Complexation of Amino Acids with CrO_3 with reference to L-Phenylalanin.

Anil Kumar Pandey
Department of Chemistry, J.N College, Dhurwa
Ranchi, Jharkhand

Abstract:

L-phenylalanin is an Amino acid. When it was treated with CrO_3 then complexation reaction was observed. By using TG&DTA and IR analysis expected structure of complex was analysed and thermal degradation provides information about loss of groups from complex.

(Key Words: L-phenylalanin, Amino acids, Oxidation, Complexation.)

Introduction:

Amino acids are one of the essential constituent of naturally occurring proteins. Oxidizing agents attack amino acids at free amino groups. Initially either an α-imino acid(I) is formed which subsequently will be hydrolysed to a α-keto acid (II) and ammonia or the oxidation is accompanied by decarboxylation giving rise to final production of next lower aldehyde(III) and ammonia

Chemicals Used:

L-phenylalanine, Chromium Trioxide, Acetone, etc. (The chemicals used were of A.R.grade) **Experimental procedure**

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Oxidation of L-phenylalanin with CrO_3 was carried out in substrate : oxidant 1:1 and 1:2 molar ratios.

The desired strength of the oxidant was achieved by taking 0.01 mole of the substrate and 1g and 2g of CrO_3 respectively.

The substrate was mixed with minimum volume of distilled water (E-Merk). Solid CrO_3 was added with constant and vigorous stirring. Precaution was taken to avoid reaction being violent at the time of mixing the reagent. The solution of the substrate and the oxidant where mixed with constant and vigorous stirring for about 1.5 hour at 75^0C (No reaction was initiated at room temperature). The mixture wat left overnight for complete reaction. The product where collected as sample AC1 and AC2.

(in substrate : oxidant 1 1:1 and 1:2 molar ratio respectively.)

The solid obtained was then powdered in mortar and pestle. Successive washings with distilled water. Ethanol, dioxin and finally with acetone removed soluble impurities. The light brown complex obtained was insoluble in water and acetone. The product was dried and collected in an air tight bottle.

FTIR Analysis of Products :

The FTIR curves of AC1 and AC2 contain almost all the peak which are expected for their formulation. The FTIR band assigned for various groups are listed below.

AC1	AC2	Band Assignment	References
1133.10	1137.20	(NH_2)	3a
1080.72	1081.08	(NH_2)	3a
551.75	524.05	V(Cr – N)	3b

490	490	V(Cr – N)	3b
419.5	418.7	V(Cr – O)	3e
290	280.3	V(Cr – O)	3e

Thermal Analysis of AC1 :

The complex having empirical formula $CrC_9H_{13}NO_6$ (AC1) shows exothermic loss up to temperature 176.05^{0}C with mass loss 21% (theoretically 20.63%) . The TG curve showed mass loss 63.999%
(theoretical 63.615%) in the temperature range 176.05^{o}C-600^{o}C attributed to the loss of benzoate. Escape of molecules like NH_3, H_2O and O_2 are expected which is indicated by mass loss of 81.999% (theoretical 81.639%) giving Cr.

Code	Temperature(^{0}C)	% mass left in experiment	Weight left Experimental (Theoretical)	Empirical Formula Predicted	Loss in Formula wt. Experimental (Theoretical)	Cumulative % Loss in Formula wt. Experimental (Theoretical)	Group loss (Theoretical)
AC1	R.T.		283.199	$CrC_9H_{13}NO_6$			
	Upto 176.05^{0}C	79	223.727 (224.155)	$CrC_7H_{10}NO_4$	59.471 (59.044)	21% (20.630%)	CH_3COO
	Upto 600^{0}C	36	101.951 (103.040)	CrH_5NO_2	121.776 (121.115)	63.999% (63.615%)	C_6H_5COO
	>1000^{o}C	18	50.975 (51.996)	Cr	50.976 (51.044)	81.999% (81.639%)	H_2O, NH_3, $½O_2$

Table : Thermal Analysis of AC1

Proposed Empirical formulation of AC1 is $[CrO(C_6H_5COO)(CH_3COO)(NH_3)(H_2O)]$ and the sequence of thermal degradation may be interpreted as follows

$[CrO(C_6H_5COO)(CH_3COO)(NH_3)(H_2O)] \longrightarrow [CrO(C_6H_5COO)(NH_3)(H_2O)] \longrightarrow [CrO(NH_3)(H_2O)$

$2Cr \longrightarrow$

Thermal Analysis of AC2 :

The complex having empirical formula $Cr_2C_9H_{15}NO_9$ (AC2) shows exothermic loss up to temperature 500^{0}C with mass loss 50% (theoretically 51.445%) . The TG curve showed mass loss 61.999% (theoretical 64.173%) in the temperature range 500^{o}C-869.87^{o}C attributed to the loss of ammonia and oxygen. Escape of molecules like H_2O and O_2 are expected above 1000^{o}C which is indicated by mass loss of 71% (theoretical 73.0031%) giving Cr.

Code	Temperature(^{0}C)	% mass left in experiment	Weight left Experimental (Theoretical)	Empirical Formula Predicted	Loss in Formula wt. Experimental (Theoretical)	Cumulative % Loss in Formula wt. Experimental (Theoretical)	Group loss (Theoretical)
AC2	R.T. Upto 500^{0}C		385.208	$Cr_2C_9H_{15}NO_9$			
		50	192.604 (187.034)	$Cr_2H_5NO_4$	192.604 (198.174)	50% (51.445%)	CH_3COO, C_6H_5COO,H_2O
	Upto 869.87^{0}C >1000^{0}C	38	146.379 (86.01)	CrH_2O_2	46.224 (49.028)	61.999% (64.173%)	NH_3,O_2
		29	111.710 (103.992)	2Cr	34.577 (34.014)	71% (73.003%)	H_2O, ½O_2

Table : Thermal Analysis of AC2

Proposed Empirical formulation of AC2 is $[Cr_2O_3(C_6H_5COO)(CH_3COO)(NH_3)(H_2O)_2]$ and the sequence of thermal degradation may be interpreted as follows

$[Cr_2O_3(C_6H_5COO)(CH_3COO)(NH_3)(H_2O)_2] \longrightarrow [Cr_2O_3(NH_3)(H_2O)] \longrightarrow [Cr_2O(H_2O)]$

$2Cr \longrightarrow$

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