ABSTRACT
The result of oxidation of Menadione by CericSulphate ion are summarized below:
(1) The specific rate is related to concentration of CuSO₄ by relationship.
K = K₀ + Conc. Cu²⁺
(2) The reaction is first order, both in Ce (IV) and Menadione.
(3) The mole ratio of reaction is two moles of Menadione to one mole Ce(SO₄)₂.
(4) Final reaction product has been identified as rearrangement products of epoxide of Menadione.

Keywords---- Menadione, TLC, Physicochemical

I. INTRODUCTION
It is evident, therefore, that enough attention has not been paid to the study of oxidation of certain chemicals with medicinal values having both positive and negative inductive effect by various oxidants like Ceric Ammonium Sulphate. The investigation of oxidation of Menadione (2-Methyl-1, 4-napthaquinone, Vitamin K₃) are likely to offer many new aspects which may add immensely to our knowledge in the fields of both kinetics and medicines.

To isolate and identify the oxidation products of menadione by physicochemical methods. The oxidation products will be separated by solvent extraction and thin layer chromatography (T.L.C) methods. Identification of these products will be done by elemental analysis mixed melting point, infrared (I.R) spectrophotometer, Gas Chromatography (G.L.C/G.S.C), High Pressure Liquid Chromatography and Thin Layer Chromatography (T.L.C). Polarography will be employed wherever possible to determine the number of electrons transferred during the oxidation process.

II. STUDY OF REACTION PRODUCT
It was observed that reaction mixture change colour with time. Thus then solution changes from yellow to reddish brown and finally a brownish precipitate and supernatant liquid is formed to be colourless. After that no further changes in colour were observed with time, indicating thereby that reaction had probably completed. This led to the conclusion that the oxidation of Menadione proceeds through different stages and the compound found in the intermediate stages may positively be separated out. Hence, the separation of these intermediate compounds was in the following manner.

To a solution of 5.0 grams of Menadione in 100ml of Methyl Alcohol (Methanol) a solution of 5.0 grams of Cerium (IV) in 2N Sulphuric Acid was made with constant stirring in a 1000ml of Borosil glass beaker. Then, the reaction starts immediately.

The mixture was allowed to stand at room temperature for 2 hours and the distilled over. This compound was then extracted with ether and then it is crystallized, IR Spectroscopy, H.P.L.C, T.L.C. and G.L.C.

III. IDENTIFICATION OF REACTION PRODUCT

BY THIN LAYER CHROMATOGRAPHY (T.L.C)
Number of methods using different mobile phase has been tried to separate out and identify the reaction product. Finally the following method was found successfully to separating different components of reaction mixture.

IV. PARAMETERS OF T.L.C. CHROMATOGRAPHY

| Absorbant Layer | : | Silica Gel HF 254 |
|-----------------|:|------------------|
| Layer of Thickness | : | 0.25 mm |
| Format | : | 10 x 10 cm |
Preparation and Drying: The coated plates were 105°C for 30 mins prior to use.
Separation Technique: Ascending
Chamber Saturation: The chamber was lined on three sides with filter paper and saturated for 30 min.
Length of run: 8 cm
Solvent composition and total volume: Cyclohexane and Ether (80:20) 100 ml
Preparation of the samples: 0.5% w/v of sample
Preparation of Ref. Standard: 0.5% w/v of Ref. Std in Cyclohexane
Amount applied: 10 µl

V. DETECTION

After removal of plate, it was dried in current of hot air, and examined under UV Lights at 254 nm. Sample chromatograph shows two spots while in reference std – only one was seen. The spot at higher Rf values due to Menadione while at Lower Rf value is due to oxidised form of Menadione that seems to be rearrangement of epoxide of oxidized form Menadione.

The plate was sprayed with a mixture of one volume HCl acid, four volumes of 0.25% w/v of 1.10 – phenothroline hydrochloride in ethanol. The spot at lower Rf value showed a change of colour to orange, a confirmatory test for Menadione.

BY I.R. SPECTROPHOTOMETER

VI. STANDARD PREPARATION

Ref. Std of Vitamin K₃ equals to 15 mcg/ml in n-Heptane.

CONDITIONS OF CHROMATOGRAPHY

Test Plate: HPTLC pre-coated plates, silica gel 60 F254
Format: 10 x 10 cm thickness, 200 µm
Sporting: 15 µl (CAMAG Automatic sampler-ATS-3)
Amount per spot/band: 600 mg
Separation Technique: Ascending
Development Chamber: Normal TLC Chamber, saturated with mobile phase:
Mobile Phase: Chloroform-Cyclohexane (55+45 v/v)
Relative Humidity: 50%
Temperature: 20°C
Migration Distance: 70 mm
Detect: (UV) Densitometric scanning (CAMAG densitometer: CAT-II)
Mode: Absorbance wave length: 290 nm
Slit Dimension: 0.2 x 5 mm
HRF: Vitamin K₃ Acetate-50

REFERENCES