# CHAPTER–V

# DETECTION OF THE FINAL PRODUCTS

# CHAPTER - V

## DETECTION OF THE FINAL PRODUCTS :

A knowledge of the end products formed in the reaction subjected to kinetic study is an important step in the education of the mechanis of the reaction.

After keeping for 24 hours, the reaction mixture was treated with potassium chloride to precipitate out the silver catalyst. The solution was filtered. Then the filtrate was boiled to destroy any peroxydisulphate remained in the solution. The solution was further concentrated and the test were performed as follows :

### Test for ammonia :

Test with P-nitrobenzenediazonium Chloride :

This test for ammonia was carried out in the case of crotonamide. In the case of pyruvamide, the solution was prepared in acetic acid and hence, ammonia was not detected due to acidic medium of pyruvamide.

A good sensitive test for ammonia depends on the red colouration with P-nitrobenzenediazonium Chloride (I), when it is shaken with the solution containing an ammonium salt and 10 % sodium hydroxide added drop by drop. A coloured ammonium salt of P-nitrophenylnitrosoamine (II) is formed.

Procedure :

A drop of slightly acid or neutral test solution is placed on the spot plate followed by a drop of Reiglers' reagent. Then a very small amount of calcium oxide is added. In the presence of ammonium salts, a red zone forms at once around calcium oxide. In the absence of ammonium salts the reaction is slow. For very amounts, a blank test should be carried out with a drop of water.

Thus the first oxidation in the case of crotonamide was ammonia but in the case of pyruvamide no ammonia was detected. The other oxidation products were formaldehyde (in the case of pyruvamide and crotonaldehyde in the case of crotonamide.

Test for formaldehyde :

(1) Aqueous solution + Tollens or Fehling's Solution ⟶ grey/black or red ppt. shows the presence of formaldehyde.

(2) Aqueous Solution + resorcinol + Con $H_2SO_4$ ⟶ red layer at the junction of two liquids. White ppt. changes to violet red in the aqueous layer.

The above tests were carried out according to Feigl.[198]

Tests for crotonaldehyde :

(1) To 1 cc of schiff's reagent add a few drops of O.S., Shake. A deep red colour is obtained.

(2) To 1 cc of Tollen's reagent add a few drops of O.S. and shake, A grey or black ppt. is obtained.

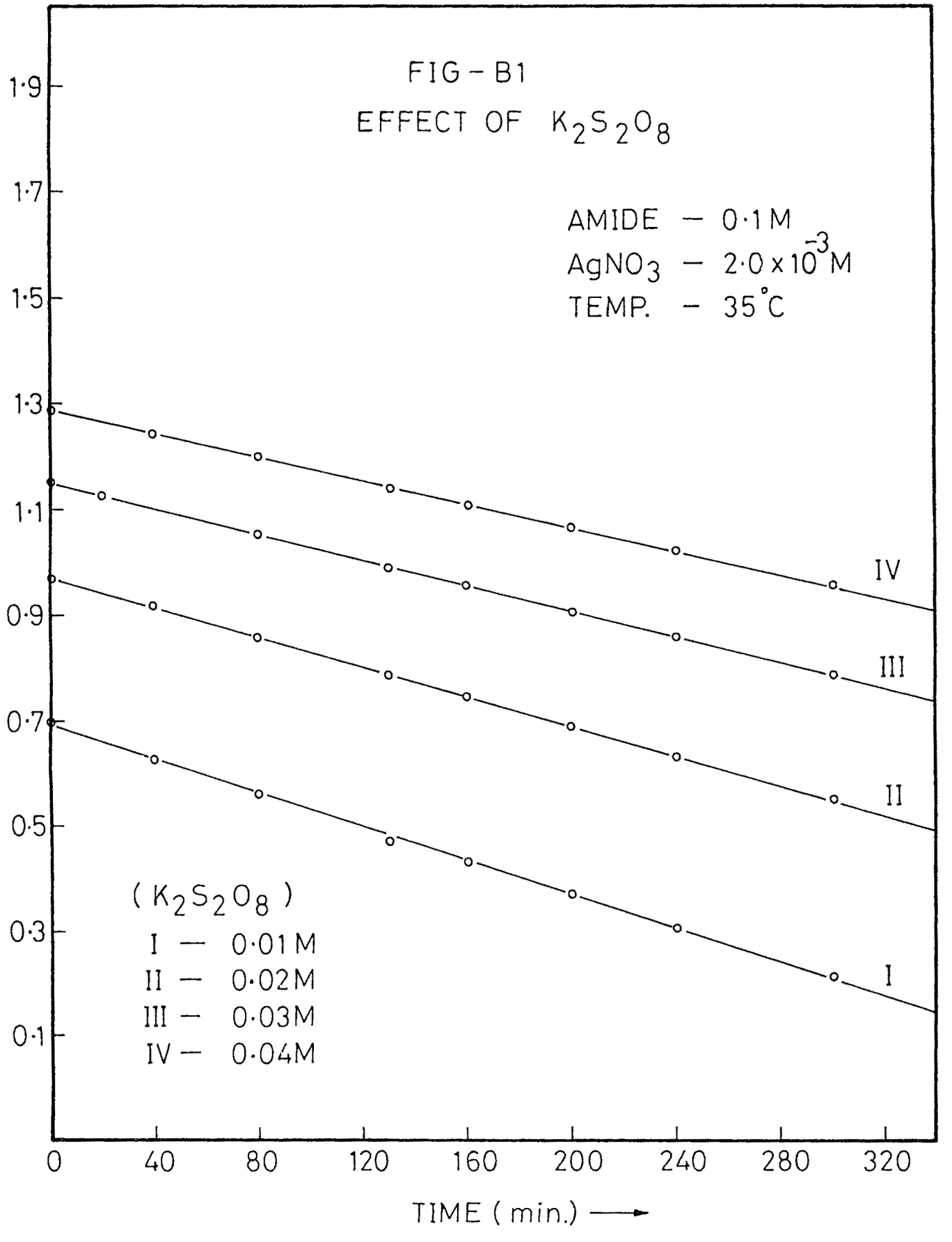


FIG - B1
EFFECT OF $K_2S_2O_8$
AMIDE — 0·1 M
$AgNO_3$ — $2·0 \times 10^{-3}$ M
TEMP. — 35°C
1·9
1·7
1·5
1·3
1·1
0·9
0·7
0·5
0·3
0·1
IV
III
II
I
( $K_2S_2O_8$ )
I — 0·01 M
II — 0·02 M
III — 0·03 M
IV — 0·04 M
0
40
80
120
160
200
240
280
320
TIME ( min.) ⟶

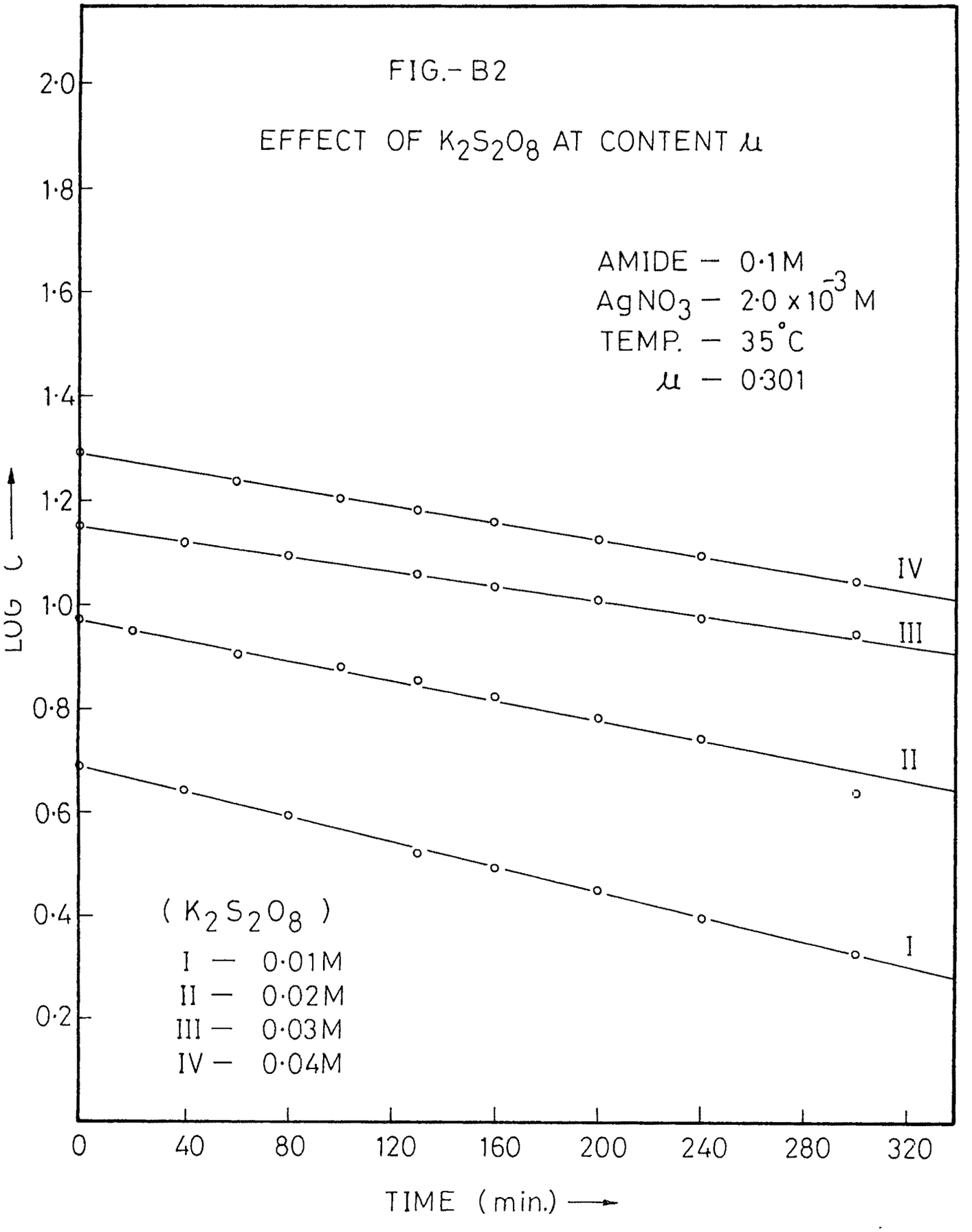


FIG.- B2
EFFECT OF $K_2S_2O_8$ AT CONTENT $\mu$
AMIDE — 0·1M
$AgNO_3$ — $2·0 \times 10^{-3}$ M
TEMP. — 35°C
$\mu$ — 0·301
2·0
1·8
1·6
1·4
1·2
1·0
0·8
0·6
0·4
0·2
LOG C
IV
III
II
I
( $K_2S_2O_8$ )
I — 0·01M
II — 0·02M
III — 0·03M
IV — 0·04M
0
40
80
120
160
200
240
280
320
TIME (min.)

