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KINETIC INTERPRETATION OF
BATCH FLOTATION OF A
MOLYBDENITE ORE

By

Deepak Malhotra

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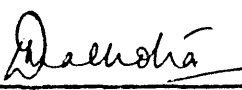
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A Thesis respectfully submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science in Metallurgical Engineering.

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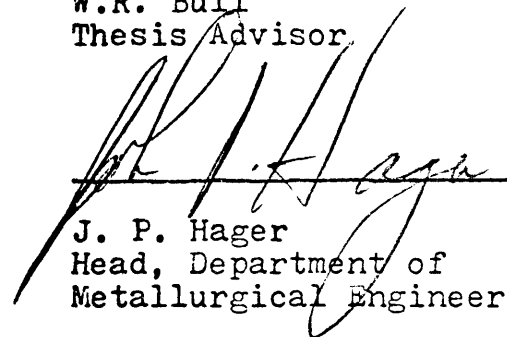
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ABSTRACT

Batch flotation tests were carried out in a laboratory flotation cell using high-grade molybdenite ore as the feed material. As a result of the analysis of the kinetics of flotation of molybdenite ore, two different mathematical models of the flotation process are proposed. One is the use of a second order equation to describe the system. The results can be interpreted in terms of a single parameter, the rate coefficient. The other is the "2-spike" model based on two rate coefficients - one for a fast-floating proportion, the other for a slow-floating proportion of the material.

Due to poor reproducibility of the results, no conclusions could be drawn regarding the effect of the operating variables on the flotation process.

TABLE OF CONTENTS

	Page
ABSTRACT	iii
TABLE OF CONTENTS	iv
LIST OF FIGURES	vii
LIST OF TABLES	x
LIST OF APPENDICES	xi
ACKNOWLEDGEMENTS	xii
INTRODUCTION	1
SURVEY OF THE LITERATURE	3
Basic Kinetic Equations	3
The First Order Equation	6
Second Order Equation	7
Non-Integral Order	9
Holland-Batt's General Approach	10
Factors Affecting the Rate of Flotation	11
Bubble Mineral Aggregation Mechanism.	11
Cell Operation	12

	Page
Flotation Reagents	13
Particle Size	13
Liberation	14
Flotation Rate Constant K	16
Concept of R_{∞}	22
EXPERIMENTAL APPARATOS AND PROCEDURE	23
Sample Preparation	23
Crushing	23
Sampling	23
Grinding	24
Batch Flotation Tests	25
Equipment	25
Automatic Froth Removal System.	25
Pulp Level Control	29
Control of Air Input to Flotation Cell	31
Procedure	31
Analysis of the Samples	33
EXPERIMENTAL RESULTS	33
DISCUSSION OF RESULTS	46
Feed Assay	46
Determination of Value of R_{∞}	49
First Order Rate Equation	51
Second Order Rate Equation	54
Non-integral Order	59

	Page
Models Using Distributed Rate Constants	60
Effect of Air Flow Rate and Grind on the Rate of Flotation	64
CONCLUSIONS	68
BIBLIOGRAPHY	85
APPENDICES	89

LIST OF FIGURES

Figure	Page
1. Typical variation of $W'(K)$ with time.	19
2. General view of equipment	26
3. Photograph of flotation cell showing the automatic scrapers and air control system	27
4. Constant head chamber and the automatic froth removal system	28
5. System for controlling air input to flotation cell.	32
6. Recovery-time curves for different grind times and 5.6 l/min air flow rate	39
7. Recovery-time curves for different air flow rates and for grind time of 7 minutes	40
8. Recovery-time curves for tests 4 and 8 conducted under identical conditions (13 min grind and 5.6 l/min air flow rate)	41
9. Recovery-time curves for tests 2, 6 and 7 conducted under identical conditions (7 min grind and 5.6 l/min air flow rate)	42
10. Recovery-time curves for tests 9 and 10 conducted under identical conditions (7 min grind and 7.64 l/min air flow rate)	43
11. Recovery-time curves for tests 12 and 13 conducted under identical conditions (7 min grind and 4.62 l/min air flow rate)	44

Figure	Page
12. Recovery-time curves for tests 2 and 14 for 7 minute grind and 5.61 l/min air flow rate. . .	45
13. Plot of $\ln \frac{C-C_{\infty}}{C_0-C_{\infty}}$ against time for tests 4 and 8 conducted under identical conditions (13 min grind and 5.61 l/min air flow rate). . .	70
14. Plot of $\ln \frac{C-C_{\infty}}{C_0-C_{\infty}}$ against time for tests 2, 6 and 7 conducted under identical conditions (7 min grind and 5.61 l/min air flow rate) . . .	71
15. Plot of $\ln \frac{C-C_{\infty}}{C_0-C_{\infty}}$ against time for tests 1 through 5 ⁰ at 5.61 l/min of air flow rate . . .	72
16. Kelsall's graphical method for obtaining slow and fast floating fractions for any mineral	73
17. Experimental and theoretical curves using modified Kelsall's graphical procedure for obtaining "2-spike" model for test 1	74
18. Experimental and theoretical curves using modified Kelsall's graphical procedure for obtaining "2-spike" model for test 4	75
19. Theoretical (as predicted by "2-spike" model) and experimental recovery-time curves for test 4.	76
20. $\frac{t}{R}$ vrs t curves for tests 1 and 4	77
21. $\frac{1}{t}$ vrs $\frac{1}{R}$ plots for tests 1 to 5 using Hukki's modified second order equation	78
22. $\frac{1}{t}$ vrs $\frac{1}{R}$ plots for tests 12, 13 and 14 using Hukki's ⁽⁸⁾ modified second order equation	79
23. Curve-fitting technique of Harris and Chakravarti ⁽²²⁾	80
24. Graphical technique for obtaining parameters r, m and n for Harris' 3-parameter model	81

Figure	Page
25. Species Distribution: plot of $\Psi(K,0)$ vrs K for tests 6, 9, 11 and 12	82
26. Species distribution: plot of $\Psi(K,0)$ vrs K for different values of m for test 12 ($r=0.17$, $n=1.12$, $m=5.53$)	83
27. Hatched region showing the 95% confidence limits for tests 2, 6 and 7 conducted under plentical conditions (7 min grind and 5.61 l/min air flow rate)	84

LIST OF TABLES

Table	Page
1. Average size distribution of the flotation feed. .	35
2. Size distributions for different grind times . . .	36
3. Summary of test conditions	38
4. Calculated feed assays	48
5. Calculated values of R_{∞}	50
6(a) Experimental and predicted values of recovery for test 1 using "2-spike" model	55
6(b) Experimental and predicted values of recovery for test 4 using "2-spike" model	56
7. R_{∞} and k_2' values obtained using Arbiter's second order equation	58
8. The values of r, n and m of the 3-parameter or "2-spike" model	63
9. The values of r, m and n of the 3-parameter or "2-spike" model for different grinds at air rate of 5.61 l/min	65
10. The values of r, m and n of the 3-parameter or "2-spike" model for different air flow rates (and for 7 minute grind)	66

LIST OF APPENDICES

	Page
A. Derivation of the Equation Used by Hukki ⁽⁸⁾	89
B. Use of Reagents in the Flotation of Molybdenite. .	90
C. Determination of Molybdenum in Ores and Mill Products	92
D. Cumulative Recovery and Grade for Tests 1 through 14	96
E. Wet and Dry Screening Analyses of Tailings from tests 1 to 8	111
F. Values of $\frac{C-C_{\infty}}{C_0-C_{\infty}}$ for tests 1 through 14	112

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INTRODUCTION

Since the introduction of the flotation process to the concentration of minerals, great advances have been made in its application and in the efficiency of the separations performed. This has been caused mainly by progress in the chemistry of flotation. A large number of chemical compounds have been developed which offer considerable selectivity. To a lesser extent, better design of flotation machines has also enhanced the efficiency of operation.

The advances in the physical aspects of the flotation process, however, have largely resulted from trial-and-error, and it is only in the last decade or so that attention has been directed to "process analysis" - the development of mathematical models that describe the flotation process in terms of parameters, the magnitudes of which may be determined.

If a realistic model could be established, it could be used for computer simulation of the process. This in turn would form the basis for process optimization and on-line computer control.

This approach has been successfully used in the chemical and petroleum engineering during the past decade⁽¹⁾, but is only in its infancy in mineral processing, particularly in flotation. This is probably due to the large number of variables involved in the flotation process and to the difficulty of controlling some of these variables, e.g. feed grade, frother concentration, and even feed rate. The complexity of the flotation system makes it impossible at this moment to develop a mathematical model.

This investigation is concerned with the application of kinetic models to the data from the batch flotation of a molybdenite ore and their practical usefulness to plant operators as an aid to optimization of flotation circuits.

The problem was approached experimentally by using a batch flotation system by means of which it was possible to control four important variables in the flotation test - impeller speed, air-flow rate, pulp level and rate of froth removal. The experimental observations were then analyzed with the purpose of determining the applicability of kinetic equations to the molybdenite flotation system and the application of a model which characterises this flotation system.

SURVEY OF THE LITERATURE

As a result of the increased interest in the analysis of the flotation process during the last decade or so, different methods of kinetic interpretation have been introduced in the literature. The broad goal of these studies has been a better quantitative understanding of the process which should lead to improvements both in metallurgical results and flotation cell and circuit design. It should also lead to reliable methods for the scaling-up of laboratory and pilot plant results to full size operation without the need for expensive and time-consuming mill tests on commercial machines. Finally, this type of quantitative information will assist the automation of flotation cells and circuits, as well as control by computers, which will yield higher levels of grade and recovery through reduction of the variability of present-day results.

Basic Kinetic Equations

Grunder and Kadur⁽²⁾ were the first to draw attention to the analogy between the flotation process and a chemical

reaction. Chemical kinetics is concerned with interactions between atoms, molecules or ions, and flotation kinetics deals with interactions between air bubbles and particles.

Before the development of these kinetic equations is undertaken, it is important to note that the analogy between flotation kinetics and chemical kinetics was based on the concept of distinct species (i.e. pure minerals). The applicability of these equations to other cases, particularly that of a ground ore containing middlings, will be dealt with later.

