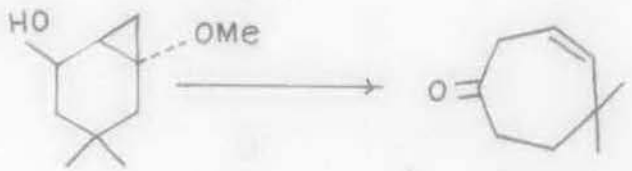
CHAPTER - VII

Potassium t-butoxide treatment on cyclopropyl ketones.

Section-A : Aims and objects:

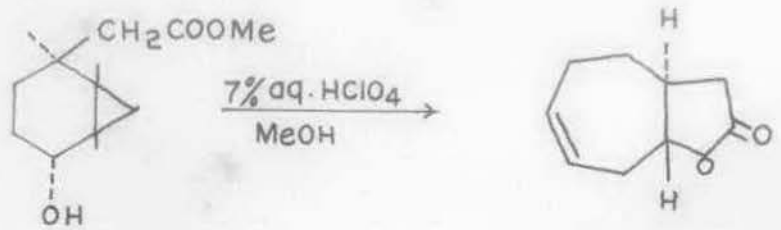
It is reported that cycloprane derivatives undergo different type of rearrangements when these were treated with basic or acidic substances and even with some solvent or by heating only.

E.Wenkert et al (168) found that cyclopropane derivative [A] produced [B] in presence of HCl



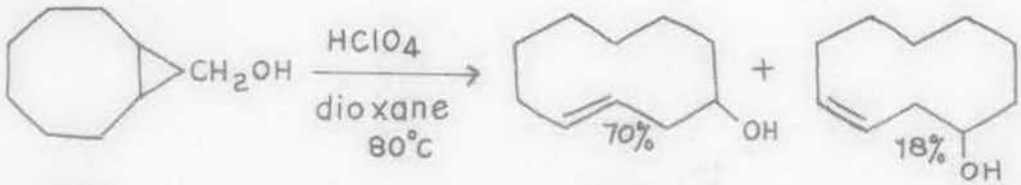
[A] [B]

When [C] (169) suffered a rearrangement to produce [D] in presence of aqueous $HClO_4$.



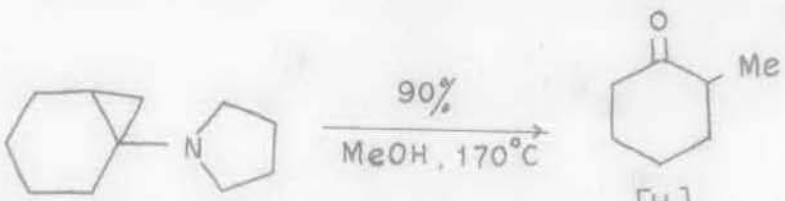
[C] [D]

Similarly when [E] (170) was treated with $HClO_4$, F was produced.



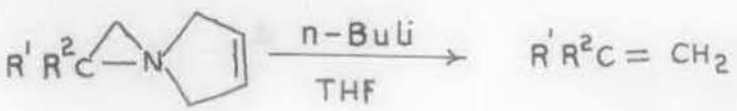
[E] [F]

M.E.Kuchne and J.C.King (171) found that at 170°C in presence of 90% methanol, the cyclopropane derivative [G] produced [H]



[G] [H]

When [I] (172) was treated with n-BuLi in THF [J] was formed.

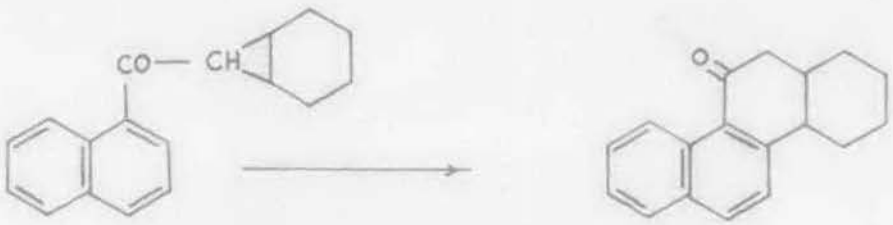


[I] [J]

Simple heating of [K] produced [L]

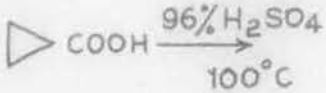
[K] [L]

In presence high boiling solvent compound [M] was converted into [N].



