CHAPTER 6

GENERAL CONCLUSIONS AND SCOPE FOR FUTURE WORK IN GEL GROWTH OF SINGLE CRYSTALS

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As mentioned in the preface, the present work deals with the growth of potassium perchlorate and sodium chloride single crystals in silica gels. The detailed systematic investigations carried out in these directions have been described and discussed in the preceding chapters [Chapters 3 to 5]. A brief review of the present work and some general conclusions along with the scope for future work in gel growth are presented in this chapter.

6.1 GENERAL CONCLUSIONS

From the present work it reveals that transparent single crystals of potassium chlorate and sodium chloride can be grown by diffusing KNO_3 and $HClO_4$ through silica gel.

The present study of the growth of potassium perchlorate crystals reveals that the morphology of the crystals grown in silica as well as in gelatin gels are highly sensitive to the concentration of reactants and the crystallization temperature. It is also very clear from the observation of cusps that the crystals grow by the displacement of gel.

Growth of $KClO_4$ crystals using 250 ml beakers, test tubes and U-tubes reveals that test tubes are more suitable to grow good quality single crystals in terms of intercrystalline separation and crystal size. In the test tubes it is observed that the crystals at greater depths from the gel-solution interfaces grow more slowly, less in number and are more transparent because of the smaller concentration gradients than those near the top. The lattice parameters and interplanner spacing values of these crystals are in well agreement with the values reported in the literature and confirm that the water of crystallization is 0.5.

From the study of the effect of gel parameters on nucleation and growth of $KClO_4$ crystals, it has been observed that while high pH [greater than 7] and high density [greater than 1.03 sp.gr.] gels have been found to produce opaque crystals whereas good quality single crystals have been obtained at low density and low pH. Increase in gel ageing and the height of the intermediate neutral gel reduced the nucleation centres considerably. Further, a study of the effect of concentration programming on the growth of $KClO_4$ crystals indicates that the size and quality of the crystals can be improved by using concentration programming.

The effect of acid impurities on the growth of sodium chloride crystals showed that there is a decrease in nucleation density at higher acid concentrations which is due to the fact that an increase in concentration increases the aqueous solubility of sodium chloride. But of all the acid impurities, perchloric acid impurity has been found to result in the best quality NaCl crystals in terms of transparency and well defined shapes.

6.2 SCOPE FOR FUTURE WORK

A general assessment of the method and its potentialities is not yet possible. But recent experiments have yielded results which should prove useful to other workers in this field. A great deal of work remain to be done to document and explain the detailed nature of the gel structure which will be displaced by the growing crystal as in case of potassium chlorate or incorporation of gel into new solid by crystal growth in the gel interstices as in the case of calcite . Many industrially important crystals such as ZnS, CdS, PbS, ZnTe, PbSe, CdSe, $CaCO_3$, $CaWO_4$, Caf_2, etc. can be grown in various types of gels with various acid set gel and by adding impurities in order to decrease the nucleation density and to grow various shapes of highly perfect single crystals. It is worth while to make an attempt of comparative studies on the growth of crystals like AgI, PbI_2, HgI_2 etc. by reaction method and complex dilution method. Growth of various metal crystals is to be tried by using suitable chemical reducing agents only a few water soluble crystals (KDP, ADP, TGS) have been grown in gels so crystal growth of other water soluble materials is to be tried. With the gel method, it is also possible to produce naturally occurring habits of the crystals by controlling the experimental parameters in the laboratory and hence gel growth can be used as a tool in understanding geologic conditions.

Out of a large number of grown single crystals, properties of only a few have been studied. Comparative studies of gel grown single crystals with those grown by other methods are worth attempting in order to probe into and take the advantage of characteristics and peculiarities of gel grown crystals the further development of the gel method will certainly depend on the extent to which its mechanism can be understood and controlled.

CAPTIONS OF FIGURES

Figure 1.1 Solubility of KBr and NaCl as a function of temperature.

Figure 1.2 Solubility versus temperature for various saturation conditions.

Figure 1.3 Method of production of seed crystals.

Figure 1.4 Growing of large single crystals using seed crystal with variation of solubility by evaporation of by change of temperature.

Figure 1.5 Basic principles of pulling method.

Figure 1.6 Liquid encapsulation czochralski method for growing volatile compounds.

Figure 3.1 Schematic diagram of a beaker-single tube system.

Figure 3.2 Schematic diagram of modified special apparatus used for the growth of potassium perchloride crystals.

Figure 3.3 Crystal growing in test tubes with the solutions inside the and above the gel respectively.

a) KNO_3 and $HClO_4$

b) $HClO_4$ and KNO_3

Figure 3.4 Schematic diagram for crystal growth in an U-tube.

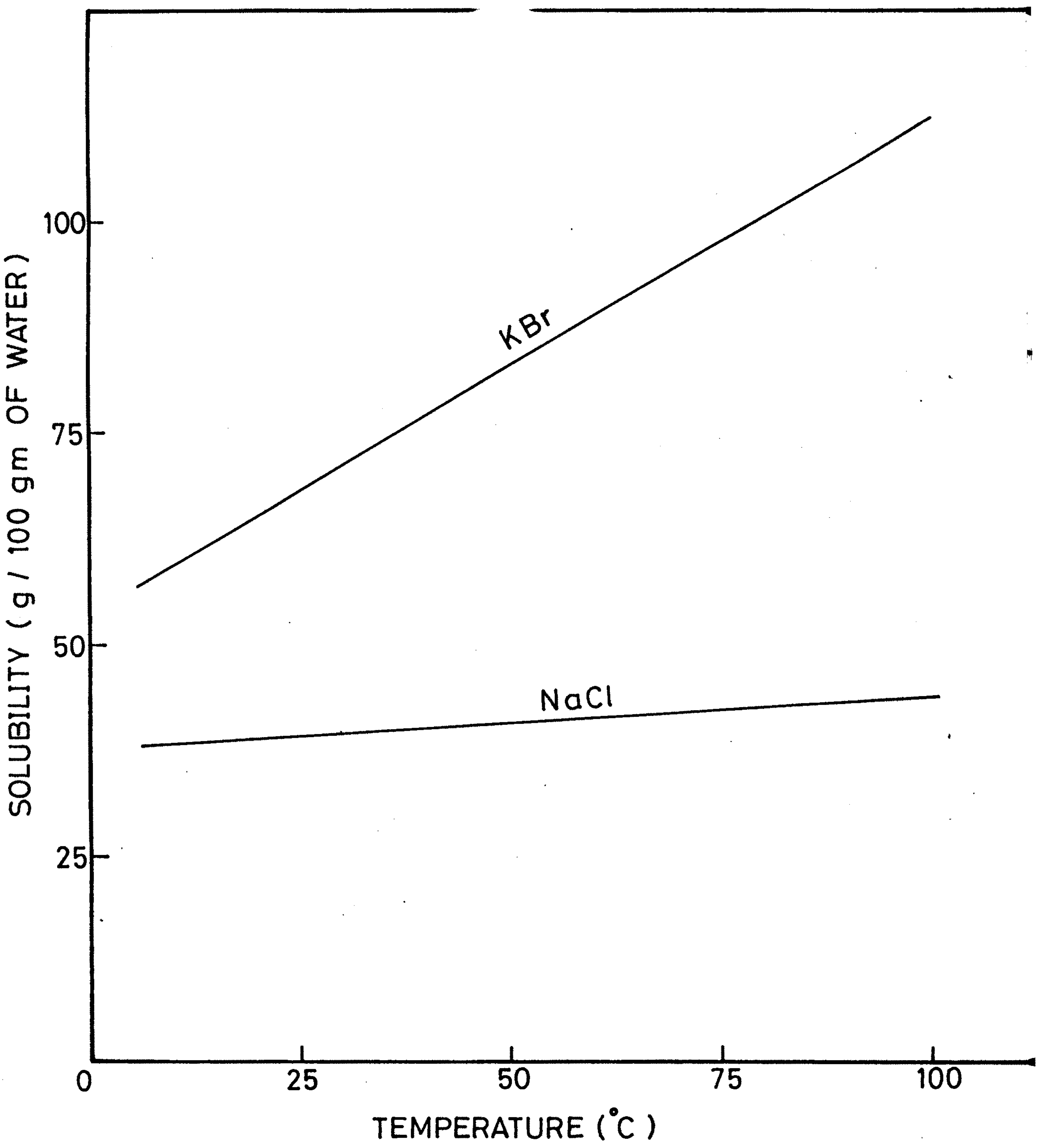
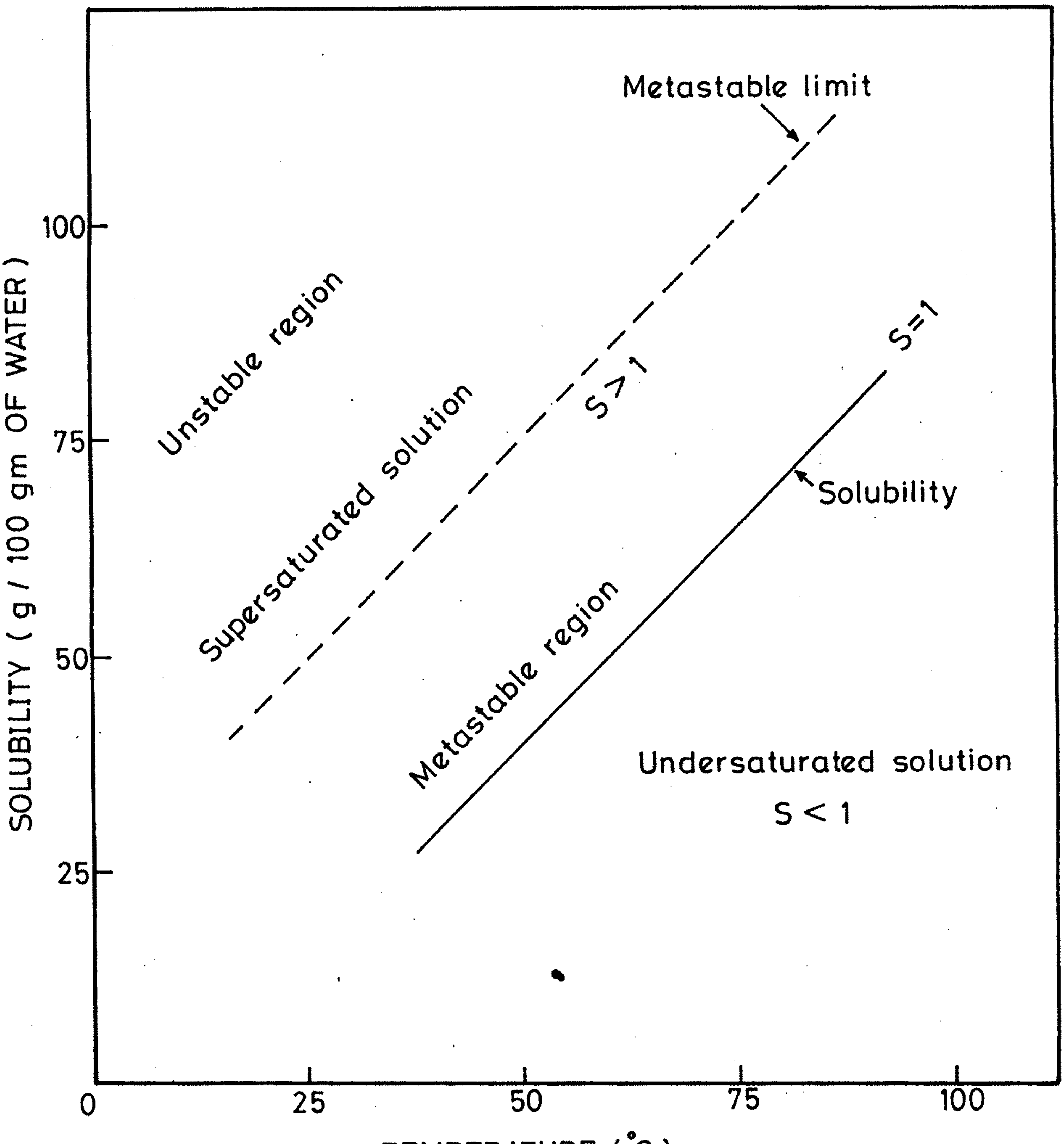
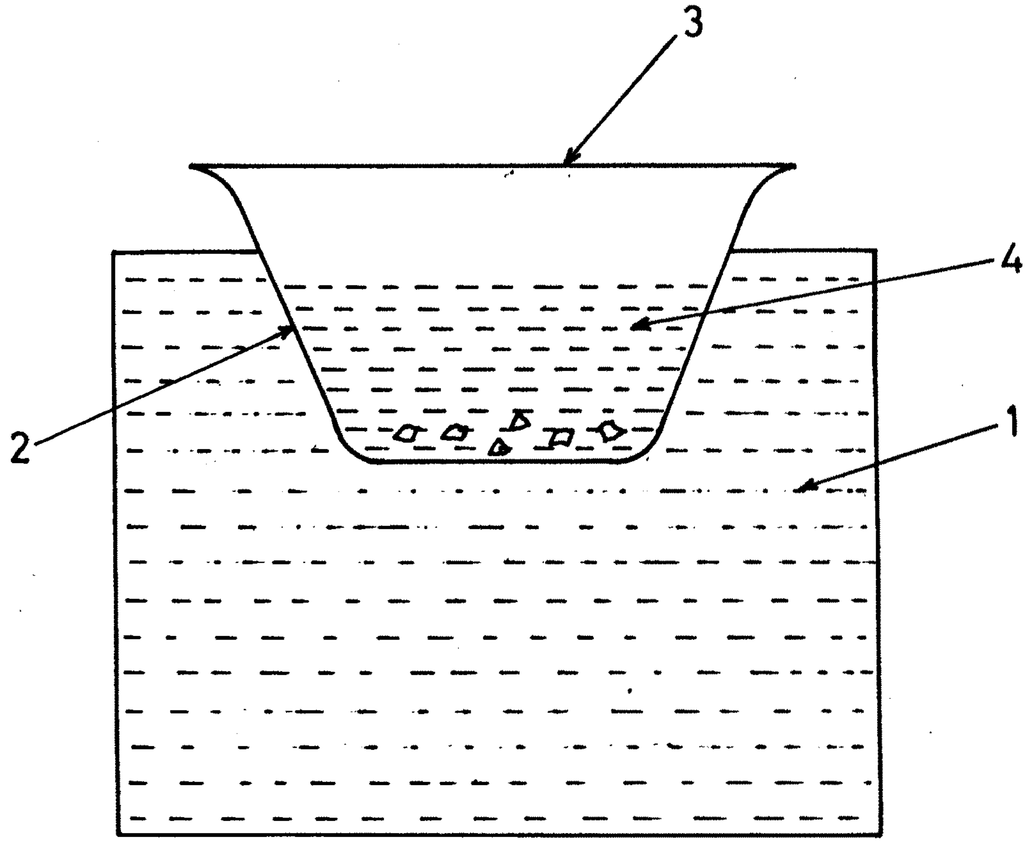