From the view point of the chemical kinetics analogy a general equation representing the kinetics of the flotation process has been postulated⁽³⁹⁾:

$$\frac{dC_j(t)}{dt} = -k_j \prod_{i=1}^m [C_i(t)]^{n_{ji}} \quad (1)$$

where $C_j(t)$ is the concentration of the j^{th} constituent of floatable material which is composed of r different species; $C_i(t)$ are the concentration variables of m different kinetic species of the system; $m > r$ since some components are not considered floatable; n_{ji} specifies the order of the reaction with respect to the j^{th} constituent and k_j is the specific flotation rate, also called the flotation rate constant or flotation rate coefficient.

As it stands, equation (1) is too complex for general

use, but, for any given test, if we assume that reagent concentrations etc. remain constant, the equation simplifies to:

$$\frac{dC}{dt} = - k \cdot C^{n_s} \cdot C_a^{n_a} \quad (2)$$

where C is the concentration in gms/litre of pulp of the species under consideration; C_a is the concentration of air in the cell pulp, expressed as volume air/unit volume of pulp; and n_s and n_a are the order of the reaction with respect to solids and air bubbles respectively. k is a complex function of all the factors affecting the process except solids concentration and bubble concentration. In a well-controlled laboratory test the rate of aeration can also be kept constant so that Eq.(2) further simplifies to:

$$\frac{dC}{dt} = - K \cdot C^n \quad (3)$$

where C is the concentration in grams/litre of pulp of the species under consideration; t is the time; n is the order of the reaction, and K is the flotation rate constant. K is a complex function of the chemical conditions of the pulp and the physical characteristics of cell operation and design⁽⁴⁾.

The First Order Equation (2, 9, 10, 11, 17, 33)

With $n=1$ in Eq.(3), integration leads to:

$$C = C_0 e^{-Kt} \quad (4)$$

where C_0 = the concentration in grams/litre of pulp of the species in the cell at zero time.

C = the concentration in grams/litre of pulp of the species in the cell at time t . Eq.(4) may be rewritten:

$$\ln \frac{C}{C_0} = -K.t \quad (5)$$

and if experimental values of $\ln \frac{C}{C_0}$ are plotted against t , the graph should be a straight line of slope $-K$ if $n=1$.

Even if flotation is continued for a prolonged time, some apparently floatable material may still remain in the pulp. This proportion should be excluded from the terms C and C_0 , and a modified equation appears to have more validity -

$$\ln \frac{C-C_\infty}{C_0-C_\infty} = -K.t \quad (6)$$

where C_0 = the concentration of the species under consideration in the cell at zero time in grams/litre of pulp, C = the concentration of that species at time t in grams/litre of

pulp, and C_{∞} = the terminal concentration (concentration remaining unfloated in the cell after prolonged flotation) of that species in grams/litre of pulp. In terms of recoveries Eq.(6) may be written:

$$R = R_{\infty} (1 - e^{-Kt}) \quad (7)$$

where $R = \frac{C_o - C}{C_o}$
 = fractional recovery up to time t of the species under consideration, and

$$R_{\infty} = \frac{C_o - C_{\infty}}{C_o}$$

= fraction of that species which is floatable.

For any given conditions, K is a quantitative measure of a species being recovered in the concentrate. K can be used to compare different chemical conditions in the same cell, or different flotation cells treating the same pulp⁽⁵⁾. Each particle type in an ore (e.g. chalcopyrite and pyrite) will have its own value of K in any test, and the ratios of these values are a quantitative measure of the selectivity of the operation.

The Second Order Equation (6, 7, 8)

With $n=2$, Eq. (3) gives by integration with the limits

$t=0, C=C_0$ and $t=t, C=C$

$$C = \frac{C_0}{1 + C_0 k_2 t} \quad (8)$$

However, taking into consideration $R_\infty < 1$ (or $C_\infty > 0$) a more complicated equation than Eq.(8) is the result

$$C = \frac{C_0 + C_\infty (C_0 - C) k_2 t}{1 + (C_0 - C_\infty) k_2 t} \quad (9)$$

A modified form of Eq.(9) was obtained by Arbiter⁽⁶⁾ from the equation

$$\frac{dR}{dt} = k_n' (R_\infty - R)^n \quad (10)$$

By integration with $R=0$ at $t=0$

$$R = \frac{R_\infty^2 k_2' t}{1 + R_\infty^2 k_2' t} \quad (11)$$

For graphical representation Arbiter wrote

$$\frac{t}{R} = \frac{1}{R_\infty^2 k_2'} + \frac{t}{R_\infty} \quad (12)$$

If a second order equation is valid, a plot of $\frac{t}{R}$ vrs. t should result in a straight line. However, he warns that

the method of plotting becomes insensitive at longer flotation times since, when R is large and changing very slowly, one is effectively plotting t vrs. t . Hence the agreement of the earlier points with the straight line is a necessary condition for the graphical test to be significant.

Morris⁽¹²⁾ criticises this method of plotting on the basis that the value of R_{∞} found by extrapolation can exceed 100%. On the other hand, Hukki⁽⁸⁾ suggests a more sensitive method of plotting whereby the data presented by Arbiter⁽⁶⁾ demonstrates the superiority of a second order plot. He suggests that $\frac{1}{t}$ be plotted against $\frac{1}{R}$ (see Appendix A). The slope of the second order plot could be used as a useful FLOTATION RATE INDEX, the numerical value of which increases with improving performance, i.e. 0.01 for very slow; 0.1 for slow; 1 for normal and 10 for very fast flotation.

Non-Integral Order

Although the majority of the investigators have argued either for first or second order rate equations, some have proposed non-integral orders. Horst and Morris⁽¹³⁾ have proposed a three-halves-order rate equation.

Tomlinson and Fleming⁽¹⁴⁾ argue that over a narrow

range of conditions (transitional flotation), the kinetic order of the process with respect to particle concentration would lie between zero and one. Volkova⁽¹⁵⁾ also argued that the rate equation is in general between first and second order, simplifying to first order for the flotation of single minerals, or in a very dilute pulp, and to second order for low grade ores or more concentrated pulps.

Holland-Batt's General Approach

Holland-Batt⁽¹⁶⁾ provides an alternative and more general approach to investigate the behavior of the flotation system.

For a system which proceeds according to an n^{th} order reaction

$$\frac{da}{dt} = - K_a a^n \quad (13)$$

$$\text{where } R_a = 1 - \frac{a}{a_0} \quad (14)$$

a = fractional content of ore mineral in material
being treated

a_0 = fractional content of ore mineral in feed

K_a = rate constant of ore mineral and

R_a = fractional recovery of ore mineral in concentrate .

On substituting for a from Eq. (14) into Eq.(13),

$$\Delta_t = \frac{dR_a}{dt} = a_o^{n-1} K_a (1-R_a)^n \quad (15)$$

Taking logs

$$\log \Delta_t = \log (a_o^{n-1} K_a) + n \log (1-R_a) \quad (16)$$

The values of Δ_t were determined at selected values of R_a by drawing tangents to the curve of R vrs. time.

Thus plotting Δ_t vrs. $(1-R_a)$ on a log-log basis, the slope of the plot gives the order of the process, i.e.

$$\frac{d \log \Delta_t}{d \log(1-R_a)} = n \quad (17)$$

Factors Affecting the Rate of Flotation

The important factors affecting the rate of flotation will be briefly summarized. Detailed surveys have been given by Arbiter and Harris⁽³⁾ and by Bull^(5,22).

Bubble-Mineral Aggregation Mechanism

As Eq.(2) implies we are considering the flotation process

as analogous to a chemical reaction in which, instead of collisions and reactions of ions or molecules, we have interactions between mineral particles and air bubbles. The fact that collision can take place and result in adhesion and flotation has been demonstrated by high-speed movie techniques⁽³²⁾. Collisions and attachment, however, may not be the only mechanism responsible for flotation. The precipitation of gas from solution on hydrophobic surfaces has been considered as a possible mechanism. It has been proposed that the occurrence of precipitation may be an important preliminary step which favors subsequent collision and attachment⁽²⁴⁾. In addition, particles could appear in the froth as a result of physical entrapment between laden bubbles, or by attachment of mineral particles to the bottom of the froth column by pulp agitation⁽²⁶⁾.

Cell Operation

The variables of cell operation affecting the rate of flotation are cell design, air flow rate, impeller speed, and clearance between impeller and stator. A detailed account of their effect on the flotation rate can be found elsewhere^(3,5).

Flotation Reagents

It is clear from the discussion under Bubble-Mineral Aggregation Mechanism that the rate of flotation will be affected by the type and concentration of reagents used when the functions of these reagents are considered. The function of a collector is to transform the hydrophilic surface of the mineral particles to the hydrophobic state. The function of the frother is to permit the formation of a froth which is sufficiently stable to hold the floated minerals so that they may be removed as a concentrate. Modifying agents are used to depress or activate mineral surfaces, i.e. to make a mineral surface repulsive or receptive to a specific collector.

Detailed discussion on the effect of flotation reagents on the rate of flotation can be found elsewhere^(3,5).

Particle Size

The probability of collision and attachment of a particle with a freely moving bubble must vary with the size of the particle because of 1) its projected area, 2) its inertia which will determine whether or not a particle may cut across flow lines around the bubbles, 3) the possibility of the particle being thrown off the bubble due to disruptive turbulence, and 4) the extent to which collision may distort the bubble and alter the time of contact. In any given cell,

considering the size of the bubbles produced and the disruptive forces present due to the turbulence of the pulp, there is probably a maximum size beyond which particles cannot be floated in conventional mechanical cells. This maximum size is about 35 mesh with sulphide particles and is correspondingly coarser for a lighter material like coal⁽⁵⁾. With decreasing size below about 200 mesh, recovery remains constant and then declines steadily below 10 to 20 μ due to flocculation.

In practice, these physical effects mean that different-sized particles of the same mineral, under the same chemical conditions, must be expected to have different rate coefficients. This has been confirmed in several publications^(5,26,14). Several investigators, however, have reported coefficients only for the unsized sample; this has probably led to misleading interpretations, since if the species is considered in the unsized form, the apparent rate equation may be of any order, depending upon the values of the individual rate coefficients for different-sized material and upon the size distribution of the feed.