FIG.– B3

PLOT OF LOG k VERSUS CONC. OF $K_2S_2O_8$

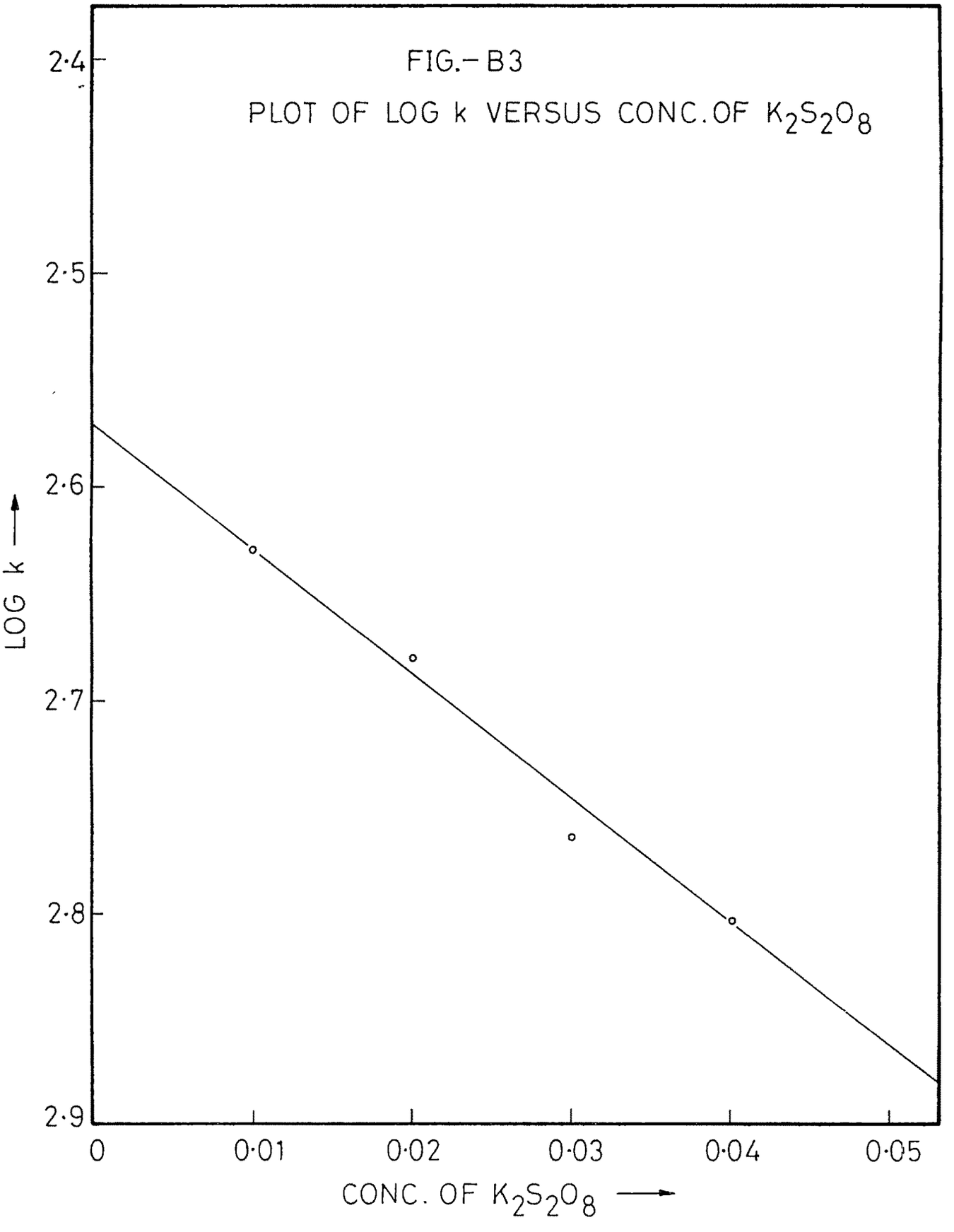
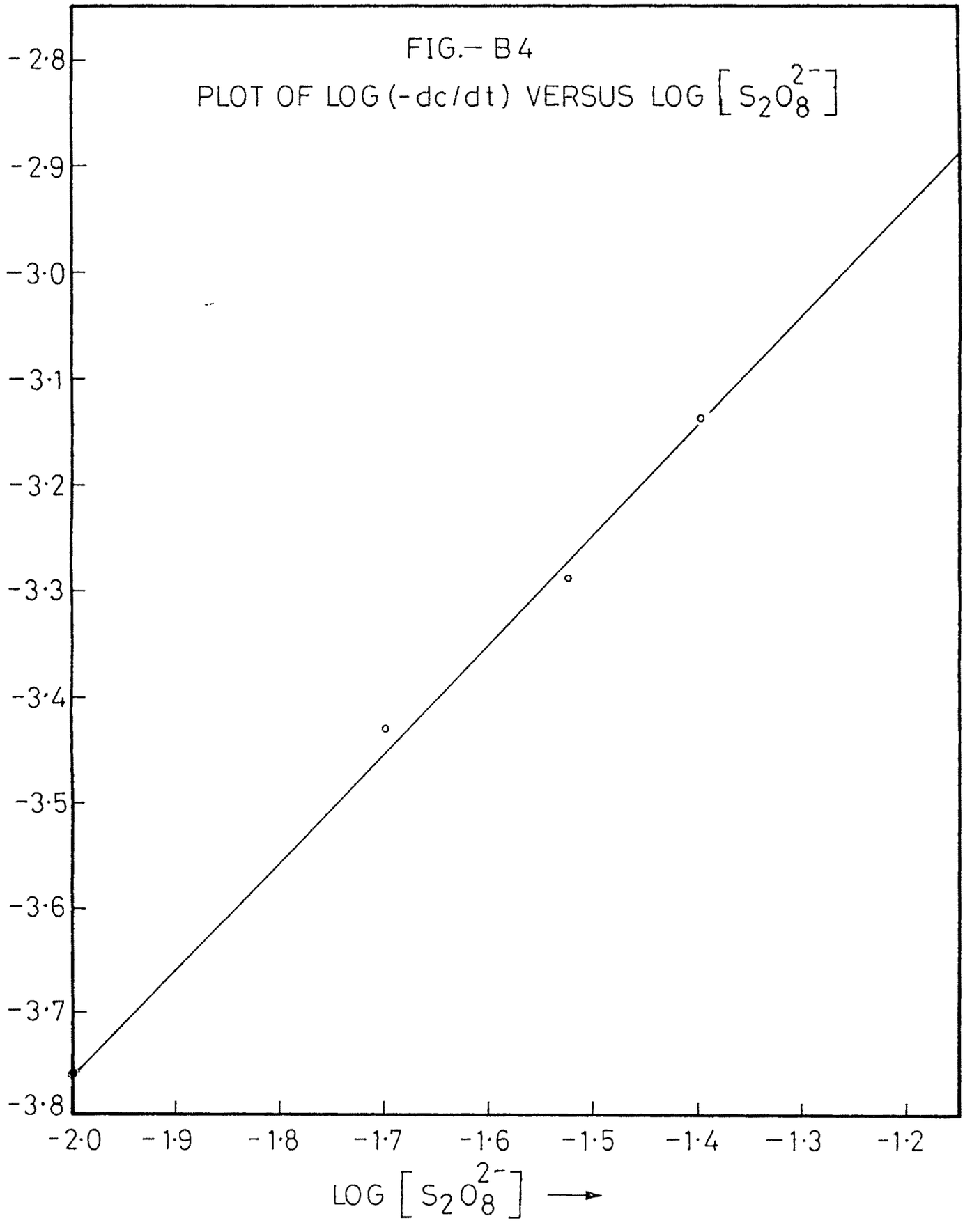


FIG.– B 4

PLOT OF LOG (-dc/dt) VERSUS LOG $[S_2O_8^{2-}]$



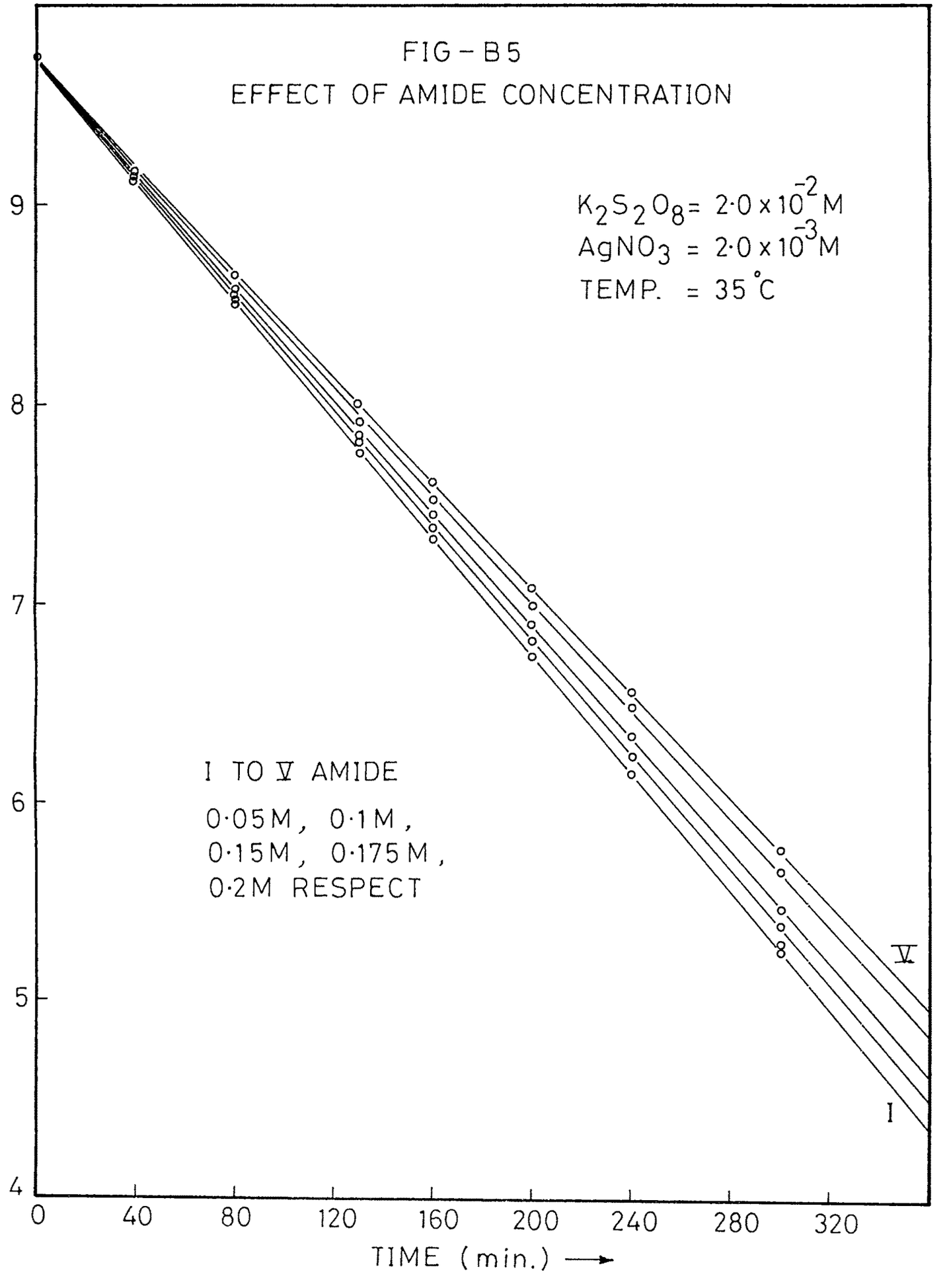


FIG – B5
EFFECT OF AMIDE CONCENTRATION
$K_2S_2O_8 = 2{\cdot}0 \times 10^{-2}$ M
$AgNO_3 = 2{\cdot}0 \times 10^{-3}$ M
TEMP. = 35 °C
I TO V AMIDE
0·05M, 0·1M,
0·15M, 0·175M,
0·2M RESPECT
V
I
9
8
7
6
5
4
0
40
80
120
160
200
240
280
320
TIME (min.) ⟶