SOLUBILITY (g / 100 gm OF WATER)
100
75
50
25
KBr
NaCl
0
25
50
75
100
TEMPERATURE (°C)

Fig. 1·1



Metastable limit
100
75
50
25
0
25
50
75
100
SOLUBILITY (g / 100 gm OF WATER)
TEMPERATURE (°C)
Unstable region
Supersaturated solution
S > 1
S=1
Solubility
Metastable region
Undersaturated solution
S < 1

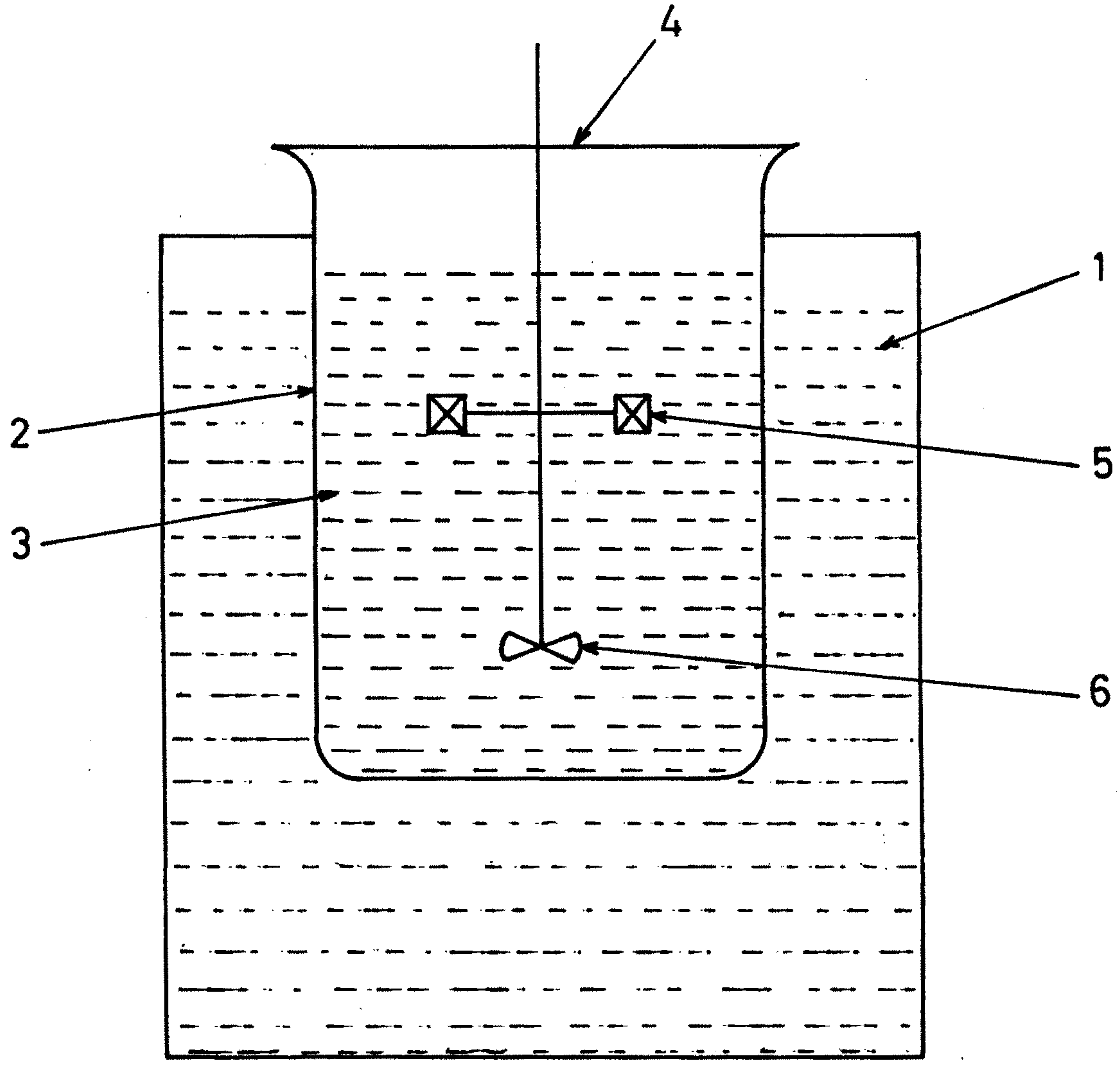
Fig. 1·2



PRODUCTION OF SEED CRYSTALS

1. Water bath of the thermostat.
2. Crystalization vessel.
3. Perfectly or only partially closed lid.
4. Supersaturated solution with the seed crystals.

