AAEC/E154

AAEC/E154

AUSTRALIAN ATOMIC ENERGY COMMISSION

RESEARCH ESTABLISHMENT
LUCAS HEIGHTS

LABORATORY DEVELOPMENT OF THE GRIND-LEACH PROCESS FOR THE H.T.G.C.R. FUEL CYCLE

PART 2. DISSOLUTION OF BERYLLIA IN NITRIC ACID SOLUTIONS

by

M.S. FARRELL
S.R. ISAACS
M.E. SHYING

June 1966

AUSTRALIAN ATOMIC ENERGY COMMISSION

RESEARCH ESTABLISHMENT

LUCAS HEIGHTS

LABORATORY DEVELOPMENT OF THE GRIND–LEACH PROCESS FOR THE H.T.G.C.R. FUEL CYCLE PART 2. DISSOLUTION OF BERYLLIA IN NITRIC ACID SOLUTIONS

by

M. S. FARRELL

S. R. ISAACS

M. E. SHYING

ABSTRACT

The development of the grind–leach process for the processing of beryllia–based fuels requires a knowledge of the dissolution of beryllia in nitric acid. A kinetic study using powdered specimens has proved suitable for the investigation of this system.

The parameters studied include particle size, agitation, temperature, acid concentration, the effect of the addition of fluoride and aluminium, and the effect of neutron irradiation of the beryllia.

The dissolution of beryllia in nitric acid is controlled by a chemical reaction at the surface of the solid and has an apparent activation energy of 18 kcal/mole.

PREFACE

This report is Part 2 of a series of reports on the laboratory development of the grind–leach process for the High Temperature Gas–Cooled Reactor fuel cycle.

> Part 1, Dissolution of Urania–Thoria Fuel Particles in Nitric Acid Solutions, has been issued as AAEC/E143.

Companion series deal with:

Development of Solvent Extraction Processes for the H.T.G.C.R. Fuel Cycle.

> Part 1. Design of a Flowsheet for the Recovery of Actinides, has been issued as AAEC/E139;

and

Economics of the H.T.G.C.R. Fuel Cycle.

CONTENTS

1. INTRODUCTION

A dispersion type fuel with a beryllia matrix as described by Smith (1964) has been chosen for the Australian high temperature gas–cooled reactor (H.T.G.C.R.) feasibility study.

The reprocessing of this beryllia matrix fuel has been discussed by Cairns (1964), who suggested an aqueous fuel cycle using a grind–leach head–end process to obtain solution of the actinides with maximum separation of the more insoluble beryllia. Figure 1 illustrates the conceptual fuel cycle proposed.

The chemical feasibility of such a fuel cycle is being examined and this is the second report of a series on the laboratory development of the grind–leach process. A more general description of this process is given in the first report of the series (Farrell and Isaacs 1965), which deals with the dissolution of $(U,Th)O_2$ fuel particles in nitric acid solutions.

This report considers the effects of comminution, irradiation, and solution variables on the dissolution rate of sintered beryllia in nitric acid solution. The effects of the structural variables of the beryllia such as density, grain size, and impurity content on the dissolution rates were not examined.

2. EXPERIMENTAL

2.1 Materials and Reagents

The beryllia was prepared by the A.A.E.C. Fuel Element Development Section.

Brush UOX grade beryllia was mixed with 2 $^{w}/_{o}$ gum tragacanth, 10 $^{w}/_{o}$ glycerine, and 17 $^{w}/_{o}$ water in a Winkworth mixer for 4 hours. The beryllia mix was then extruded at 2800 p.s.i. through a ½–in diameter steel die, air dried for 48 hours, oven dried for 24 hours at 80 °C and finally at 150 °C for a further 24 hours. After drying, the beryllia was baked in air for 4 hours at 800 °C to destroy the binder. Finally it was sintered in nitrogen at 1500 °C for 2 hours. The sintered density of the beryllia was 2.89 g/cm^3. The grain size was variable, but was mainly 5–15μ with some large grains up to 125μ.

Two batches of the beryllia rods, 3/8 in dia. x 4 in long, were irradiated in the A.A.E.C.'s high flux materials test reactor HIFAR, and a third batch was retained. Both batches were irradiated in the same rig (X–95) and position in HIFAR, but to different doses, 3 x 10^{20} and 6 x 10^{20} nvt (integrated fission neutron dose).

Both control and irradiated beryllia was manually crushed in a percussion mortar until all the material passed a –52 BSS sieve. The beryllia was crushed carefully, using a high load in the mortar and recycling through the –52 BSS sieve frequently to minimize the production of fines. The crushed –52 BSS size beryllia was wet screened through a set of standard Endicott stainless steel sieves, and the material remaining on each sieve was dried and weighed. The resulting comminuted, sized, and dried beryllia was used in the dissolution rate experiments.

The surface areas of the various sieve fractions of the comminuted material were measured by the B.E.T. (Kr^{85}) gas adsorption method (Harding 1962).

The reagents used in the dissolution rate experiments were of analytical grade (A.R.) quality except for the aluminium nitrate which was laboratory grade (L.R.).

2.2 Apparatus

In the dissolution experiments a glass dissolver was used when the dissolvent did not contain fluoride, and Teflon or stainless steel (18–8–1) dissolvers were used when fluoride was present. Each dissolver was equipped with a paddle agitator with speed control (checked with a stroboflash). Electrical resistance heating was used, and the temperature was controlled to within ± 1 °C.

2.3 Procedure

A known mass of dry beryllia (1 to 3 g) was added to 200–250 ml nitric acid, preheated to the required temperature, and the solution was stirred at constant speed.

Samples of the solution were withdrawn at time intervals chosen from an estimate of the expected dissolution rate. Either glass or Nalgon pipettes were used to extract the samples. The volume of the reagent was kept constant by adding fresh dissolvent equivalent to the volume of the sample.

The samples were centrifuged to remove any solids and the supernate was analysed for beryllium using a spectrophotometric method (Pakalns 1964). The mass dissolved was calculated from these analyses, corrected for sample losses.

3. RESULTS AND DISCUSSION

3.1 Dissolution Kinetics using Powders

Comparison of the average dissolution rates for the same per cent. reaction was used to determine the effect of each variable on the dissolution of powdered beryllia in nitric acid solution. The use of powdered material overcomes the problems associated with the preparation of homogeneous massive specimens. Further, the results are more readily applicable to the comminuted fuel being used in the grind–leach process.

Average dissolution rates can be determined with a high degree of accuracy. Normally the dissolution curves used to determine rates are non–linear because of the decrease in mass of solids present in the solution as the dissolution proceeds. It is difficult to determine either initial or instantaneous dissolution rates from such curves.

Schortmann and DeSesa (1957) used average dissolution rates over a fixed per cent. of the reaction to study the dissolution of powdered UO_2 in aqueous Na_2CO_3 solutions. They demonstrated that the non–linearity of the rate curves obtained from extensive dissolutions of powders, coarse or fine, is no problem provided the surface area varies with per cent. reaction in the same manner for each experiment. Thus the effect of a variable can be correlated with the rates at a particular per cent. reaction.

As stated by Schortmann and DeSesa, "the average particle size of a fine powder at any per cent. reaction is a function of the per cent. reacted and not of the time required to reach that degree of completion, nor of the amount of material initially charged to the dissolver".

Schortmann and DeSesa's method was consequently investigated to determine its applicability to the dissolution of the beryllia in nitric acid. If the average particle size of beryllia powder at any per cent. reaction is a function of the per cent. reacted and is independent of the reaction time, then the effect of a variable determined at a number of different per cent. dissolution values should be identical. To test the beryllia/nitric acid system, temperature was chosen as the variable and the Arrhenius plots were calculated.

The rate curves (Figure 2) were of a similar shape. From these curves average rates were calculated for 10, 20, and 30 per cent. reaction. The rate for 30 per cent. dissolution at 105 °C is an extrapolated value. The Arrhenius plots from each set of data show no significant difference in slope (Figure 3). Consequently comparison of average dissolution rates over a fixed per cent. dissolution is a suitable method for the determination of the effect of variables on the dissolution of beryllia in nitric acid.

Changes in other parameters resulted in families of curves similar to that of temperature (Figure 2). The only exceptions were the curves obtained for the effect of particle size (Figure 4) and irradiation (Figure 17). The shapes of these curves up to 25 per cent. dissolution were considered sufficiently similar to permit the conclusions that were drawn from them.

3.2 Effects of Particle Size and Specific Surface Area

The comminution of the beryllia matrix in the grind–leach process necessitates a study of the effect of particle size or specific surface area on the dissolution rate of the beryllia in nitric acid.

The dissolution of a series of sieve fractions of varying particle size was studied under identical conditions (Figure 4). The specific dissolution rate increases markedly as the particle size decreases (Figure 5). B.E.T. (Kr^{85}) surface areas were determined on each sieve size and enabled rates to be expressed as $mg/cm^2.min$. The results (Table 1) show that the increased dissolution of the beryllia results only from an increase in available surface area.

Care must therefore be taken in the comminution step of the grind–leach process to maintain a minimum beryllia surface area relative to that of the fuel particles to minimize the dissolution of the beryllia.

Solntsev and Tolmachev (1961) used powders to investigate the mechanism of the dissolution of solids in aqueous solutions. They examined reactions whose rates depend on the rate of reaction at the surface of the solid, and reactions whose rate depends on the diffusion of the reacting substances. The relationship

$$M_O^{1/3} - M_T^{1/3} = k_1 T \qquad (1)$$

(M_O = initial mass, M_T = mass at time T)

was derived for chemically controlled reactions, and the relationship

$$(M_O^{1/3} - M_T^{1/3})^2 = k_2 T \qquad (2)$$

for diffusion controlled reactions.

Solntsev and Tolmachev showed that (1) and (2) apply even to irregularly shaped particles of the type used in this investigation (Figure 13).

Using values from dissolution of beryllia in nitric acid, both $M_O^{1/3} - M_T^{1/3}$ and $(M_O^{1/3} - M_T^{1/3})^2$ were plotted against time. Linear relationships were obtained with $M_O^{1/3} - M_T^{1/3}$ and curved relationships with $(M_O^{1/3} - M_T^{1/3})^2$. Curves 2 and 4 of Figures 6 and 7 are typical of the plots obtained. Some deviation from the linear plots was detected for the very large and very small particles (Curves 1 and 3, Figure 6).

These results indicate that the dissolution kinetics of beryllia in nitric acid solutions are controlled by a chemical reaction at the surface of the solid.