FIG – B6

PLOT OF CONCENTRATION OF AMIDE VERSUS AMIDE/k

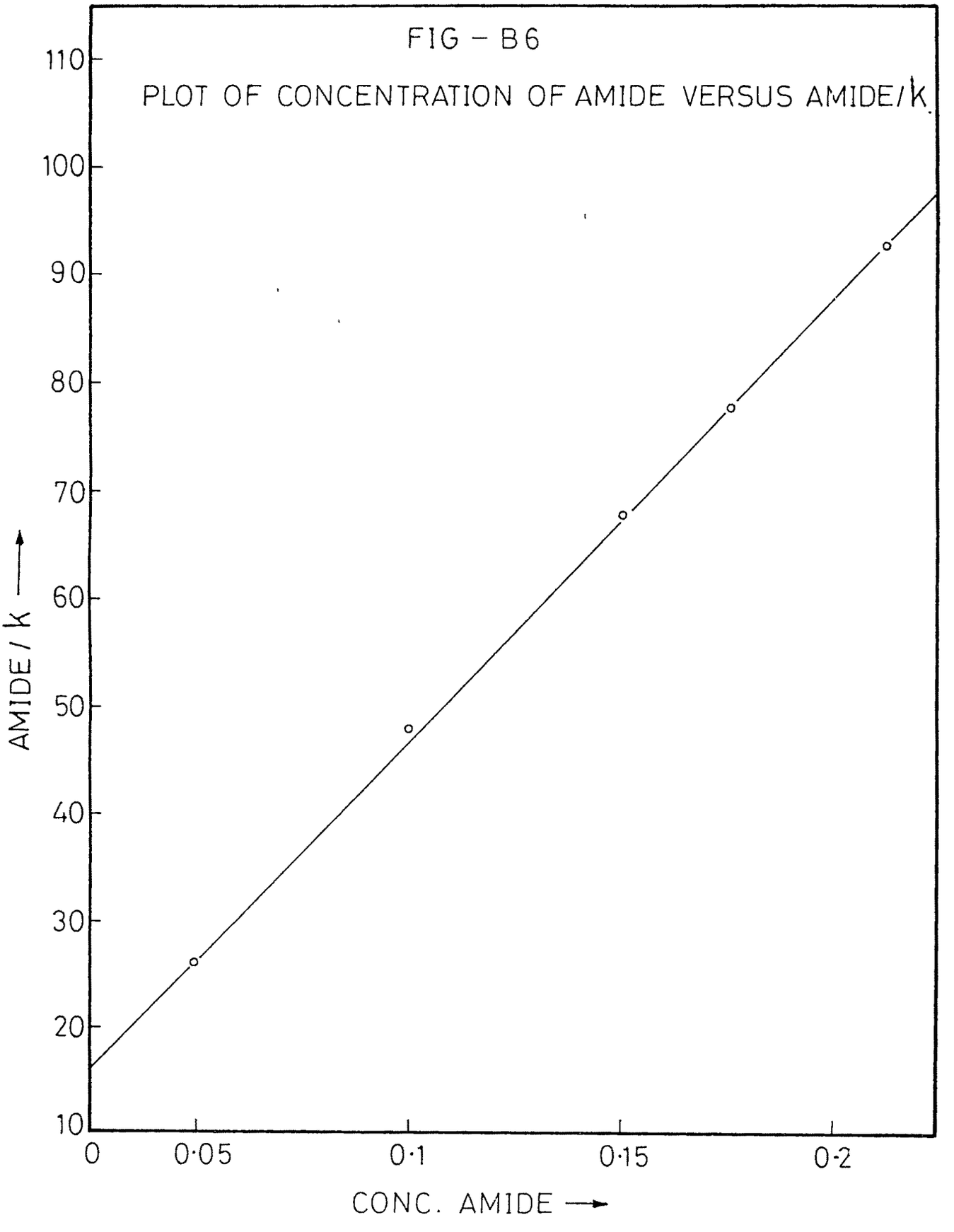
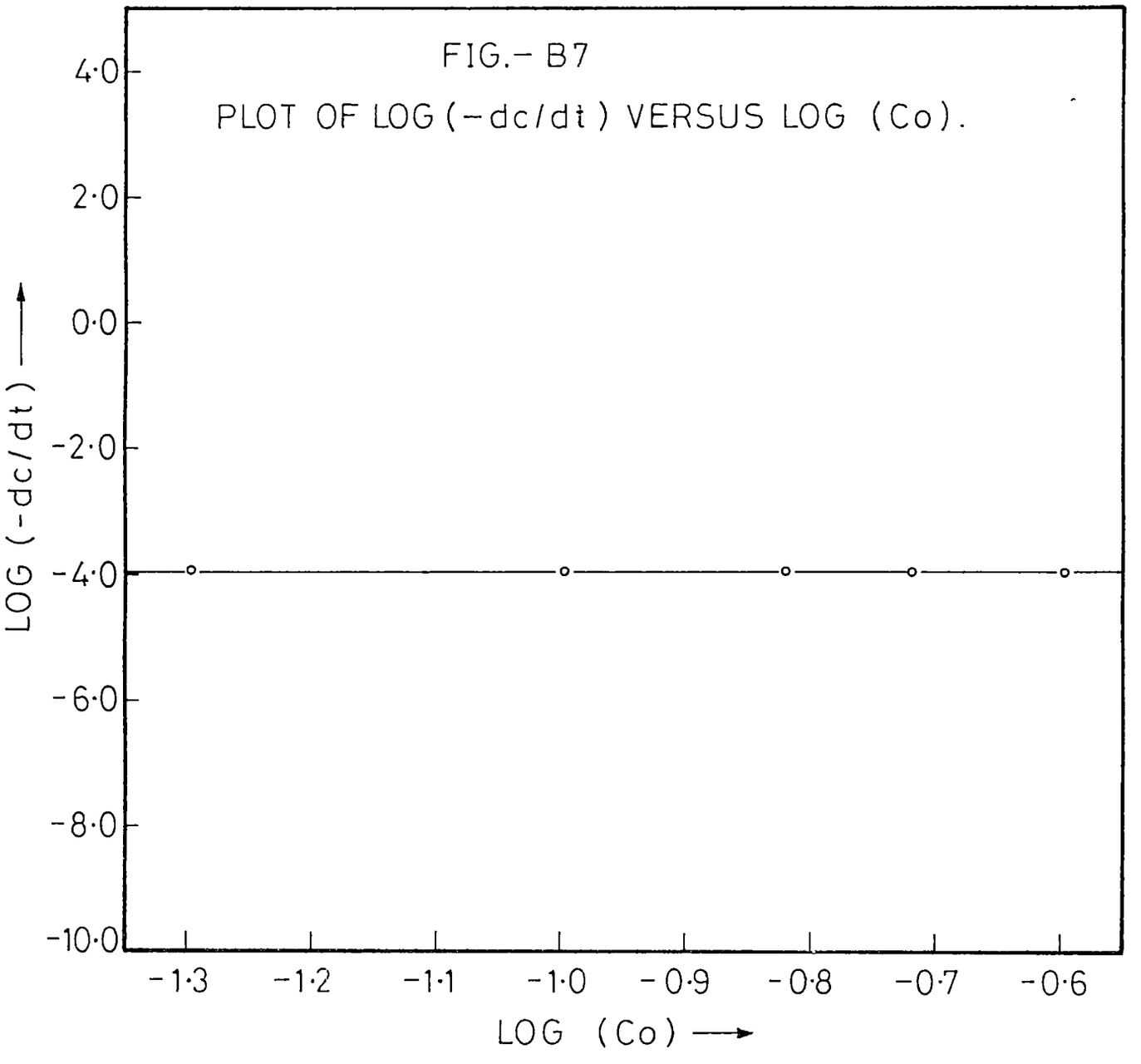


FIG.- B7

PLOT OF LOG (-dc/dt) VERSUS LOG (Co).



