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# RADIOLYSIS OF 2-PROPANOL IN PRESENCE OF 2-HEXANOYL FURANE; THE YIELDS OF PRINCIPAL PRODUCT IN SOLUTION

BY

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## دراسة النواتج الأساسية لتشعيع الكحول الأيزوبروبيلى فى وجود ٢ ـ هيكسانويل الفيوران

خلاصة : قد تم تعيين المركبات الأساسية التى تكونت نتيجة لتعرض ٢ ـ هيكسانويل الفيوران فى الكحول الأيزوبروبيلى لأشعة جاما ، وقد وجد أن أهم تلك المركبات وأكثرها تكونا هو ٢ ـ هيكسانويل ٥ ـ ( ٢ ـ هيدروكسى ٢ بروبيل ) فيوران . وقد تم دراسة تأثير الجرعه الاشعاعيه المكتسبه على كمية هذا الناتج ، وقد تم دراسة ومناقشة ميكانيكية تكوين المركبات المكتشفه التى تكونت بالاشعاع فى المحلول .

## ABSTRACT

2- hexanoyl, 5- (2-hydroxy 2-propyl) furan (HHPF), has been investigated as the principal product of radiolysis of 2-hexanoyl furan (HF), in 2-propanol. The effect of absorbed dose on the product yields has been examined. Possible mechanisms for the formation of radiolytic products are discussed.

## INTRODUCTION

Ketones are now considered and known as electron and hydrogen atom scavengers during their radiolysis in alcoholic medium [1-4]. The radiolytic products are believed to be formed as a result of interaction between radicals created from alcohol and ketone molecules during $\gamma$-radiolysis. The main radicals formed due to $\gamma$-radiation of 2-propanol have been discussed and identified by different authors [5-11].

In the present work the radiolytic species formed from the $\gamma$-radiolysis of (HF) ketone have been predicted via the radiolytic product.

## EXPERIMENTAL

### Materials:

Treatment of pure 2-propanol to eliminate the traces of acetone was carried out according to the method described by Gilles [7]. After treatment, the purity of 2-propanol was examined by gas chromatograph using several coloumns with different polarities. 2-hexanoyl furan was prepared by the procedure described by Galli [12] for the preparation of phenyl ketones. The ketone was purified by different techniques including; several vacuum distillation and separation of the ketone by thin layer chromatography. (HF) and (HHPF) ketones were analysed by the following instruments:

IR Spectrophotometer. (Perkin-Elmer-457).
NMR T60. (Varian).
UV Beckman Spectrophotometer (Acta III).
Gas chromatograph (HP 3390 A).

The optimum coloumn for the analysis of (HF) and (HHPF) ketones was :

Coloumn packing SP 1000, (10%), 60-80 mesh; L=3m, D=2.5 mm., $T_{coloumn}$ =180°C., $T_{detector}$ = 250°C., $T_{injector}$ =250°C. and the flow rate of carrier gas, (He), =20 ml/min.

### Experimental procedure:

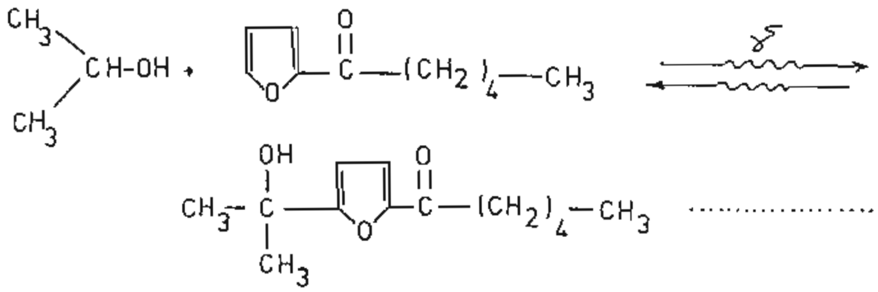
3.5 ml pyrex glass ampoules were utilized as containers for irradiation of prepared solutions. The ampoules were, at first, washed with chromic acid and rinsed thoroughly several times by distilled water. After the ampoules have been oven dried they were pre-irradiated for one hour.