[M] [N]

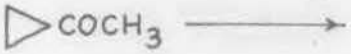
Treatment of 96% H_2SO_4 on [O] and [P] (173) produced [Q] and [R] respectively.



[o]

$CH_3COCH_3 + HO-SO_3(CH_2)_3COOH$

[q]

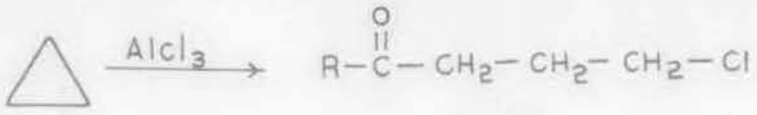


[P]

$HOSO_3(CH_2)_3COCH_3$

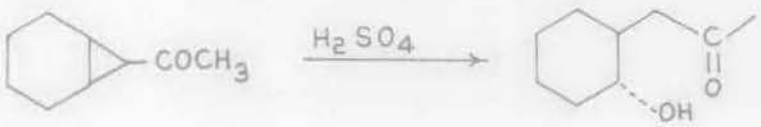
[R]

When simple cyclopropane was treated with $AlCl_3$, it was rearranged to [S] (174).



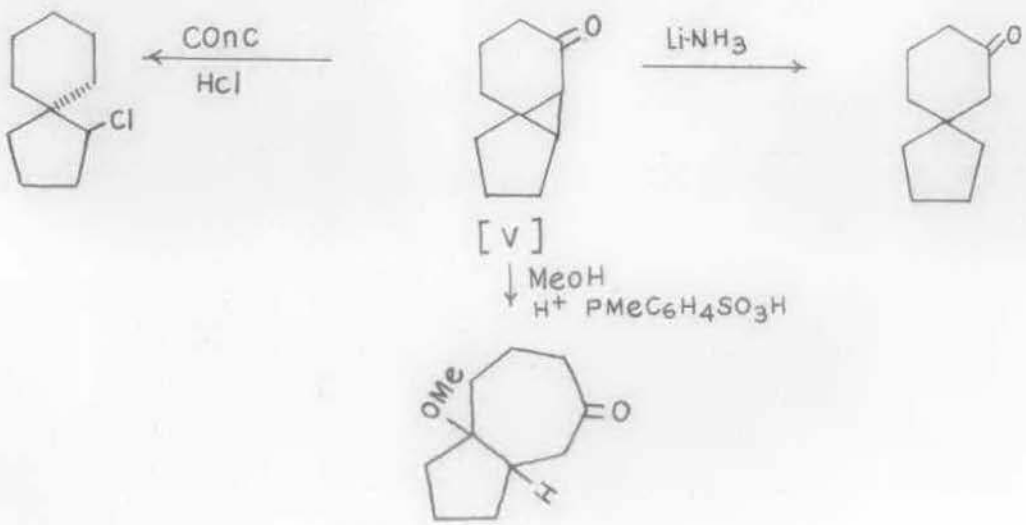
[S]

When cyclopropyl ketone [T] was treated with H_2SO_4, [U] was formed.



[T] [U]

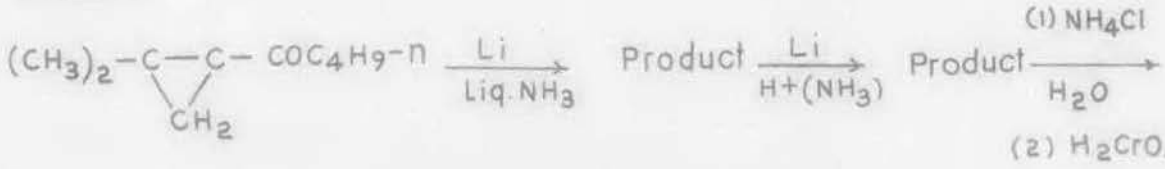
When cyclopropane derivative [V] (175) was treated $LiNH_3$, methanolic solution of p-toluene sulphonic acid or conc. HCl, it rearranged to different products.