3.3 Effect of Temperature

The Arrhenius plots (Figure 3) are linear and indicate that there is no change in reaction mechanism with temperature over the range investigated. The apparent activation energy for the overall reaction,

$$BeO_{(s)} + 2HNO_3 \longrightarrow Be(NO_3)_2 + H_2O$$

is 18 kcal/mole. This value is similar to that (19 kcal/mole) obtained by Farrell and Isaacs (1965) for the dissolution of thoria in nitric acid using fluoride as a catalyst. A value of 15 kcal/mole was obtained by C.E.N. – Belgo–Nucleaire (1962) for the fluoride–catalysed dissolution of PuO_2 in nitric acid. Hence a variation in the leaching temperature is unlikely to affect the selectivity of the grind–leach process although in the present case the effect of the addition of a small quantity of fluoride to the nitric acid dissolvent on the activation energy was not examined.

3.4 Effect of Agitation

The effect of agitation is important in the design of the leaching vessel and also as an indication of whether the reaction is a diffusion controlled process. The dissolution curves at 60, 400, and 1500 rev/min (Figure 8) show that there is no significant difference in the dissolution rate with varying degrees of agitation. The greater randomness of the 60 rev/min run is due to poorer mixing and consequent inhomogeneity of the solution when sampled.

This reaction is clearly insensitive to agitation, although paddle stirring is not an ideal method of determining boundary layer effects.

3.5 Effect of Nitric Acid Concentration

The dissolution rates of beryllia powder (−200 +300 BSS) were measured in nitric acid solutions of various concentrations, other conditions being constant. The results (Figure 9) indicate that the dissolution of beryllia is fairly insensitive to changes in nitric acid concentration; below 8M there is an approximate first order dependency and between 8 – 15M the dissolution rate is independent of nitric acid concentration.

3.6 Effect of Fluoride Concentration

To leach thoria from a beryllia matrix with nitric acid the reaction must be catalysed with fluoride (Bond 1958). Farrell and Isaacs (1965) found that a small addition (0.001M fluoride) increased the dissolution of thoria by a factor of approximately 200.

The addition of 0.001M F^- to 13M nitric acid only increases the specific dissolution rate of beryllia by a factor of approximately two, suggesting that the fluoride does not behave catalytically in this reaction.

A log–plot of initial fluoride ion concentration versus beryllia dissolution rate (Figure 10) shows a linear relationship but with a change of slope at about 0.07M F^-. The apparent order of reaction with respect to fluoride ion concentration in the range 0.001 – 0.07M F^- is 0.4. The dissolution of thoria from $(U,Th)O_2$ fuel particles also shows an apparent order of reaction of 0.4 with respect to fluoride over the same range.

Hydrofluoric acid dissolves massive beryllia rapidly whereas nitric acid of an equivalent concentration dissolves it at a slower rate (Ekstrom et al. 1962). Apparently above 0.07M F^- the hydrofluoric acid dissolution predominates.

3.7 Effect of Aluminium Concentration in the Presence of Fluoride

The addition of Al^{3+} as aluminium nitrate inhibits the dissolution of beryllia in 13M HNO_3 + 0.05M F^- over the range 0.05 – 0.5M Al^{3+} (Figure 11). The effect of Al^{3+} addition is more marked for beryllia dissolution than for thoria dissolution in the same dissolvent (Farrell and Isaacs 1965). This is because the stabilities of the fluoro–complexes of aluminium are greater than those of beryllium but lower than those of thorium as determined by Babko and Shimadina (1959).

Other cation effects have not been studied but preliminary measurements on the presence of dissolved beryllium have indicated that there is only slight inhibition of the dissolution of beryllia in nitric acid–fluoride solution.

3.8 Effect of Neutron Irradiation

Irradiation of beryllia causes anisotropic growth of the beryllia grains. The essential properties of the beryllia, thermal conductivity and strength, are not adversely affected until microcracking takes place, strength increasing up to this stage (Hickman 1966). Beryllia is only acceptable in a reactor to some safe point prior to microcracking. Consequently material irradiated to a point below the microcracking stage is required, to determine the effect of the increased strength on comminution and the effect of high intergranular strain on the dissolution properties.

The beryllia rods irradiated for these experiments were microcracked (Figures 12 and 13). Nevertheless some data were obtained using this material.

Both batches of irradiated material were more readily crushed than the control material while the more highly irradiated material required the least effort. Comparative screen analysis (Figure 14) shows that the mass per cent. fines (−300 BSS) increased markedly with increased irradiation.

The fines fraction from each batch was analysed for particle size distribution using the Sharples Micromerograph. The results (Figure 15) show that the mass per cent. of the $< 10\,\mu$ material was greatest for the unirradiated material. The loss of intergranular strength due to irradiation permitted the ready comminution of the massive material to its original grain size. The greater effort required to crush the control material, because of its higher intergranular strength, resulted in the fracture of the grains themselves.

The dissolution characteristics of the irradiated beryllia were examined. The rates obtained (Table 2) for both large particles (−52 +72 BSS) from Figure 16, and for small particles (−200 +300 BSS) from Figure 17, show that although there was no significant difference between the dissolution rates of the unirradiated and irradiated material of smaller particle size, the irradiated material of larger particle size dissolved twice as fast. These results are consistent with grain boundary effects rather than a change in the chemical reactivity of the beryllia, and this is confirmed at least partly by the increase in specific surface area of the irradiated material (Table 2). If dissolution rates depend primarily on chemical reactivity then the difference between the rates of irradiated and unirradiated material should be the same irrespective of particle size, but if rates depend on intergranular properties then this difference in rate should decrease with decrease in particle size because the number of grains per particle decreases, as has occurred in this case.

Irradiation beyond the point of microcracking produces a more readily comminuted material, but there is no evidence of a change in chemical reactivity.

4. CONCLUSIONS

(i) Comparison of average dissolution rates over a fixed per cent. dissolution is a suitable means of determining the solution variables of the dissolution of beryllia powders in nitric acid solutions.

(ii) The dissolution rate of beryllia in nitric acid solution expressed as mg/cm^2(B.E.T.).min is independent of the mean particle size over the range 250 to $25\,\mu$.

(iii) The dissolution rate is independent of nitric acid concentration above 8M, below which there is an apparent first order dependency.

(iv) Addition of fluoride ion increases the dissolution rate. The relationship between dissolution rate and fluoride concentration is similar to that of thoria up to 0.07M, after which hydrofluoric acid attack apparently predominates. Aluminium ion complexes with the fluoride and consequently reduces dissolution rates. The effect of added aluminium is greater for beryllia than for thoria dissolution under the same conditions.

(v) Irradiation of the beryllia up to 6×10^{20} nvt (integrated fission neutron dose) resulted in microcracking. The resultant material was more readily comminuted and contained an increased percentage of fines which would increase the dissolution rate of the beryllia in the grind–leach process. Although the irradiated beryllia had suffered severe damage, no apparent change in chemical reactivity was detected in the region investigated.

(vi) The dissolution of beryllia in nitric acid is kinetically controlled by a chemical reaction at the surface of the solid. It is consistent with the relationship $M_O^{1/3} - M_T^{1/3} = k_1 T$ proposed by Solntsev and Tolmachev (1961), and its reaction rate is independent of the degree of agitation. Further it is temperature sensitive with an apparent activation energy of approximately 18 kcal/mole.

5. RECOMMENDATIONS FOR THE GRIND-LEACH PROCESS

The selective leaching of urania and thoria from a beryllia matrix, after comminution, will depend on the relative size distribution of the components. Particle size will be a problem in the grind–leach process only if the actinide particles are less readily abraded. Comminution and leaching studies on the highly irradiated fuelled beryllia to practical levels of burnup are necessary to determine the relative particle size distributions and the feasibility of the selective leaching process.

Temperature change will not affect the selectivity of the leaching process. If fuel of smaller initial particle size (50 μ) is chosen, fine grinding will be necessary and temperature reduction may be used to maintain reasonable dissolution times.

Variation of the effective fluoride concentration between 0.01 – 0.05M in 13M nitric acid will not affect the selectivity of the leaching process. Some effects of foreign cations, in the presence of fluoride, have been determined. However, as the fuel oxides dissolve during a nitric acid leach, beryllium, uranium, and thorium will complex the fluoride. Leaching trials will be necessary to show the effectiveness of the fluoride present and to indicate if further additions of fluoride are necessary during the leaching process.

Nitric acid concentration should be maintained at approximately 13M.

6. ACKNOWLEDGEMENTS

Preliminary dissolution data was obtained by J.R. Coady and A. Ekstrom. The surface areas were determined by A. Berzins and the particle size distributions by C. LaRiviere. The beryllia was prepared and irradiated by the Fuel Element Development Section and Materials Physics Section respectively. Analytical methods were developed by T.M. Florence and P. Pakalns of the Analytical Chemistry Section.

The assistance of all these is gratefully acknowledged. The advice of Dr. R.C. Cairns (Fuel Cycle Development Section) is especially acknowledged.

7. REFERENCES

Babko, A.K. and Shimadina, L.G. (1959). – Russian J. of Inorganic Chemistry 4(5): 482.

Bond, W.D. (1958). – ORNL–2519.

Cairns, R.C. (1964). – J. Nucl. Materials 14: 55–68.

C.E.N. – Belgo–Nucleaire (1962). – EURAEC – 708.

Ekstrom, A., Farrell, M.S., and Temple, R.B. (1962). – AAEC/TM155.

Farrell, M.S., and Isaacs, S.R. (1965). – AAEC/E143.

Harding, B.C. (1962). – AAEC/TM142.

Hickman, B.S. (1966). – Studies in Irradiation Effects Vol. I. p. 77. Gordon and Breach, New York.

Pakalns, P. (1964). – Anal. Chim. Acta 31: 576.

Schortmann, W.E., and DeSesa, M.A. (1957). – National Lead Co. Inc. WIN 85.

Smith, R. (1964). – Third Int. Conf. on the Peaceful Uses of Atomic Energy, Geneva. Paper 543.

Solntsev, V.M., and Tolmachev, Yu.M. (1961). – Radiokhimiya III (2): 187–194.

TABLE 1

DISSOLUTION RATES OF BERYLLIA POWDERS IN NITRIC ACID EFFECT OF SURFACE AREA

Sieve Size (BSS)	Mean Particle Size (μ)	B.E.T. (Kr^{85}) Surface Area (cm^2/g)	Initial Mass (g)	Dissolution Rates at 25 w/o Reacted	
				mg/g.min	10^{-3} mg/cm^2.min (B.E.T. Kr^{85} area)
−52 +72	253	1590	2.84	0.85	0.55
−72 +100	181.5	1520	3.04	0.98	0.65
−100 +150	128	1650	2.96	1.11	0.69
−150 +200	90	2290	2.80	1.27	0.60
−200 +300	64.5	2870	2.32	1.49	0.57
−300	−53*	9660	3.16	4.82	0.60

* Mean particle size = 25 μ (from a particle size analysis using the Sharples micromerograph – see Figure 15).