Liberation

Particles of an ore can consist of a single mineral, in which case they are termed free particles, or they may consist of two or more minerals in which case they are locked particles.

The degree of liberation of a certain mineral or phase is the percentage of that mineral or phase occurring as free particles of the total of that mineral occurring in the free and locked forms. These locked particles behave, broadly speaking, in manners intermediate between those of their constituent minerals. In flotation, the proportion of each mineral at the surface is the controlling factor. The locked particles grade from those consisting mostly of gangue with only a few specks of mineral to those consisting mostly of mineral with only a few specks of gangue.

The development of the kinetic equations based on the chemical kinetics analogy was restricted to pure minerals or mixtures of pure minerals. When dealing with an actual ore, as in the present investigation, the strict analogy becomes invalid. The value of the concentration of the species (in this case molybdenite) is derived only from chemical analysis, which detect the Mo species regardless of its mineralogical form. Since the MoS_2 species must exist in both a size range of free particles and in a wide range of type of locked particles, the application of kinetic equations must be considered to be an empirical description of the process.

However, if any of these equations can be shown to describe the process with acceptable accuracy, they may be of practical use. This is the main objective of this work.

At this point it would be worthwhile to define some terms used in the empirical kinetic equations in the present investigation. They are:

C_0 = initial concentration in grams/litre of pulp of Mo as derived from chemical analysis

C = concentration in grams/litre of pulp of Mo at time t as derived from chemical analysis and

C_∞ = the terminal concentration in grams per litre of pulp (concentration remaining unfloated in the cell after prolonged flotation) of Mo as derived from chemical analysis.

Flotation Rate Constant K

The kinetic theory for which the equations have been developed above makes a basic assumption that the value of K is constant at different cell concentrations of the floatable material. As mentioned earlier, the flotation rate constant K is a complex function of the chemical conditions of the pulp and of the physical characteristics of cell operation and design.

Some recent publications^(18,19) have introduced the concept of reverse transfer of particles from froth to pulp

and the reverse rate coefficient K' (K being essentially a forward rate coefficient) as an important parameter. Its consideration involves the use of equations of greatly increased complexity which, for practical purposes, may prove unnecessary when the reverse rate coefficient is very low⁽²²⁾.

Harris and Chakravarti⁽²³⁾ report that "even with a narrow size range of single mineral, a plot of $f(t)$ vs. time ($f(t)$ is the fraction of floatable solids left in the cell at time t) can show substantial curvature, which indicates that a distribution and not a single species is involved." Sutherland⁽²⁴⁾ laid the foundation for the mathematical procedures for dealing with species distribution, although his physical model (induction period distribution) was an over-simplification. Kelsall⁽²⁵⁾ gave the first practical graphical method for estimating the distribution of species. Imaizumi and Inoue⁽²⁶⁾ reported that "theoretical consideration of flotation mechanisms based on flotation systems, have led to the conclusion that the first order rate equation, in which the rate constant K_1 is not necessarily single-valued but variant (distributed), is the only form which represents plausibly the flotation rate function under normal conditions." They developed a graphical method for estimating the distribution of species but later⁽²⁷⁾ rejected it in favor

of a procedure developed by Woodburn and Loveday⁽²⁸⁾.

Woodburn and Loveday⁽²⁸⁾ introduced the distributed-K theory as the basis for their model of the flotation process. They assumed, as most investigators did, ideal mixing of all particles in the flotation cell.

The distribution of "K" values in a population of particles may be described by a frequency function $W(K)$ where $W(K)dK$ is the weight fraction of material with a flotation rate constant between K and $K+dK$. The rate of removal of any individual fraction is then characterized by the first order rate equation:

$$\frac{d(W(K)dK)}{dt} = -KW(K)dK \quad (18)$$

where $W(K)$ is a bounded function containing unit area.

For any individual fraction, the integrated form of Eq.(18) yields the quantity left after a time t , in a batch reaction. Thus after time t , fraction of material remaining in the cell with a floatability constant between K and $K+dK$ is given by

$$W'(K)dK = \frac{W(K)dK * e^{-Kt}}{\int_0^{\infty} W(K) * e^{-Kt} dK} \quad (19)$$

i.e. the frequency distribution in the cell changes because the more floatable material is removed at a faster rate than the less floatable material. The limiting value of $W'(K)$ is thus a discontinuous function at $K=0$ (Fig. 1 indicates typical variation⁽²⁹⁾ of $W'(K)$ with time).

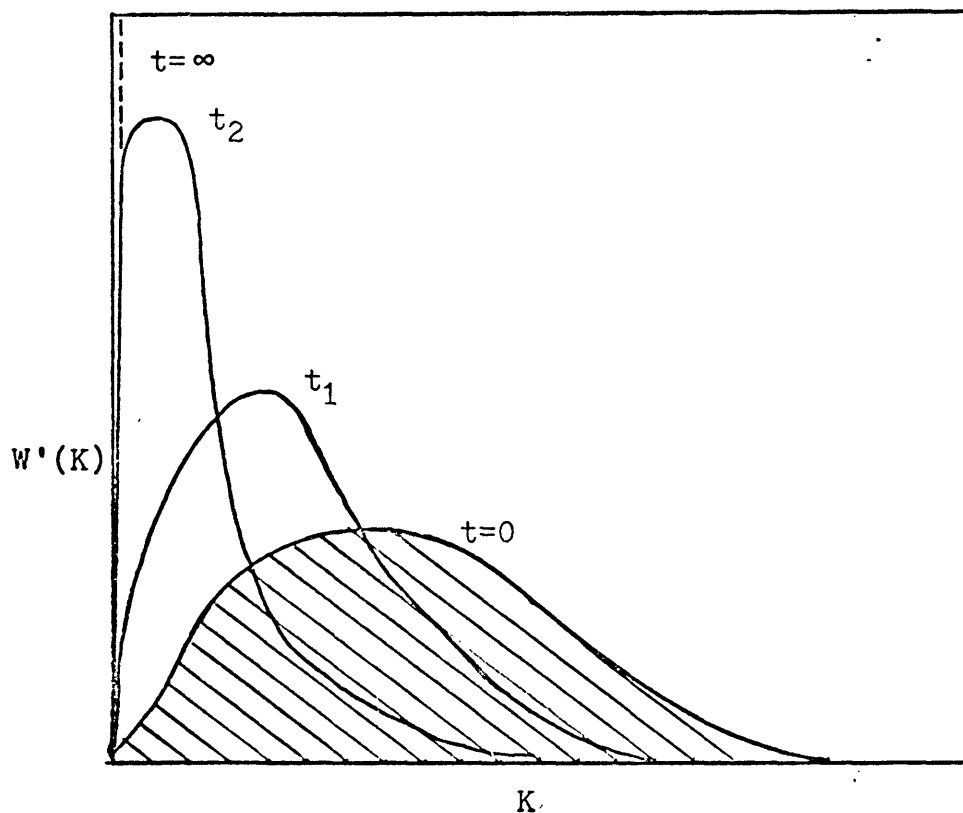


Fig. 1 Typical variation of $W'(K)$ with time

The overall rate of removal of material may be expressed in terms of an average value of K :

$$\frac{dW}{dt} = - K_{av} \cdot W \quad (20)$$

where K_{av} is defined as:

$$K_{av} = \int_0^{\infty} K \cdot W(K) \, dK \quad (21)$$

$$= \frac{\int_0^{\infty} K \cdot W(K) \cdot e^{-Kt} \, dK}{\int_0^{\infty} W(K) \cdot e^{-Kt} \, dK} \quad (22)$$

Woodburn and Loveday assumed that the floatability distribution may be fitted by a gamma function of the form

$$W(K) = c \cdot K^a e^{-bK} \quad (23)$$

$$\text{As } \int_0^{\infty} W(K) \, dK = 1, \quad c = \frac{b^{a+1}}{\Gamma(a+1)}$$

The distribution $W(K)$ can thus be described by parameters "a" and "b". Hence knowing the parameters "a" and "b", the performance of a flotation process could be characterised.

Later Loveday⁽²⁹⁾ found that an improved fit could be obtained by using a truncated gamma function of the form

$$W(K) = c \cdot K^a e^{-bK} (1 - H(K_{\max})) \quad (24)$$

where

$$H(K_{\max}) = 0; \quad 0 \leq K \leq K_{\max}$$

$$H(K_{\max}) = 1; \quad K > K_{\max}$$

Recent investigators^(20,21) have combined the concept of distributed-rate constants with that of a two-phase flotation model⁽¹⁹⁾ in order to obtain an overall model, more powerful and flexible than previous ones.

Recently, Harris and Chakravarti⁽²³⁾ suggested a method for finding n and K_m by plotting $\log \log \frac{1}{f(t)}$ vrs. $\log t$ on a Rosin-Rammler grid and fitting to it the theoretical curve reproduced as a transparent overlay. In case the fit is not good, the species distribution may be bimodal. The species distribution can be described, in this case, by a 3-parameter model:

$$f(t) = \frac{r}{(1+t)^n} + \frac{1-r}{(1+t)^m} \quad (m > n) \quad (25)$$

where r = fraction of floatable solid having the lower average rate constants and

n and m are arguments of the gamma function species distribution.

Once the parameters n , m and r are known, the species distribution can be plotted as $\Psi(K,0)$ vrs. K in order to display its detailed structure. The investigators have shown that several different species distributions can generate similar recovery vrs. time curves (and $f(t)$ vrs. t curves). Thus extreme close fitting between the model and the data must be assured to avoid the prediction of a spurious species distribution. However this three parameter model is much more flexible for fitting batch flotation data than the 3-parameter truncated gamma function.

Concept of R_∞

The concept of R_∞ , or of terminal concentration C_∞ , which is equivalent to the terminal floatability R_∞ , was first introduced by Morris⁽⁹⁾.