Solutions of (HF) in 2-propanol were prepared as 0.1 and 0.01 M. 2.5 ml of each solution was . pipetted into ampoules. Gasses in these solutions were stripped off in a high vacuum line by the conventional freeze-melt technique. In this work from four to five cycles were made under a vacuum of $10^{-5}$ -$10^{-6}$ torr. Finally the ampoules were sealed off under vacuum. Irradiation of the ampoules containing solutions was carried out using a $^{60}Co$ (GAMMACELL TYPE 220) source from Atomic Energy of Canada Ltd. The absorbed dose rate (0.305 KGy/hr) was determined using freshly prepared Frick dosimeter solutions; $[G\ (Fe^{+3}) = 15.6\ (100\ eV)^{-1}]$ [13]

Qualitative determination of (HF) and (HHPF) ketones were made instrumentally as discussed before, and they were determined quantitatively via a standard curve. As (HHPF) is considered as the principal product; the other compounds results were not considered.

## RESULTS AND DISCUSSION

Fig. (1) shows the dependence of (HHPF) yields as a function of the absorbed dose with (HF) concentrations 0.1 and 0.01 M. The curves illustrate that the yield decreases as the absorbed dose increases.. At higher doses, (D >150 KGy), the rate of change of yield G with the dose tends to be smaller. This may be attributed to the following equilibrium:

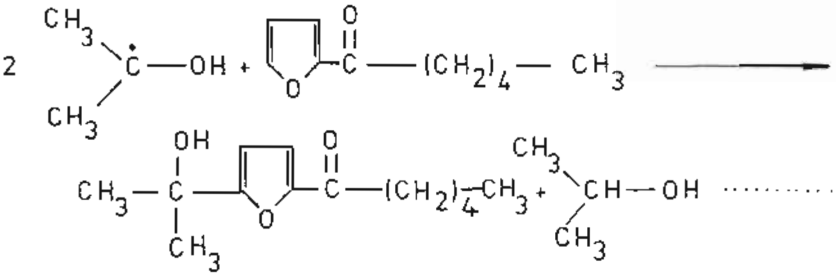


........................( 1 )

The rate of formation of (HHPF) at higher doses is less than that at lower ones due to gradual increasing and accumulation of this product in the medium. Moreover, the effect of γ-radiation on that product cannot be neglected.

At doses greater than 70 KGy, the radiation yield increases with the increase of (HF) concentration. This may, also, be attributed to the higher concentration of the ketone species which may interact with the solvent species to form (HHPF) ketone. This assumption may be supported from the results shown in Fig. (2). This figure illustrates the radiation yield of the disappeared ketone at different doses. It is, also, indicated from Fig. (2), that the loss in (HF) ketone at 0.1 M is higher than that at 0.01 M. Therefore, it may be expected that the concentration of created species in 0.1 M would be higher than in the case of 0.01 M solution.

The principal reaction which produce (HHPF) may be given as follows :



............(2)

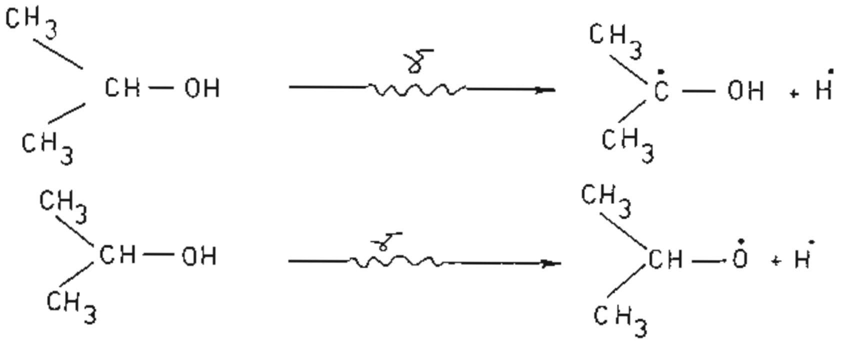
From reaction (2), it is obvious that, as the concentration of (HF) ketone increases the concentration of (HHPF) ketone would also be increased.