TABLE 2

EFFECT OF IRRADIATION ON THE DISSOLUTION RATE OF BERYLLIA

Mean Particle Size (μ)	Integrated Fission Neutron Dose (nvt)			
	Nil	3×10^{20}	6×10^{20}	
253	2.2	4.1	4.8	* Average Dissolution Rate (mg/g.min)
(−52 +72) BSS	1170	1450	1690	† Specific Surface Area (cm^2/g)
64.5 (−200 +300) BSS	1.6	1.9	1.4	* Average Dissolution Rate (mg/g.min)

* at 20 w/o dissolved. † by B.E.T. (Kr^{85}).

For dissolution conditions see Figures 16 and 17.

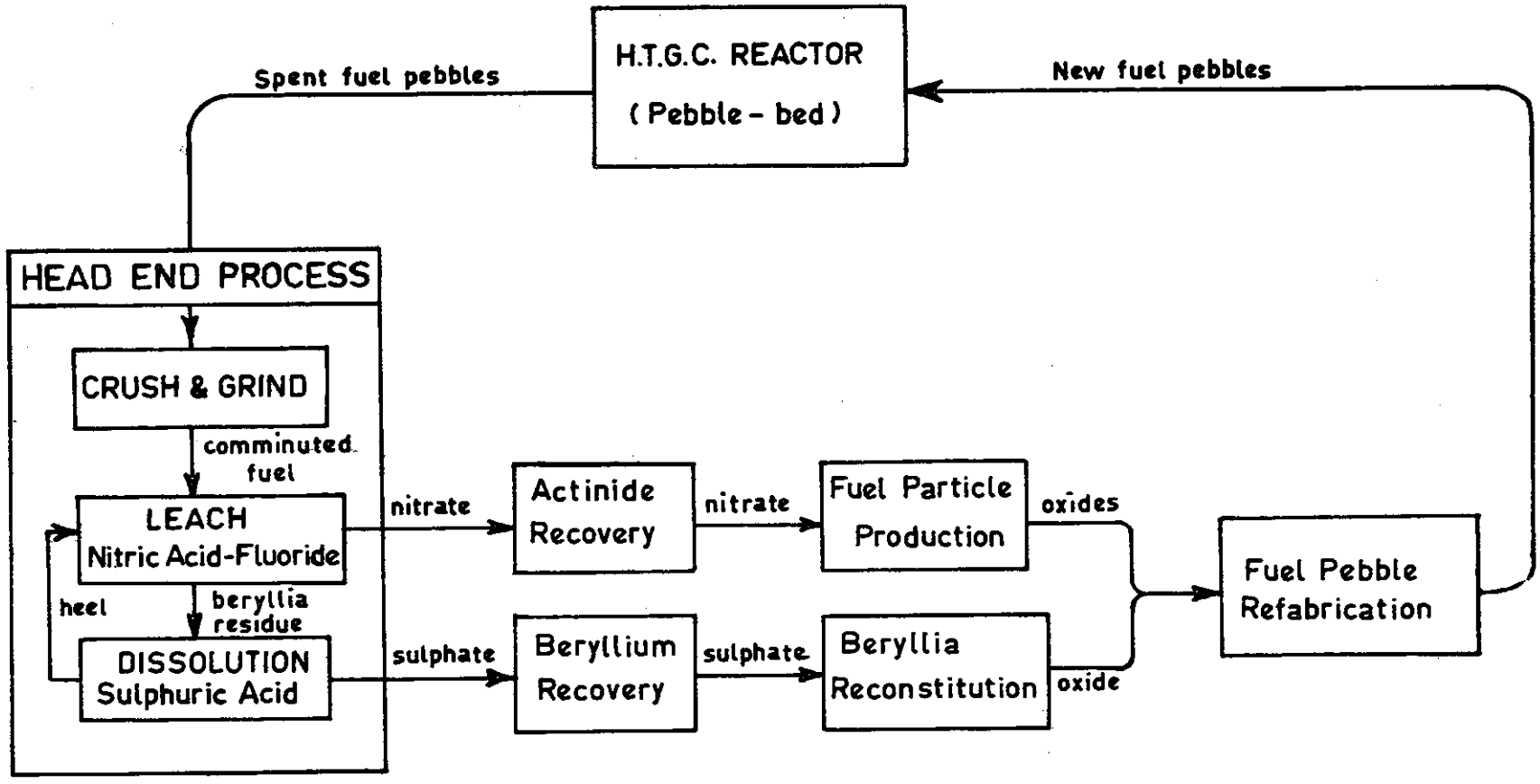


FIGURE 1. FUEL CYCLE FOR H.T.G.C. REACTOR USING A BERYLLIA-BASED DISPERSION TYPE FUEL

PB36

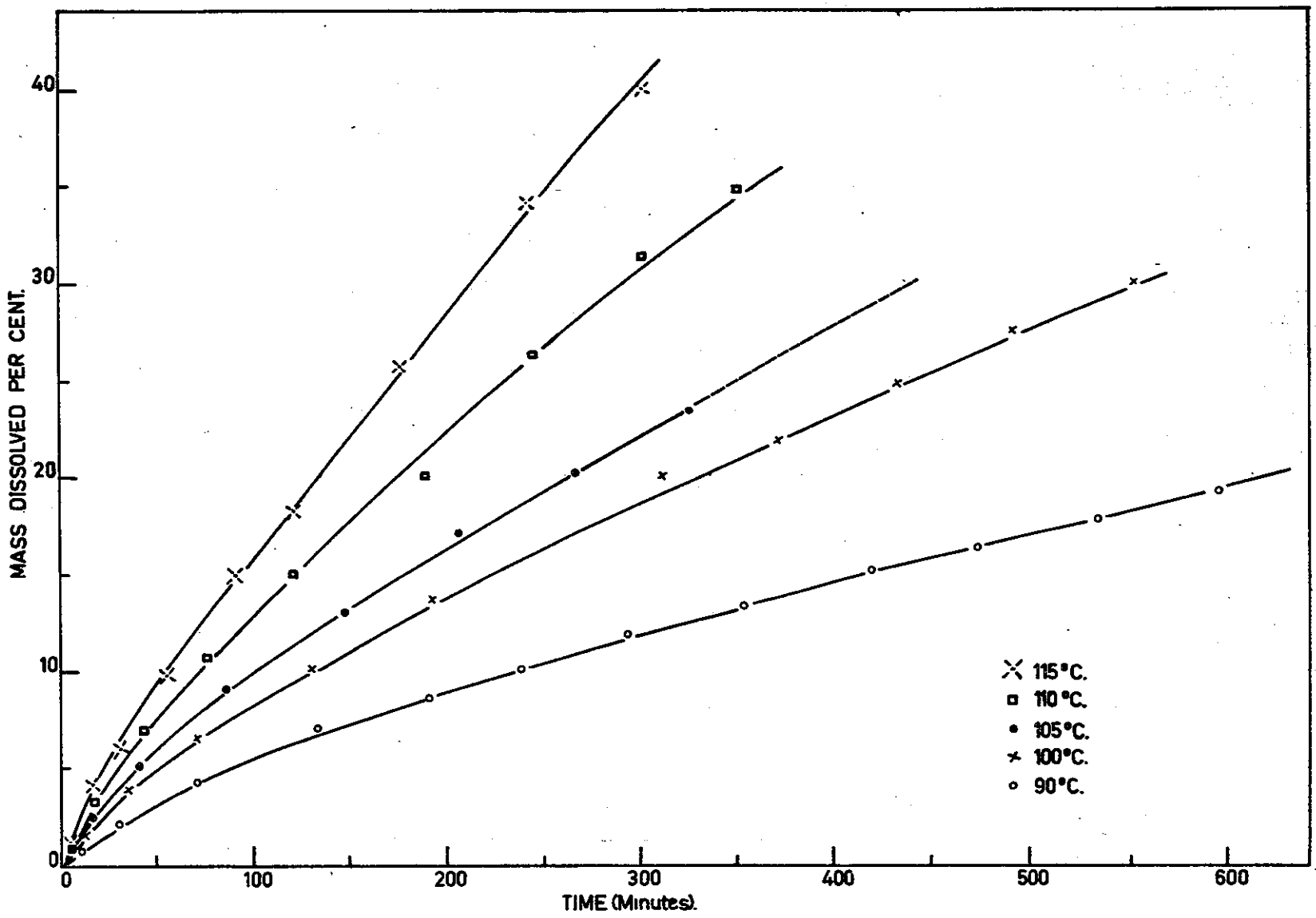


FIGURE 2. DISSOLUTION CURVES — EFFECT OF TEMPERATURE

Dissolution Conditions: Acid, 13M HNO_3; Sieve Size, -200+300 BSS; Agitation, 400 rev/min; Dissolver, glass

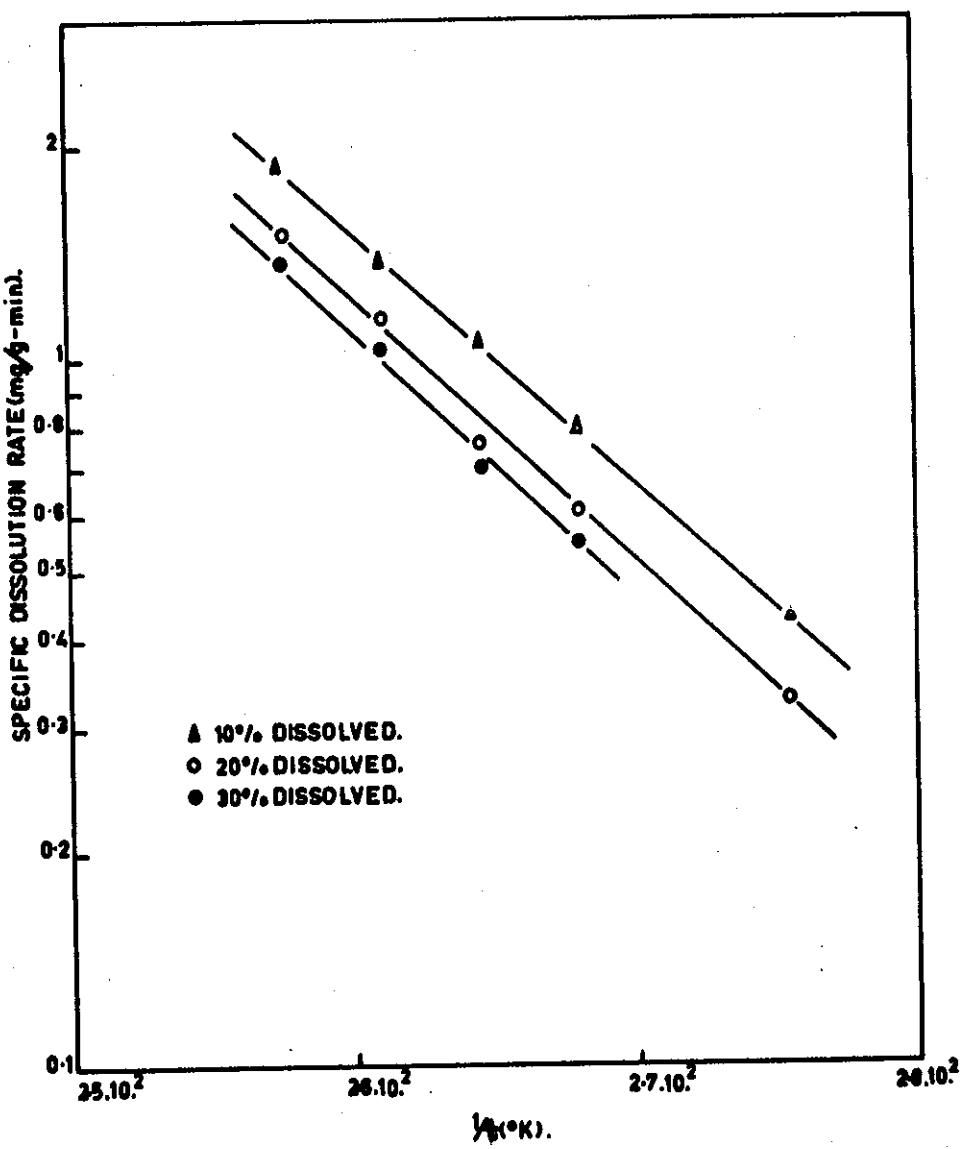


FIGURE 3. ARRHENIUS PLOTS FOR AVERAGE RATES TO 10, 20 AND 30% DISSOLUTION
Dissolution Conditions: Acid, 13M HNO_3; Sieve Size -200+300 BSS;
Agitation, 400 rev/min; Dissolver, glass

