Characterization of Aluminium Ethoxide by IR and GLC Analysis

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Abstract– **Aluminium ethoxide was prepared by the direct reaction of aluminium metal and dry ethanol using mercury (1) Chloride as catalyst. The final product was analyzed by IR & GLC. The results of these analyses indicated the formation of polymeric aluminium ethoxide.**

Keywords– **Aluminium Ethoxide, IR, GLC and Terameric**

I. INTRODUCTION

Aluminium alkoxides have been used as catalysts and intermediates in a variety of pharmaceutical and industrial processes [1], [2]. The literature survey showed the detailed structure of aluminium alkoxides is not completely known. The structural chemistry of aluminium alkoxides posse many problems, owing to the tendency of aluminium to attain coordination numbers of 4, 5, or 6 depending on the steric require ments of Ligands [3]. The proposed structure for aluminium ethoxide was tetrameric [4], [5]. The mass spectrum of aluminium ethoxide and the proposed mass fragmentation pattern appears to indicate that in addition to tetrameric and other lower species, some pentameric species are present and the fragmentation scheme of Fig. 1 can be deduced form different mass ion peaks [3].

Improvements in spectroscopic and chromatographic instrumentation in recent years have given encouraging results when applied to the elucidation of the constitution of aluminium alkoxides. Therefore, the aim of the present work is to elucidate the structure of aluminium ethoxide prepared by direct route using IR & GLC analysis.

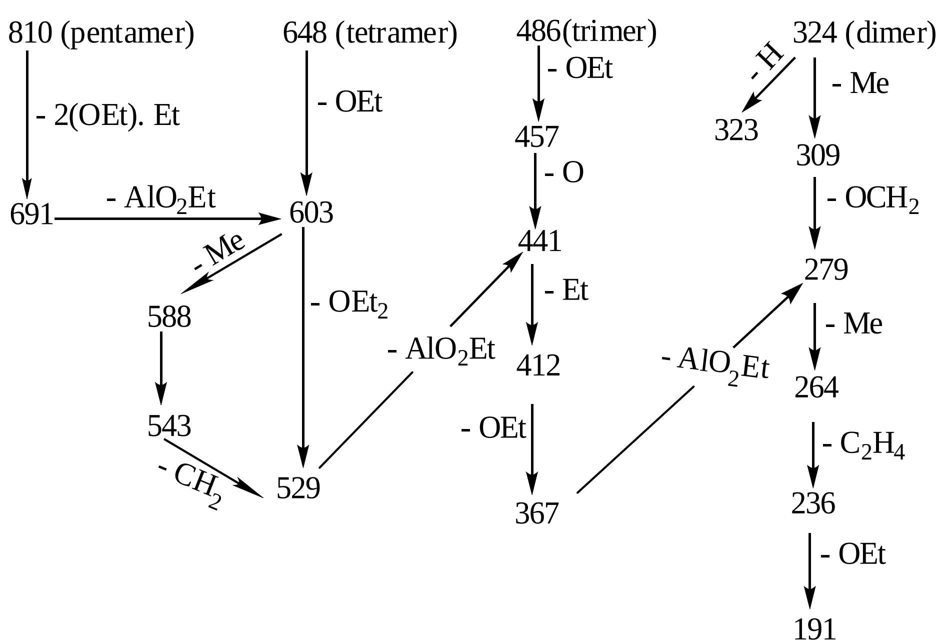

Fig. 1: Proposed mass fragmentation of pattern of $[Al(OEt)_3]_n$

II. EXPERIMENTAL

A. Chemicals

All chemicals used were of analytical grade type.

B. Apparatus

1) Infrared spectra

A Perkin Elmer spectrophotometer has been used to record infrared spectra of each prepared sample over the range of (4000 – 600) cm^{-1}. A small amount of the prepared sample was spotted on tow sodium chloride plates which were pressed together to give athin film. Liquids were used directly; solids were dissolved in absolute athanol. Absorption frequencies are given in cm^{-1}.