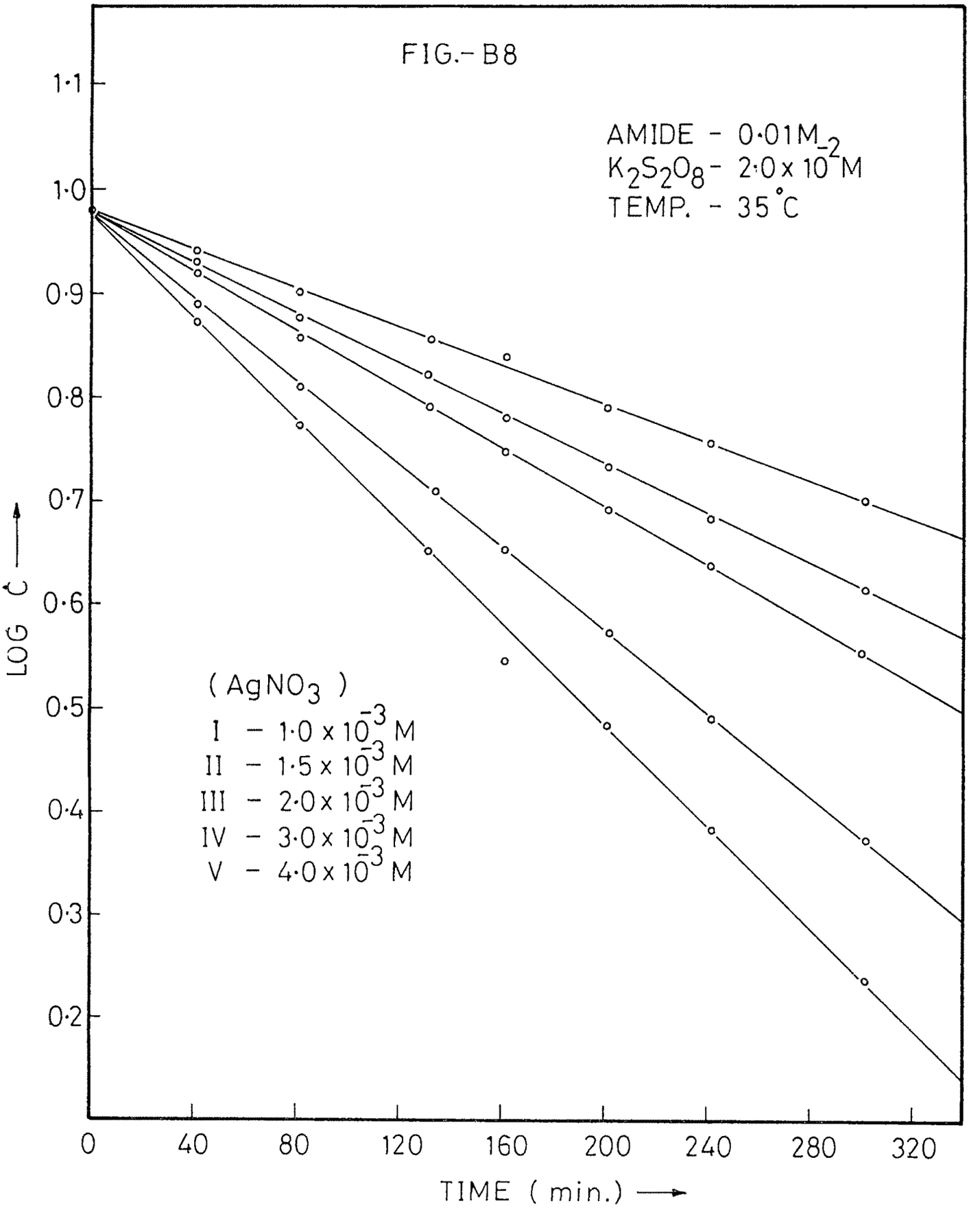
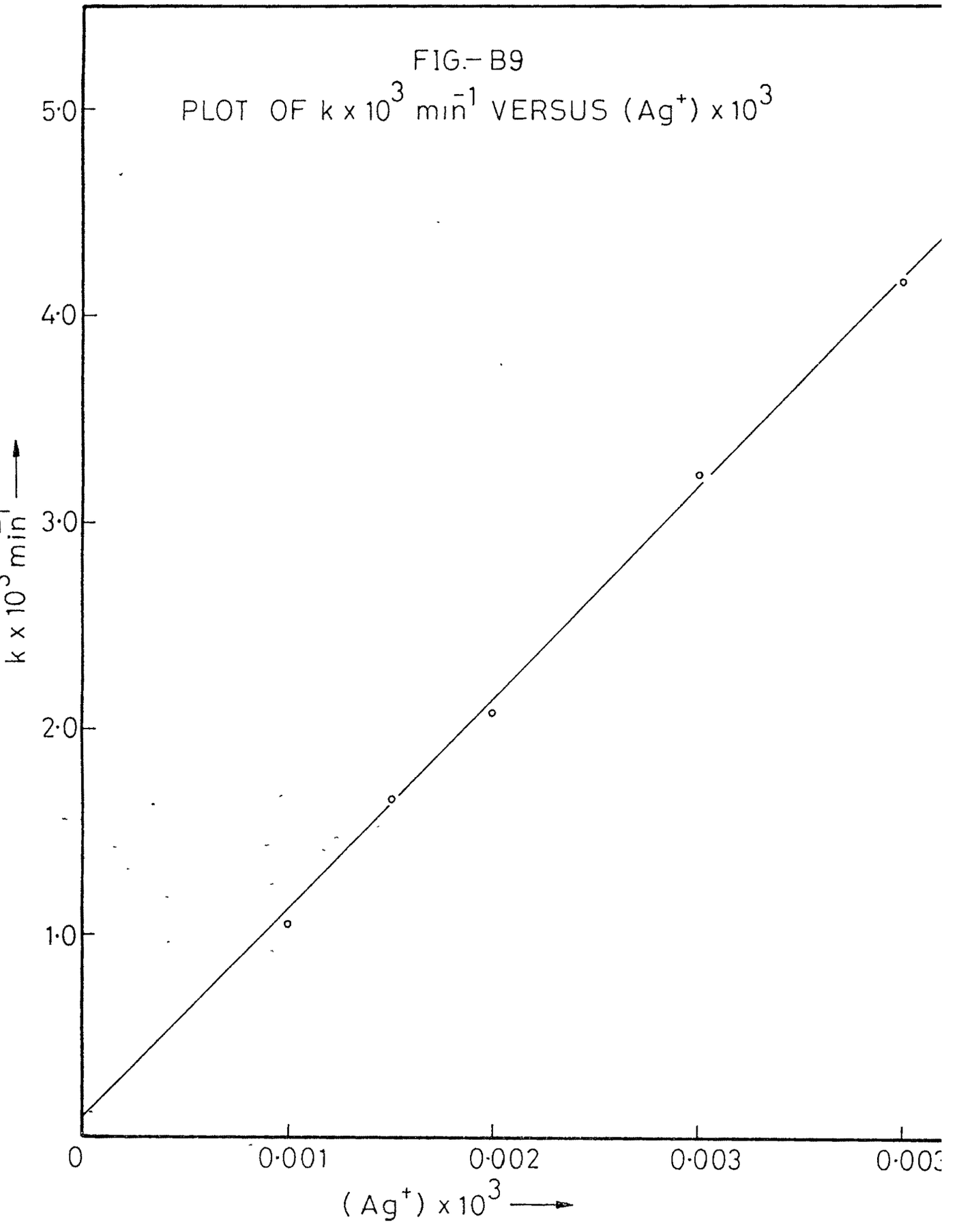


FIG.- B8
AMIDE - 0·01M
$K_2S_2O_8$ - $2{\cdot}0 \times 10^{-2}$ M
TEMP. - 35 °C
( $AgNO_3$ )
I - $1{\cdot}0 \times 10^{-3}$ M
II - $1{\cdot}5 \times 10^{-3}$ M
III - $2{\cdot}0 \times 10^{-3}$ M
IV - $3{\cdot}0 \times 10^{-3}$ M
V - $4{\cdot}0 \times 10^{-3}$ M
LOG C
1·1
1·0
0·9
0·8
0·7
0·6
0·5
0·4
0·3
0·2
0
40
80
120
160
200
240
280
320
TIME ( min.)

FIG.– B9

PLOT OF $k \times 10^3$ $min^{-1}$ VERSUS $(Ag^+) \times 10^3$



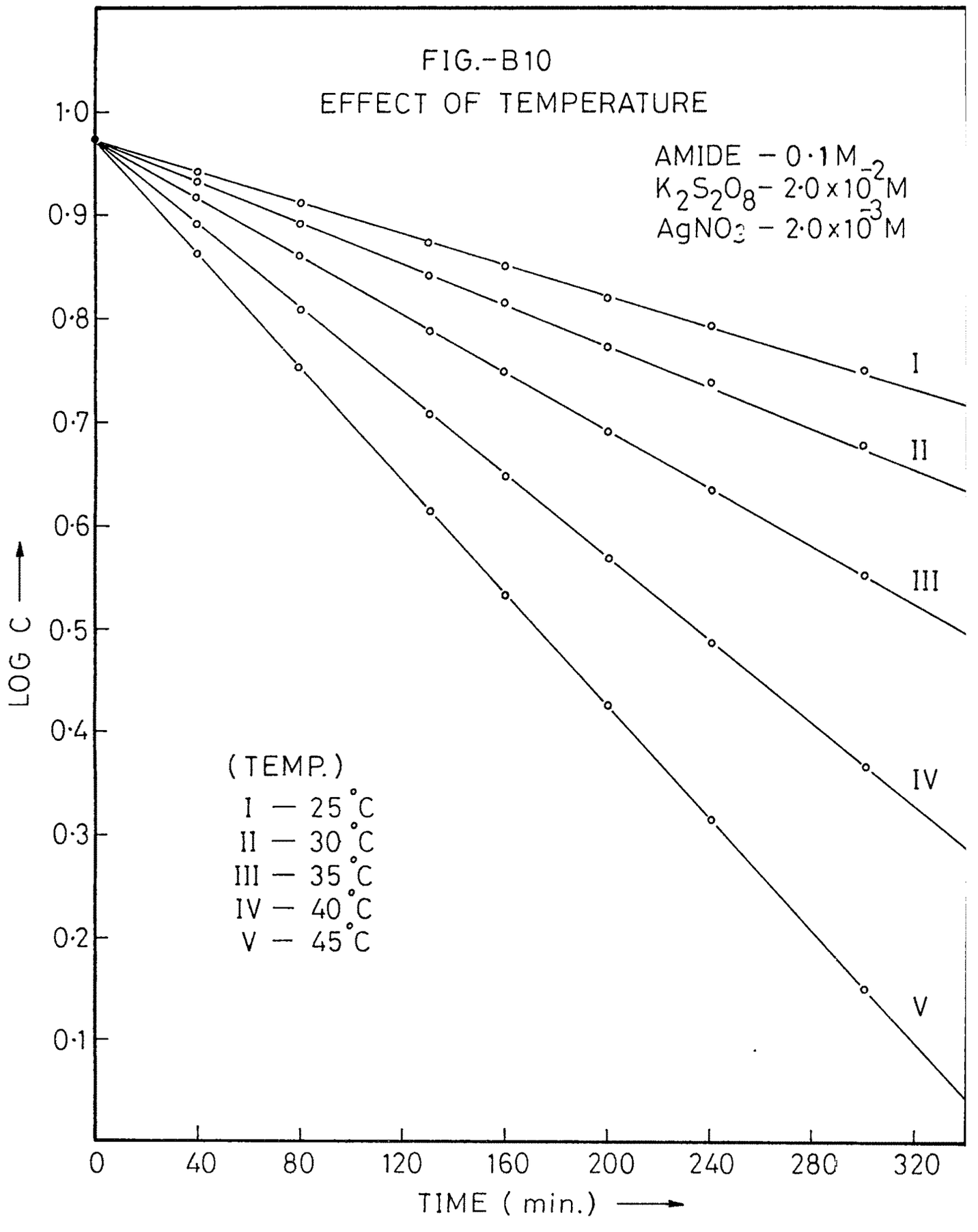


FIG.-B10
EFFECT OF TEMPERATURE
AMIDE – 0·1M
$K_2S_2O_8$ – $2{\cdot}0\times10^{-2}$M
$AgNO_3$ – $2{\cdot}0\times10^{-3}$M
I
II
III
IV
V
(TEMP.)
I — 25°C
II — 30°C
III — 35°C
IV — 40°C
V — 45°C
LOG C
1·0
0·9
0·8
0·7
0·6
0·5
0·4
0·3
0·2
0·1
0
40
80
120
160
200
240
280
320
TIME ( min.)