To interpret the formation of (HHPF) ketone the following three mechanisms are proposed :

(1) Radical initiation .
(2) Capture of solvated electrons .
(3) Participation of the excited state.

Similar mechanisms were proposed in the case of radiolysis of benzophenone and cyclohexanone in 2-propanol [1,2,3,14].

In the radical initiation mechanisms, the following principal reactions between the formed radicls from (HF) ketone and 2-propanol are proposed :



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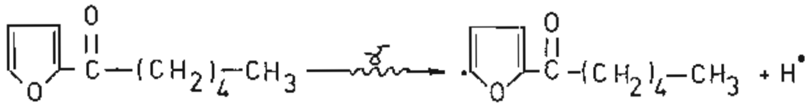
.........(4)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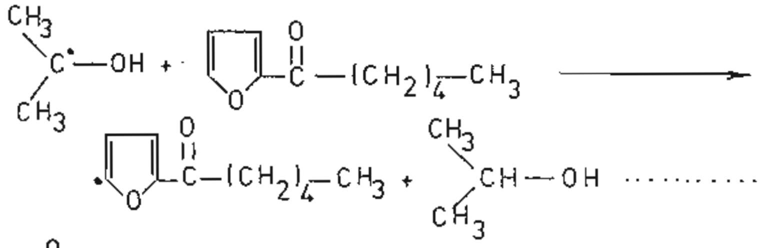
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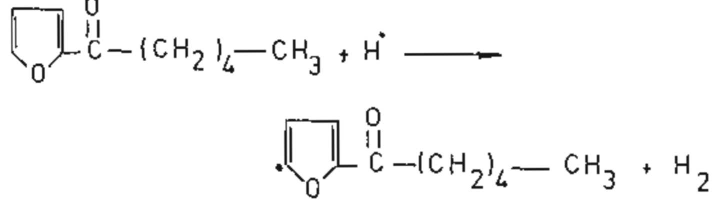
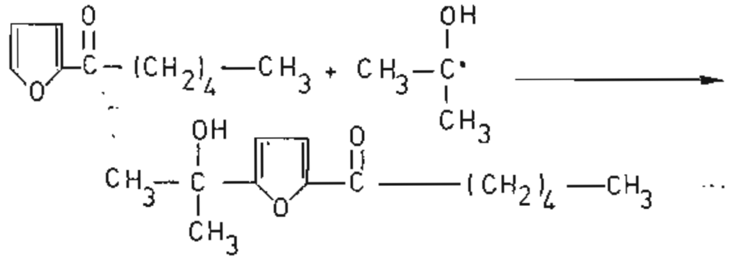
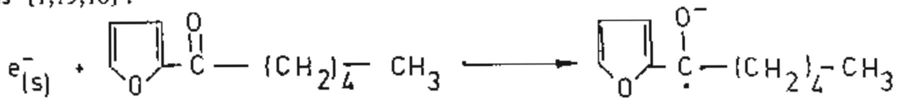
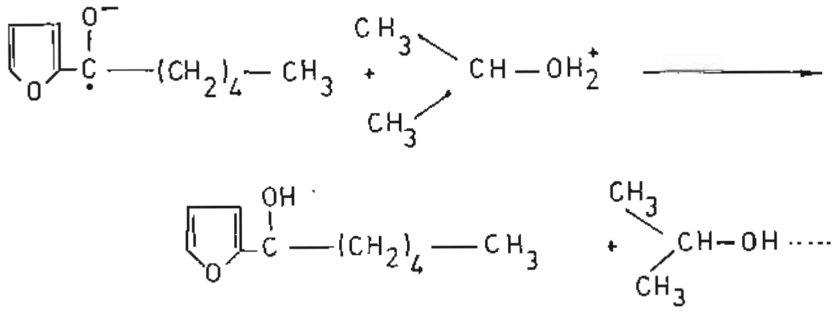

