text{terminal}}$ is 0.80 in fair accord with the results recorded for other halide complexes. The IR spectra of the *trans*-(CrIm$_4$Cl$_2$)Cl heated up to 370°

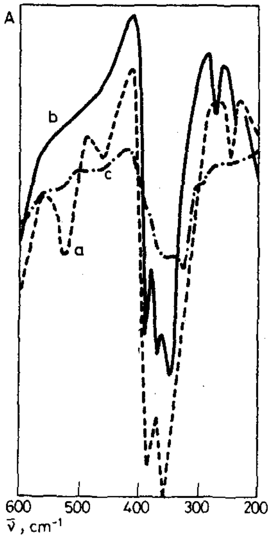


Fig. 8 IR spectra; (a) *trans*-$(CrIm_4Cl_2)Cl$; products resulting from it by heating, (b) up to 320 °C; (c) up to 370 °C

shows poorly defined bands, which could be due to a decreased symmetry caused by a higher degree of polymerization.

The different behaviour of the *cis*- and *trans*-$(CrIm_4Cl_2)Cl$ can be attributed to the different trans influence of the chlorine ligands in both complexes. That is, in the case of the cis complex both chlorine ligands produce the same trans influence and presumably, although not necessarily [8], the trans effect. However, in the case of the trans complex the chlorine ligands are trans to each other and the formation of the tetramer happens in a different way. Similar results have been reported for imidazole-palladium complexes [9]. We have also studied the thermal decomposition of *trans*-$(Cr2MIm_4Cl_2)Cl$, Fig. 9. The first step of the decomposition is clearly defined on the TG curve from which an experimental weight loss of 24.70% is obtained. This amount is equivalent to the evolution of three imidazole molecules for two chromium atoms. The first process is followed by two exothermic reactions, the first one is clearly defined. Its weight loss corresponds nearly to the elimination of one imidazole molecule for two chromium atoms. Under these conditions, one can postulate that the first step happens according to reaction (6) or to reactions (10) and (11) overlapped, giving a tetramer in one only step.

The different thermal behaviour of the three analogous trans complexes is noteworthy. This can perhaps be due to bulkier size of the *N*- and 2-methyl substituted imidazole molecules and as a result the corresponding complexes will show different inclinations to polymerization.

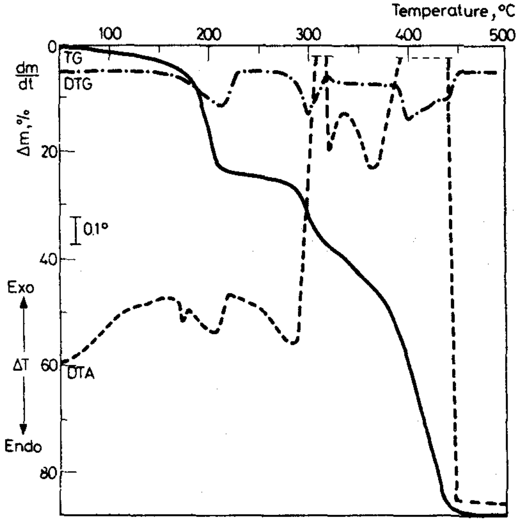


Fig. 9 Thermoanalytical curves for *trans*-$(Cr2MIm_4Cl_2)Cl$

Imidazole and 2-methylimidazole complexes exhibit a small endothermic effect with its maximum placed between 135° and 190° before the TG curve shows any sign of decomposition. There are two possible explanations: breaking of hydrogen bonds, which could be expected to form between the hydrogen bonded to the nitrogen atom in the imidazole ligand of a complex and a halogen atom of a next-neighbour complex. The other explanation could be a phase transition. However, since this small first peak does not appear on the DTA curves of the N-methylimidazole complexes and $(CrIm_6)Cl_3$, Figs 3 and 6, we believe that the first explanation is more likely Moreover, the existence of hydrogen bonds is confirmed by the presence, in the infrared spectra of the imidazole complexes [2] of bands at 3350 and 2800 cm^{-1} which can be attributed to the νN—H stretching vibration an also at 1170 and 700 cm^{-1} which can be attributed to the deformation modes δCNH within the molecular plane and νCNH out of the plane respectively, that can be assimilated to hydrogen bonding analogous to that operating in solid imidazole [10].