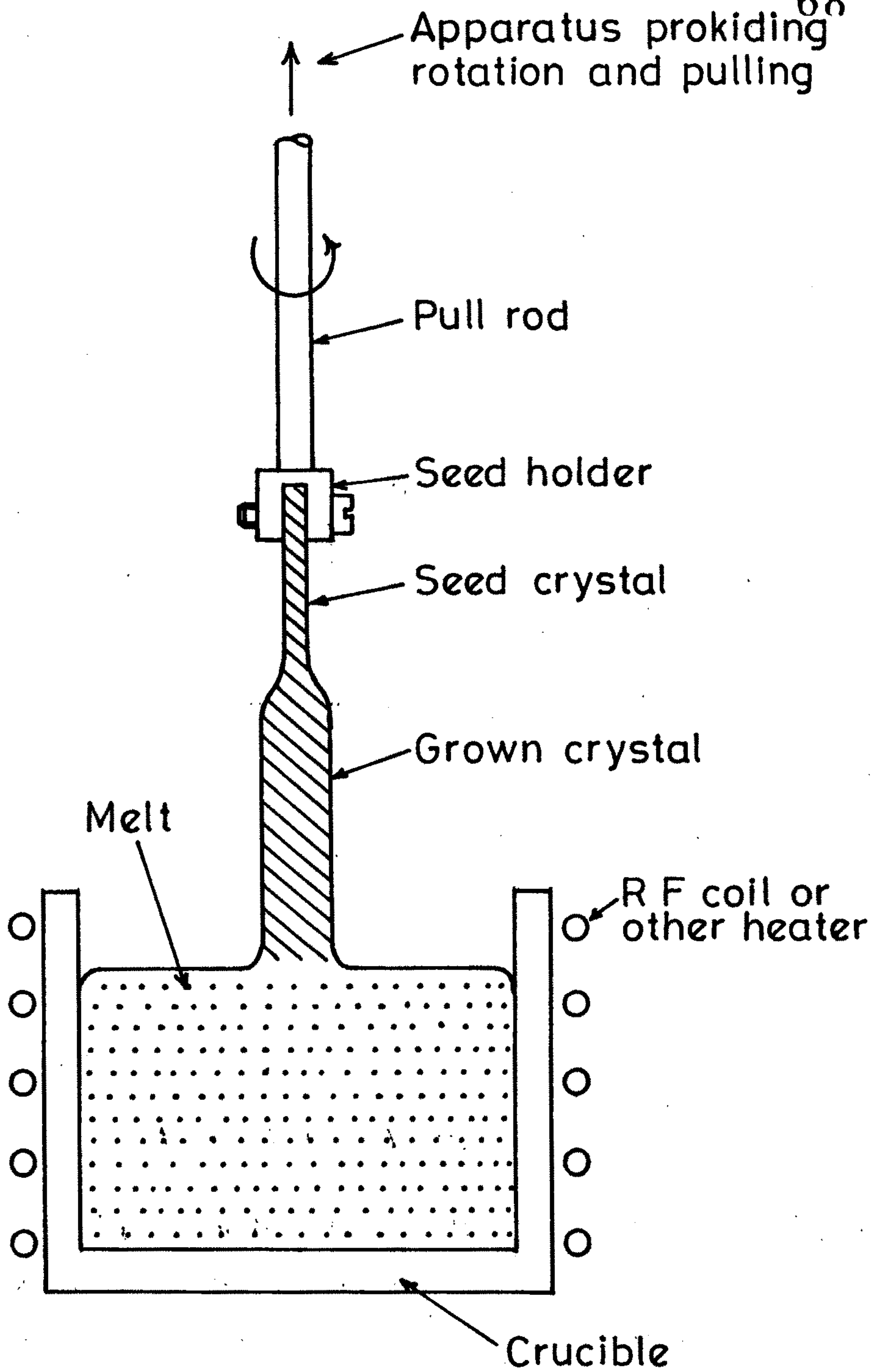
Fig. 1·3



GROWTH APPARATUS

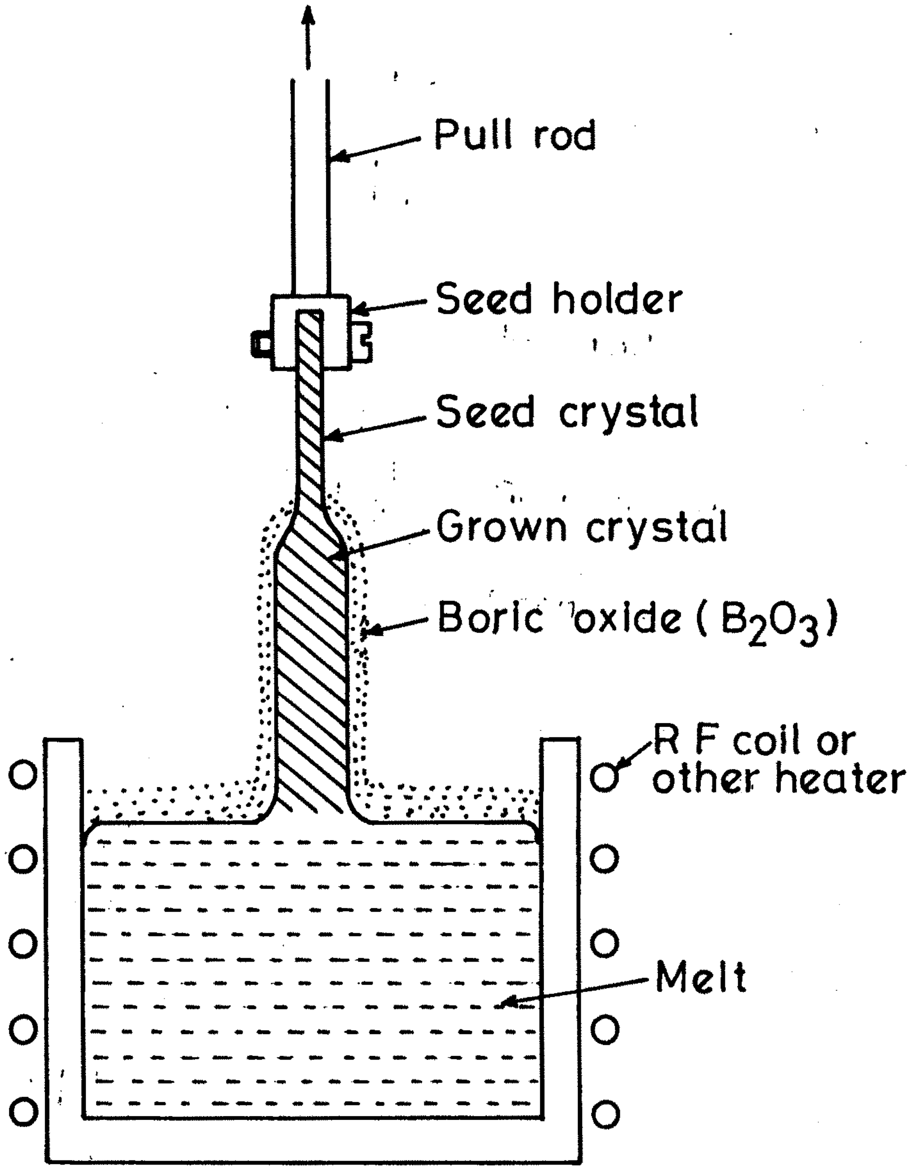
1. Water bath of the thermostat.
2. Crystallization vessel.
3. Slightly supersaturated solution.
4. Perfectly or only partially closed lid.
5. Rotatably mounted seed crystals.
6. Stirrer.

Fig. 1·4



SCHEMATIC OF CRYSTAL PULLING OR CROCHRALSKI METHOD.

Fig. 1·5



LIQUID ENCAPSULATION CROCHRALSKI METHOD.