(7)


(8)

In the electron capture mechanism, it is well known that ketones can capture electrons forming the corresponding alcohols and pinacols according to the following reactions [1,15,16]:


(9)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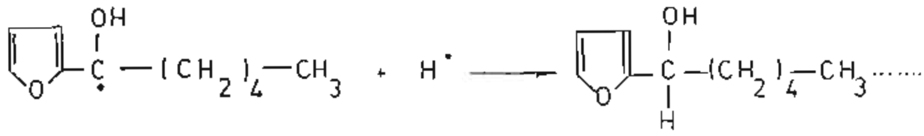
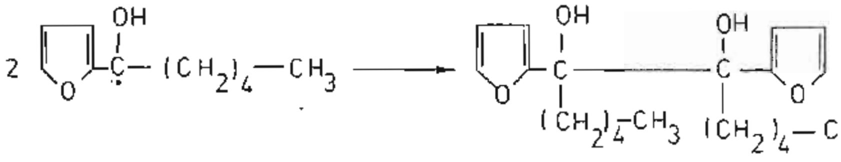

......(11)

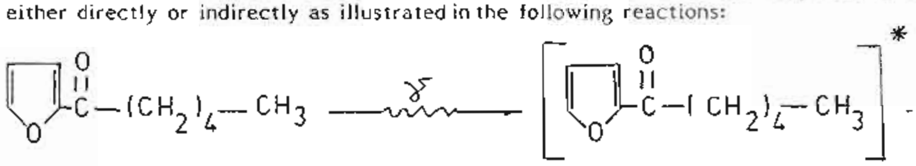
( Corresponding alcohol )


............ (12)

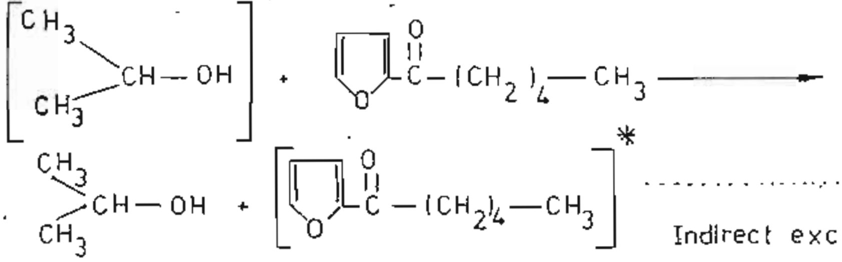
( Corresponding pinacols )

The radiolytic products: (HHPF), (FH) and (FD) had been detected in the irradiated medium and their mass spectra are shown in Figures: (3), (4) and (5) respectively.

In the excitation mechanism the molecules of ketones are supposed to be excited either directly or indirectly as illustrated in the following reactions:

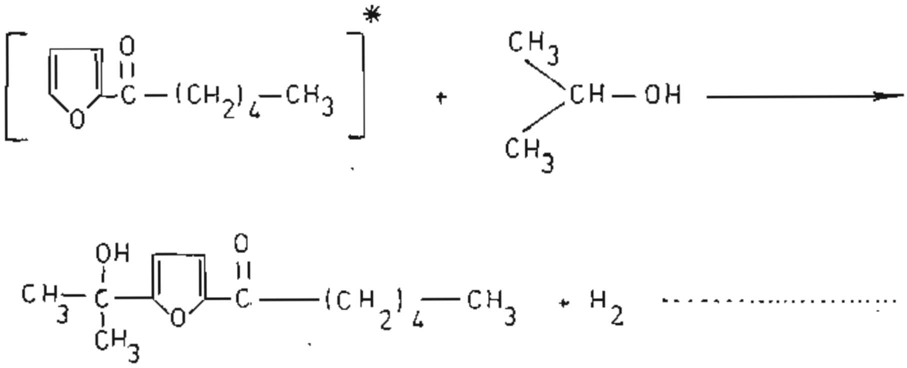

........ (13)