FIG.- B 11

PLOT OF LOG k VERSUS $1/T \times 10^3$

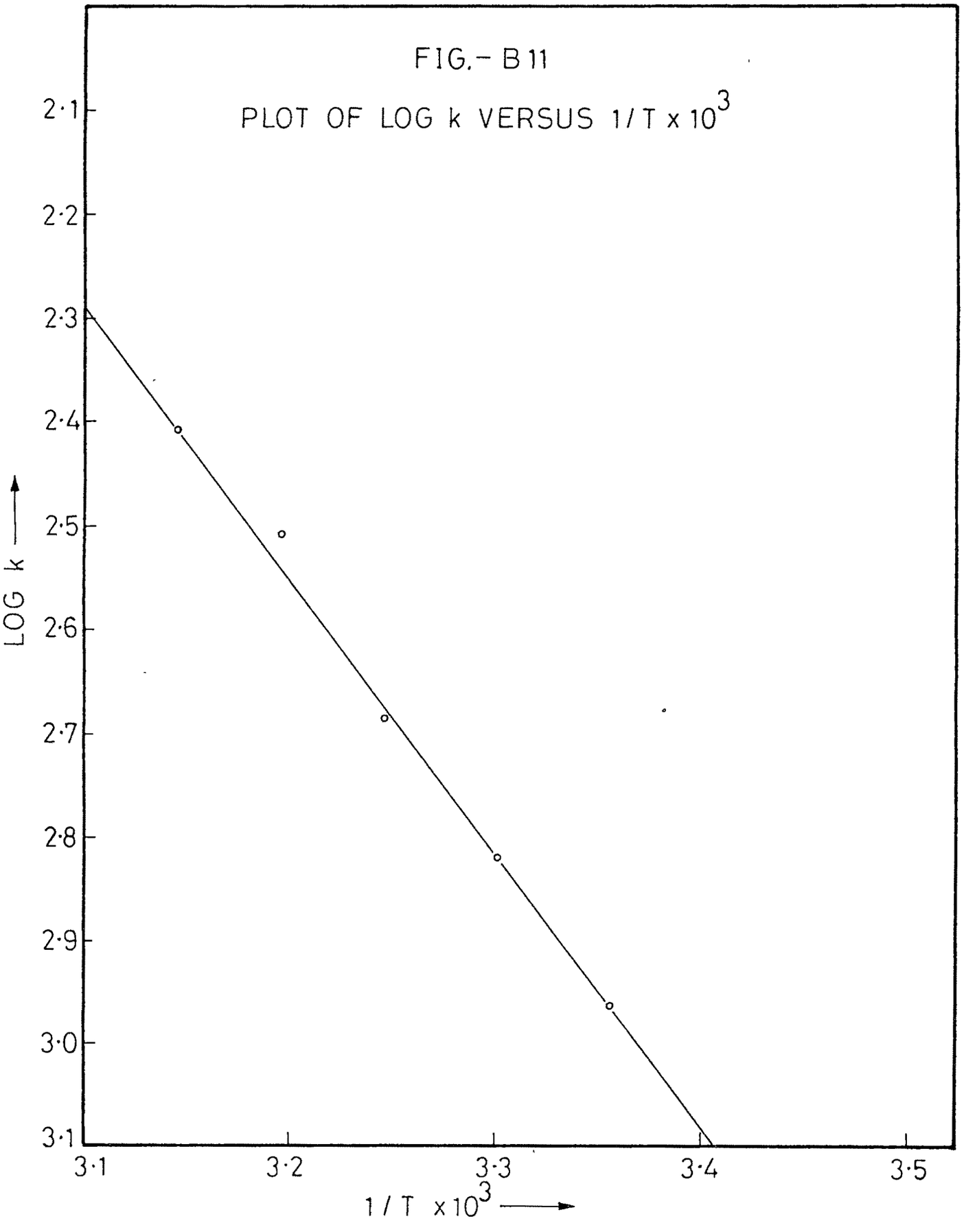
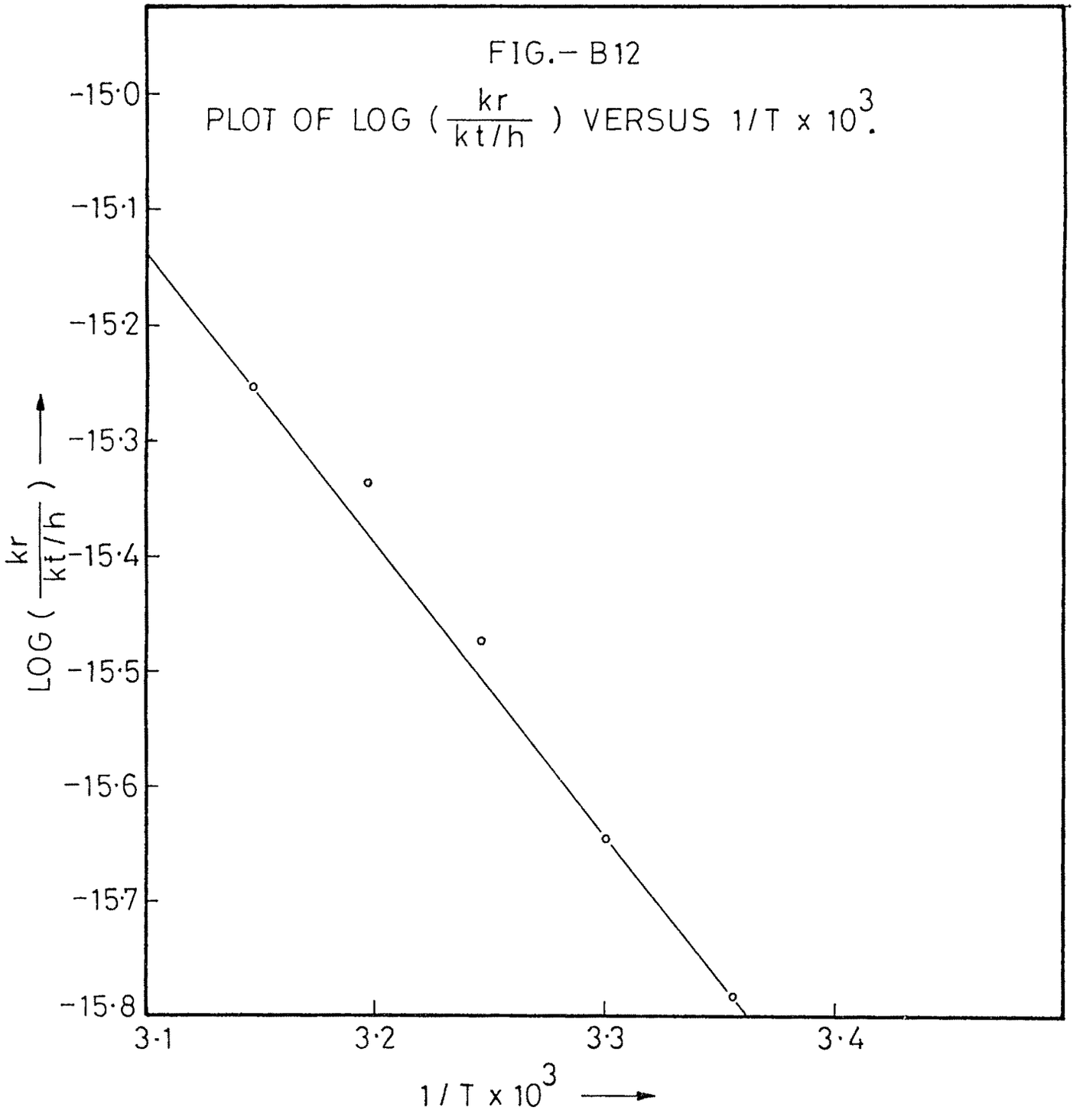


FIG.– B12

PLOT OF LOG ( $\frac{kr}{kt/h}$ ) VERSUS $1/T \times 10^3$.