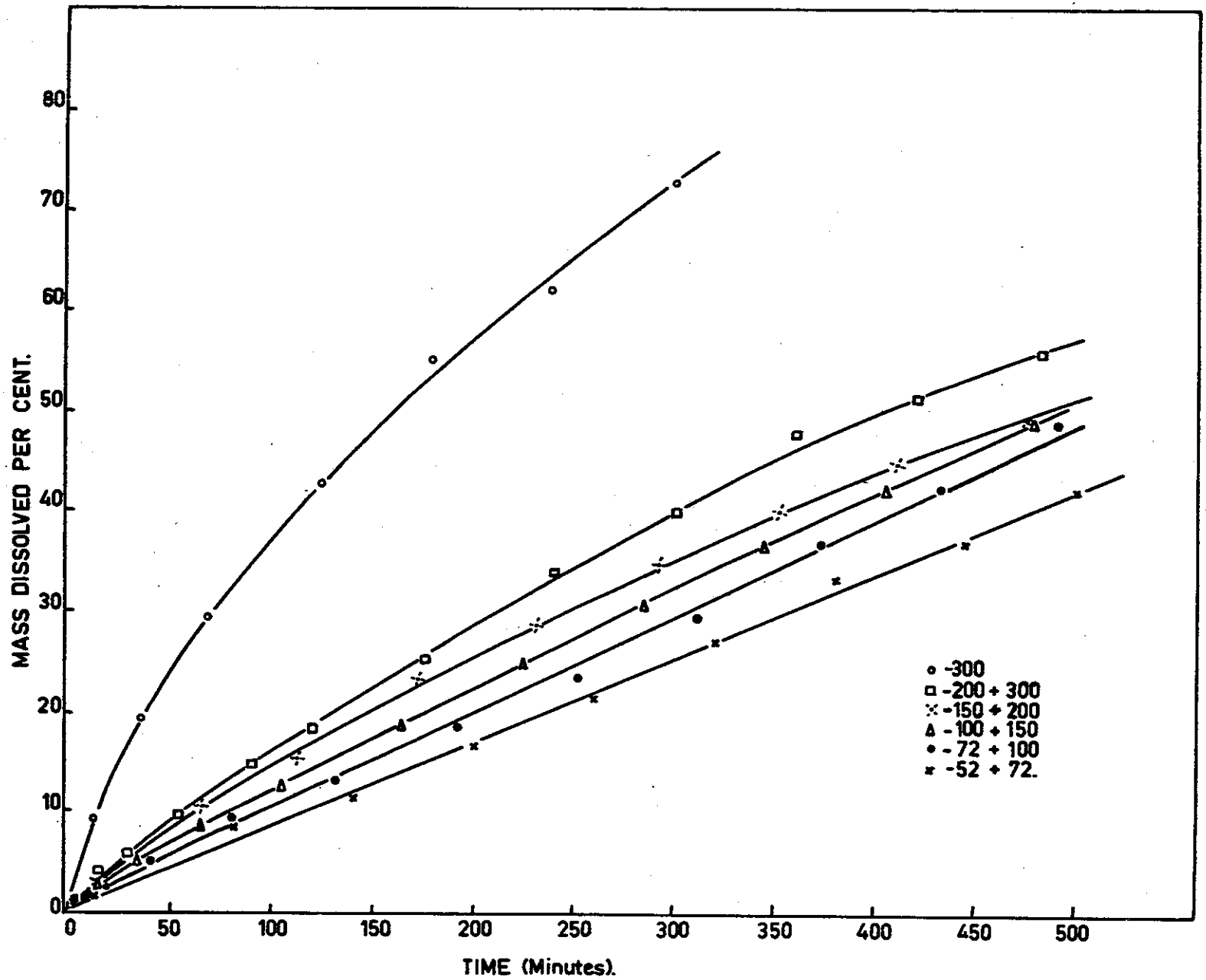


FIGURE 4. DISSOLUTION CURVES — EFFECT OF PARTICLE SIZE

Dissolution Conditions: Acid, 12M HNO_3; Temperature, 115°C; Agitation, 400 rev./min; Dissolver glass

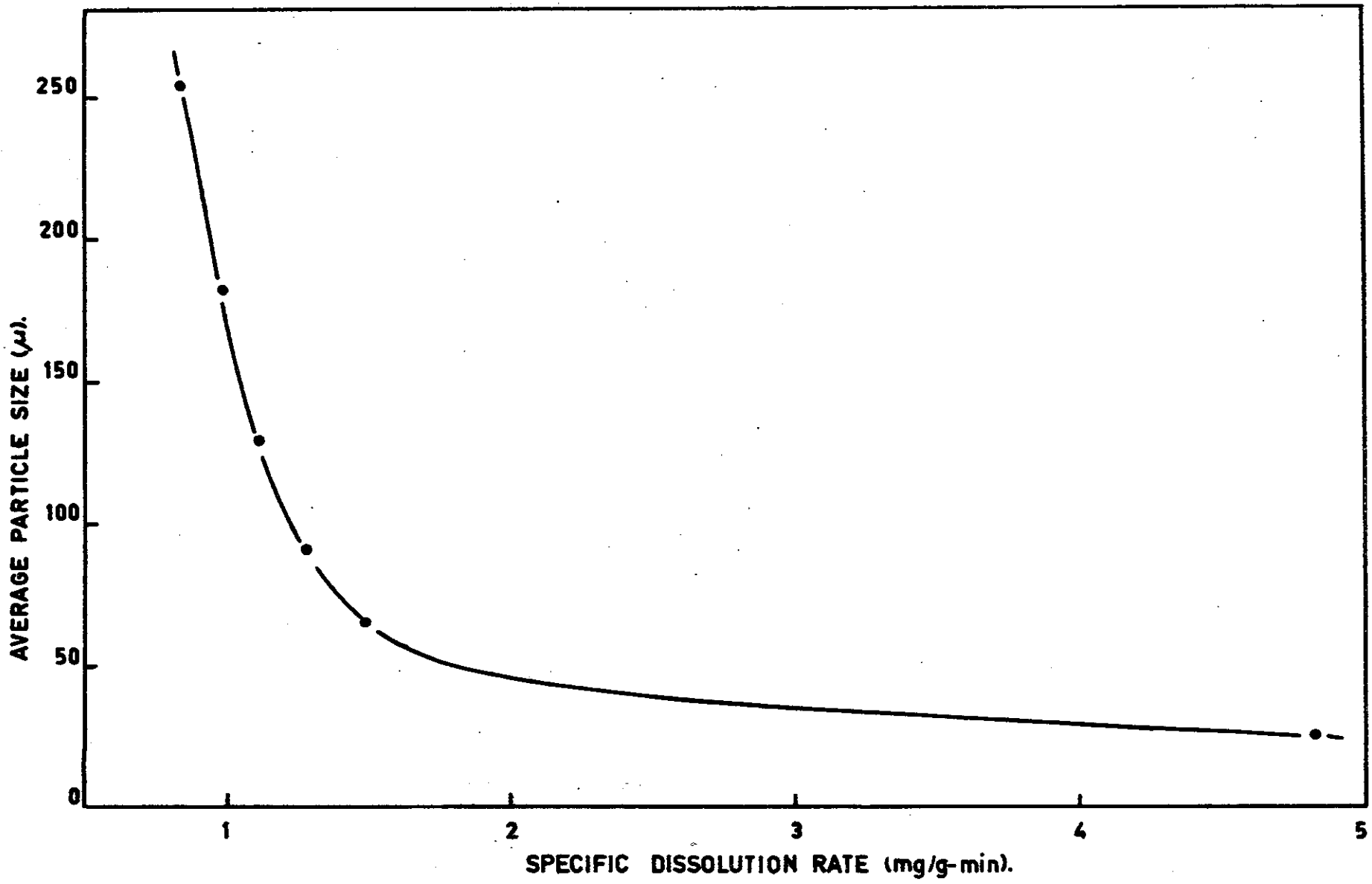


FIGURE 5. EFFECT OF PARTICLE SIZE ON DISSOLUTION RATE
Dissolution Conditions: Acid, 13M HNO_3; Temperature, 115° Agitation, 400 rev/min; Dissolver, glass