A simple reductive cleave of cyclopropyl ketones has been (176) effected by irradiating a methanolic solution of a cyclopropyl ketone and tri-n-butyl hydride with ultraviolet.

Metal reductions have also been used to cleave C-C bonds in the case of cyclopropane derivatives (177), specially cyclopropyl ketones (178).

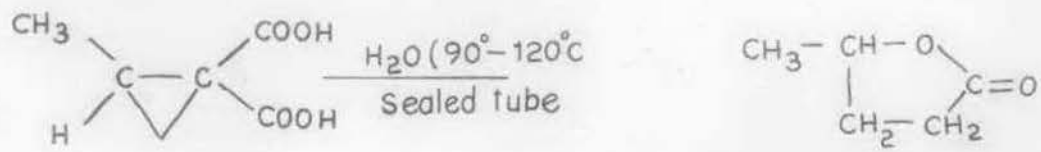
When following cyclopropyl ketone (179) was subjected to Li in liquid NH_3 and thereby other reagents it broke into two parts.



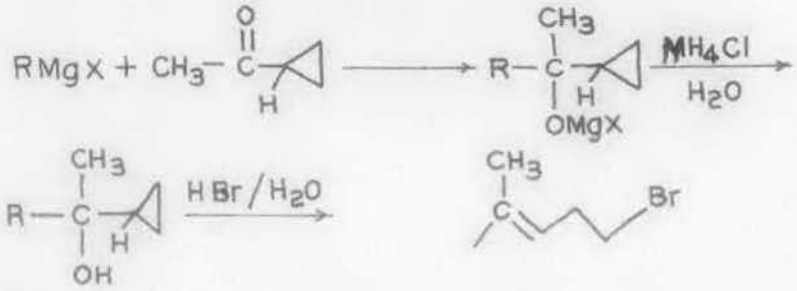
$(CH_3)CH \cdot CH_2 \cdot CH_2 \cdot CO C_4H_9\text{-}n$
8%

$+(CH_3)_3 \cdot C \cdot CH_2 \cdot CO\, C_4Hq\text{-}n$
18%

When following cyclopropane (180) was treated with H_2O at 90°-$120^\circ C$ in a sealed tube it suffered rearrangement.



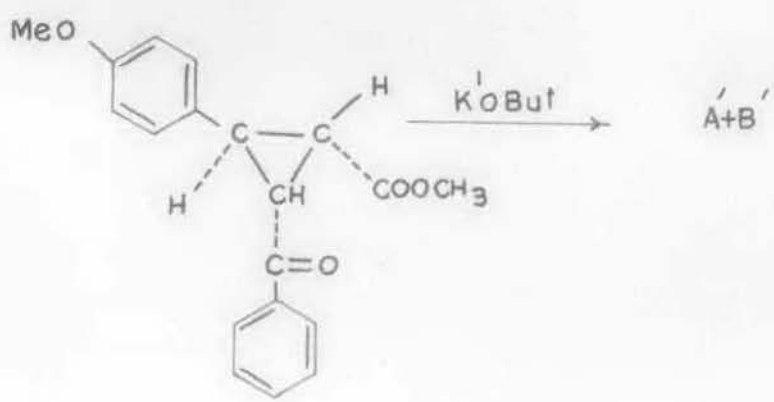
By the cleavage of cyclopropane derivative (181) Homollylic bromides and iodides were prepared.



So we were proposed to see the effect of potassium tert. butoxide on the cyclopropyl ketones. It was our attempt to note what type of change occurred when cyclopropyl ketones were treated with potassium t-butoxide. With this view in mind we treated potassium tert. butoxide on 1-benzoyl-2-carbomethoxy-2' (p-methoxy) phenyl cyclopropane.