2) Gas Liquid chromatography

A Hewett Packard Gas chromatography series 5840 GC with flame ionization detector filled at 250 ºC was used to obtain chromatograms. The carrier gas nitrogen, and the column used is OV17 which is 1.5 m long, fitted at temperature range of 50 – 300 ºC. GIC analysis of absolute ethanol has taken as reference compound (see Fig. 2).

III. PREPARATION OF ALUMINIUM ETHOXIDE

Aluminium powder about 1g was introduced into a 3 – neck round bottom flask. Dry ethanol (100 cm^3, 0.789 g/cm^3, 1.72 mol) was added in addition to atiny amount of mercury (I) chloride. The reaction mixture was stirred at temperatune of 78 ^oC and refluxed for 40 hours. On heating the reaction mixtune gas evolution was observed. Then the mixture was filtered to separate unreacted aluminium powder. The filtrate was distilled to remove excess ethanol until white gelatinous solid product was obtained. The product was about 5.3g.

IV. RESULTS AND DISCUSSION

Aluminium ethoxide was prepared by direct reaction between aluminium metal and Liquid ethanol using mercury (I) chloride as catalyst. The final product was analysed by IR & GLC (see Fig. 3, Fig. 4, Table 1 and Table 2). The resutts of IR analysis were in a good agreement with reported values in Literature [3], [6]. The results of GLC analysis showed the presence of two compounds (Fig. 4 and Table 2). Compound (1) is aluminium ethoxide and compound (2) indicates the existence of polymeric aluminium ethoxide due to the appearance of broad peak. The absent of peak for ethanol may be attributed to the high temperature of apparatus compared with its low boiling point.