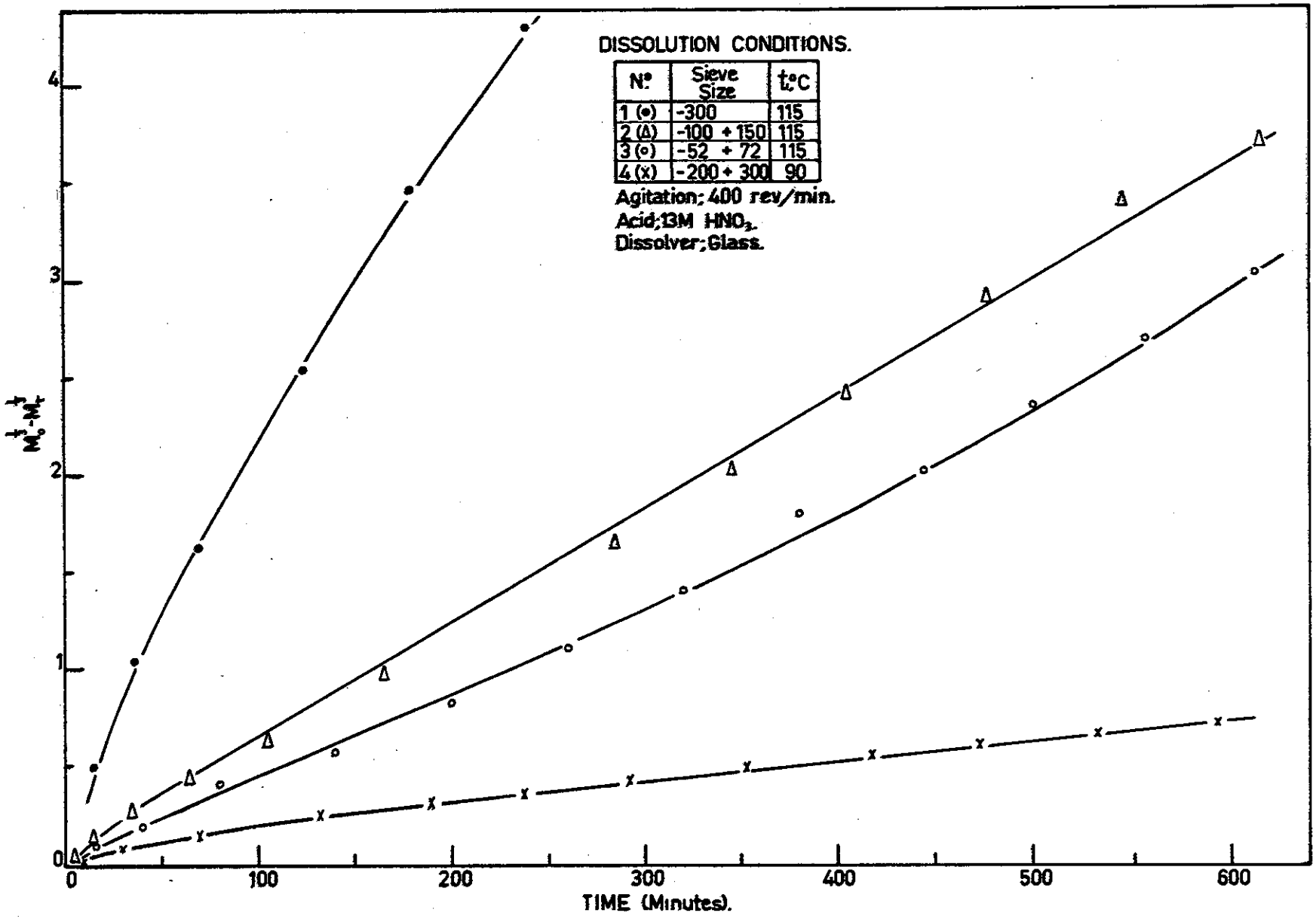


FIGURE 6. LINEAR NATURE OF RELATIONSHIP $M_o^{1/3} - M_T^{1/3} = k_1 T$

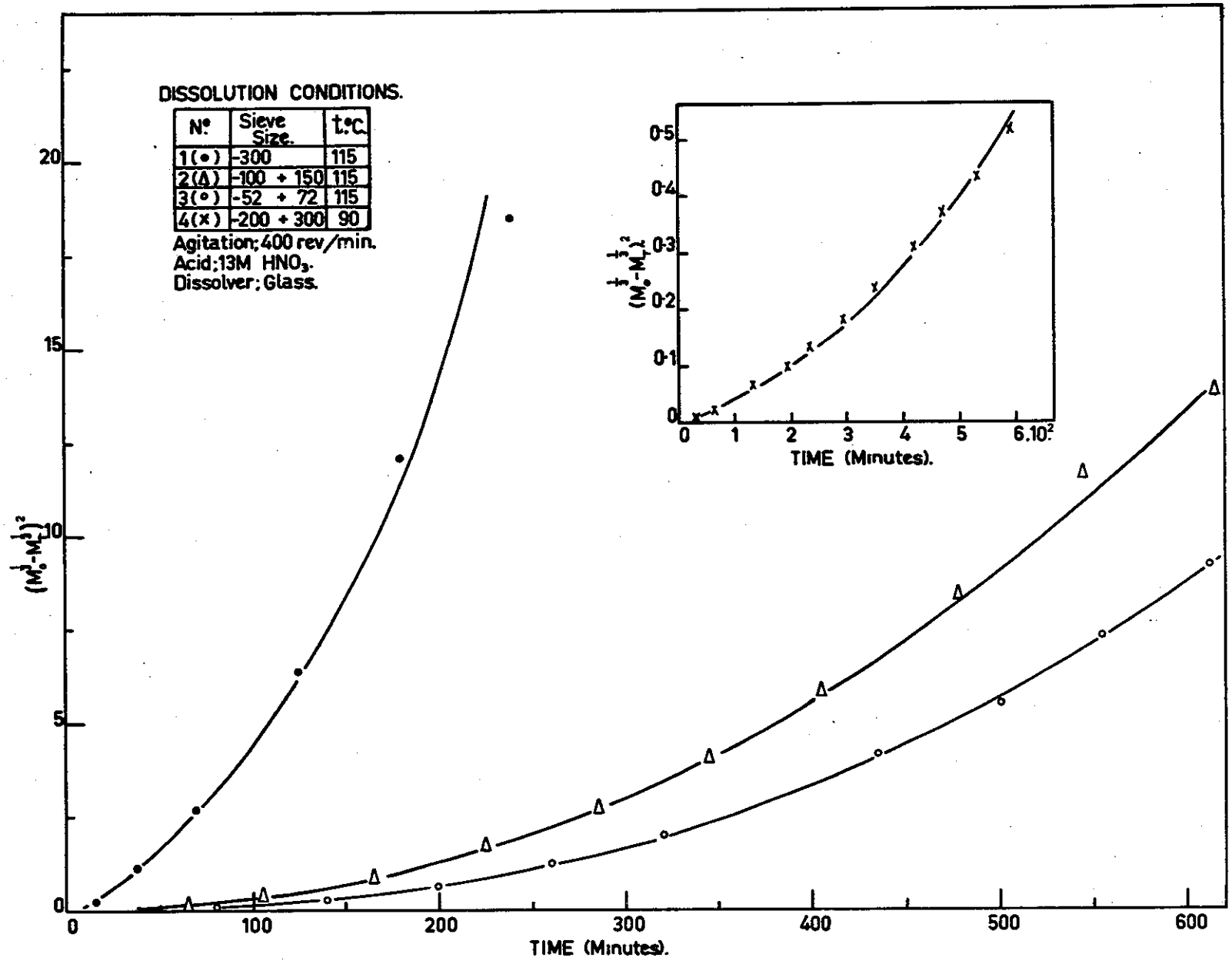


FIGURE 7. NON-LINEAR NATURE OF RELATIONSHIP $(M_o^{1/3} - M_t^{1/3})^2 = k_2 T$

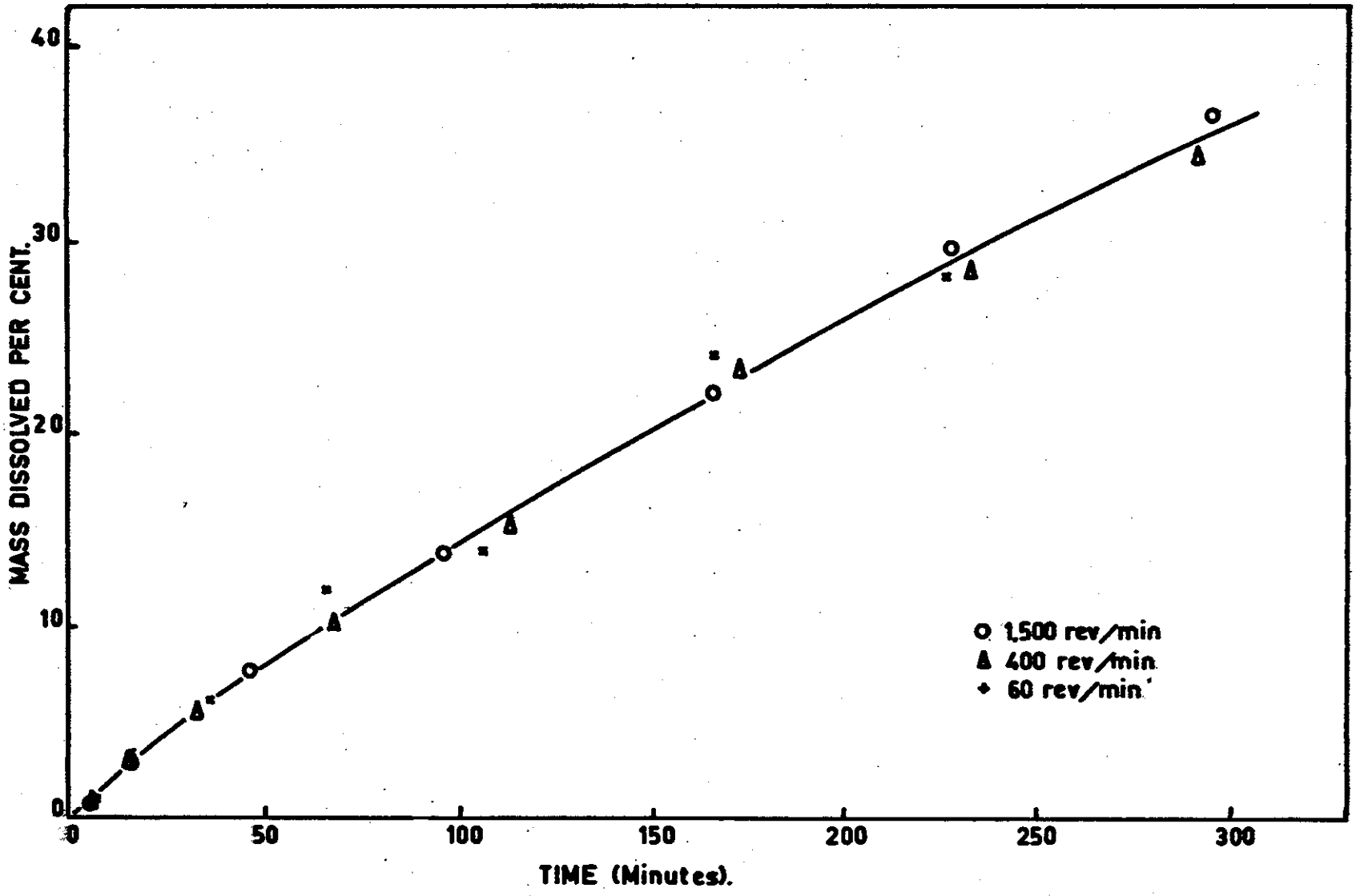


FIGURE 8. EFFECT OF AGITATION ON DISSOLUTION

Dissolution Conditions: Acid, 13M HNO_3; Sieve Size, -150+200 BSS; Temperature, 115°C; Dissolver, glass