Fig. 1·6

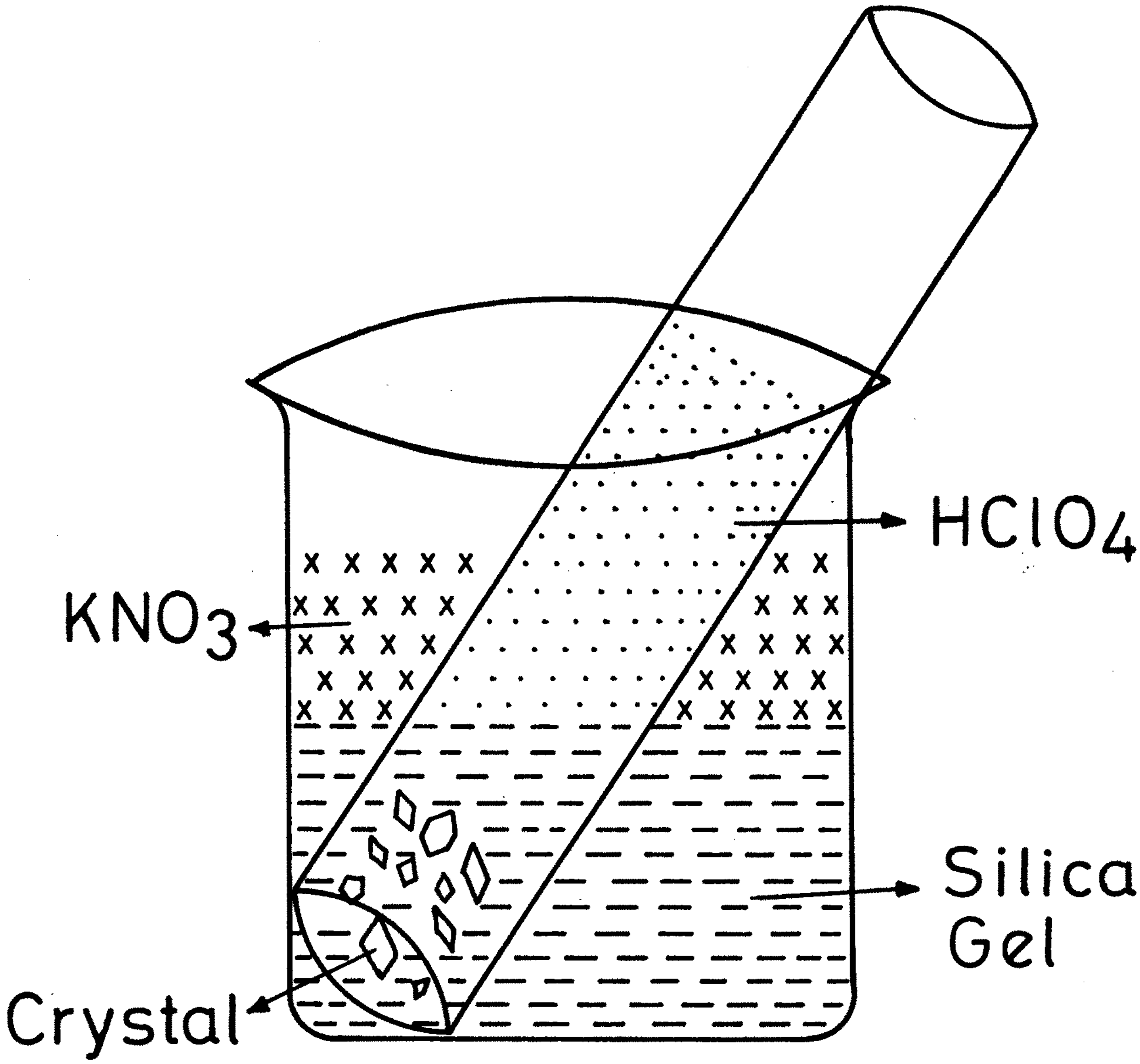


Fig. 3·1

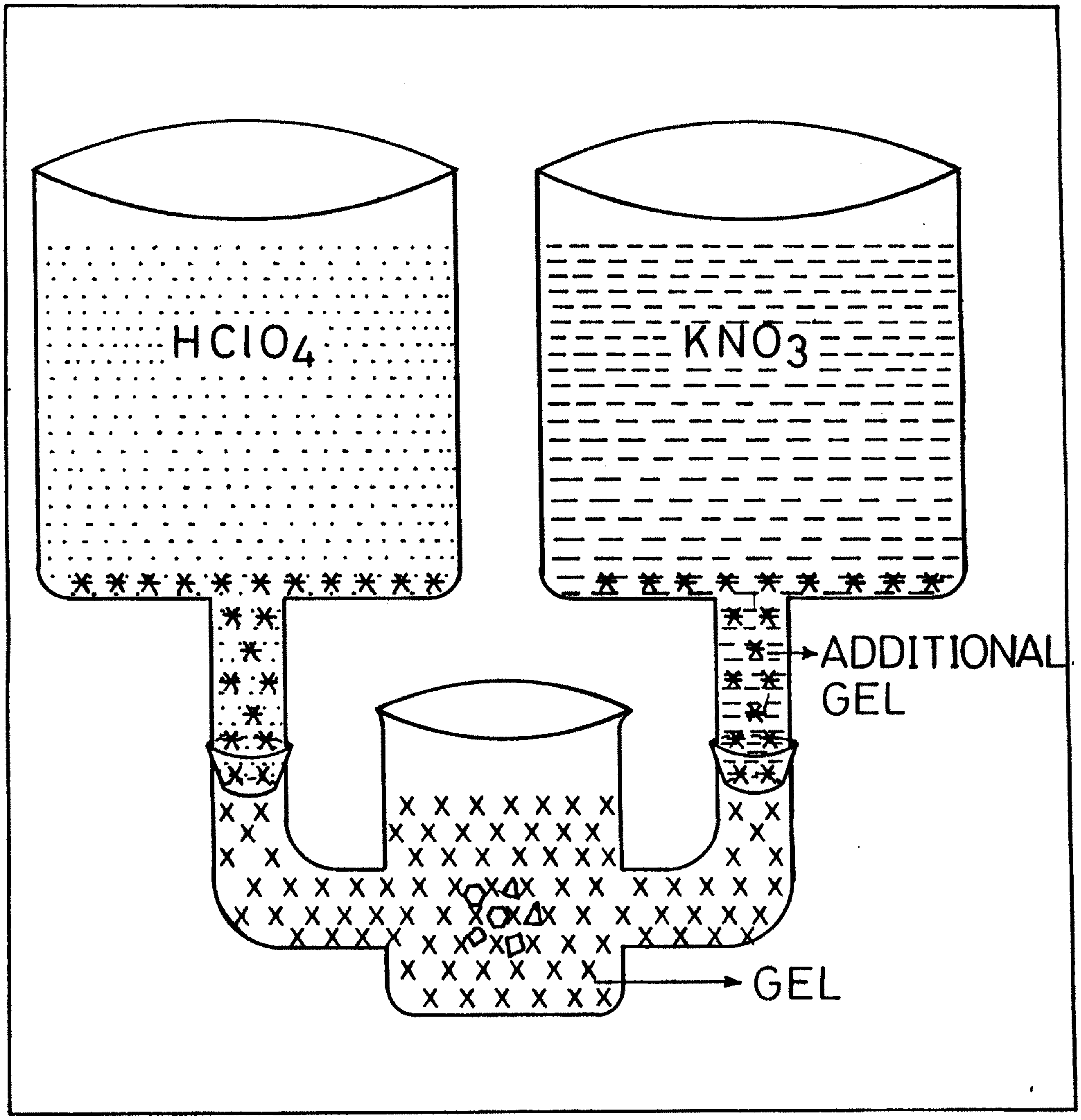


Fig. 3·2

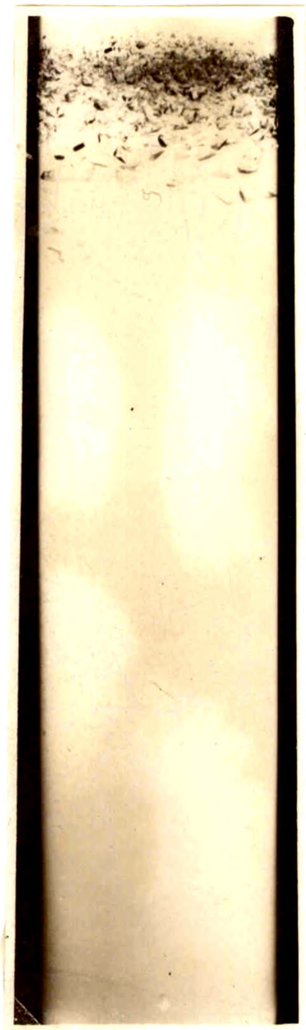


Fig 3·3 <a> <b>

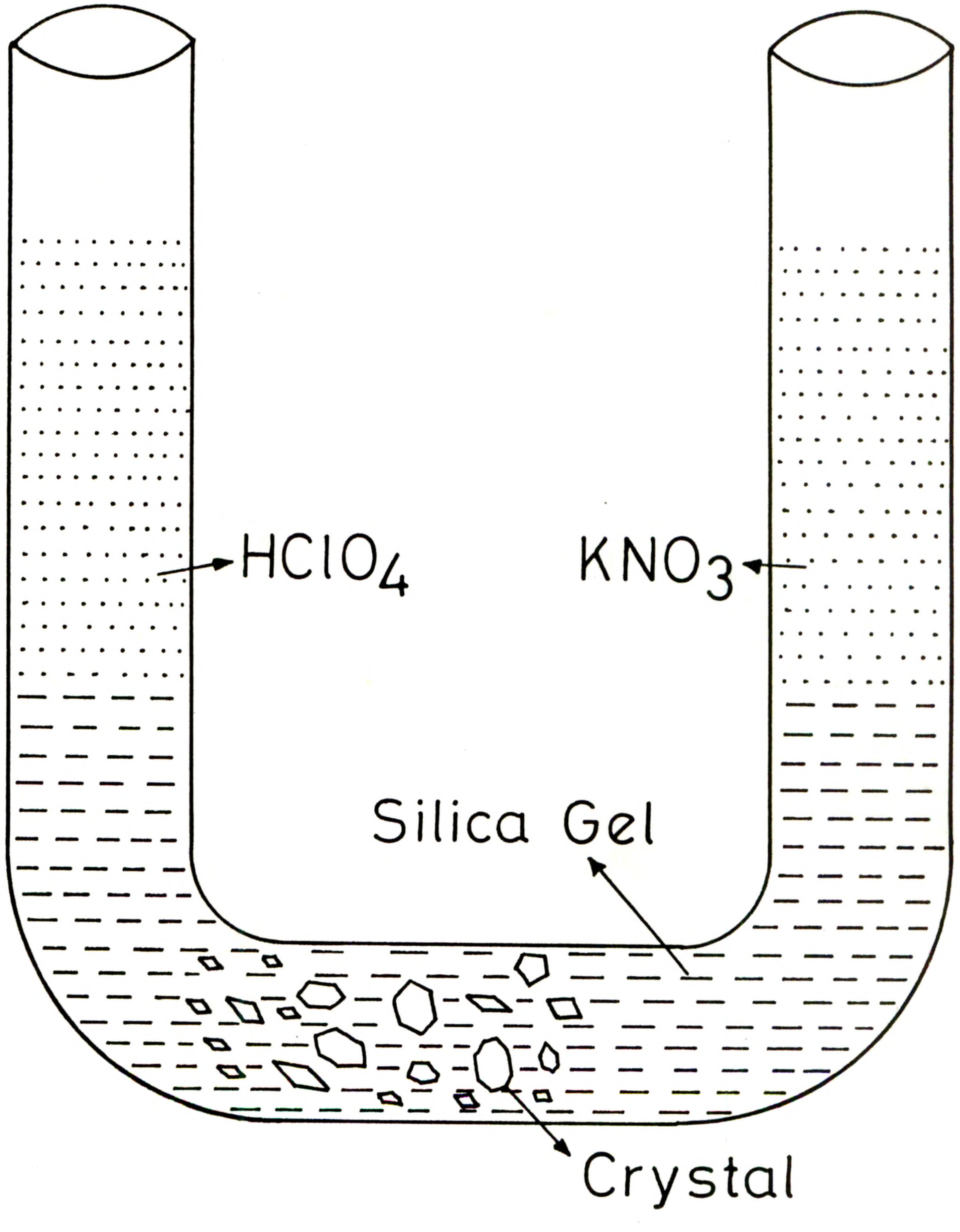


Fig. 3·4

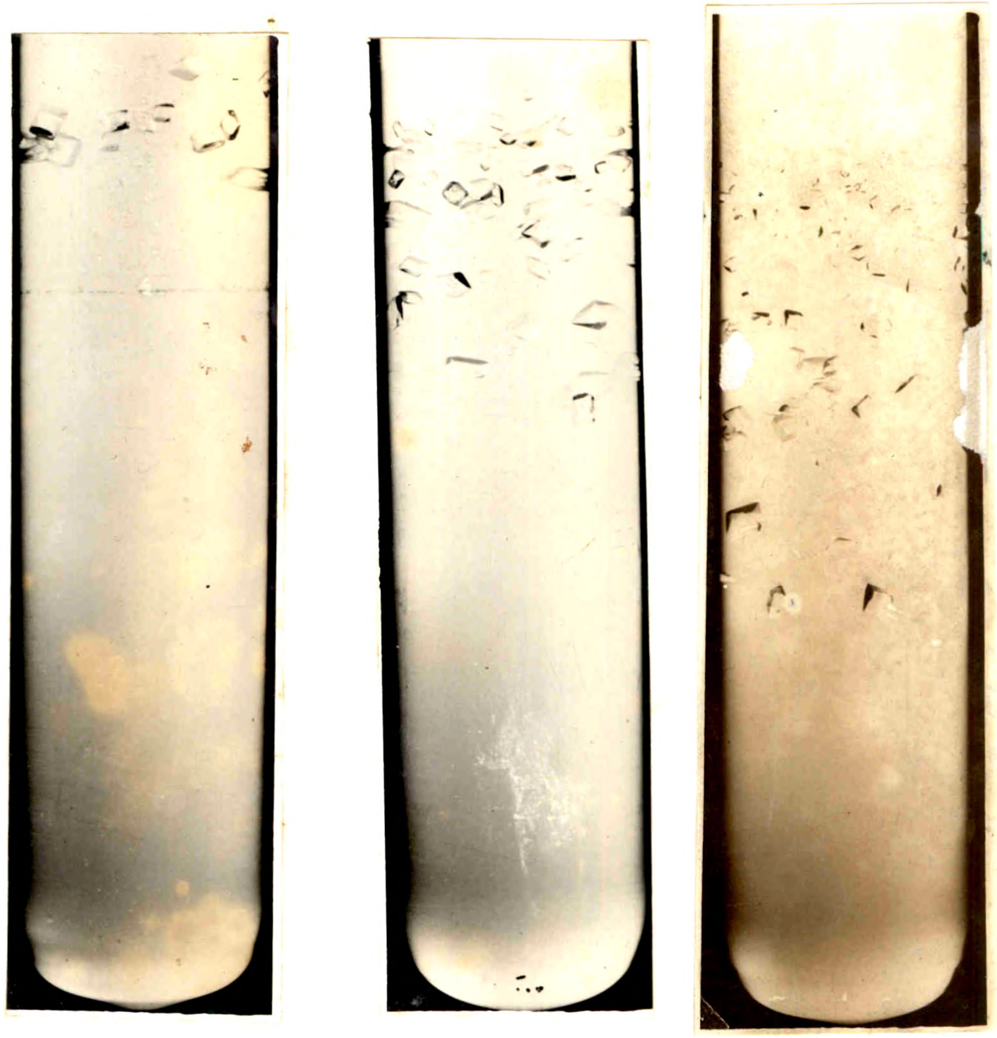