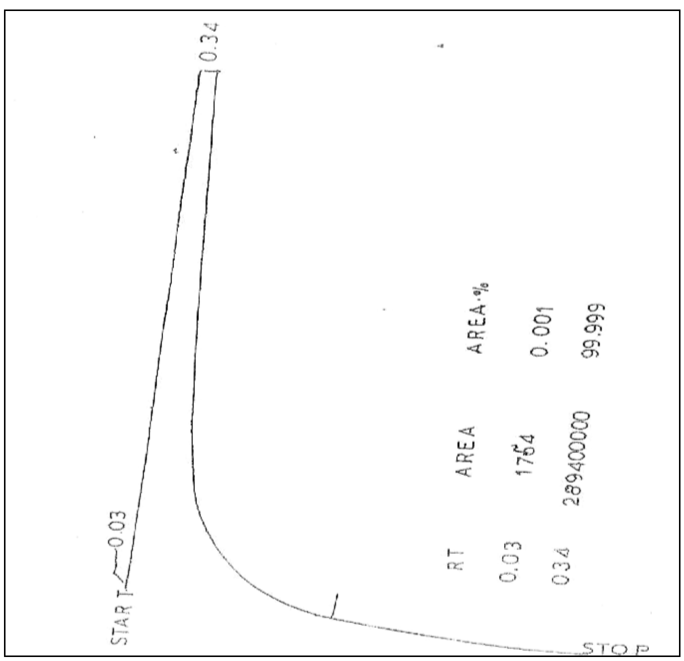

Fig. 2: GLC analysis for absolute ethanol

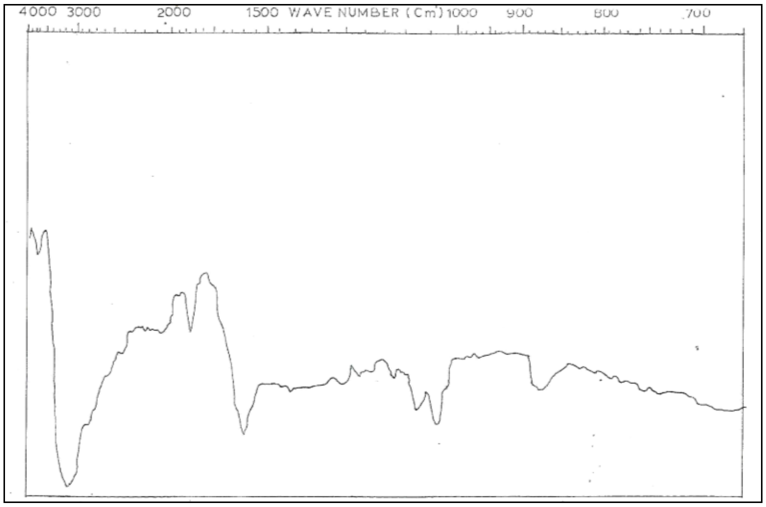

Fig. 3: infrared spectra of the product obtained from the reaction of Aluminium metal and dry ethanol using mercury (I) chloride as catalyst

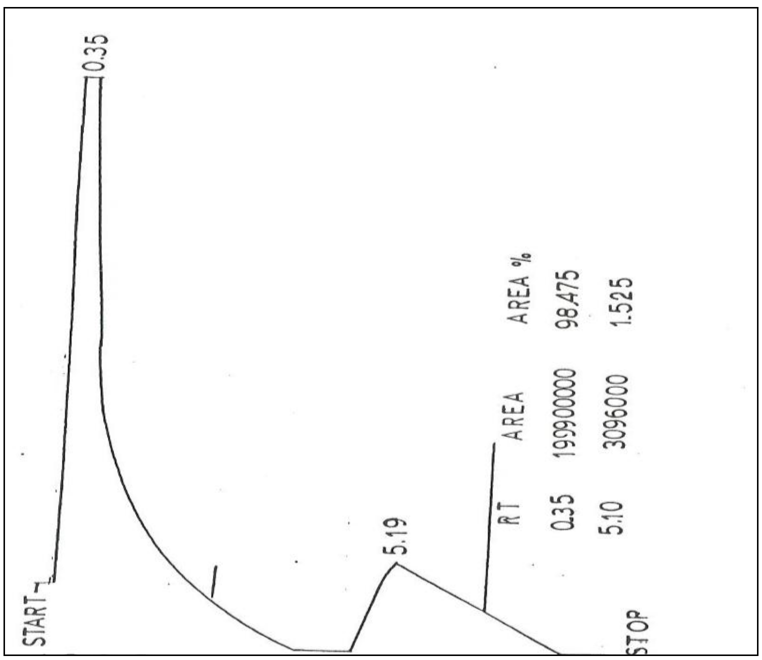

Fig. 4: GLC analysis of the final product obtained from the reaction between Aluminium metal and dry ethanol using mercury (I) chloride as catalyst

Table 1: Infrared spectral analysis of the product obtained from the reaction between aluminium and dry ethanol using mercury (I) chloride as catalyst

Frequency cm^{-1}	Assignment
3300 b	O – H stretching
2900 w	C – H (asymmetric and symmetric stretching
2500 w	C – H (asymmetric and symmetric stretching
2155 vw	C – H (asymmetric and symmetric stretching
1900 s	C – H (asymmetric and symmetric stretching
1610 m	C – H (bending in CH_3)
1250 b	C – H (bending)
1050 m	C – O stretching
870 b	Al – O – Al asymmetric stretching

b = Broad, s= Strong, m= Moderate, w= weak, vw= very weak

Table 2: GLC analysis of the final product obtained from the reaction between aluminium powder and dry ethanol using mercury (I) chloride as catalyst

Rt	Area % Assignment
0.53	98.475 compound (1)
5.10	1.525 compound (2)

V. CONCLUSION

Aluminium ethoxide is electron deficient compound and it has tendency to form auto complex. This has been supported by the results of GLC analysis for the product which shows the presence of broad peak. This was due to the existence of polymeric aluminium ethoxide.

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