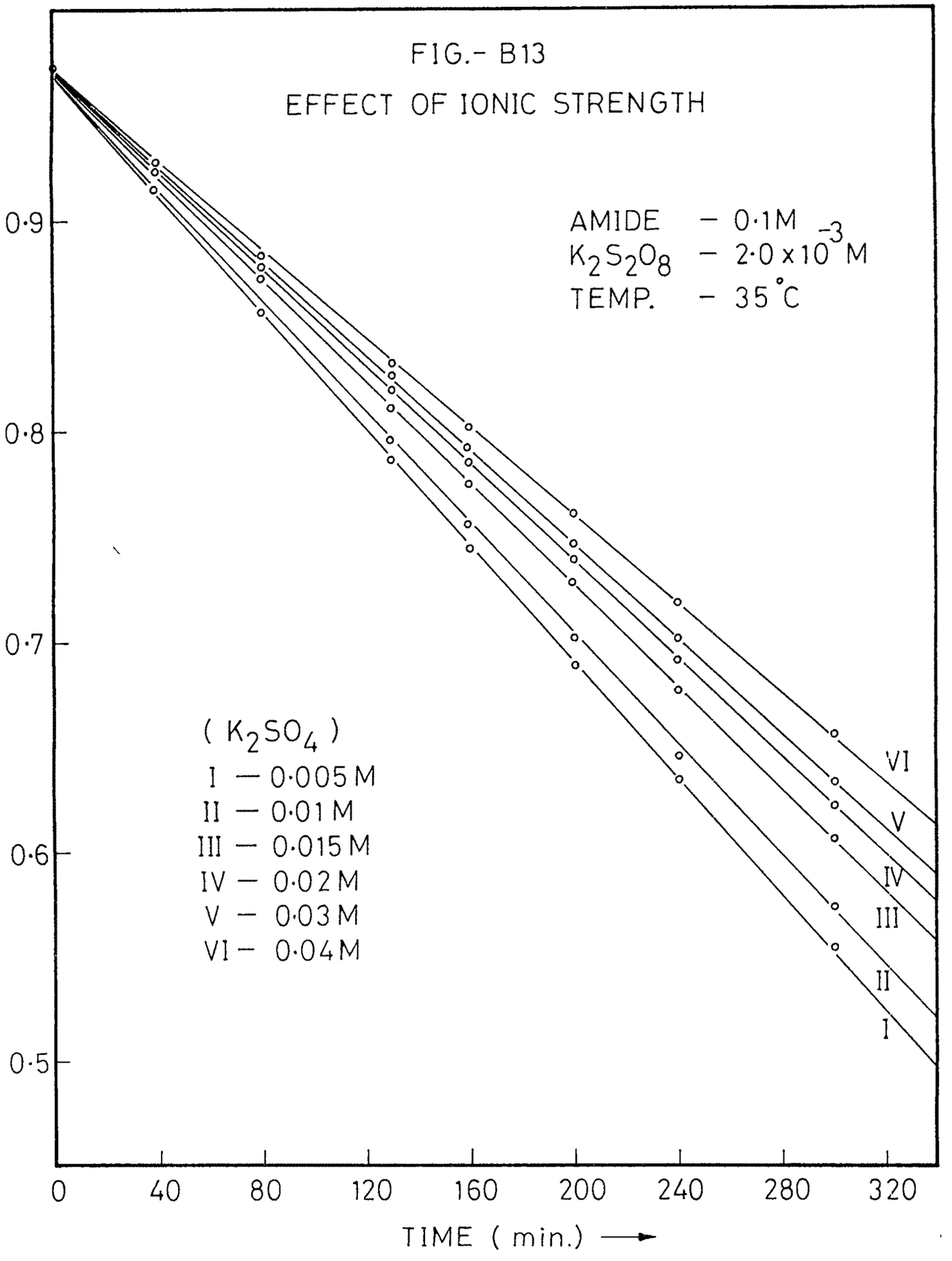
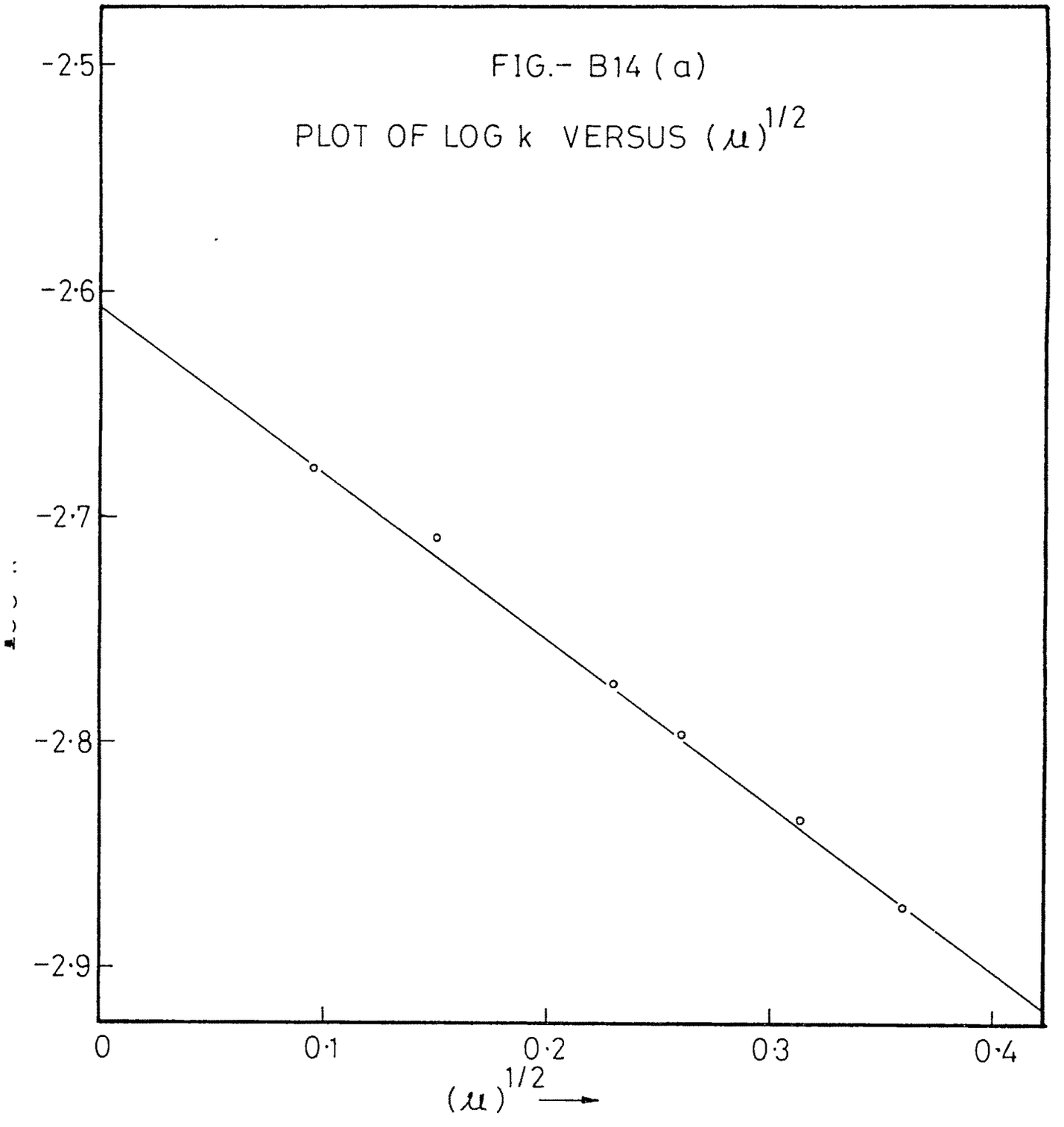


FIG.- B13
EFFECT OF IONIC STRENGTH
AMIDE - 0·1M
$K_2S_2O_8$ - $2·0 \times 10^{-3}$ M
TEMP. - 35°C
( $K_2SO_4$ )
I — 0·005 M
II — 0·01 M
III — 0·015 M
IV — 0·02 M
V — 0·03 M
VI — 0·04 M
VI
V
IV
III
II
I
0·9
0·8
0·7
0·6
0·5
0
40
80
120
160
200
240
280
320
TIME ( min.) ⟶

FIG.- B14 ( a)

PLOT OF LOG k VERSUS $(\mu)^{1/2}$



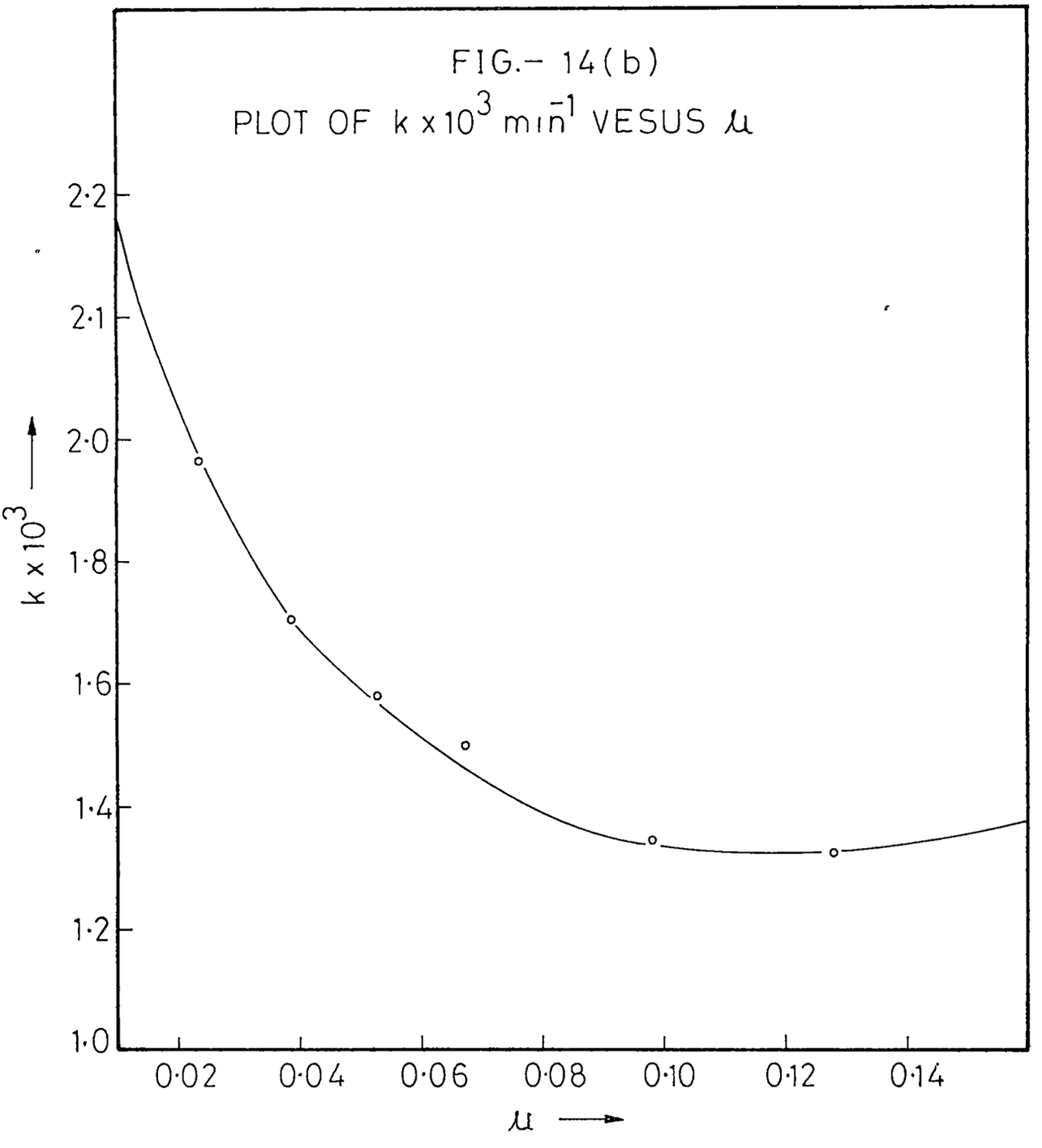


FIG.- 14(b)
PLOT OF k x 10^3 min^-1 VESUS μ
2·2
2·1
2·0
1·8
1·6
1·4
1·2
1·0
k x 10^3
0·02
0·04
0·06
0·08
0·10
0·12
0·14
μ

