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

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Abstract:

L-phenylalanin is an Amino acid. When it was treated with TBC 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, TBA, 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,TBA(Tert.Butyl Alcohol), Acetone, etc. (The chemicals used were of A.R.grade)

Experimental procedure :

Oxidation of L-phenylalanin with TBA 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 in TBA respectively.

The substrate was mixed with minimum volume of distilled water (E-Merk). Solid CrO_3 in TBA 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 ATC1 and ATC2.

(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 ATC1 and ATC2 contain almost all the peak which are expected for their formulation. The FTIR band assigned for various groups are listed below.

ATC1	ATC2	Band Assignment	References
1135.35	1135.99	(NH_2)	4a
1081.05	1080.83	(NH_2)	4a
540.70	538.31	V(Cr – N)	4b

490	490	V(Cr – N)	4b
419.5	418.7	V(Cr – O)	4e
281.0	282.6	V(Cr – O)	4e

Thermal Analysis of ATC1 :

The complex having empirical formula $CrC_9H_{13}NO_6$ (ATC1) shows exothermic loss up to temperature 160.67^{0}C with mass loss 13.999% (theoretically 14.947%) . The TG curve showed mass loss 34.999% (theoretical 37.208%) in the temperature range 160.67^{o}C-388.77^{o}C attributed to the escape of molecules like NH_3, H_2O & O_2. Finally above 864.44^{o}C oxidative dissociation is indicated by mass loss of 64.499% (theoretically 66.798%) giving mixed oxides of Cr.

Code	perature(^{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)
ATC1	R.T.		301.170	$CrC_9H_{13}NO_6$			
	Upto 160.67^{0}C	86	259.006 (224.155)	$CrC_8H_{12}NO_4$	42.163 (45.017)	13.999% (14.947%)	HCOO
	Upto 388.77^{0}C Upto 864.44^{o}C	65	195.760 (103.040)	CrC_8H_7O	63.246 (67.043)	34.999% (37.208%)	H_2O, NH_3,O_2
		35.5	106.915 (99.993)	CrO or mixed oxides of Cr	88.845 (89.117)	64.499% (66.798%)	Volatile Products

Table : Thermal Analysis of ATC1

Proposed Empirical formulation of ATC1 is $[CrO(C_6H_5COO)(NH_3)(H_2O)(CH_3COO)]$ and the sequence of thermal degradation may be interpreted as follows

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

$[CrO(C_6H_5COO)] \longrightarrow$ Metal oxides of Cr

Thermal Analysis of ATC2 :

The complex having empirical formula $Cr_2C_9H_{13}NO_8$ (ATC2) shows exothermic loss of benzoate and water up to temperature 397.18^{0}C with mass loss 34.999% (theoretical 32.893%) . Finally above 866.79^{o}C oxidative dissociation is indicated by mass loss of 66.999% (theoretical 66.997%) giving mixed oxides of Cr. Here loss of mass is expected due to small molecules like NH_3, H_2O,CO_2 & O_2

Code	perature(^{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)
ATC2	R.T.		367.194	$Cr_2C_9H_{13}NO_8$			
	Upto 397.18^{0}C	65	238.676 (232.072)	$Cr_2C_2H_8O_6$	128.517 (121.115)	34.999% (32.893%)	$C_6H_5CO_2$,
	Upto 866.79C	33	121.174 (119.991)	Cr_2O or mixed oxides of Cr	117.495 (112.087)	66.999% (63.997%)	Volatile Products

Table : Thermal Analysis of ATC2

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

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

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