Fig 4·1 ‹a›　　‹b›　　‹c›

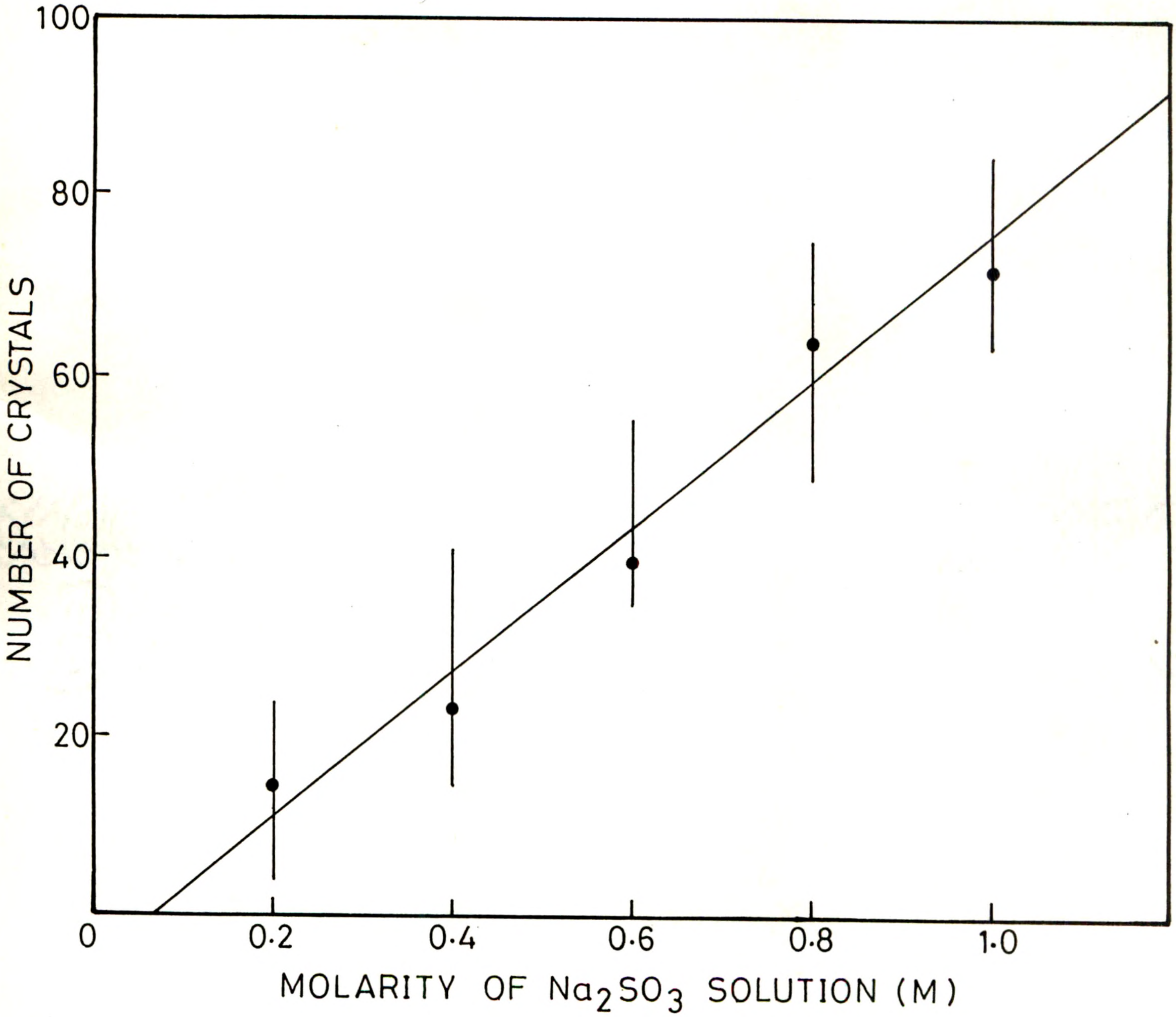

100
80
60
40
20
0
0·2
0·4
0·6
0·8
1·0
NUMBER OF CRYSTALS
MOLARITY OF Na_2SO_3 SOLUTION (M)

Fig. 4·2

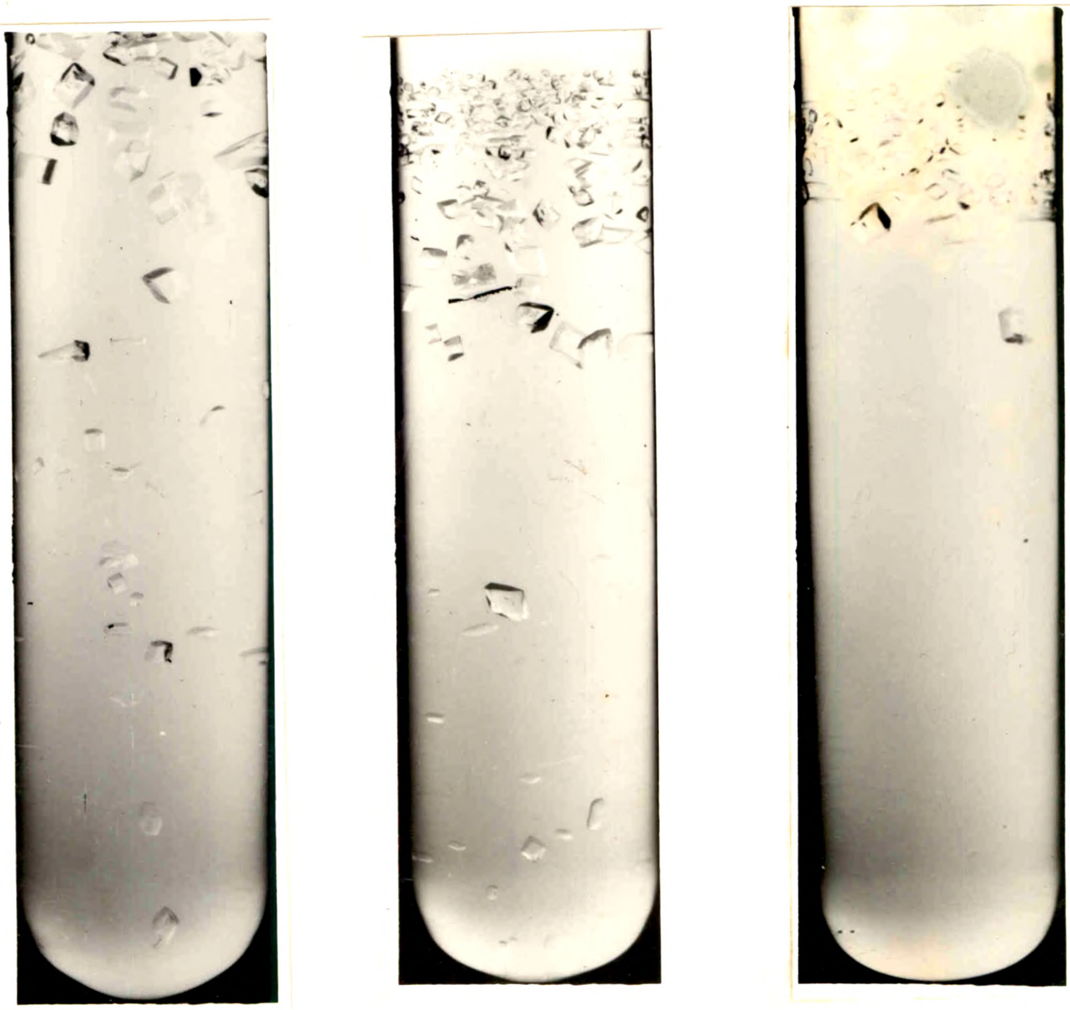
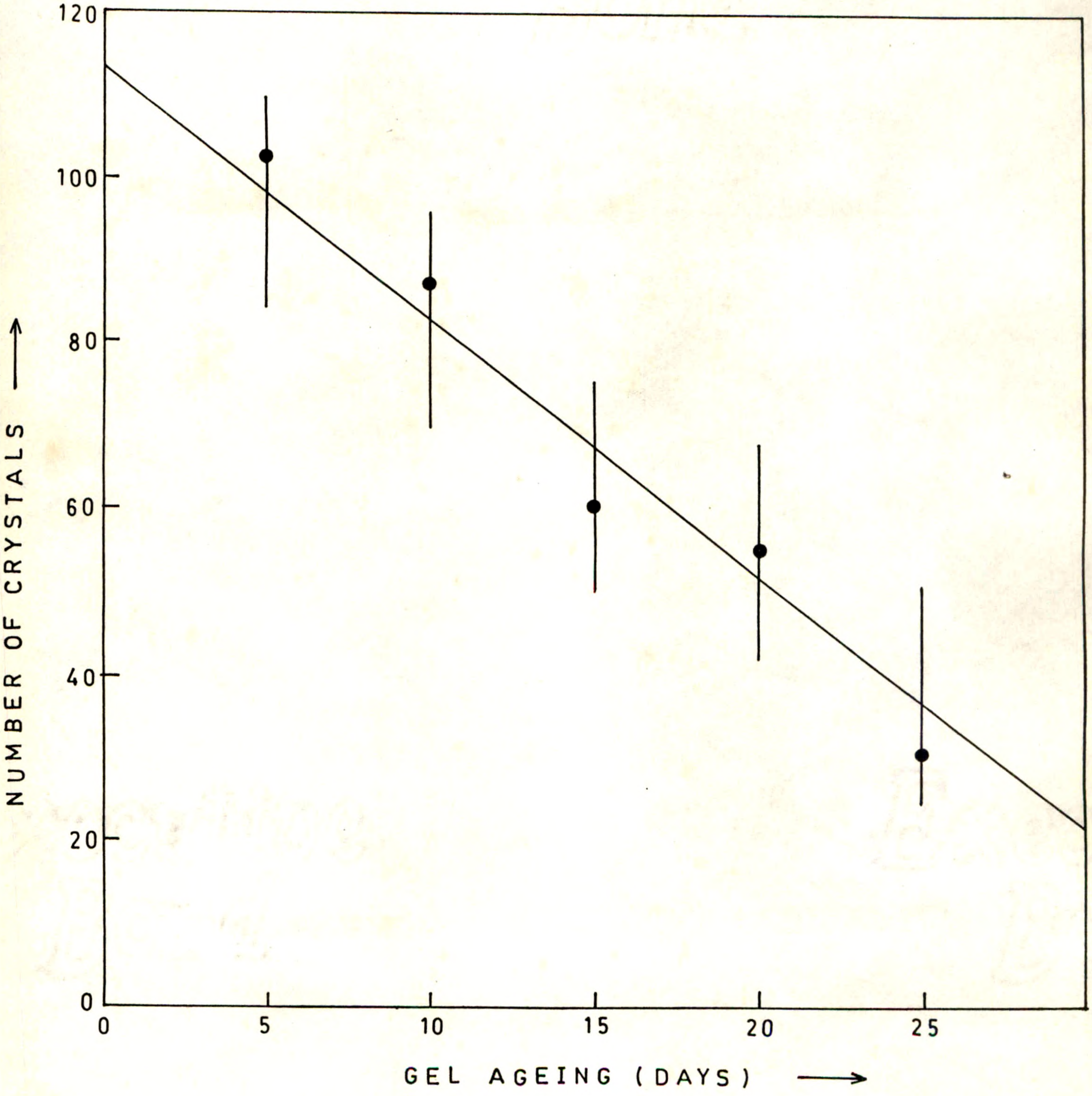