The investigators who have used the kinetic equations^(17,22,26,30,31) have assumed or calculated one value of R_∞ for each size fraction and/or for the unsized sample. Some of the investigators arrived at a value for R_∞ by batch flotation for a long period of time or until no further appearance of material in the froth^(17,31). Others estimated values for R_∞ by extrapolating the plot of percent cumulative recovery vrs. cell number⁽²²⁾. Still others used iterative calculations⁽⁹⁾.

EXPERIMENTAL APPARATUS AND PROCEDURES

Molybdenite ore (from the Climax mine, Climax, Colorado) was selected for kinetic studies of batch flotation because of its ready availability and because it was known that crushed samples did not change their flotation characteristics with time to any appreciable extent. The procedure used for batch flotation and the reagent additions were the same as used at the Climax Molybdenum Company Research Laboratories, Golden.

Sample Preparation

Crushing

The ore was crushed in a jaw and a gyratory crusher to about 1/4 in., and then to 14 mesh with rolls in closed circuit with a vibrating screen. Care was taken to avoid contamination by any grease or oil or by other materials previously crushed in the equipment. An attempt to keep dust losses minimum was also made.

Sampling

In preparing samples for flotation tests, it is important that each sample should be representative of the whole so that the results of tests can be compared with confidence.

Material coarser than about 1/2 in. is best sampled either by coning and quatering, or by alternate shoveling, around a pile. For material between 1/2 in. and about 20 mesh in size, a riffle-type sampler is most efficient. For material finer than 20 mesh, riffle sampling is good, provided dust losses are not too great. Using these guidelines, 1000 gms representative samples for flotation feed were prepared by riffling to get approximately 1000 gms and then adjusting each sample to the desired weight by adding or removing small multiple grab samples.

Grinding

Batch grinding in the laboratory, particularly in a ball mill, produces a flotation feed with a wider size distribution than closed-circuit grinding. However, Armstrong⁽³³⁾ has shown that the size distribution obtained by grinding in a batch rod mill closely approximates that obtained in a closed-circuit ball mill. Hence for the present work, a batch rod-mill was used for grinding flotation feed.

Most of the reagents used in the flotation were added to

the rod mill (see Appendix B). Grinding was done at 66% solids in a rod mill (10-in-diam x 11-in. internal height) made of steel using a rod load of 14-Kg (9-in. rods of diameters from 0.5 to 1-in.) for different times at constant speed of 60 rpm.

Batch Flotation Tests

Equipment

The apparatus used for batch flotation of molybdenite ore is shown in figures 2 and 3. The flotation equipment had automatic froth skimming paddles, a system for constant air intake and an arrangement which would maintain the pulp level constant.

Automatic Froth Removal System

Raffinot ⁽³⁵⁾ has shown from a statistical study of the variants in batch testing that the test procedure was a more important source of variation than sampling or chemical analysis. Hence, in order to reduce the variants in batch testing, an automatic froth removing system was put on to the plexiglass flotation cell. The paddles were driven by a motor and suitable pulley and belt drive assembly.

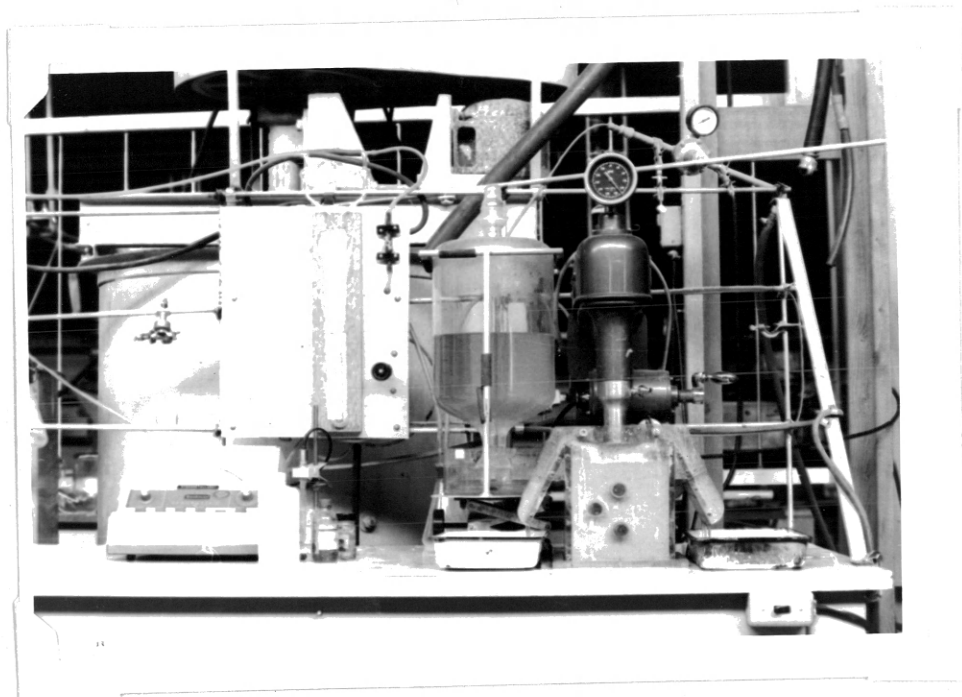


Figure 2. General view of equipment.



Figure 3. Photograph of flotation cell showing the automatic scrapers and air control system.

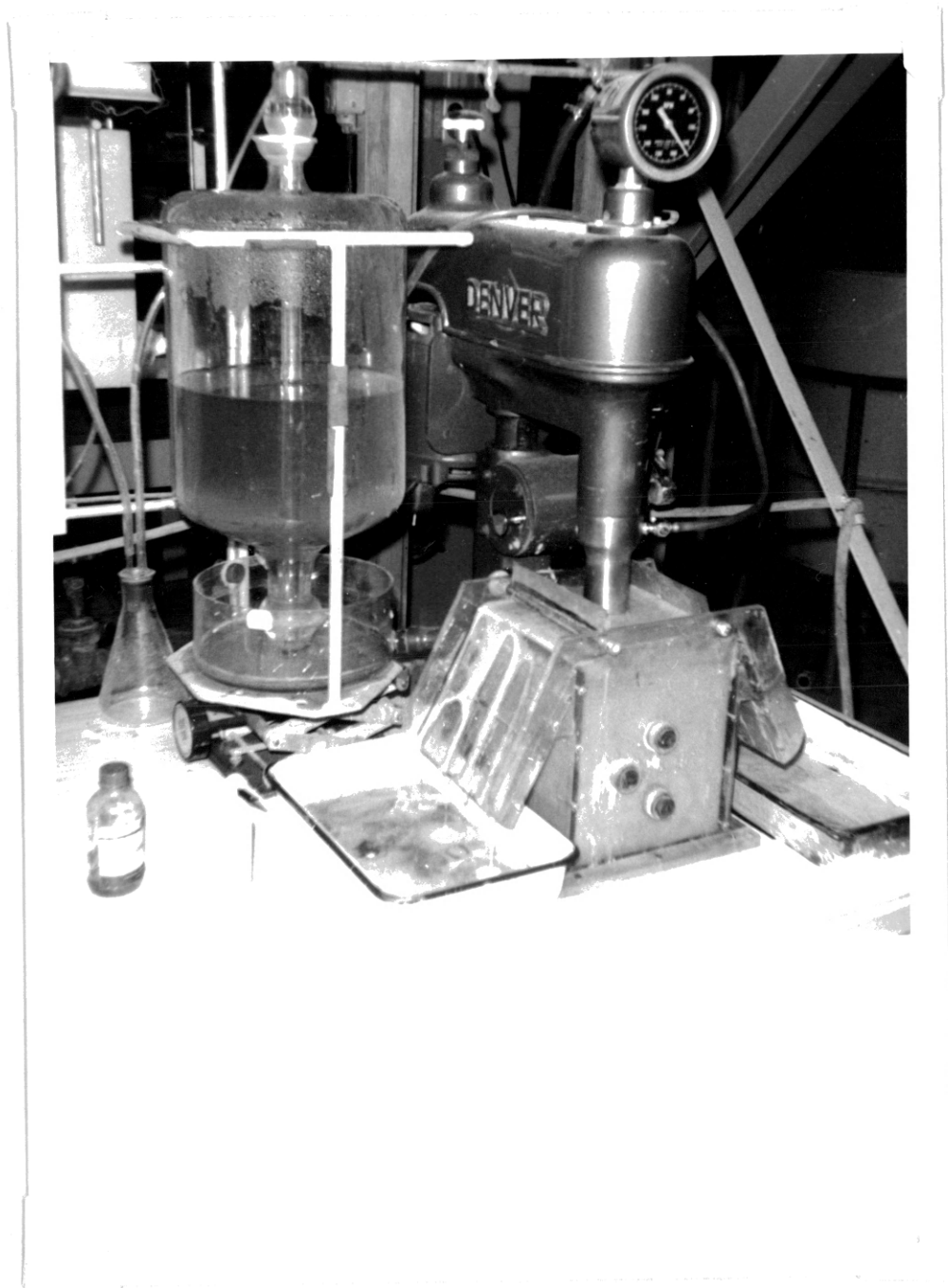


Figure 4. Constant head chamber and the automatic froth removal system.

Pulp Level Control

While the equations describing the kinetics of flotation system having variable volume can be used, the case of constant pulp volume leads to simplified equations⁽³⁾. In normal semi-batch operation, make up water with or without reagent is added intermittently by the operator. Hence the assumption that pulp volume is constant may be invalid.

The method designed for maintaining constant pulp volume was based on the Mariotte principle which is frequently used to maintain constant flow through the agency of constant hydraulic head⁽³⁶⁾. The water-reagent solution in the constant head chamber (Fig. 4) and the pulp in the flotation cell seek the same level. As flotation proceeds the level drops, and eventually an air bubble enters the neck of the bottle thus displacing make-up water. In this manner, the level, and therefore volume, are restored to their previous state. To facilitate adjustment in pulp level the constant head chamber rested on a flat surface which could be moved vertically with the help of rack and pinion device.

The bottle was filled with water, inverted and lowered into the constant head chamber. The constant head chamber was connected to the flotation cell with a flexible tubing. The orifice in the flotation cell was covered with a fine mesh cloth to prevent migration of cell contents into the feed tube.