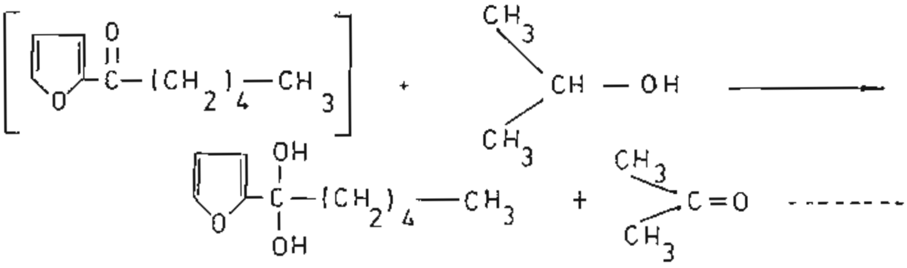
(Direct excitation)


.............................. (14)

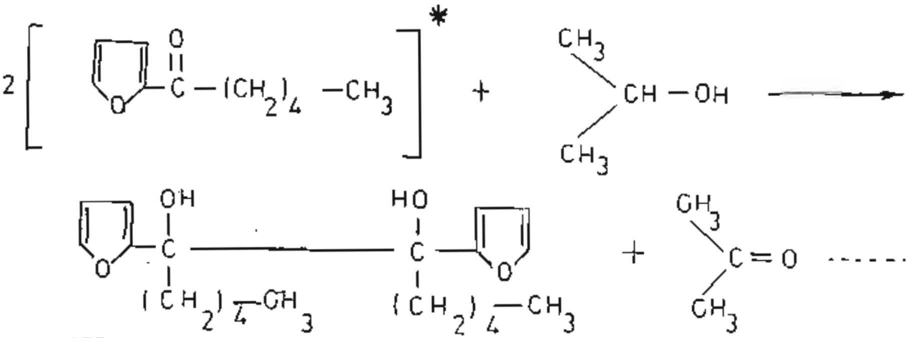
Indirect excitation)

The excited (HF) ketone molecules can produce the radiolytic products as follows :


........................ (15)



(16)



(17)

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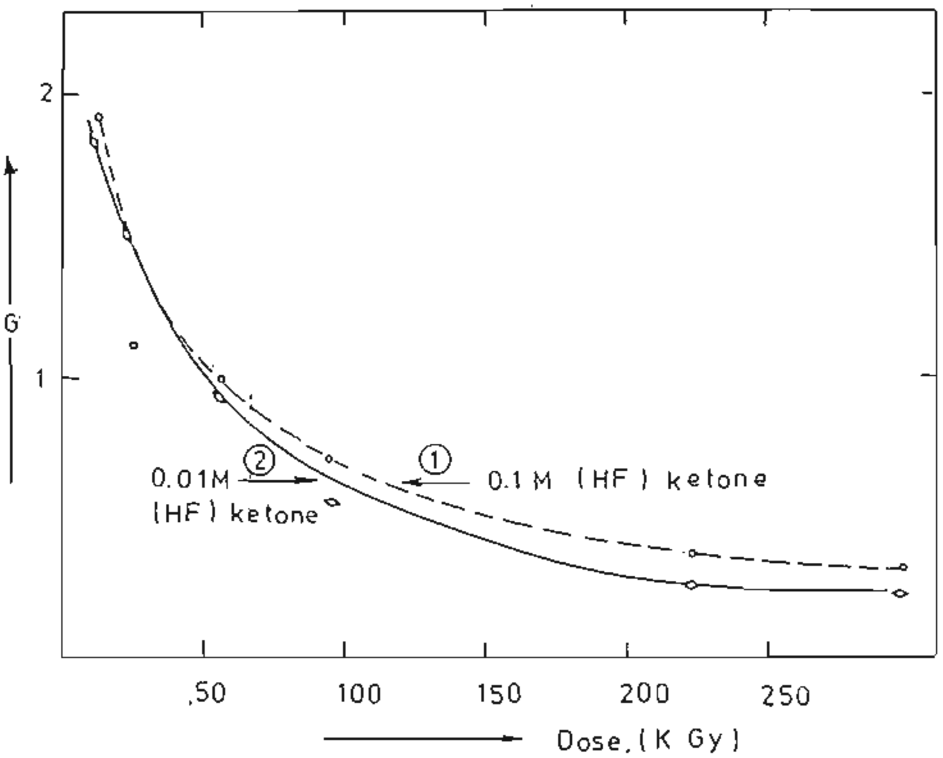