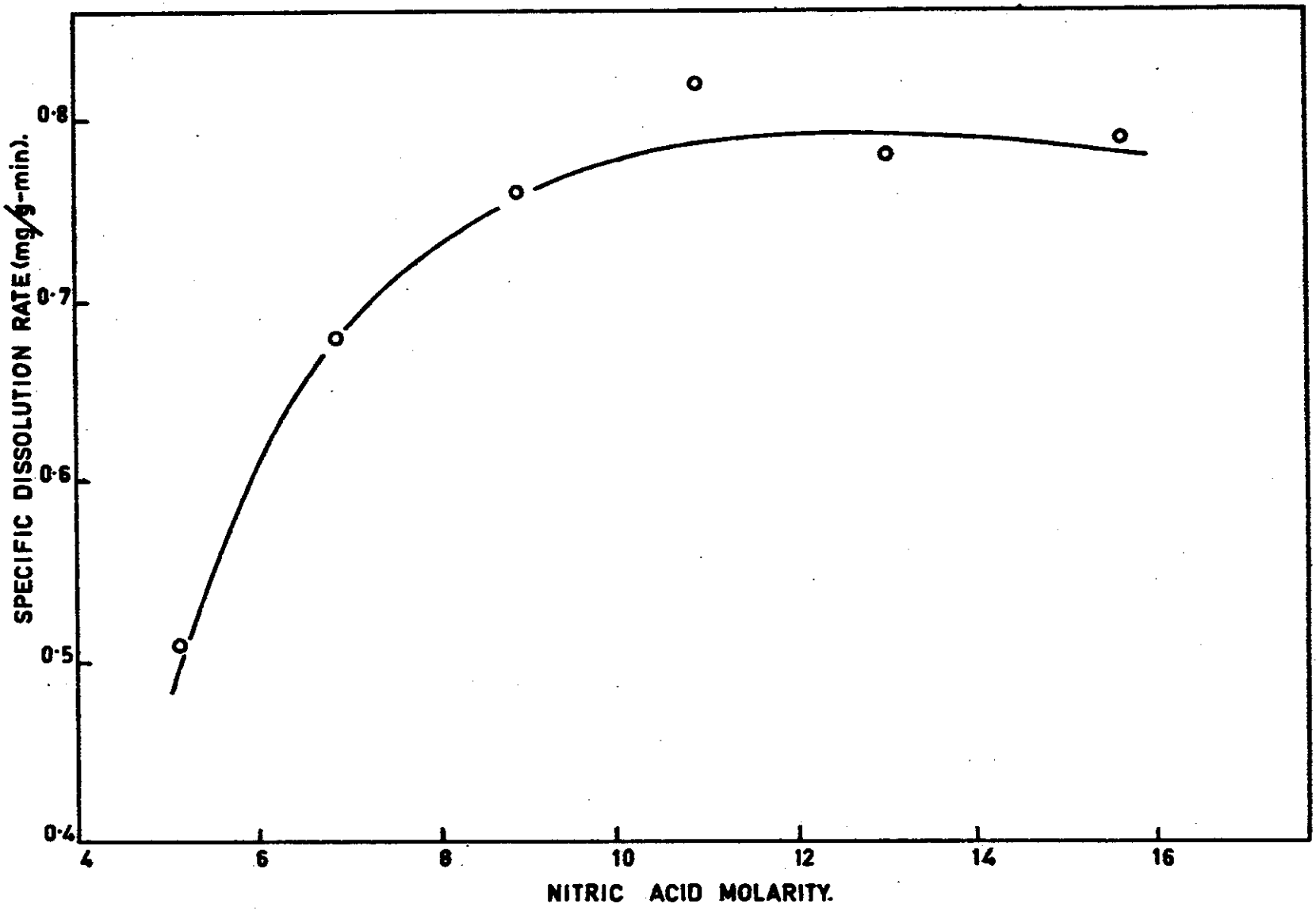


FIGURE 9. EFFECT OF NITRIC ACID CONCENTRATION ON DISSOLUTION RATE

Dissolution Conditions: Sieve Size, -200+300 BSS; Temperature, 105°C; Agitation, 400 rev/min; Dissolver, glass

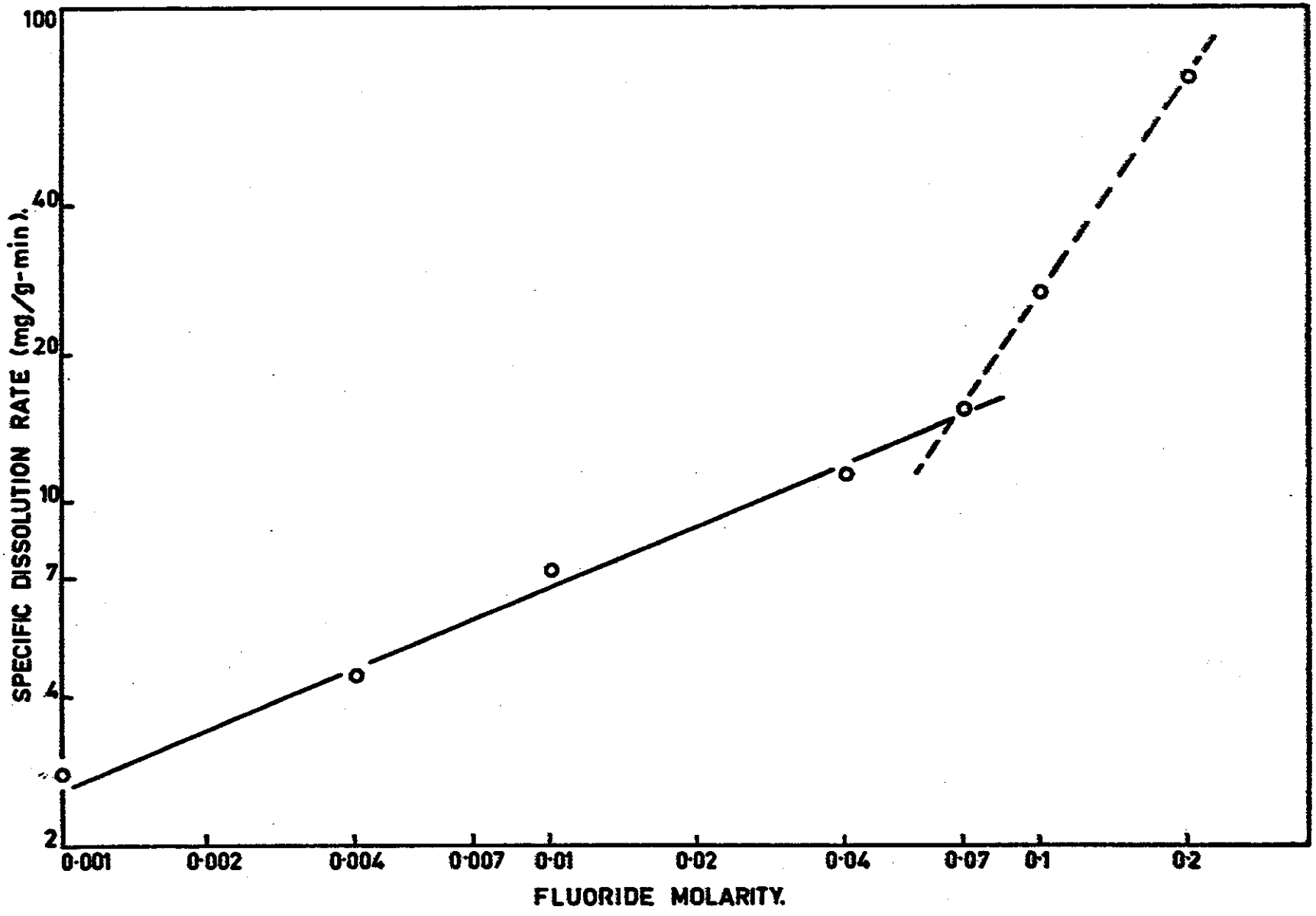


FIGURE 10. EFFECT OF FLUORIDE CONCENTRATION ON DISSOLUTION RATE
Dissolution Conditions: Acid, 13M HNO_3; Sieve Size, -200+300 BSS;
Temperature, 115°C; Agitation, 400 rev/min; Dissolver, Teflon