Section-B: Results and discussion:

When 1-benzoyl-2-carbomethoxy-2'-(p-methoxy) phenyl cyclopropane in presence of potassium t-butoxide gave two products A' and B'



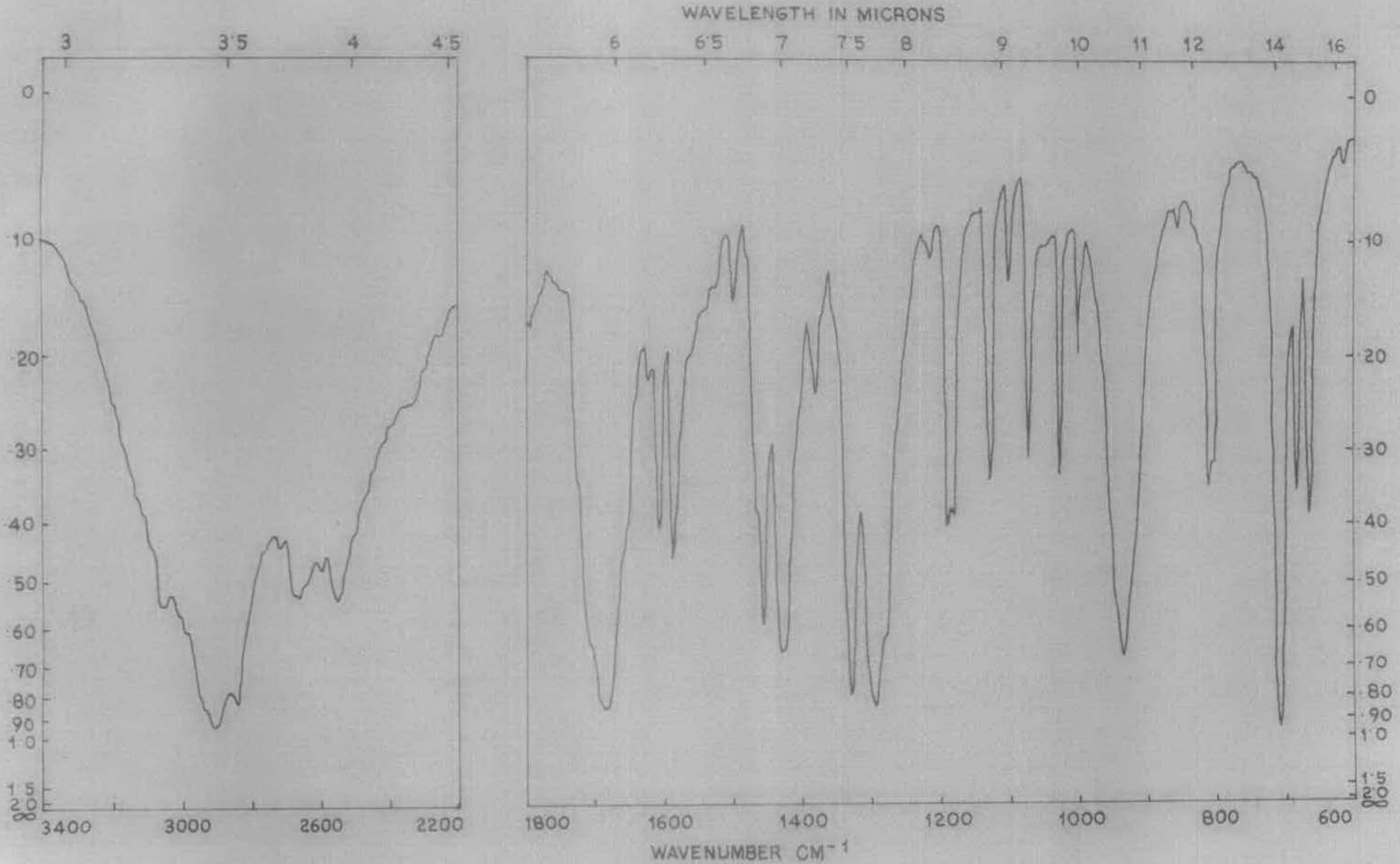

WAVELENGTH IN MICRONS
3
3·5
4
4·5
6
6·5
7
7·5
8
9
10
11
12
14
16
0
10
20
30
40
50
60
70
80
90
1·0
1·5
2·0
3400
3000
2600
2200
1800
1600
1400
1200
1000
800
600
WAVENUMBER CM⁻¹

Fig- 58.