The use of the constant head chamber for maintaining the pulp level had to be abandoned because very fine solids entered the feed tube to the cell against the counter flow of make-up water. These solids blocked the feed tube and hindered the flow of make-up water. The pulp level would fall and hence the pulp volume was not maintained constant. Also there was a loss of significant quantity of solids because of the counter-flow.

An alternative method keeping pulp level constant, though not as sophisticated, was chosen. The present method used an 0.5-in rubber tubing connected to a tap, which was within the reach of the operator, at one end. The other end was clamped to the stator shaft. The amount of water addition to the cell was monitored manually. The level of pulp in the flotation cell was thus maintained constant within close limits.

Control of Air Input to Flotation Cell

Since the amount of air introduced to the flotation cell effects the kinetics of the flotation process, a control system, as shown in fig. 5 was used to control the air input to the cell.

Air from a compressed air source was passed through a constant head low pressure regulator (consisting of a tank

with water level maintained constant) and then a surge vessel. The purpose of this surge vessel was to dampen pressure fluctuations produced by the low pressure regulator. Then the air passed through a capillary and the pressure drop across this constraint remained constant. This assured a constant supply of air to the flotation cell. In case there were any small fluctuations, they could be taken care of by the fine adjustment knob.

Procedure

The flotation feed from the rod-mill was transferred into the 5 litre plexiglass flotation cell. Water was added to the cell to make up the pulp to 25% solids. The pulp was conditioned for 3 minutes at 1500 rpm impeller speed. The pH measurement of the pulp was done using Beckman Zeromatic II pH meter for every test. A head pH of 9.0 - 9.3 was maintained. Two drops of frother (pine oil) were added and the pulp was again conditioned for 3 minutes. Timed samples were collected in pans and the sides and impeller stand pipe were washed frequently during the test including the last few minutes of flotation. The total flotation time was between 8 and 12 minutes. A constant pulp level was maintained in the cell by the continuous addition of water.

The final tail pH was measured and was usually between 8.5 and 9.1.

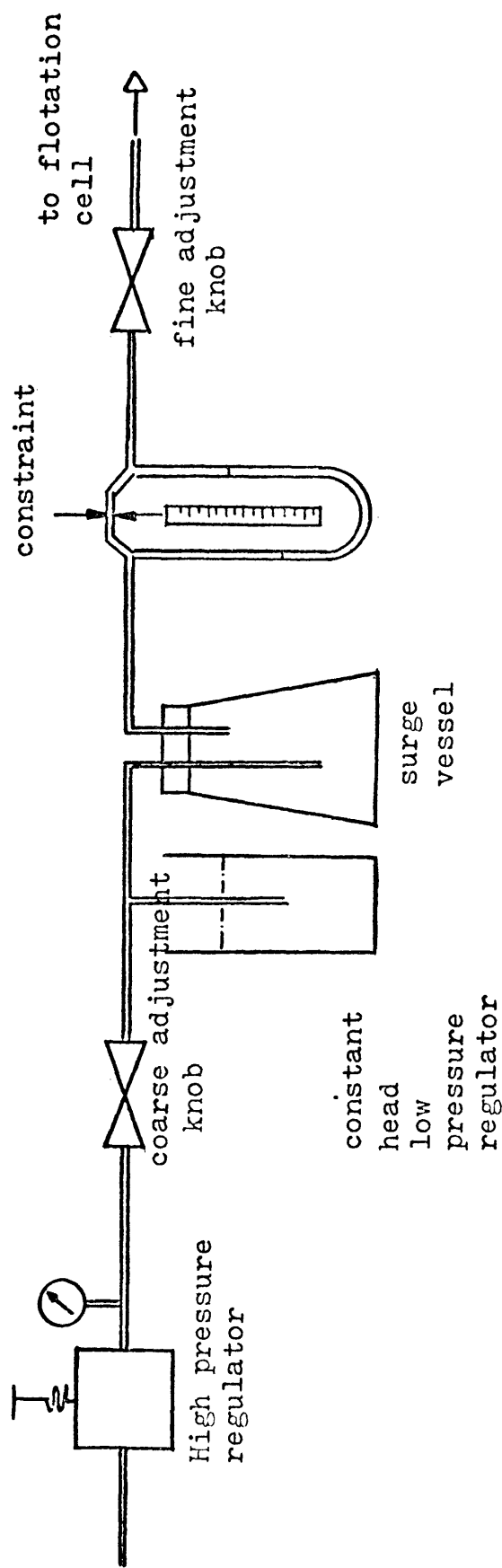


Figure 5. System for controlling air input to flotation cell.

Analysis of Samples

The timed samples obtained from the flotation tests were filtered, dried and weighed. Wet and Dry Screening Tests were done on the tailings of the flotation tests.

A representative sample of each timed sample and tailing was obtained using the micro-riffles and then these samples were pulverized.

Chemical Analysis for the evaluation of the amount of Mo in each sample was done by the method discussed in Appendix C. Replicate samples of each concentrate were analysed and in case the % Mo differed by more than 2%, another set of the same concentrate was analysed.

EXPERIMENTAL RESULTS

The sizing analysis of the rod mill feed, which was used in all the experiments, was reasonably constant from test to test. For the three randomly chosen feed samples the variation was not larger than 5% absolute for any single size fraction. This variation in size distribution is to be expected since the investigation was done on the natural ore and not a synthetic mixture. The only control the investigator had in the reproduction of size analyses was good and accurate sampling.

The average size distribution of the feed used in the rod mill is presented in Table 1.

The size analyses for grind times of 4, 7, 10, 13 and 16 minutes are given in Table 2. The +48 mesh material was not reported separately because it was generally less than 2.5% for most of the grind tests.

Table 1: Average size distribution of the flotation feed

Mesh	Percent wt retained
+48	56.7
48/65	9.2
65/100	7.2
100/150	5.8
150/200	3.5
-200	<u>17.6</u>
	100.0

Table 2: Size distributions for different grind times

Mesh	Percent weight retained				
	Grind time				
	4 min.	7 min.	10 min.	13 min.	16 min.
+65	22.54	18.44	13.49	0.63	-
65/100	20.20	19.12	17.80	9.40	2.54
100/150	12.50	13.05	13.72	19.63	13.30
150/200	8.79	10.13	11.05	15.64	18.41
-200	<u>35.98</u>	<u>39.25</u>	<u>43.93</u>	<u>54.69</u>	<u>65.76</u>
	100.01	99.99	99.99	99.99	100.01

Six representative samples of the feed to the flotation cell were assayed for Mo. The mean of the six samples was $0.313 \pm 0.03\%$ Mo.

The timed-concentrate samples obtained from the flotation tests were chemically analysed for Mo. The experimental results are reported in Appendix D.

The conditions for all the flotation tests included are summarized in Table 3. In the remainder of the tests, specific flotation tests will be referred to by their number as given in Table 3.

The experimentally observed recovery-time curves for the different grind times and air flow rates are given in figures 6 and 7. Figures 8, 9, 10 and 11 give the cumulative recovery vrs time curves for replicate tests conducted.

A test was also carried out without using collector. The experimental curve is shown in figure 12 along with the results of test 2. The wet and dry screening analyses of tests 1 to 8 are given in Appendix E.

Table 3: Summary of test conditions

Test no.	Grind time min.	Collector concentration lb/ton	Frother concentration lb/ton	Air rate l/min.	Paddle speed rpm	Impeller rpm
1	4	0.5	0.038	5.61	60	1500
2	7	0.5	0.038	5.61	60	1500
3	10	0.5	0.038	5.61	60	1500
4	13	0.5	0.038	5.61	60	1500
5	16	0.5	0.038	5.61	60	1500
6	7	0.5	0.038	5.61	60	1500
7	7	0.5	0.038	5.61	60	1500
8	13	0.5	0.038	5.61	60	1500
9	7	0.5	0.038	7.64	60	1500
10	7	0.5	0.038	7.64	60	1500
11	7	0.5	0.038	9.68	60	1500
12	7	0.5	0.038	4.62	60	1500
13	7	0.5	0.038	4.62	60	1500
14	7	No	0.038	5.61	60	1500