Fig 4·3 <a> <b> <c>



120
100
80
60
40
20
0
NUMBER OF CRYSTALS →
0
5
10
15
20
25
GEL AGEING (DAYS) →

Fig. 4·4

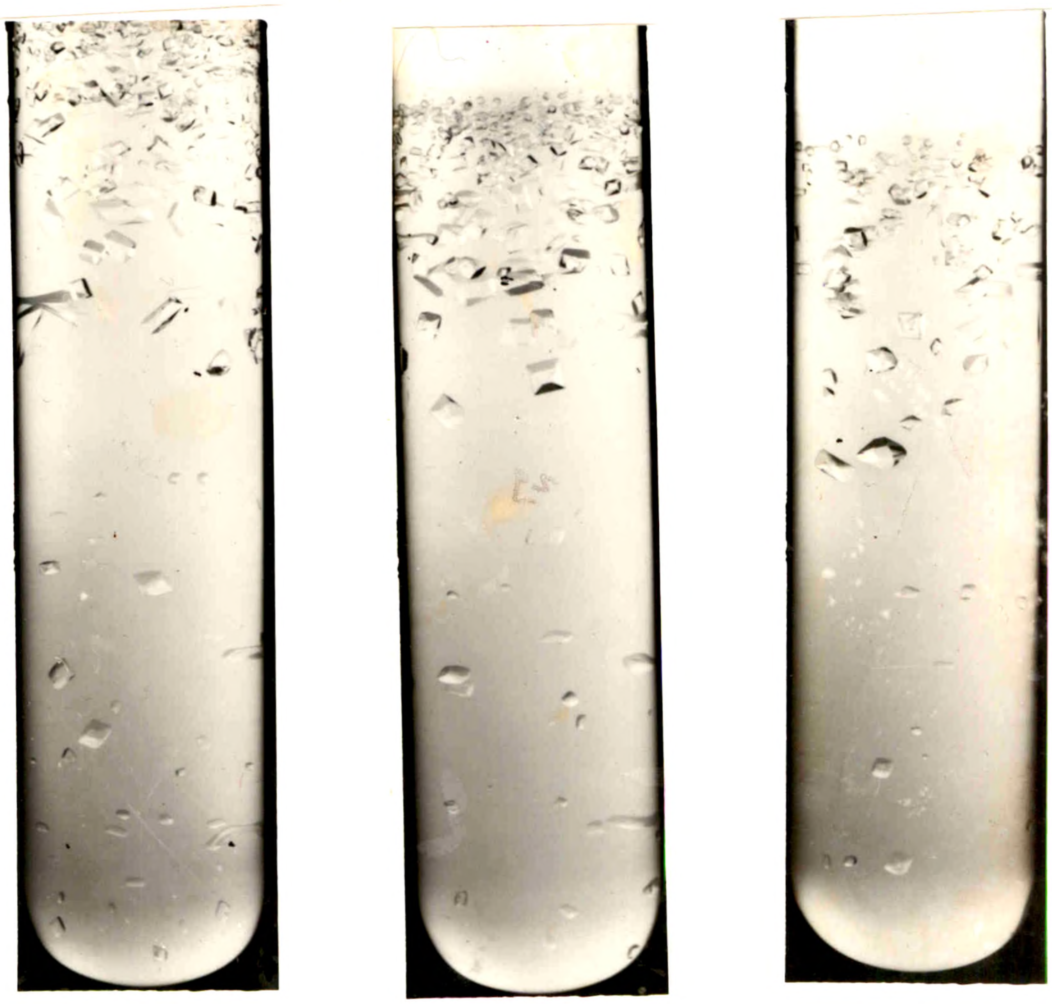


Fig 4-5 <a> <b> <c>

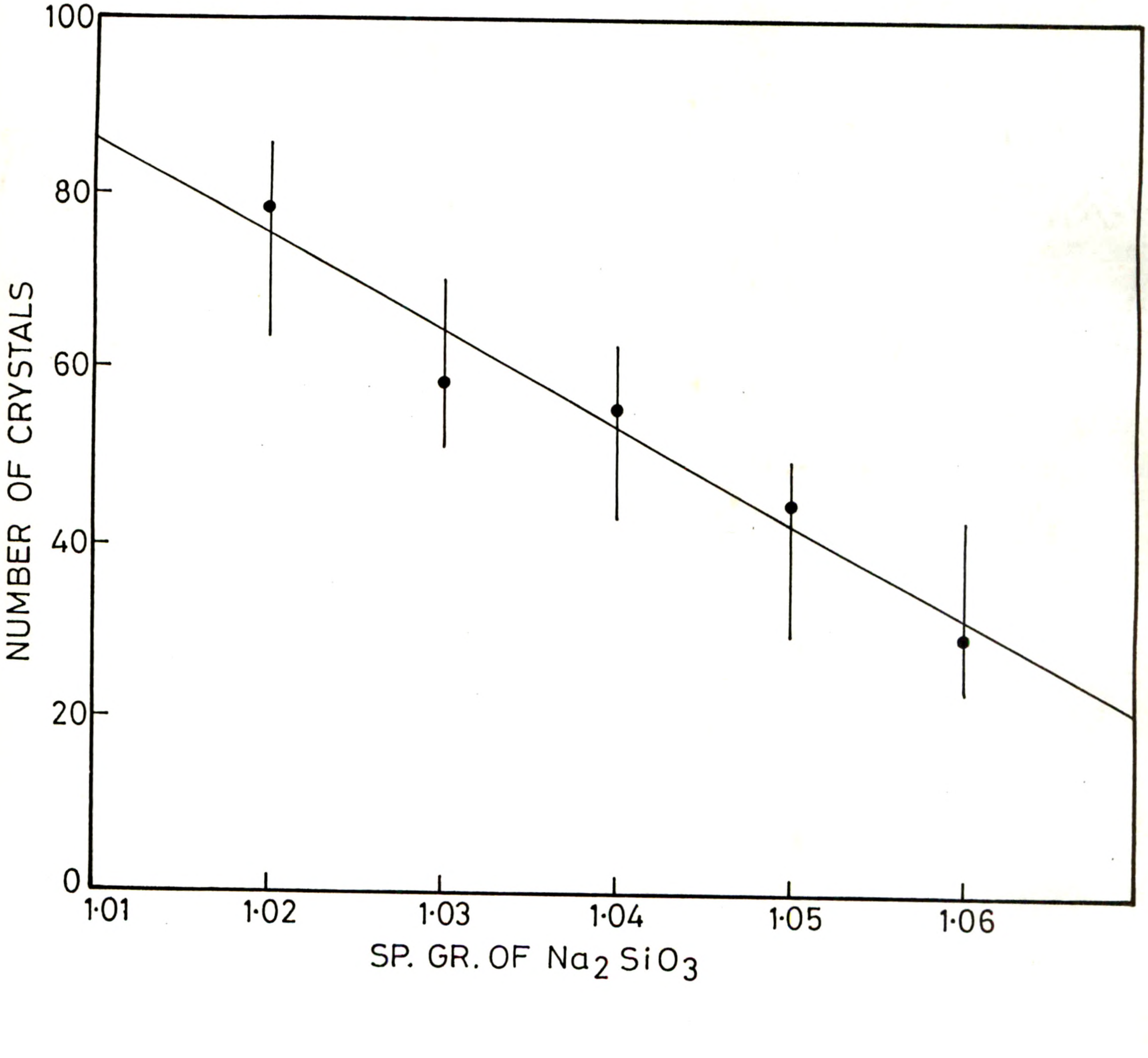

100
80
60
40
20
0
NUMBER OF CRYSTALS
1·01
1·02
1·03
1·04
1·05
1·06