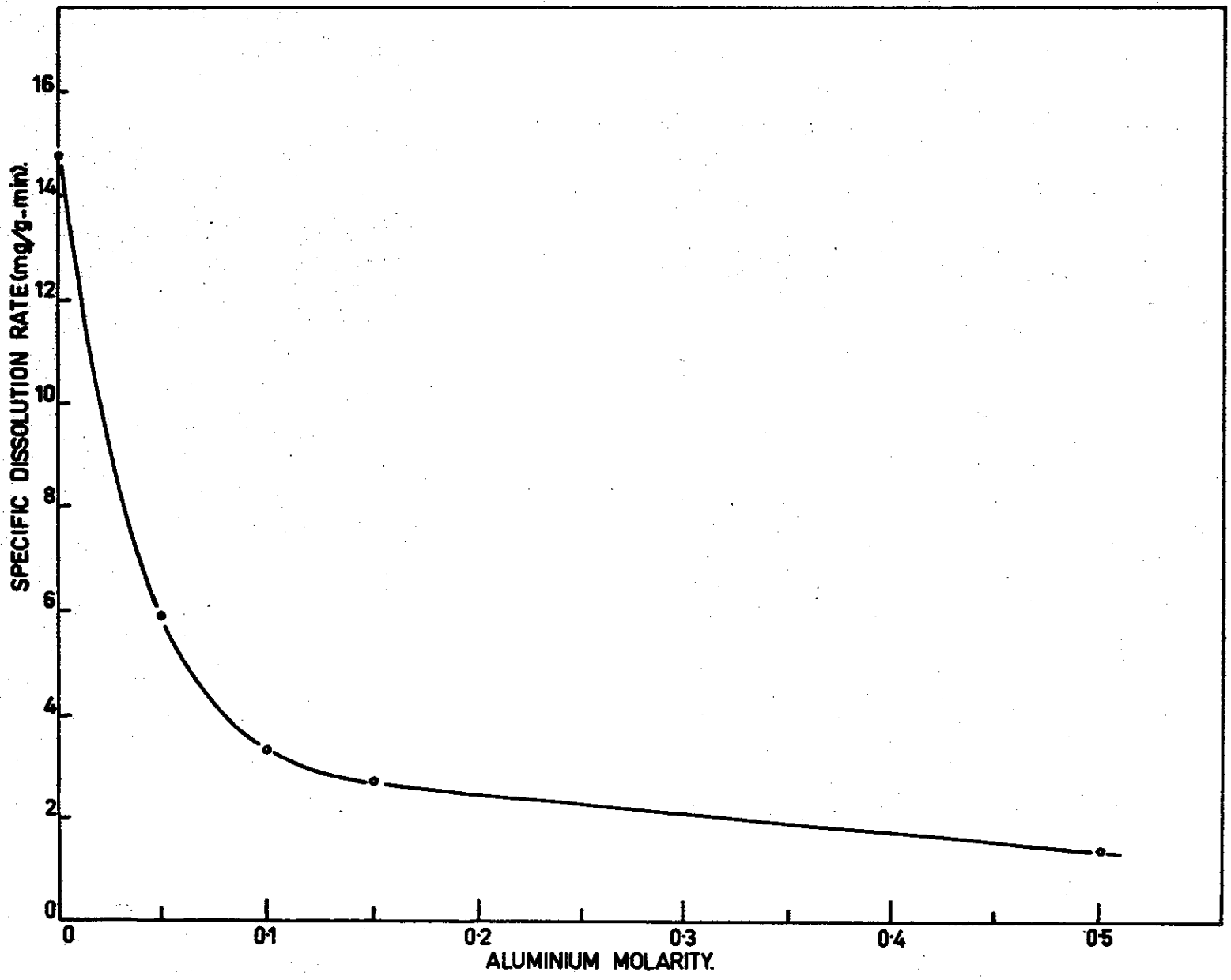
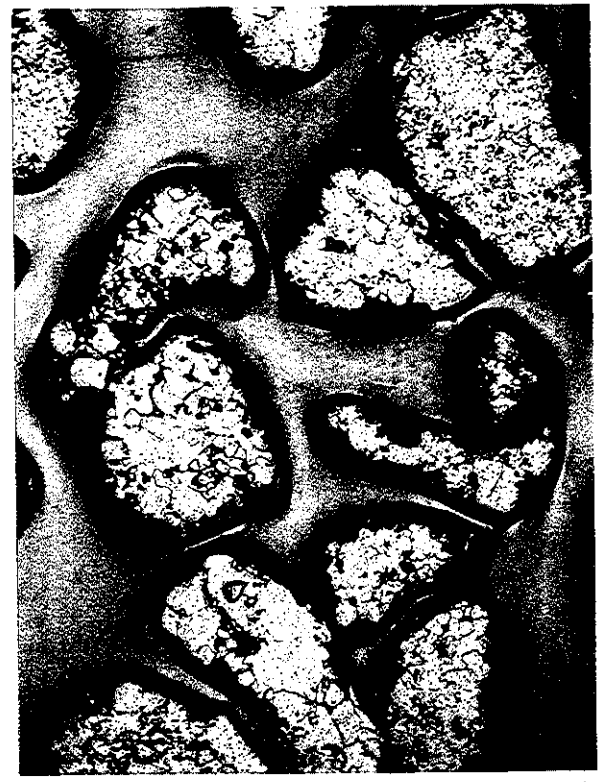


FIGURE 11. EFFECT OF ALUMINIUM IN PRESENCE OF FLUORIDE ON DISSOLUTION RATE
Dissolution Conditions: Acid, 13M HNO_3, 0.05M F; Sieve Size, -150+200 BSS; Temperature, 115°C; Agitation, 400 rev/min; Dissolver, Teflon