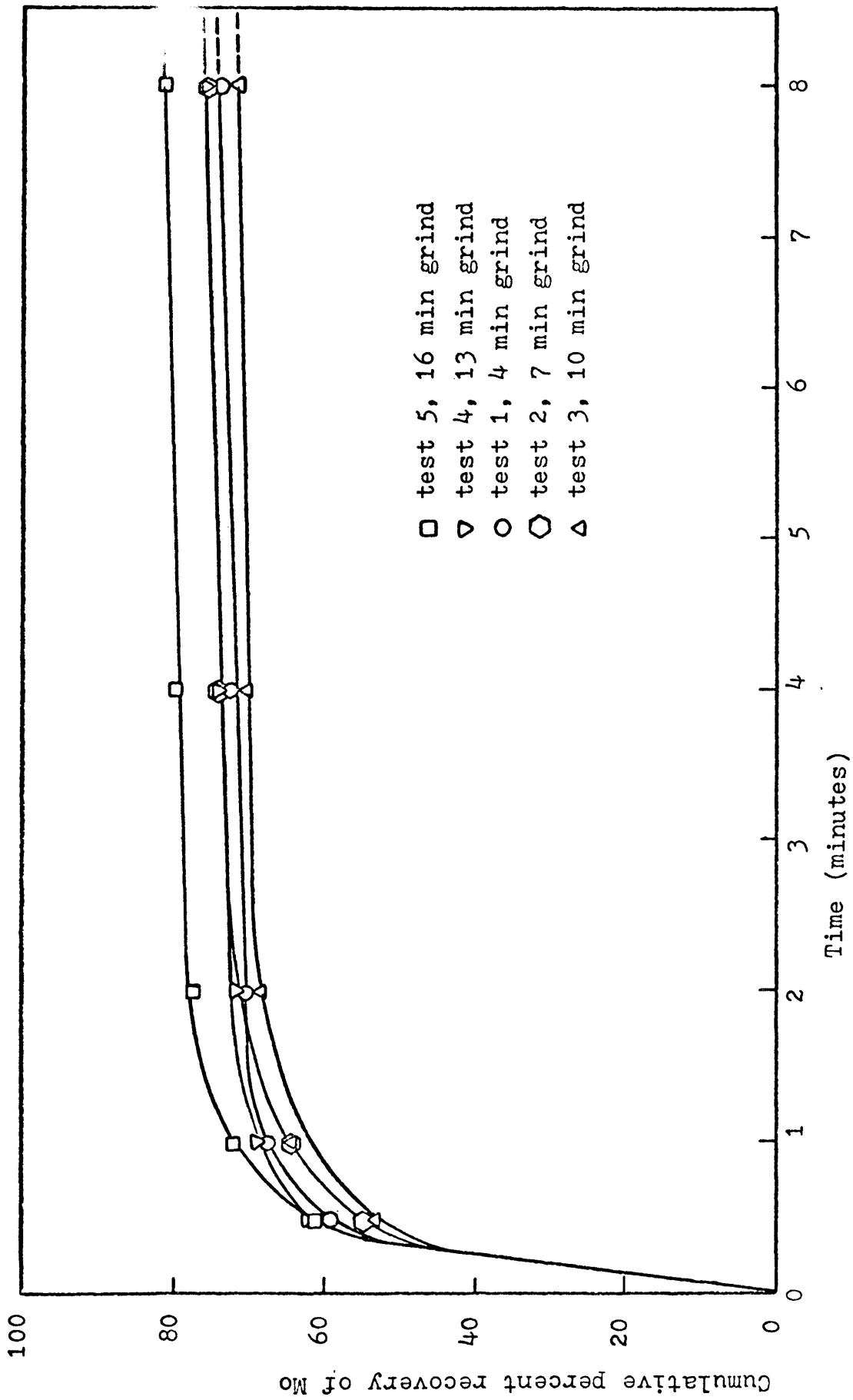


Figure 6. Recovery-time curves for different grind times and 5.61 l/min air flow rate.

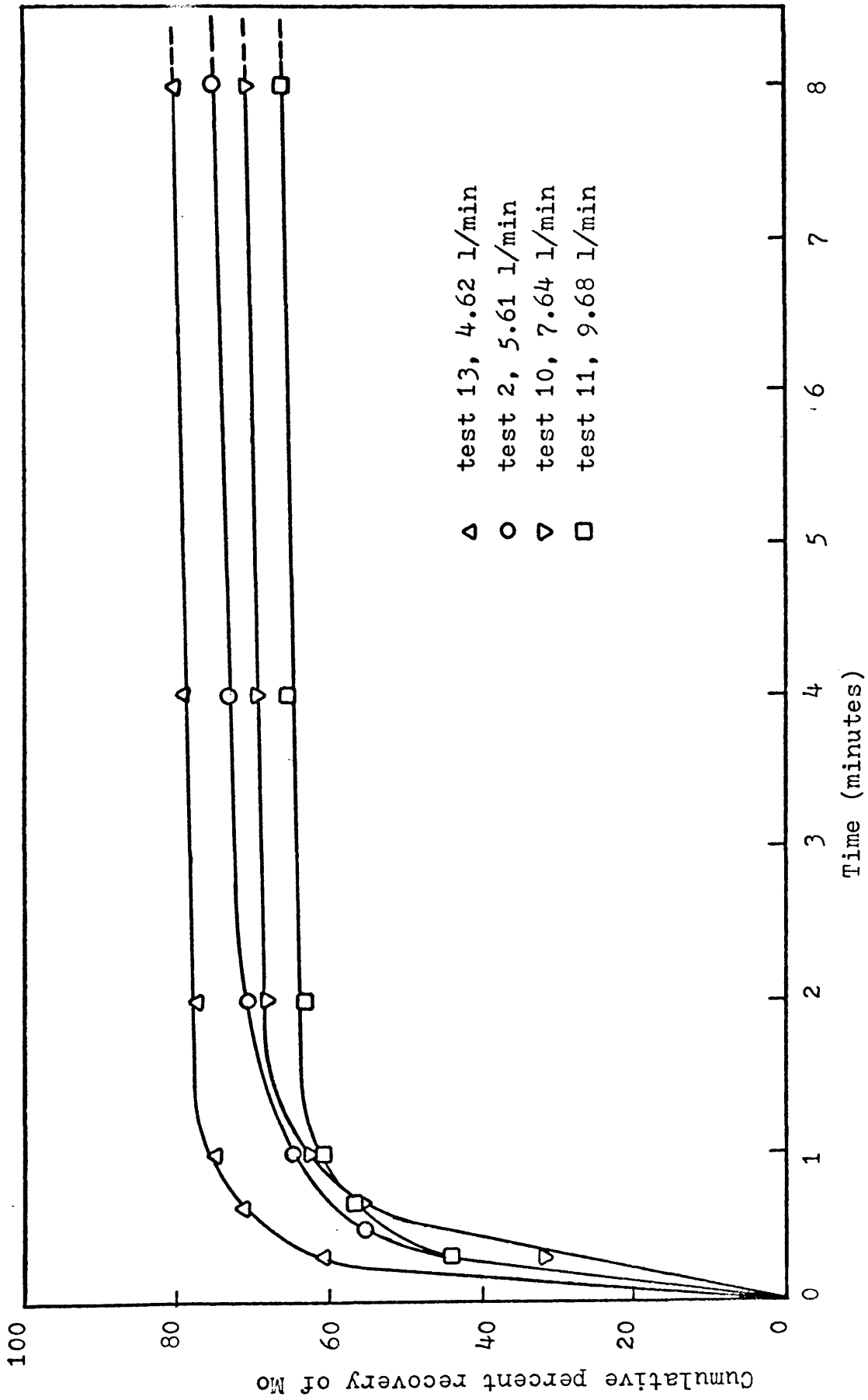


Figure 7. Recovery-time curves for different air flow rates and for grind time of 7 minutes.

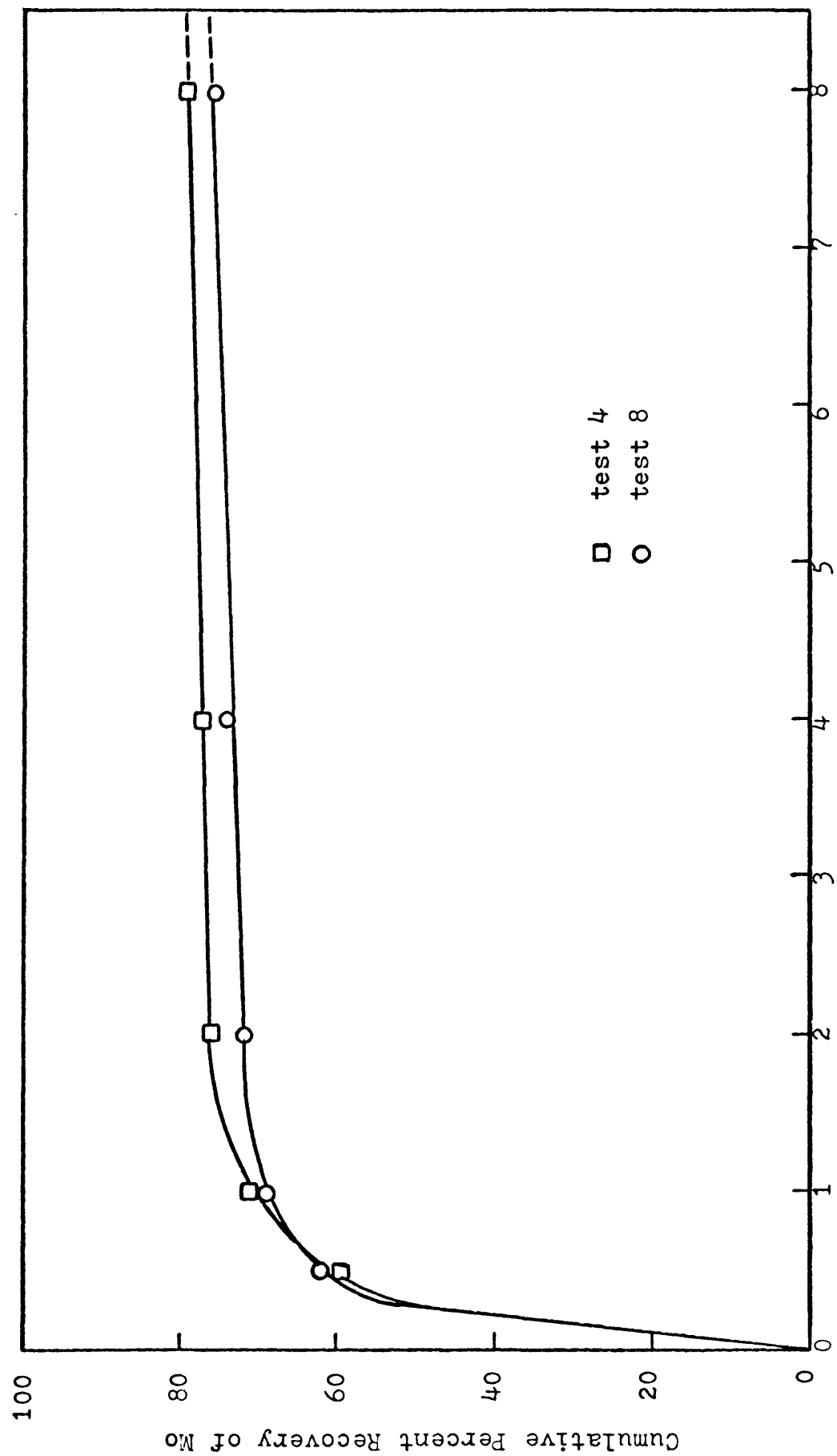


Figure 8. Recovery time curves for tests 4 and 8 conducted under identical conditions (13 min grind and 5.61 l/min air flow rate).