A' was confirmed to be an acid by the usual test of an acid function. The structure of A' was confirmed to be the structure of benzoic acid from the observation of melting point, mixed m.p. I.R. bands, elemental analysis and mass spectrum.

The possible structure of B' was established on the observation of elemental analysis, I.R. and N.M.R. spectrum.

I.R. bands of this part at 1710 cm^{-1} and 1010 cm^{-1} indicated in presence of an ester function and a cyclopropane ring.

N.M.R spectrum of the compound [B'].

From the I.R observation of the part B', it is seen that this fraction contains an aromatic system having an ester function and a cyclopropane ring.

N.M.R spectrum of this component also shows signals at δ 6.8 to δ 7.15 (phenyl proton), δ 3.75 (CH_3 of carbomethoxy group) 3.85 (CH_3 of methoxy group), δ 2.5 (cyclopropane proton), δ 1.8 (cyclopropane proton), δ 1.55 (cyclopropane proton) and δ 1.25 (cyclopropane proton).

The signals δ 2.5 for H_a, δ 1.55 for H_c and δ1.25 for H_d can be assigned.

So from the studies of elemental analysis, I.R. and N.M.R, the probable structure of this part [B'] is as below.

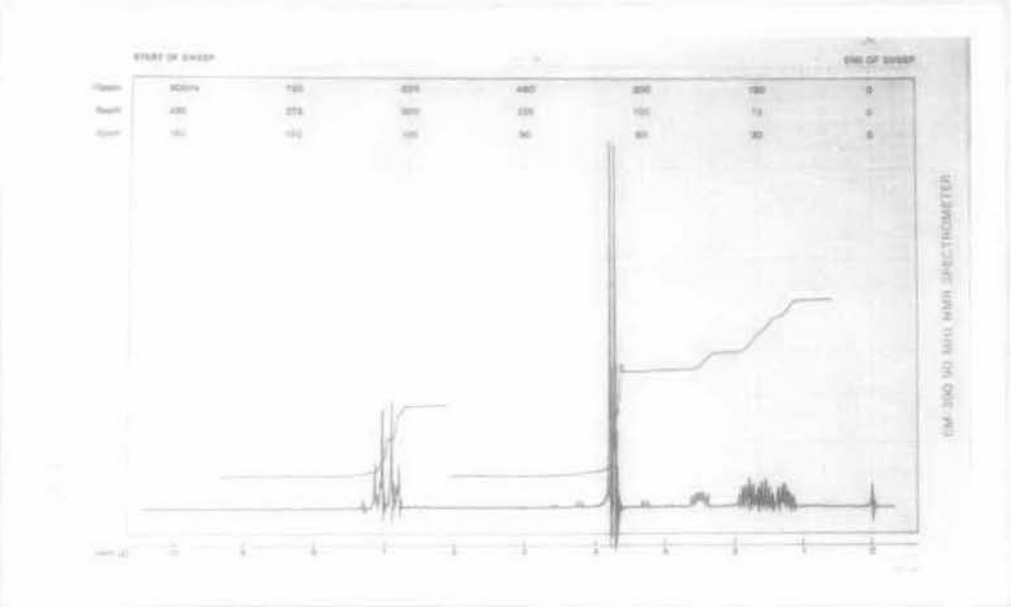


Fig. P.13. N.M.R. spectrum of B part of the p-t butoxide treatment on 1-benzoyl-2-carbomethoxy-2' (p-methoxy) phenyl cyclopropane.