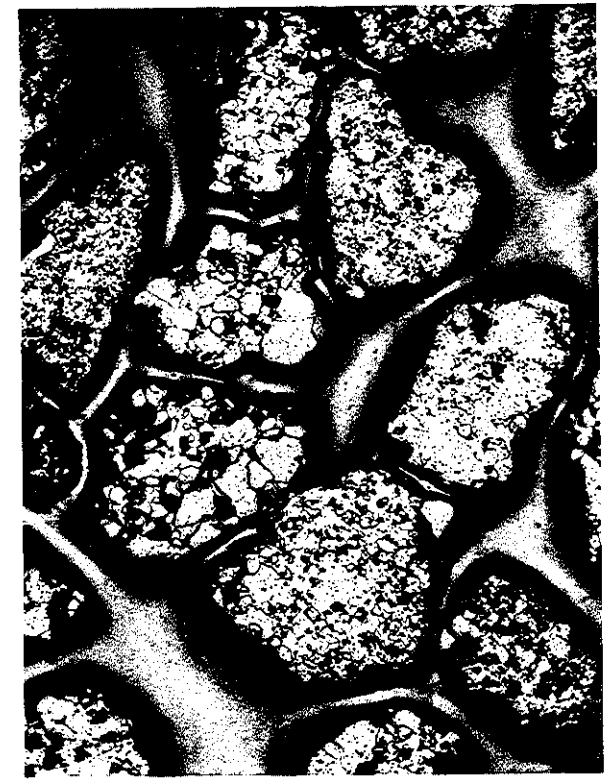
×100

CONTROL BeO
Sieve Size, -52+72 BSS



×100

IRRADIATED BeO
Sieve Size, -52+72 BSS
Irradiation Dose, 3×10^{20} nvt



×100

IRRADIATED BeO
Sieve Size, -52+72 BSS
Irradiation Dose, 6×10^{20} nvt

FIGURE 12. PHOTOMICROGRAPHS OF BERYLLIA POWDERS

×250

IRRADIATED BeO
Sieve Size, -52+72 BSS
Irradiation Dose 3×10^{20} nvt

×250

IRRADIATED BeO
Sieve Size, -52+72 BSS
Irradiation Dose 6×10^{20} nvt

FIGURE 13. PHOTOMICROGRAPHS OF BERYLLIA POWDERS

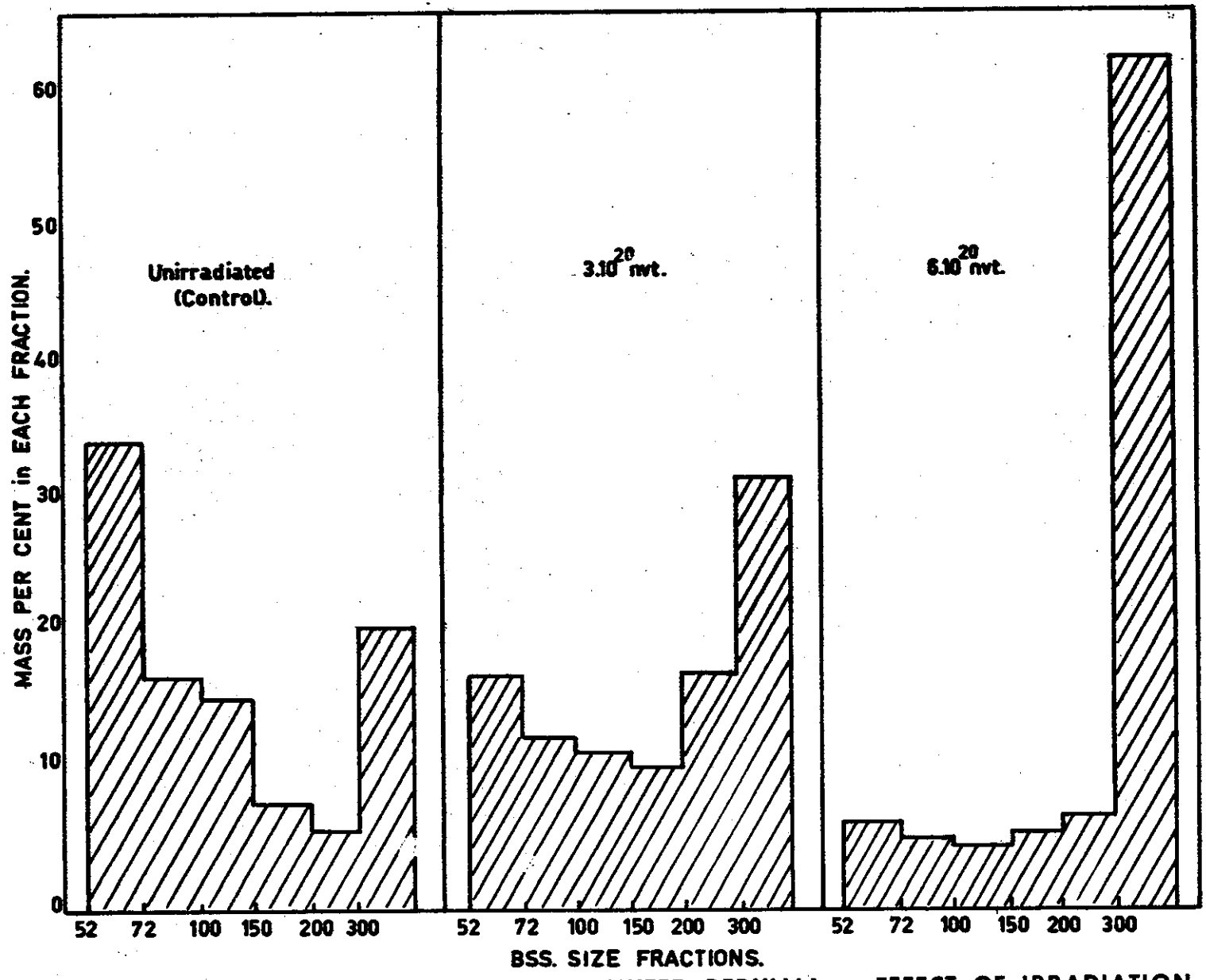


FIGURE 14. SIEVE ANALYSIS OF COMMINUTED BERYLLIA — EFFECT OF IRRADIATION

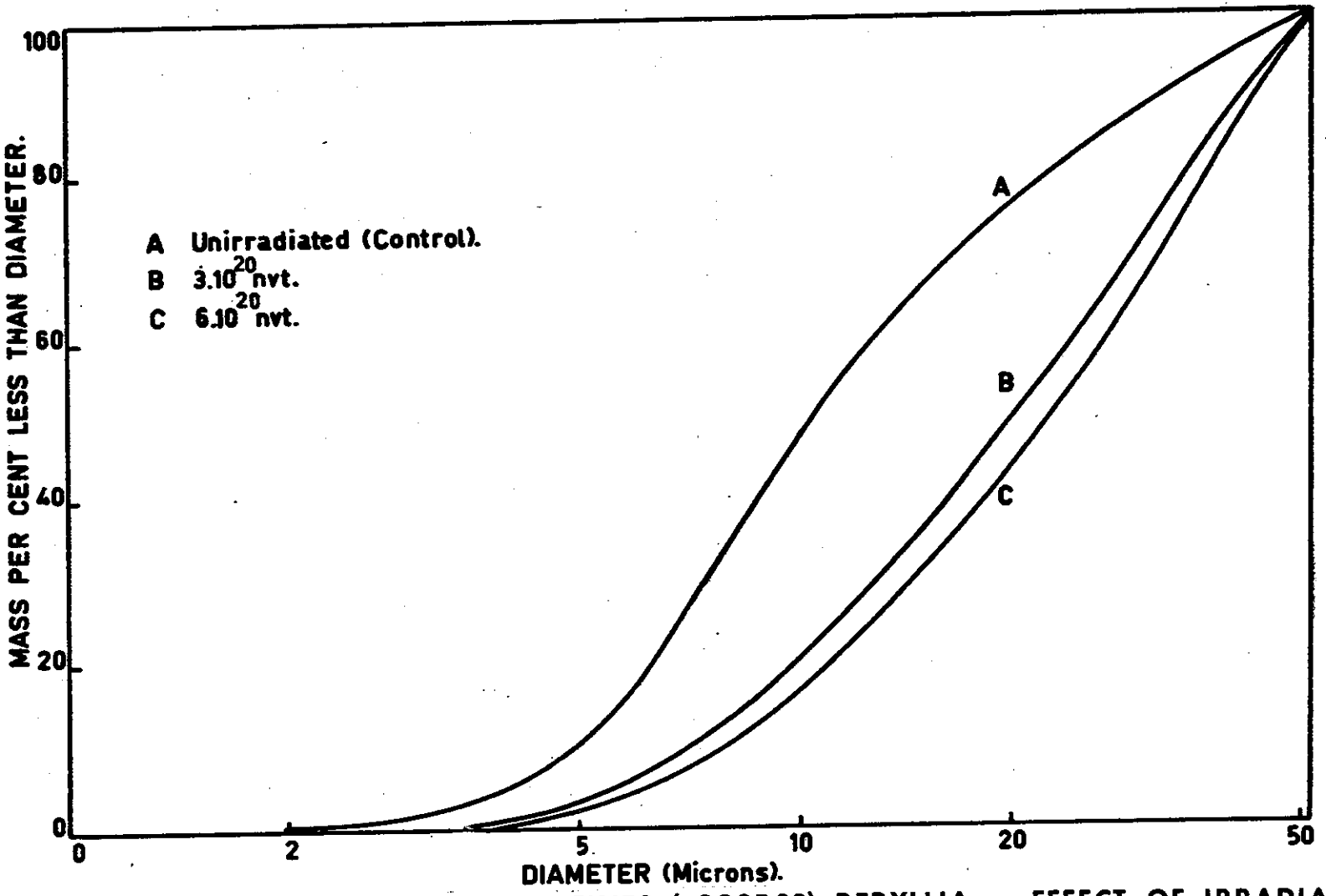


FIGURE 15. SIZE DISTRIBUTION OF FINES (-300BSS) BERYLLIA — EFFECT OF IRRADIATION

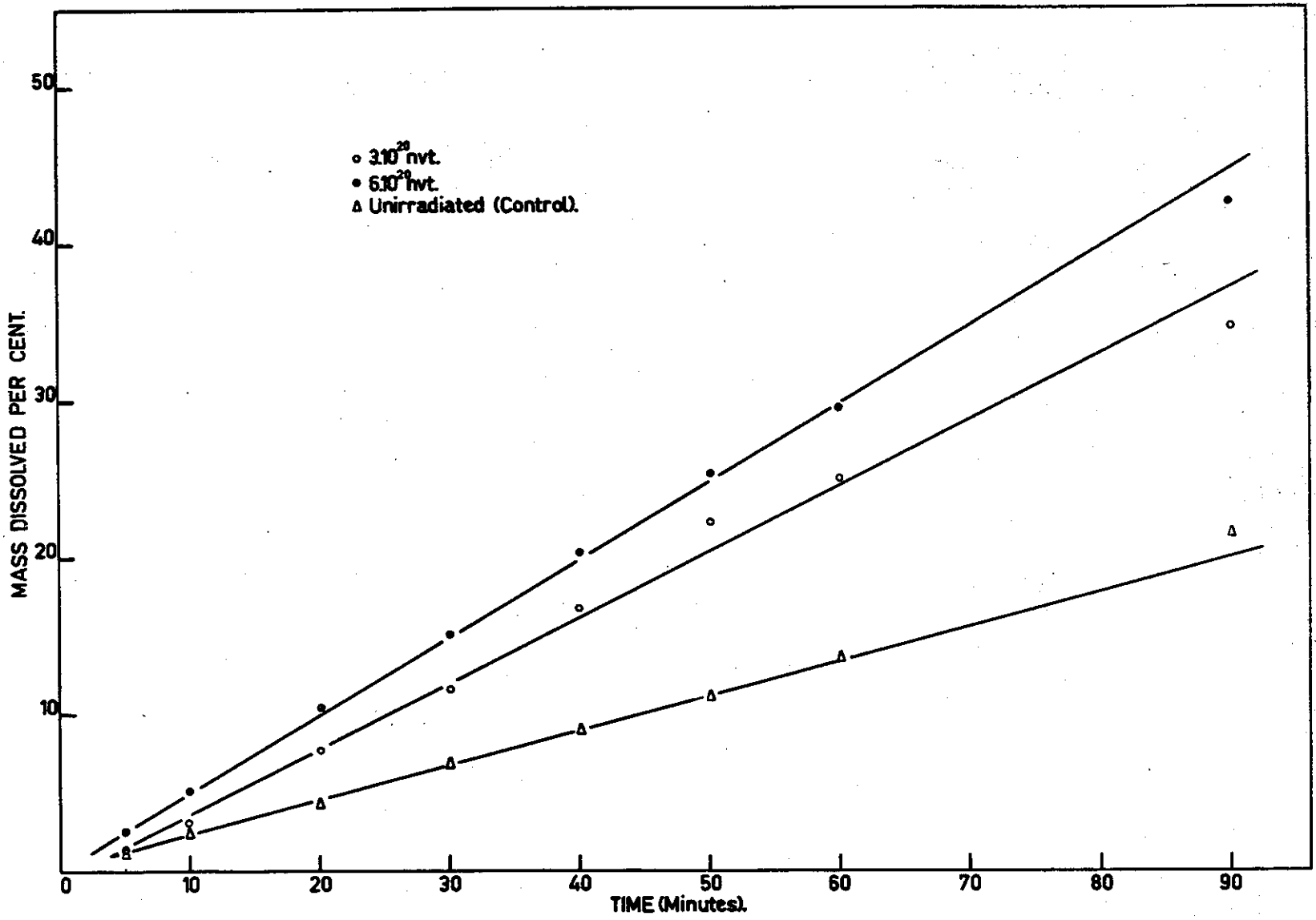


FIGURE 16. DISSOLUTION CURVES FOR BERYLLIA IN THOREX DISSOLVENT — EFFECT OF IRRADIATION
Dissolution Conditions: Acid, 13M HNO_3; 0.05M F, 0.1M Al^{3+}; Sieve Size, -52+72 BSS; Temperature, 115°C; Agitation, 400 rev/min; Dissolver, stainless steel

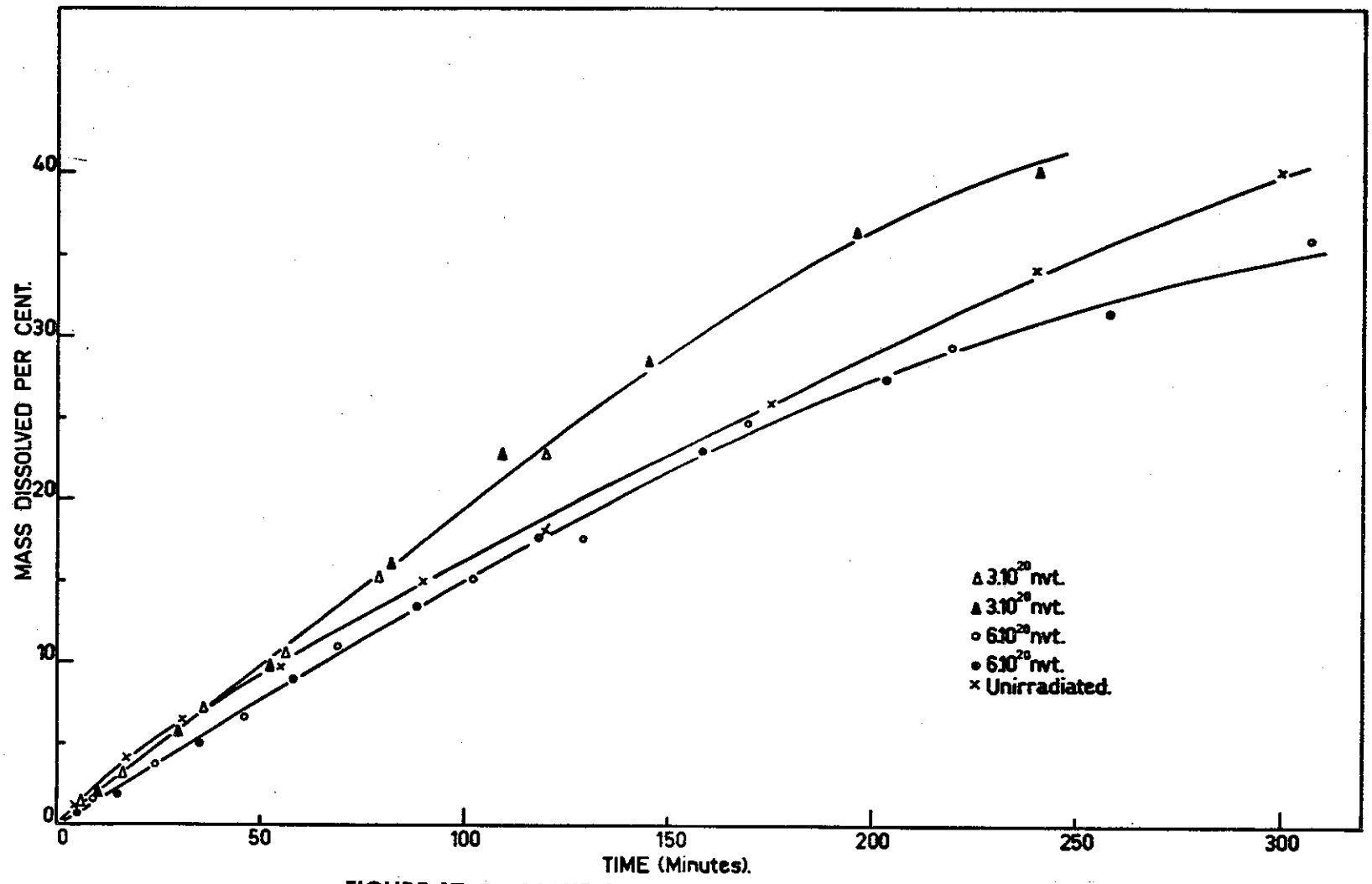


FIGURE 17. DISSOLUTION CURVES — EFFECT OF IRRADIATION

Dissolution Conditions: Acid, 13M HNO_3; Sieve Size, -200+300 BSS; Temperature, 115°C; Agitation, 400 rev/min; Dissolver, glass