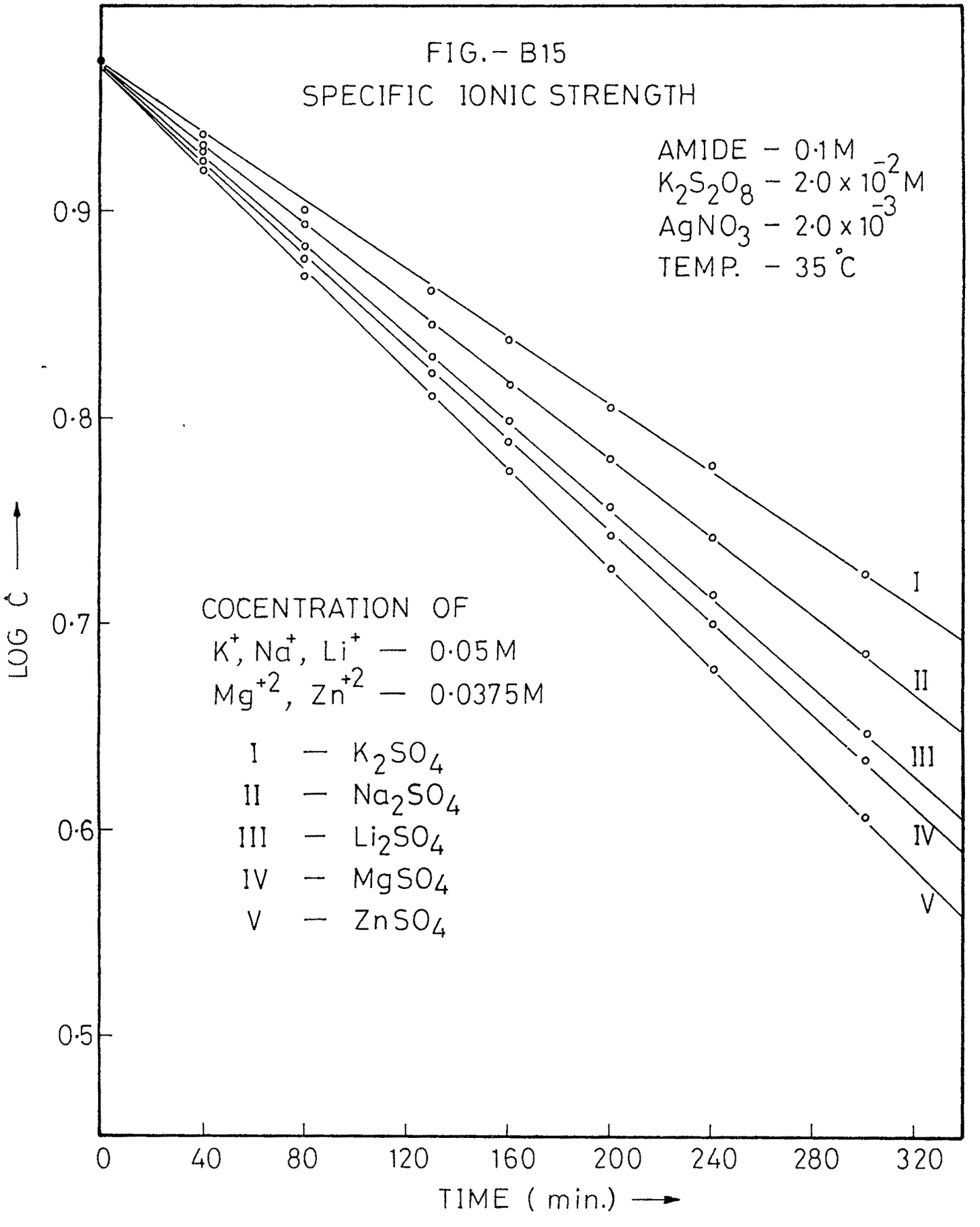


FIG.- B15
SPECIFIC IONIC STRENGTH
AMIDE - 0·1M
$K_2S_2O_8$ - $2·0 \times 10^{-2}$ M
$AgNO_3$ - $2·0 \times 10^{-3}$
TEMP. - 35 °C
COCENTRATION OF
$K^+$, $Na^+$, $Li^+$ — 0·05M
$Mg^{+2}$, $Zn^{+2}$ — 0·0375M
I — $K_2SO_4$
II — $Na_2SO_4$
III — $Li_2SO_4$
IV — $MgSO_4$
V — $ZnSO_4$
LOG C
0·9
0·8
0·7
0·6
0·5
0
40
80
120
160
200
240
280
320
TIME ( min.)

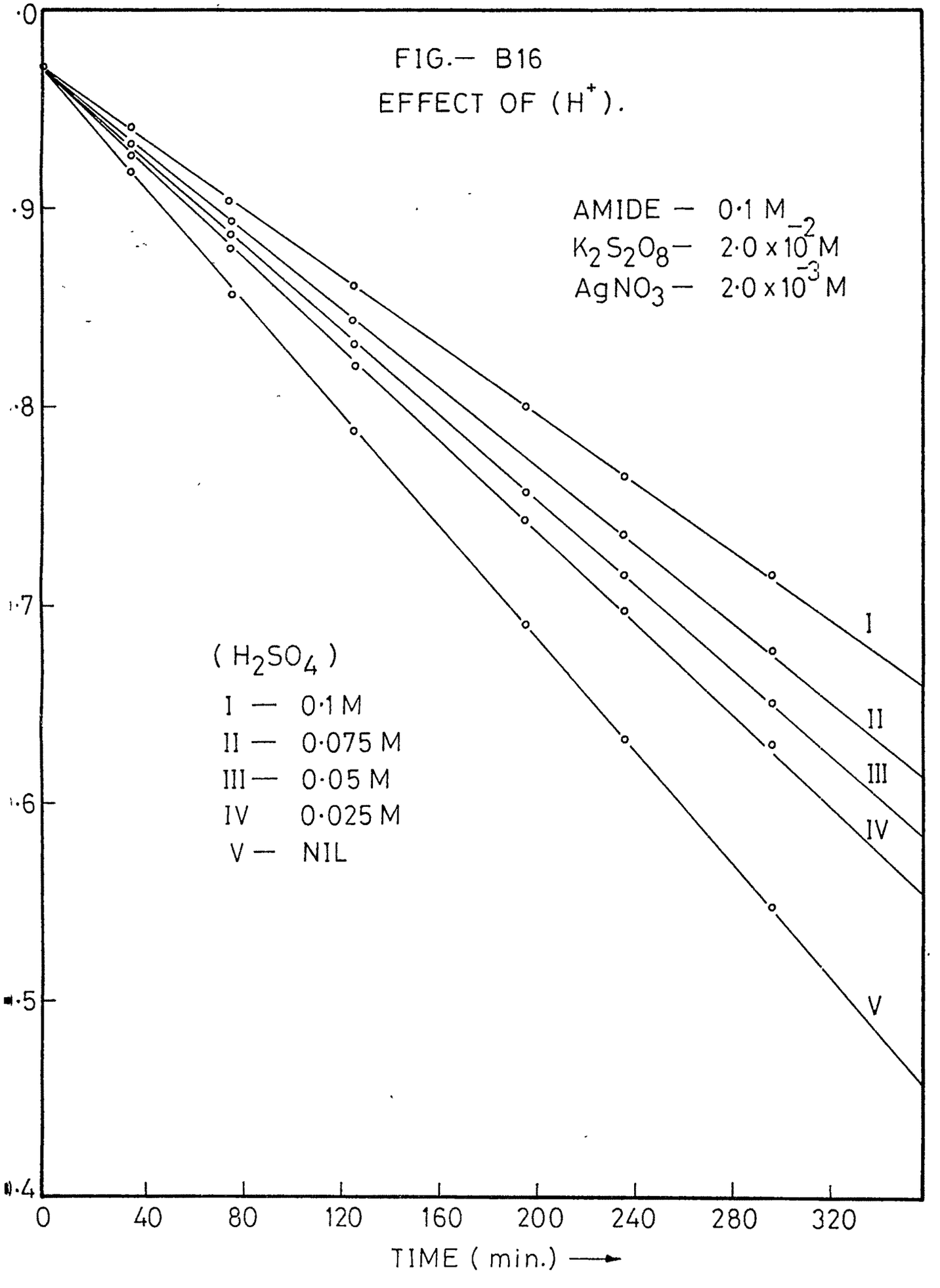


FIG.— B16
EFFECT OF ($H^+$).
AMIDE — 0·1 M
$K_2S_2O_8$ — $2{\cdot}0 \times 10^{-2}$ M
$AgNO_3$ — $2{\cdot}0 \times 10^{-3}$ M
( $H_2SO_4$ )
I — 0·1 M
II — 0·075 M
III — 0·05 M
IV 0·025 M
V — NIL
I
II
III
IV
V
·0
·9
·8
·7
·6
·5
·4
0
40
80
120
160
200
240
280
320
TIME ( min.) ⟶