Fig.(1): Radiation yield G of (HHPF) ketone as a function of absorbed dose.

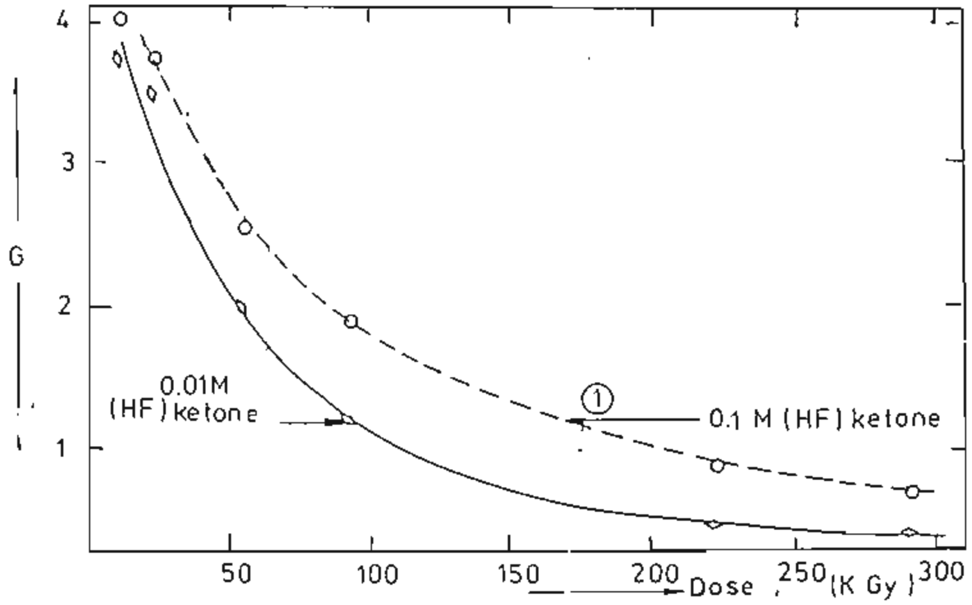

Fig.(2): Radiation yield G of the disappeared ketone as a function of absorbed dose