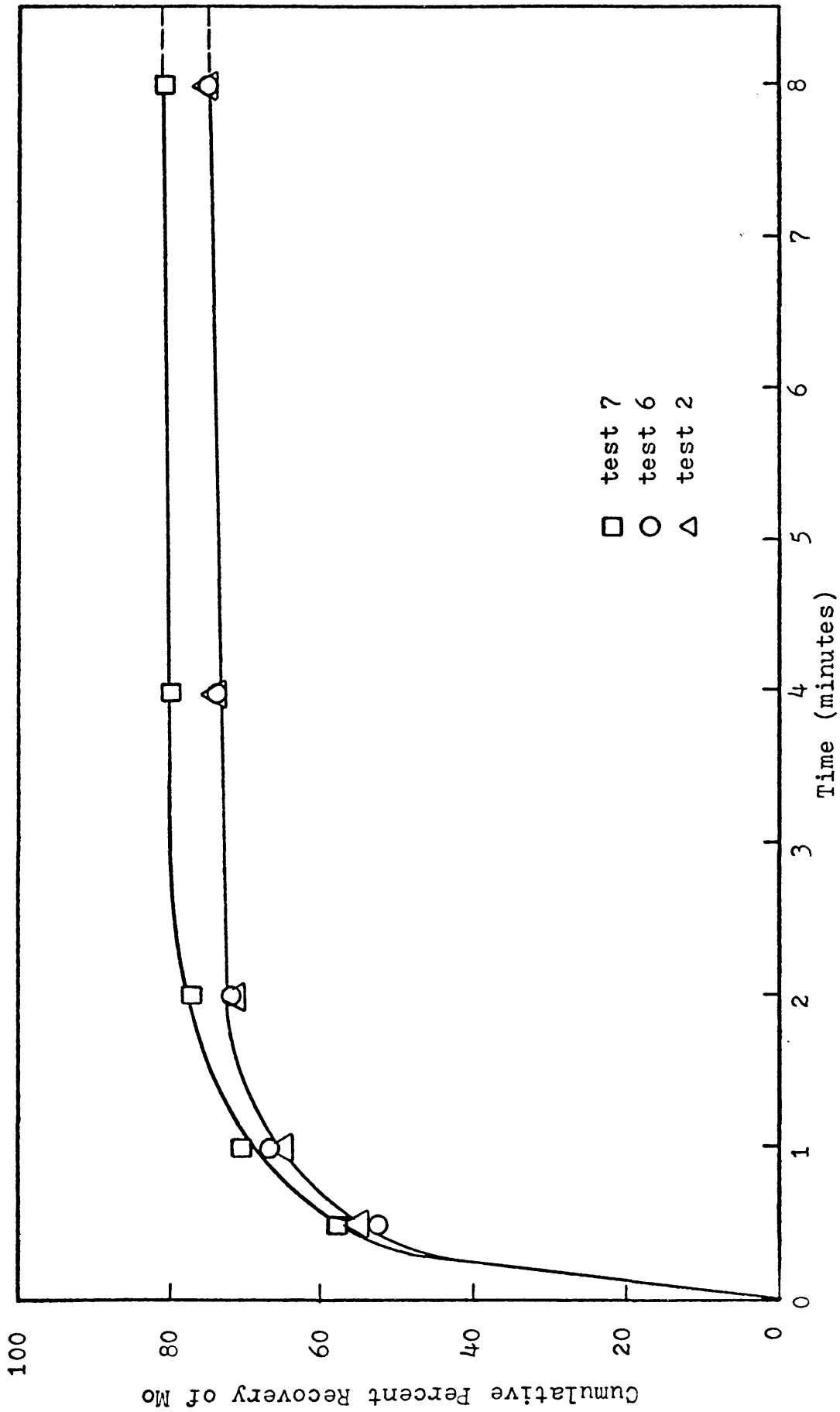


Figure 9. Recovery-time curves for tests 2, 6 and 7 conducted under identical conditions (7 min grind and 5.61 l/min air flow rate)

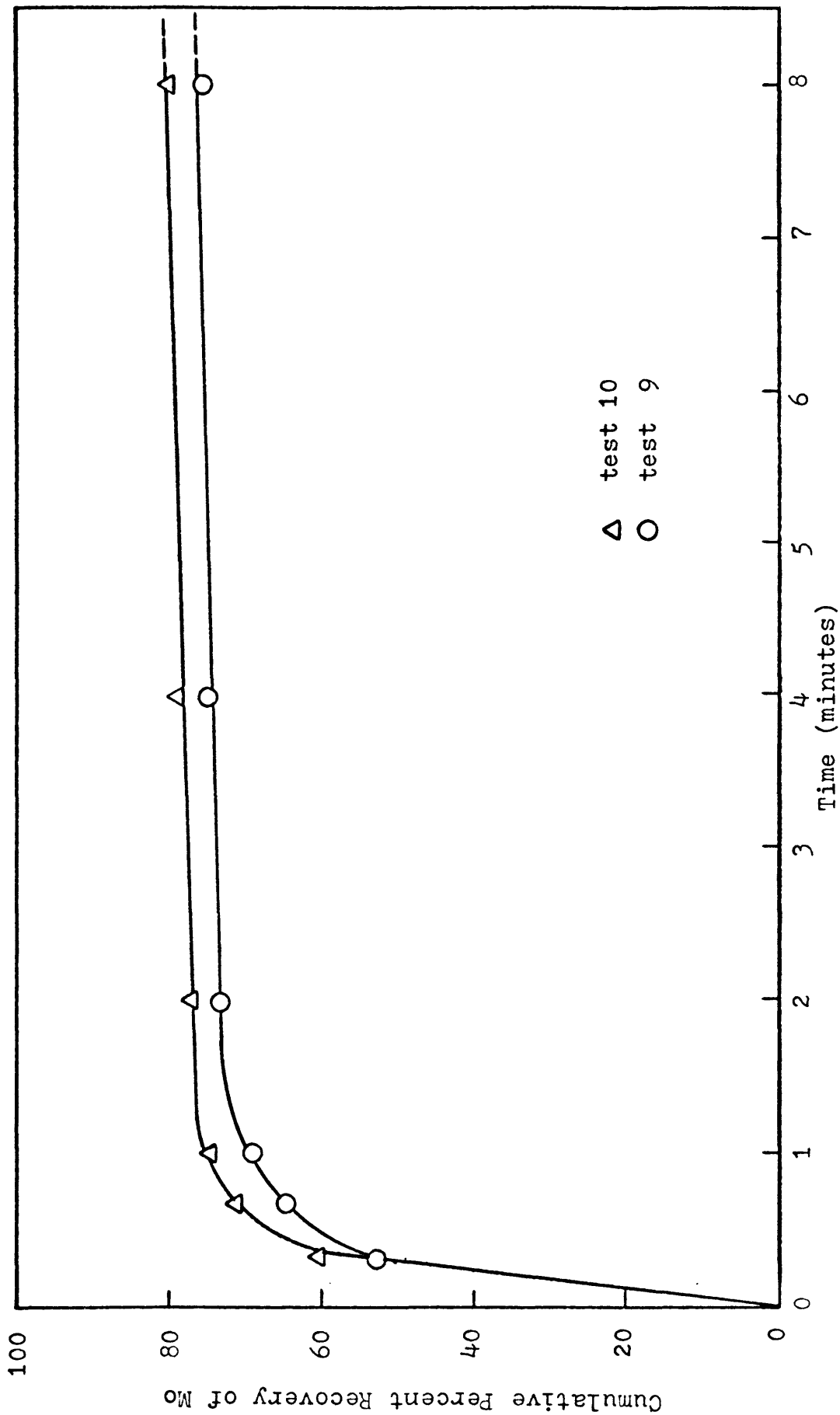


Figure 10. Recovery-time curves for tests 9 and 10 conducted under identical conditions (7 min grind and 7.64 l/min air flow rate).

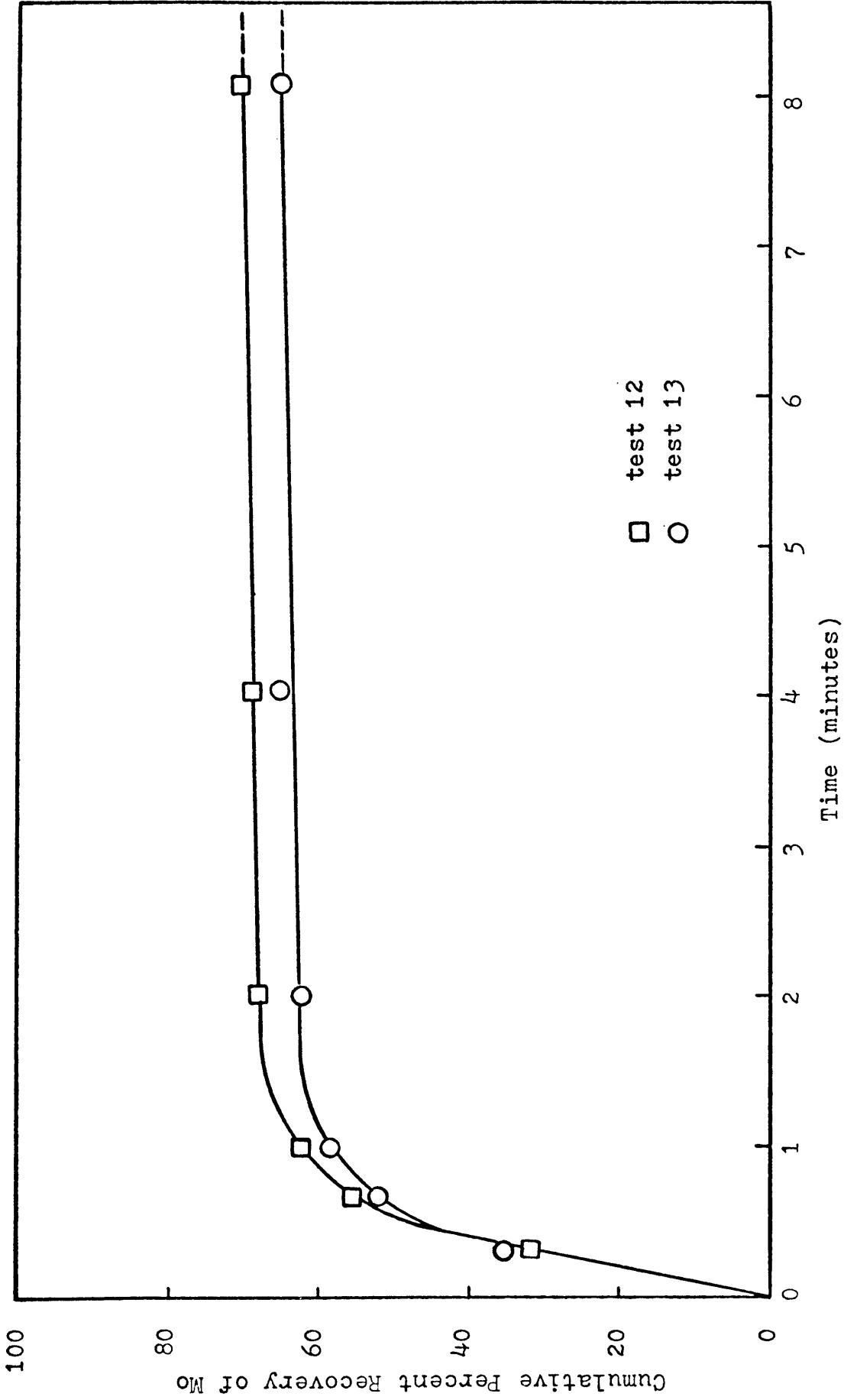


Figure 11. Recovery-time curves for tests 12 and 13 conducted under identical conditions (7 min grind and 4.62 l/min air flow rate).