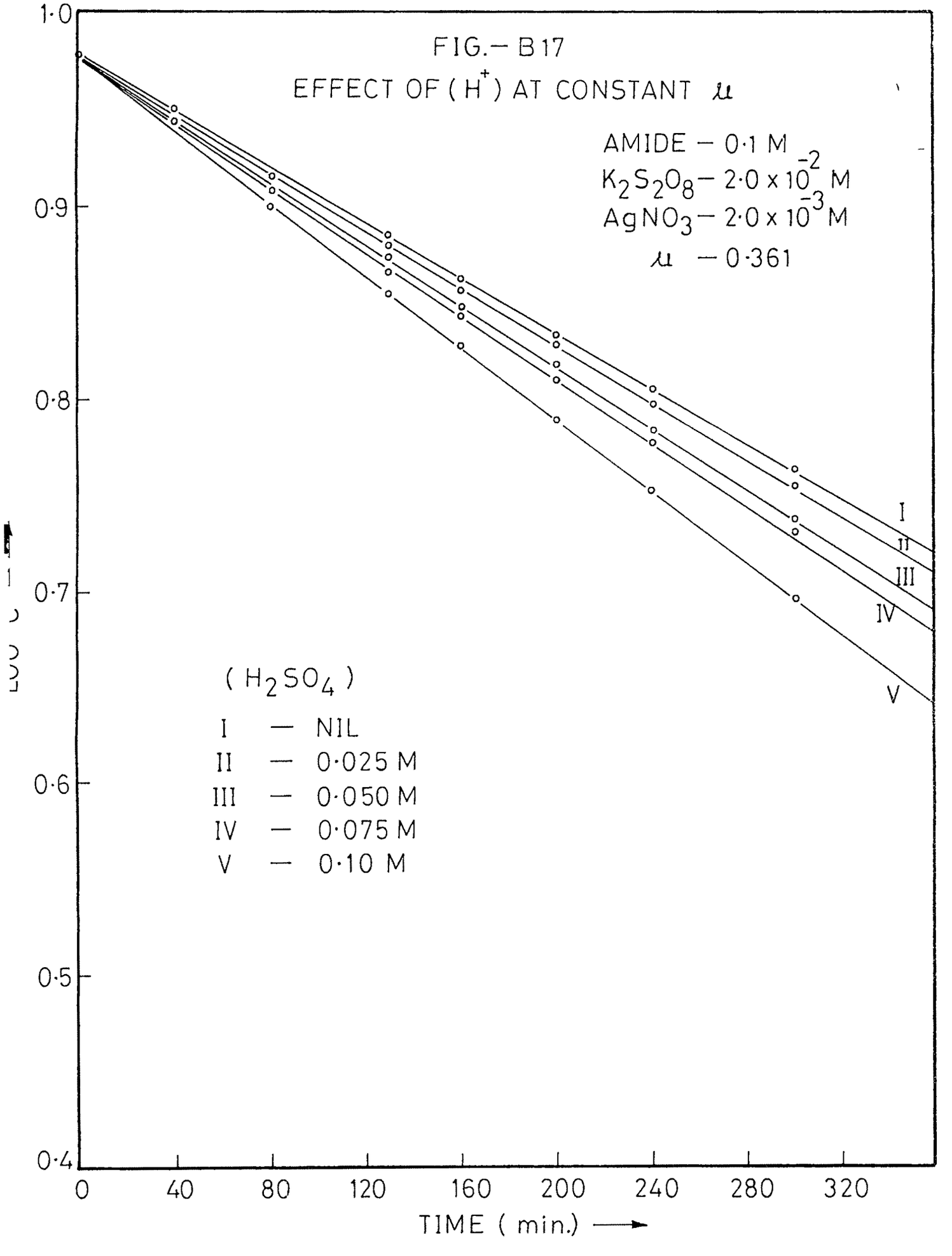


FIG.– B17
EFFECT OF ( $H^+$ ) AT CONSTANT $\mu$
AMIDE – 0·1 M
$K_2S_2O_8$ – $2·0 \times 10^{-2}$ M
$AgNO_3$ – $2·0 \times 10^{-3}$ M
$\mu$ – 0·361
( $H_2SO_4$ )
I — NIL
II — 0·025 M
III — 0·050 M
IV — 0·075 M
V — 0·10 M
I
II
III
IV
V
1·0
0·9
0·8
0·7
0·6
0·5
0·4
0
40
80
120
160
200
240
280
320
TIME ( min.) ⟶

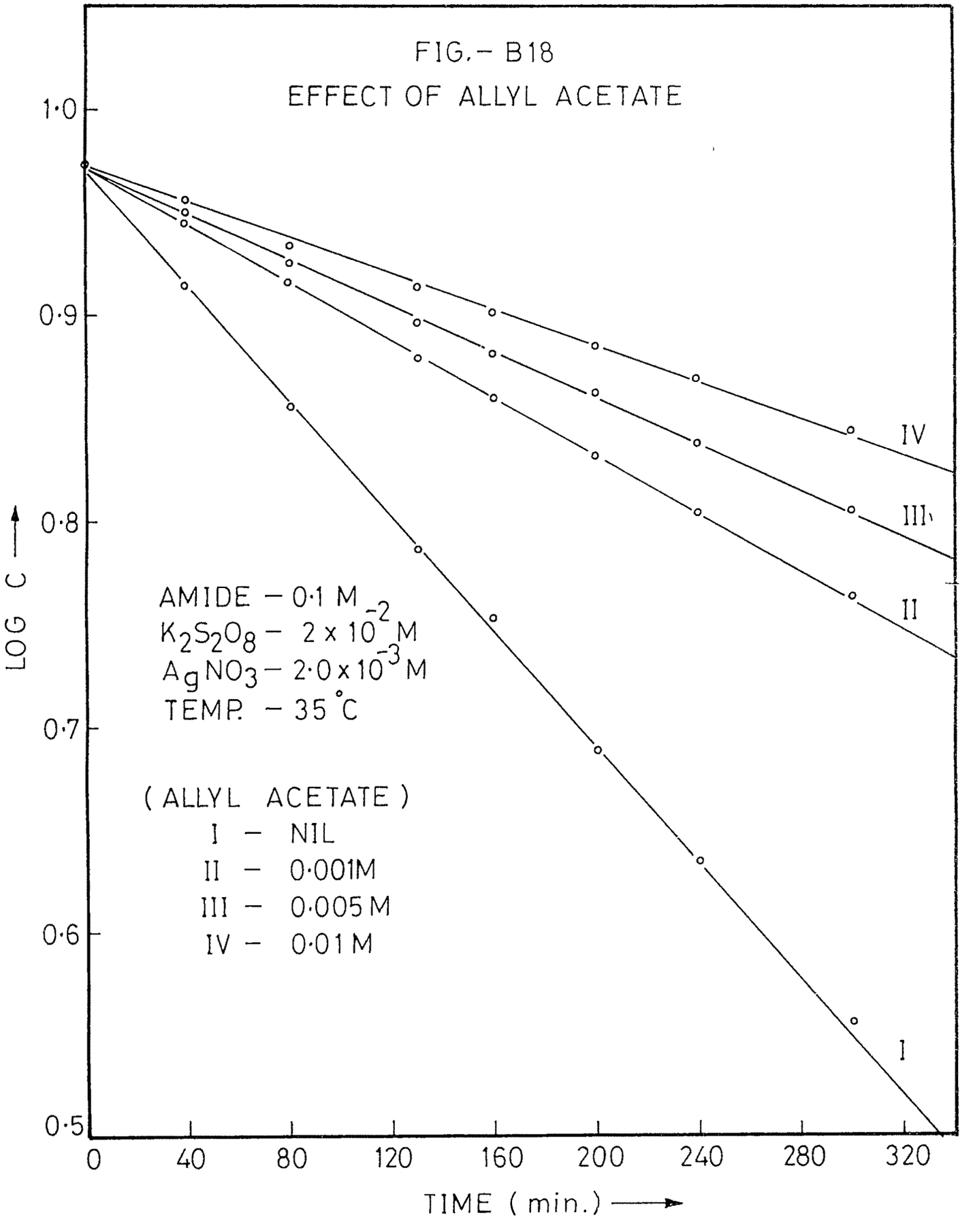


FIG.- B18
EFFECT OF ALLYL ACETATE
1·0
0·9
0·8
0·7
0·6
0·5
LOG C
AMIDE - 0·1 M
$K_2S_2O_8$ - $2 \times 10^{-2}$ M
$AgNO_3$ - $2{\cdot}0 \times 10^{-3}$ M
TEMP. - 35 °C
( ALLYL ACETATE )
I - NIL
II - 0·001M
III - 0·005 M
IV - 0·01 M
IV
III
II
I
0
40
80
120
160
200
240
280
320
TIME ( min.)

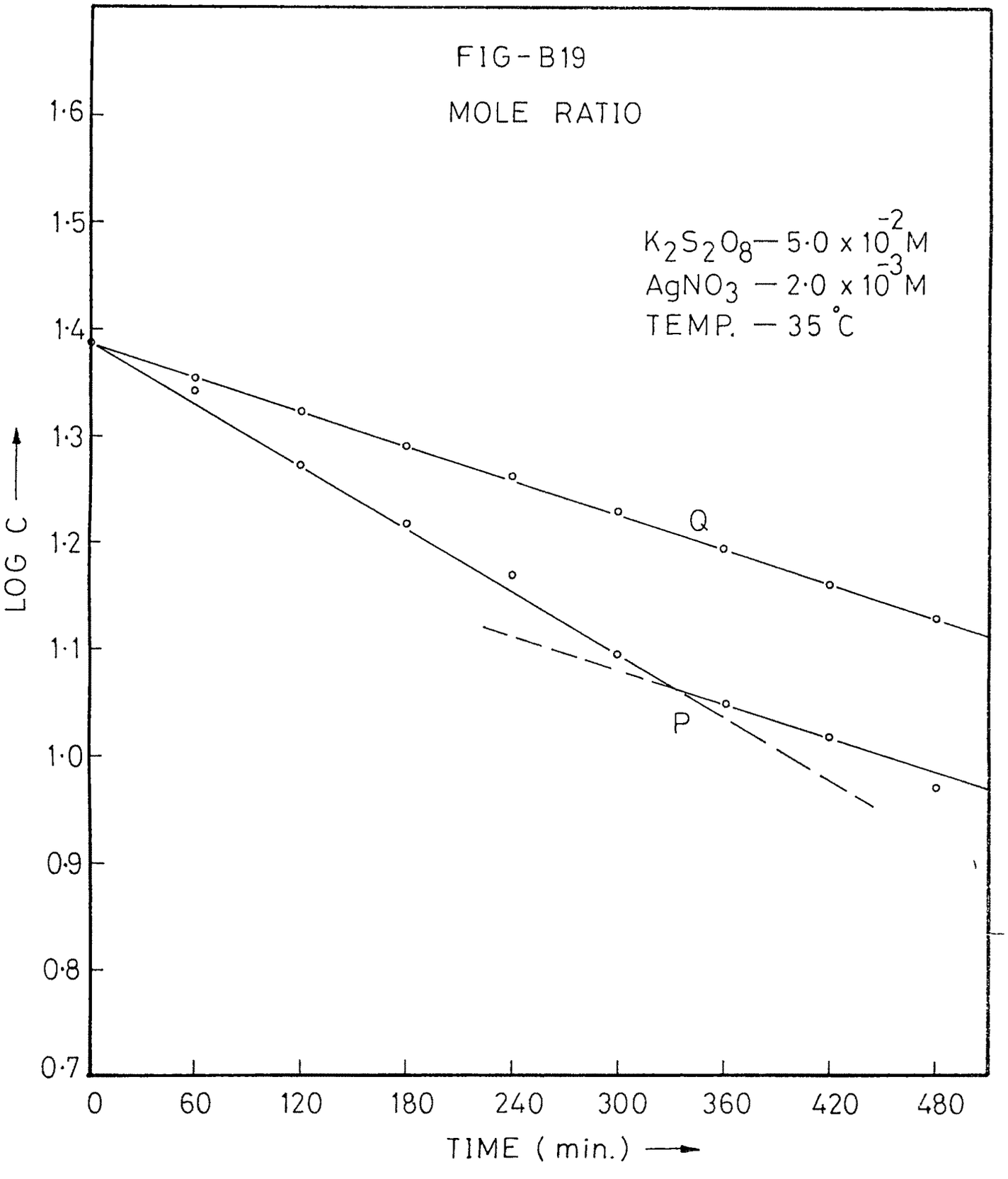


FIG-B19
MOLE RATIO
$K_2S_2O_8$ — $5.0 \times 10^{-2}$ M
$AgNO_3$ — $2.0 \times 10^{-3}$ M
TEMP. — 35 °C
Q
P
LOG C
TIME ( min.)
1·6
1·5
1·4
1·3
1·2
1·1
1·0
0·9
0·8
0·7
0
60
120
180
240
300
360
420
480