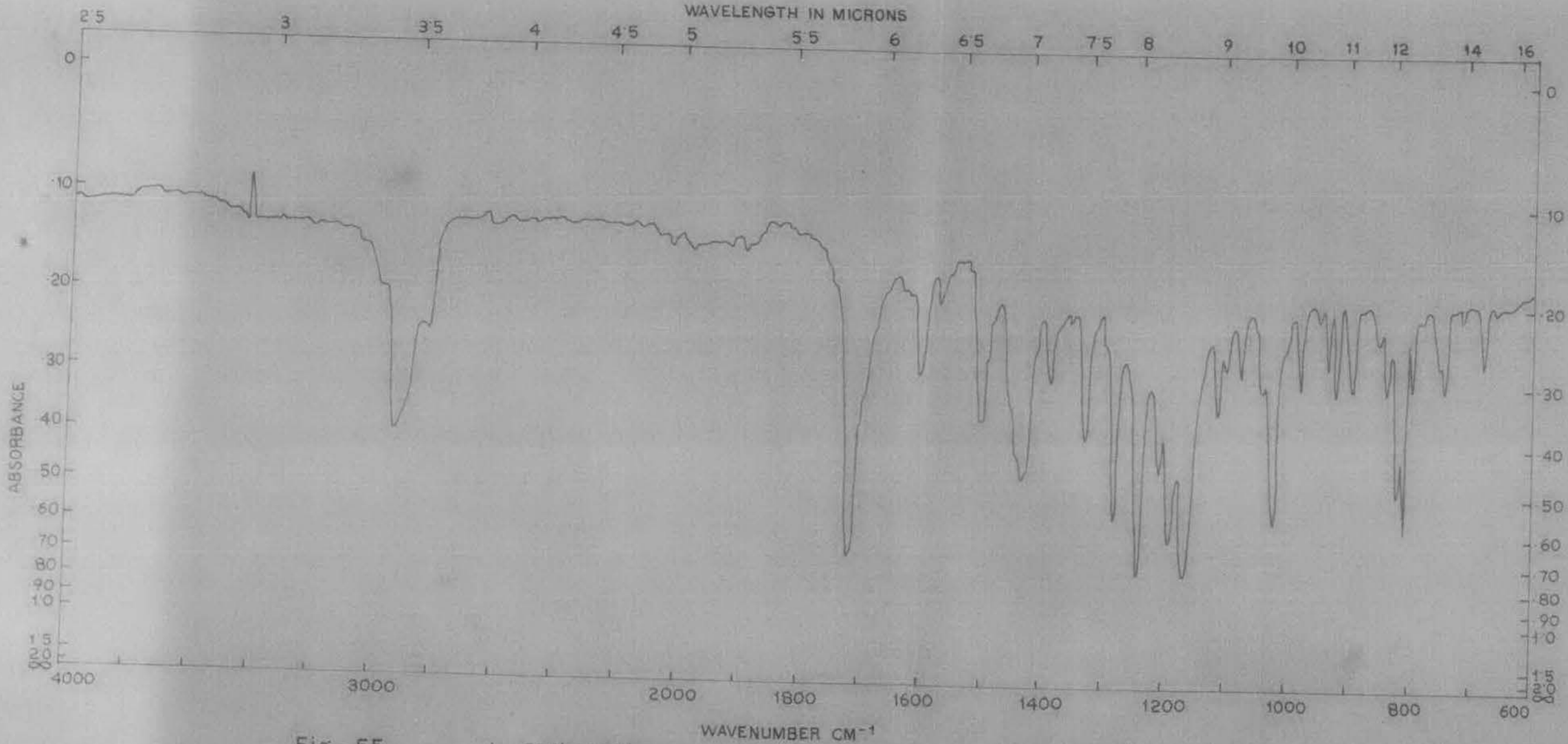


Fig- 55. 1- Carbomethoxy-2-(p-methoxy)phenyl cyclopropane.

crystallised from low boiling pet-ether. Yield-75 mg m.p. 121°C. No depression of m.p. was found when mixed m.p. with benzoic acid was seen Its I.R spectrum was identical with that of benzoic acid. (Fig.58)

Analysis found : C, 68.53%; H, 5.17%

Calculated for $C_7H_6O_2$: C, 68.85%; H, 4.91%

M/e = 122 (Fig.P 17)

(b) Ether portion: Ethereal solution was treated with dil. aqueous ammonium solution and then it was washed with water till neutral. It was dried over anhydrous sodium sulphate and ether was removed. Solid residue was crystallised from low boiling pet-ether. Colourless crystals of m.p. 46°C was filtered. Yield - 80 mg I.R. (neat) (Fig.55.).

Analysis found: C, 69.70%; H, 6.47%

Calculated for $C_{12}H_{14}O_3$: C, 69.99%; H, 6.79%

N.M.R spectrum (Fig.P 13.)

R E F E R E N C E S

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