In the case of the complexes $(CrIm_5Cl)Cl_2$, $(CrNIm_5Cl)Cl_2$ and *cis*-$(CrIm_4Cl_2)Cl$ when they are hydrated it can be observed that a small endothermic effect always occurs below 100°.

The thermal decomposition of all the complexes studied in this work with the exception of the *trans*-$(Cr2MIm_4Cl_2)Cl$ can be summarized as in Fig. 10.

In the values for ΔH_f and ΔH_L listed in Table 2 a few variations occur with respect to variations in p (the number of coordinated halogen atoms), X(Cl, Br) and Im (imidazole or substituted imidazole). First of all, from the decomposition reactions of

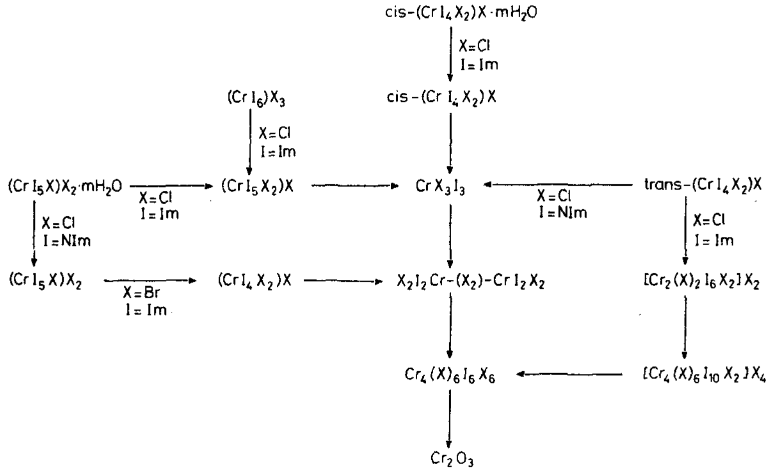


Fig. 10 Scheme of the thermal decomposition of some imidazole chromium(III) complexes in still air

the chlorides it is seen that the *N*-methylimidazole is held more strongly than the imidazole, this agrees with the major basicity of the first one and has been previously observed by Reedijk et al. [11] in similar complexes of Ni(II). Secondly when the evolution of imidazole molecules in complexes type of $(CrIm_5X)X_2$ is considered, ΔH_l increases from $X = Cl$ to $X = Br$. These results are consistent with the IR spectra [2] that shown how νCr—N slightly decreases when in position trans to an imidazole there is a more clearly electronegative ligand. The imidazole possesses some π-accepting capacity [12], the thermal behaviour of our complexes can be accounted for the different degrees of polarizability of the halogens (Br > Cl) and their different sequence in terms of their effective contribution to the metal-ligand π-dative bond.

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Zusammenfassung — Die thermische Zersetzung in Luft von verschiedenen Chrom(III)-Komplexen mit Imidazol, *N*-Methylimidazol und 2-Methylimidazol wurde im Temperaturbereich von 25—600 °C differentialthermoanalytisch, thermogravimetrisch und differentialthermogravimetrisch untersucht. Obwohl das Endprodukt der Zersetzung in allen Fällen Cr_2O_3 ist, sind doch interessante Unterschiede im Gesamtprozess der Zersetzung zu beobachten. Die Ursachen für diese Unterschiede scheinen mit dem Trans-Effekt und dem Vorliegen von Wasserstoffbrückenbindungen in den Imidazolkomplexen zusammenzuhängen. Die Enthalpien der verschiedenen Zersetzungsreaktionen wurden differentialthermoanalytisch bestimmt.

Резюме — С помощью ДТА, ТГ и ДТГ изучено термическое разложение комплексов трехвалентного хрома с имидазолом, N-метил- и 2-метил-имидазолом в атмосфере воздуха и в области температур 25—600°C. Наблюдали интересные различия в процессе полного разложения этих комплексов, хотя во всех случаях конечным продуктом разложения являлась окись трехвалентного хрома. Причины таких различий обусловлены транс-эффектом и наличием водородных связей в имидазольных комплексах. На основе ДТА определены энтальпии нескольких реакций разложения.