SP. GR. OF Na2 SiO3

Fig. 4·6

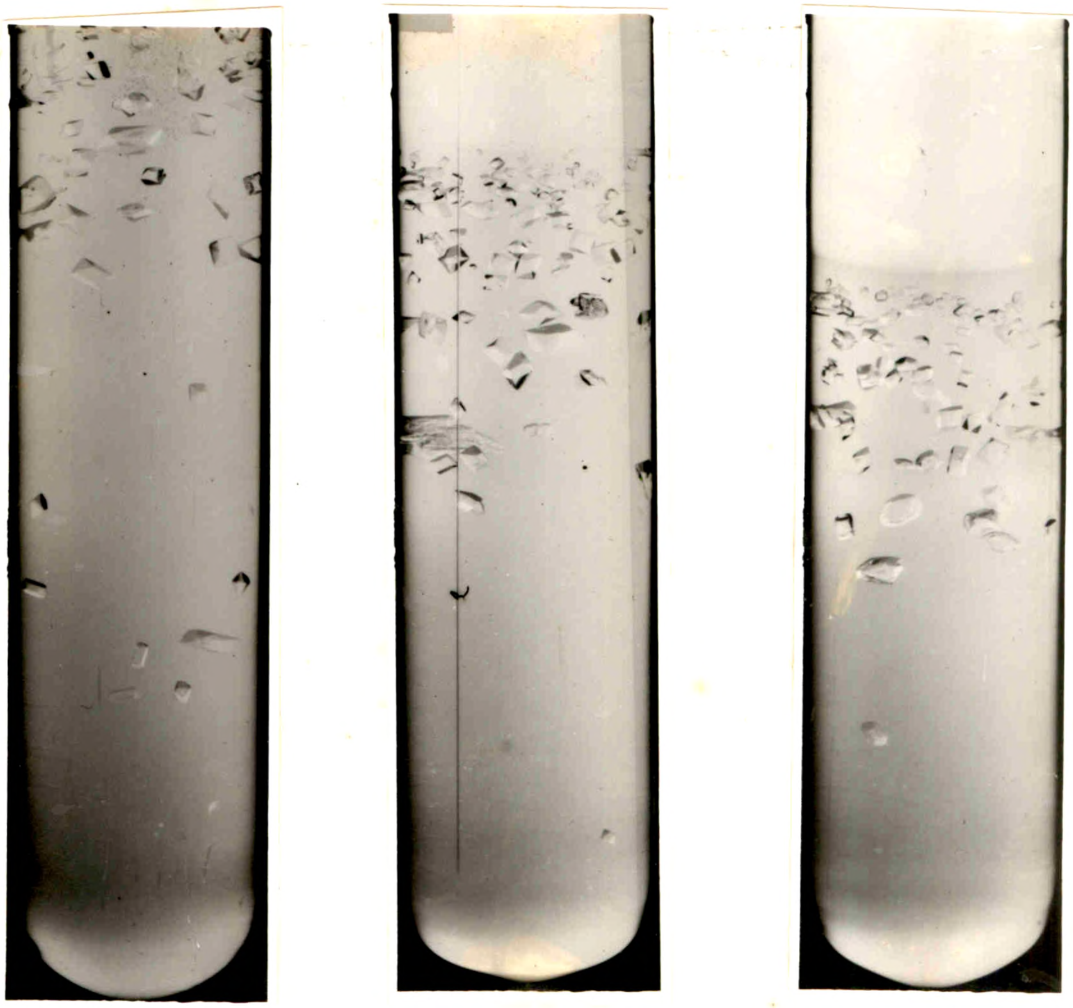


Fig 4.7 <a> <b> <c>

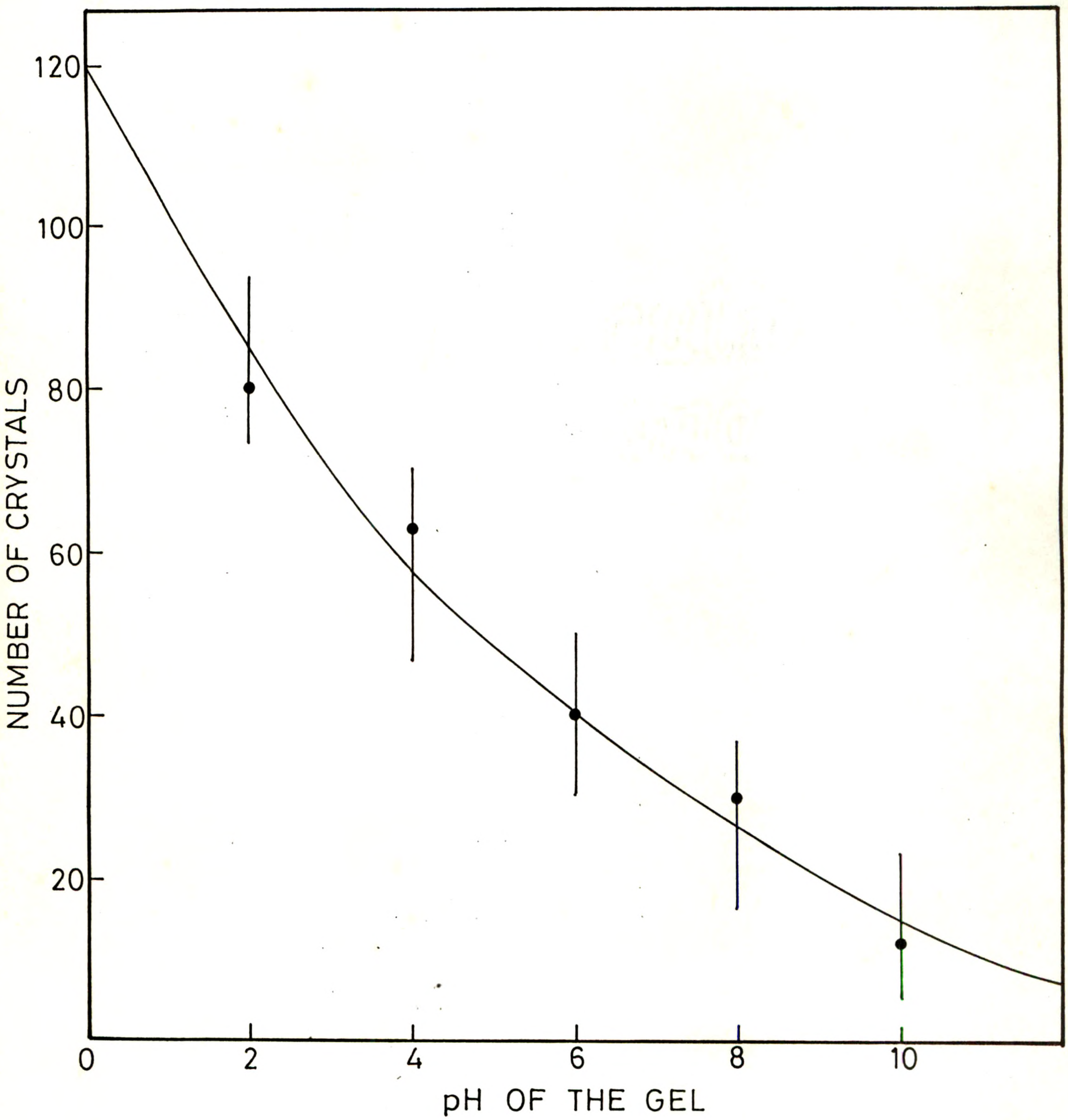


Fig. 4·8

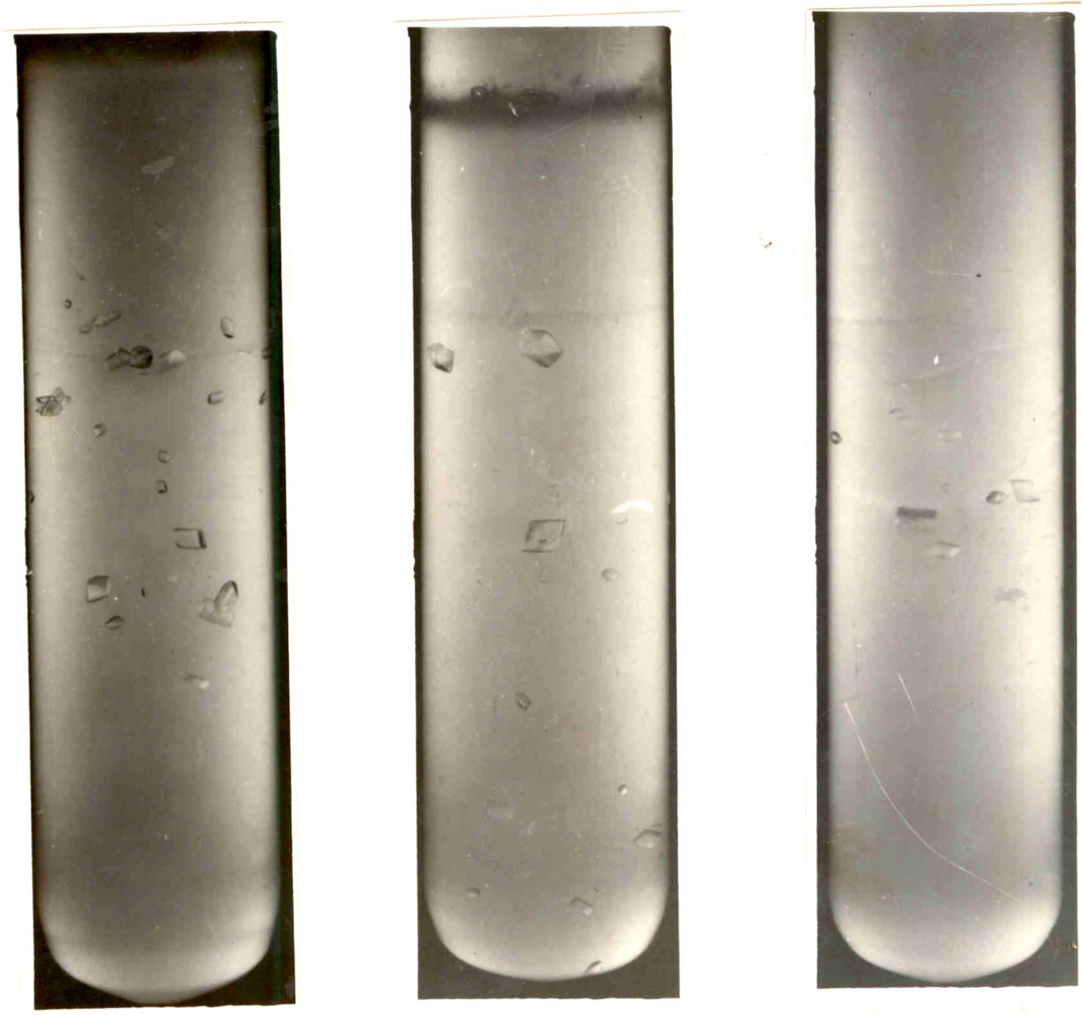


Fig 4.9 ‹a› ‹b› ‹c›

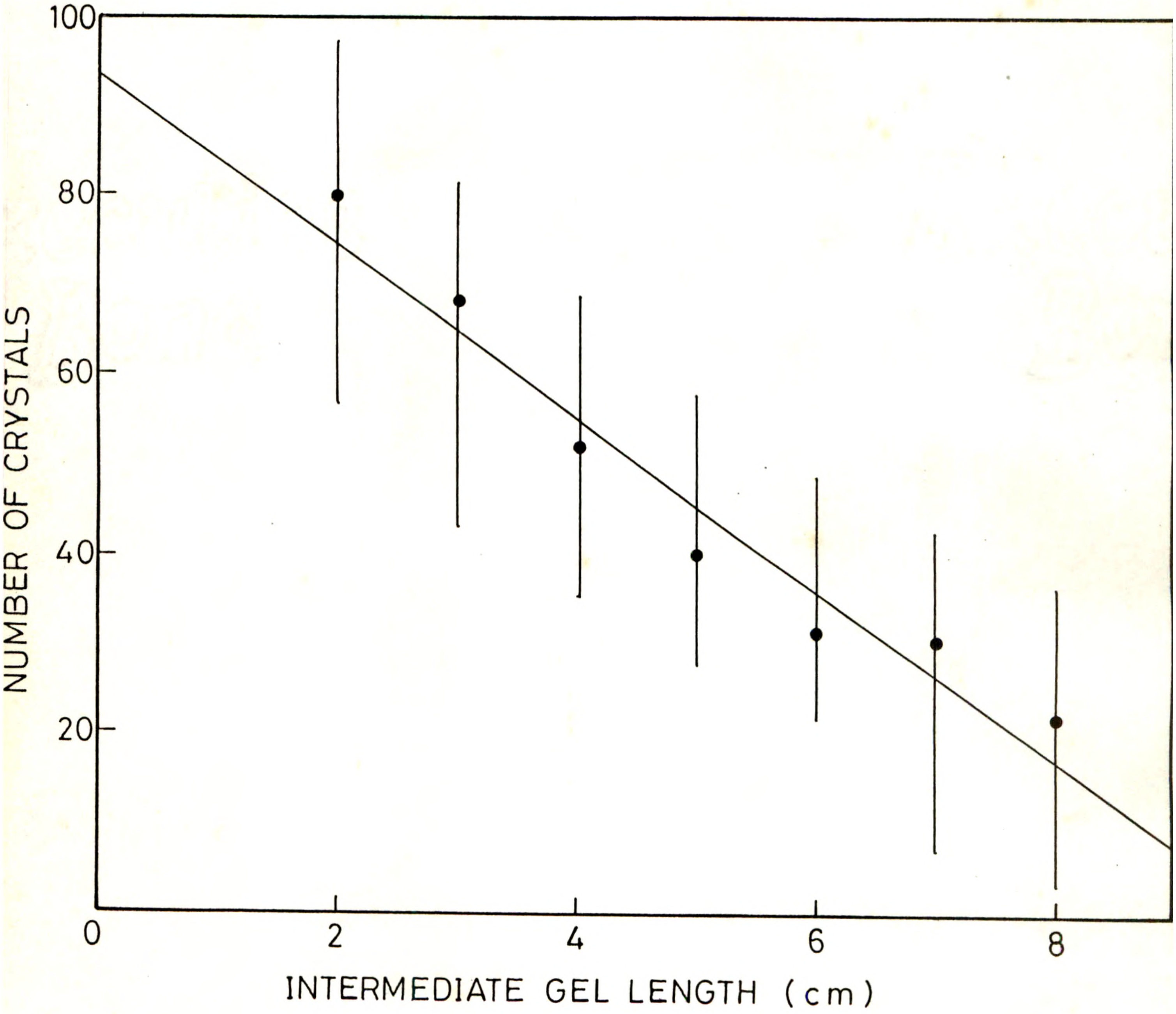

100
80
60
40
20
NUMBER OF CRYSTALS