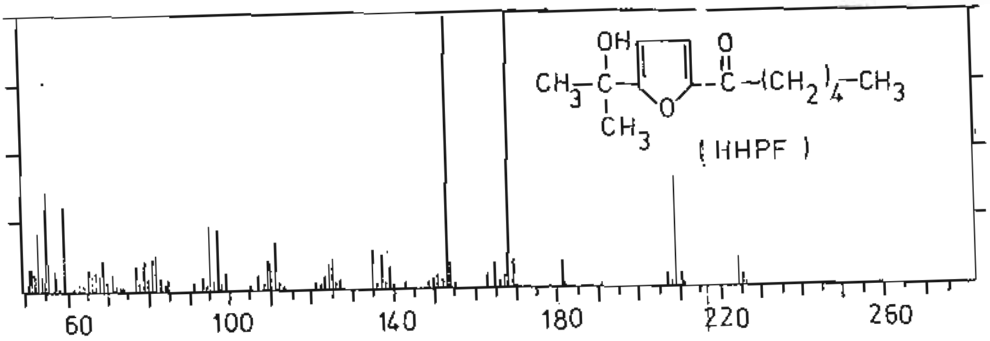

Fig.(3): Mass spectrum of 2-Hexanoyl, 5-(2-Hydroxy 2-Propyl) Furan, (HHPF)

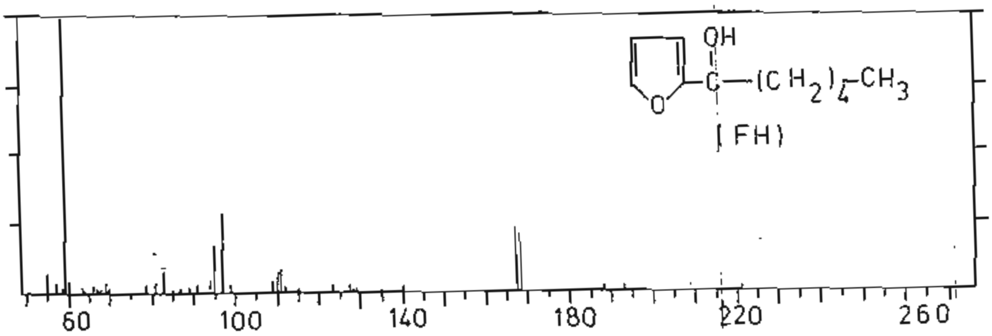

Fig.(4): Mass spectrum of 1-(2-Furanyl)-1-Hexanol, (FH).

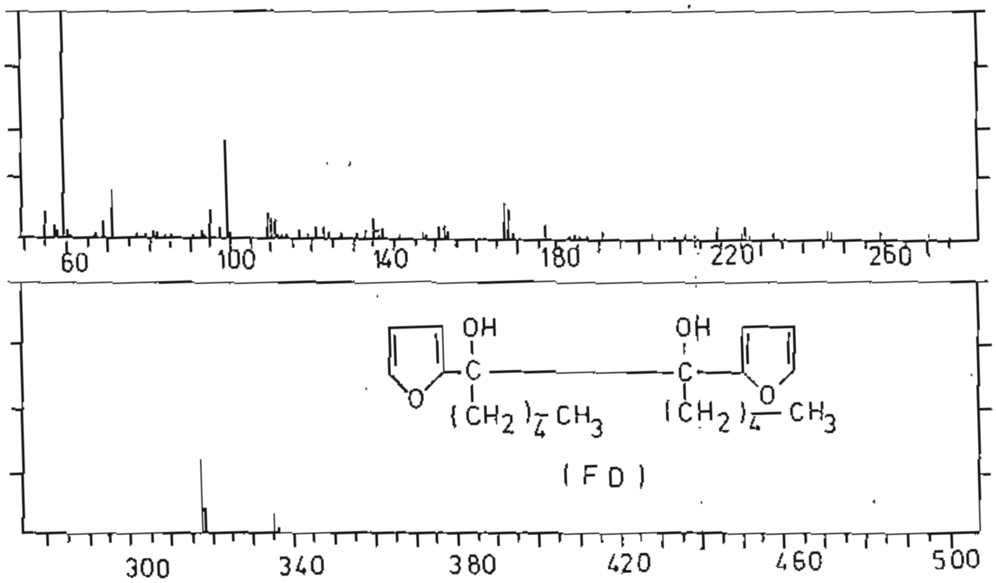

Fig.(5): Mass spectrum of 6,7-di-(2-furanyl), 6,7-decanediol, (FD).