0
2
4
6
8
INTERMEDIATE GEL LENGTH (cm)

Fig. 4·10

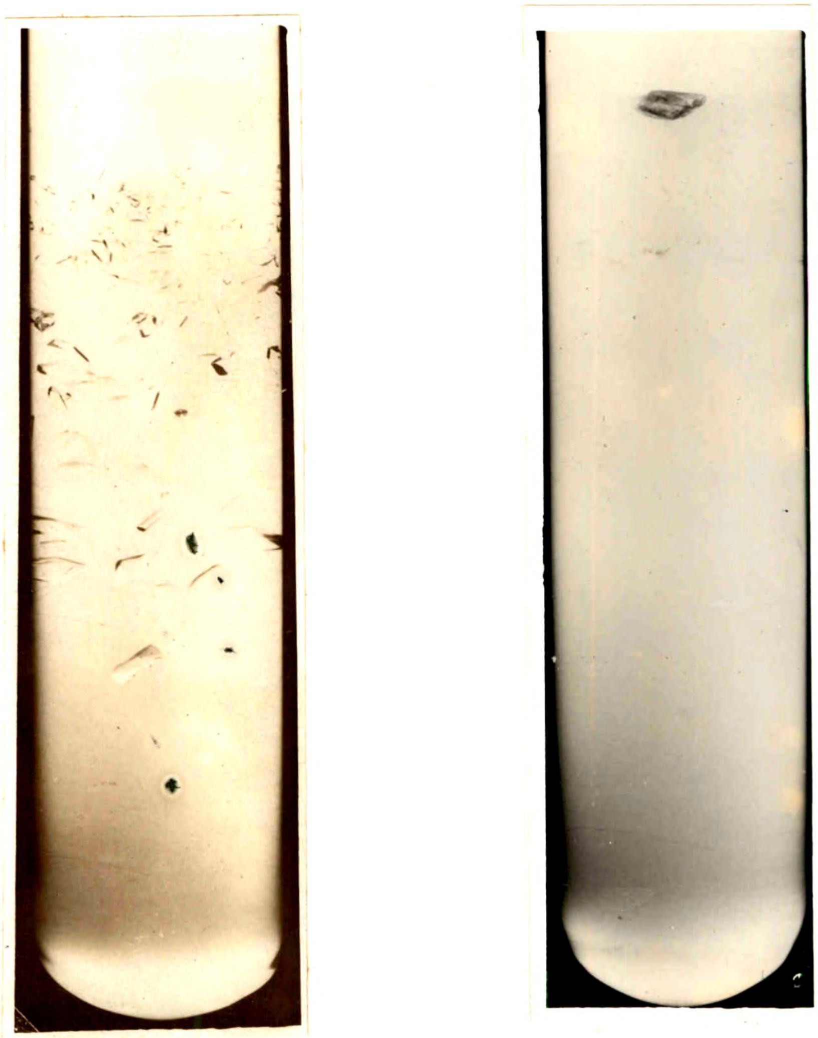
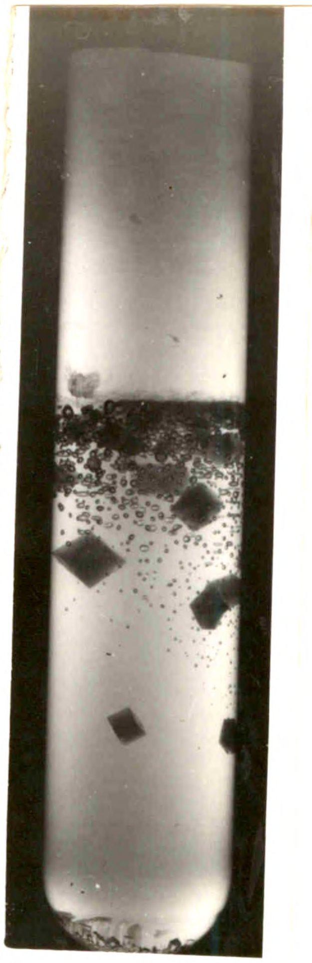


Fig 4.11 ⟨a⟩ ⟨b⟩

Fig 5.1 ⟨a⟩ ⟨b⟩



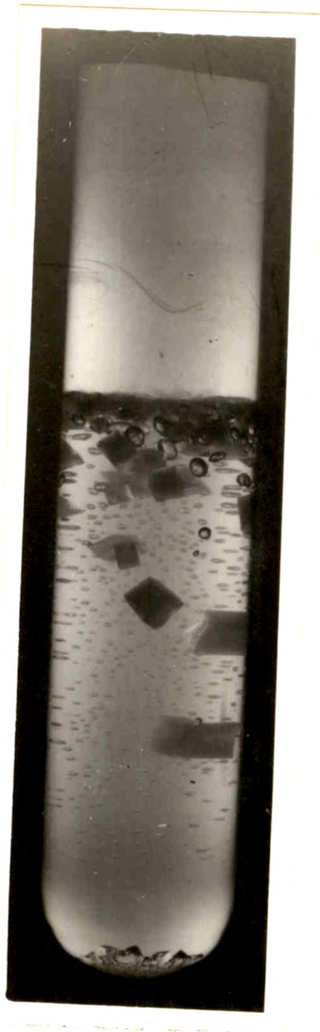


Fig 5·2 (a) (b)

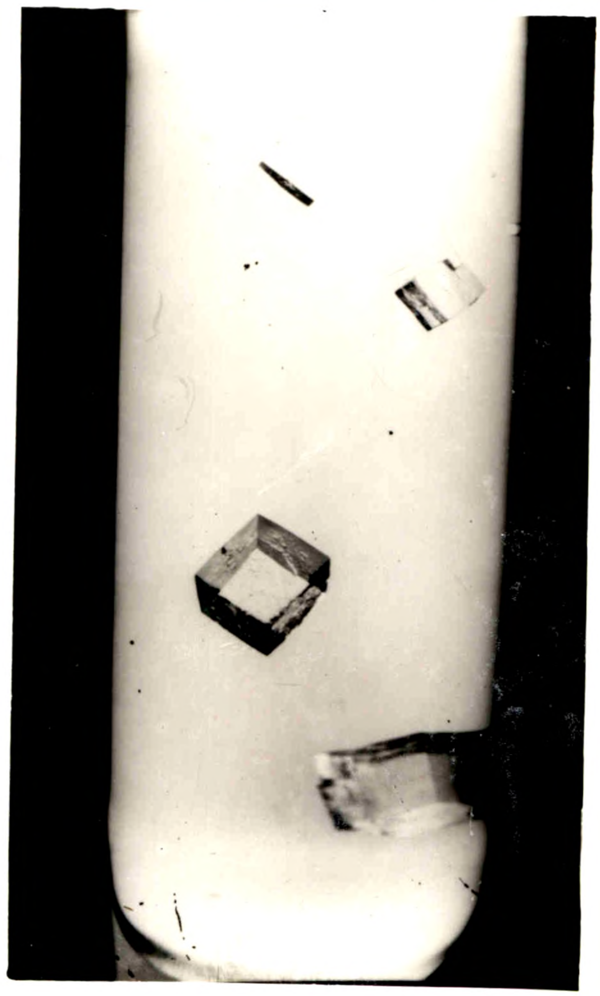


Fig 5·3