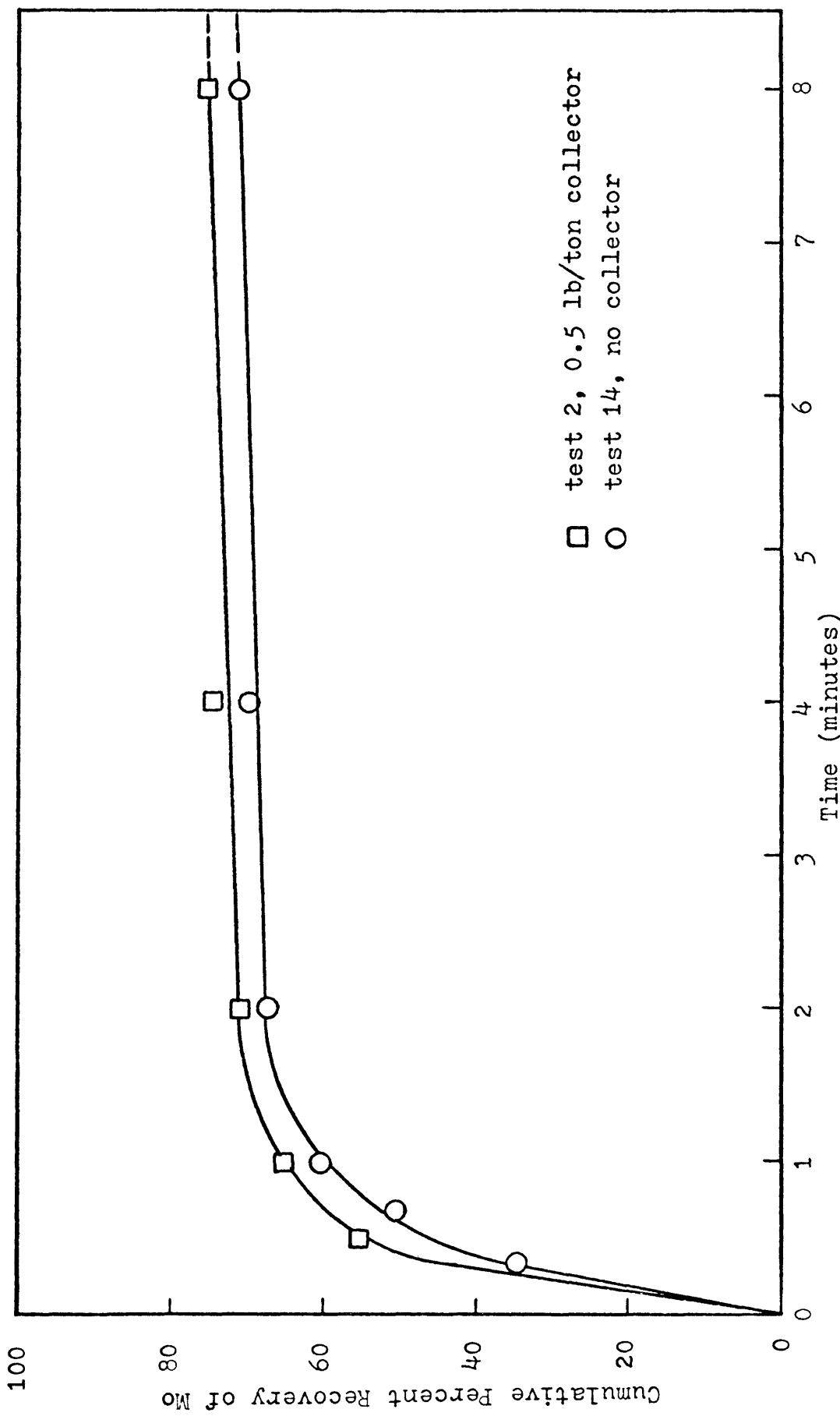


Figure 12. Recovery-time curves for tests 2 and 14 for 7 minute grind and 5.61 l/min air flow rate.

DISCUSSION OF RESULTS

Feed Assay

Molybdenite ore was chosen for kinetic interpretation of batch flotation because (a) it is relatively fine grained, and the molybdenite is homogeneously distributed throughout the gangue matrix and (b) its flotation characteristics do not change appreciably during storage after crushing to a fine size.

An ore was chosen, rather than a synthetic mixture of minerals, to investigate the applicability of kinetic equations to a practical system. A review of the literature on flotation kinetics indicates that most of the investigators made the implicit assumption that the feed solids were homogeneous. In the present investigation chemical analysis on six samples of the ore gave a mean value of $0.313 \pm 0.031\%$ Mo. The head assays were also calculated from the mass balance of the concentrate samples and the tailings. Table 4 gives the calculated feed assays for tests 1 to 14. These assays vary

from 0.23 to 0.32% Mo. The variations in the feed assays could be due to the sampling or assaying errors.

Replicate samples of each concentrate and tailings were taken for chemical analysis. If these replicate analysis did not agree to within 2%, another set of replicate tests was done. Not more than 10% of the samples had to be re-assayed for this reason. Hence, the possibility of the inaccuracy of the assay procedure leading to variations in the calculated feed assay is relatively low. It may be possible, in this case, that the implicit assumption of feed solids being homogeneous is not true. This assumption is valid in the case of a pure mineral but need not necessarily be true when working with an actual ore.

The tailing samples assayed in the range of 0.02 to 0.08% Mo. Considering the weight of the tailings (normally around 900 g) and the possibility of error in the chemical analysis of tailings, the calculated feed assays would not be expected to be as accurate as actual feed assays. The mean calculated feed assay in the present investigation was $0.28 \pm 0.026\%$ Mo. A comparison of standard deviations show that calculated feed assays are more reliable. The reason for this is that the standard deviations were calculated for different number of samples (n_1 being the number of samples of feed assayed and n_2 being the number of samples used for calculating feed

Table 4: Calculated feed assays

Test no.	% Mo
1	0.3185
2	0.3100
3	0.2590
4	0.2533
5	0.2668
6	0.2700
7	0.3160
8	0.2893
9	0.2895
10	0.2912
11	0.2337
12	0.2607
13	0.2323
14	0.2823

assays based on analyses of concentrates and tailings) in each case, i.e. $n_1 = 6$ and $n_2 = 14$. Since the range for calculated feed assay varies from 0.23 to 0.32% Mo compared with 0.27 to 0.32% Mo for feed assays chemically analysed, it was decided to use the analysed feed assay in the kinetic interpretation of flotation data.

Determination of the Value of R_∞

The recovery of any mineral rises to an asymptotic value, R_∞ , which is generally less than 100%. In the present work, the flotation rate was fast and R_∞ was approached very quickly. The values of R_∞ were obtained by extrapolation of the graphs of cumulative recovery vrs time. These values of R_∞ compared well with the values of R_∞ obtained using Arbiter's modified second order equation where $R_\infty = R$ when $1/t = 0$ or $t = \infty$. Table 5 gives the values of R_∞ calculated by the above two methods and by Arbiter's second order equation where the reciprocal of the slope of the line gives R_∞ (6).

The values of R_∞ obtained by the three methods are very close to each other. The last two methods are based on the fact that a straight line is obtained for a second order equation. This is in fact the case in the present investigation and will be discussed later in this section.

Table 5: Calculated values of R_{∞}

Test no.	R_{∞} from recovery-time curves	R_{∞} from Arbiter's 2nd order Eqn.	R_{∞} from modified 2nd order Eqn.
1	75.2	75.0	75.2
2	77.0	76.3	77.2
3	72.5	75.0	72.7
4	77.0	77.3	77.2
5	83.0	83.3	83.3
6	77.0	78.6	77.2
7	83.0	80.0	83.3
8	80.0	83.3	80.7
9	77.0	75.0	77.2
10	81.0	77.3	81.1
11	67.0	68.8	67.3
12	65.0	71.4	65.2
13	71.5	71.4	71.9
14	73.5	72.2	73.8

Since there is no universally acceptable method of extrapolating the values of R_{∞} , any of the methods could be used. However, the values of R_{∞} obtained by Arbiter's modified second order equation were used in further calculations because the values were extrapolated theoretically for $t = \infty$.

First Order Rate Equation

In order to test the modified first order kinetic theory for batch flotation, Eq. (6) can be used. The values of $\frac{C-C_{\infty}}{C_0-C_{\infty}}$ are tabulated for each test in Appendix F. Typical curves for $\frac{C-C_{\infty}}{C_0-C_{\infty}}$ versus time are shown in figs. 13, 14 and 15 for tests 1 to 7 and 11. The curves of the same general form could be drawn for the other tests from the data in Appendix F.

As can be seen from figs. 13 through 15, and as was the case for all the tests, no straight-line relationship between $\ln \frac{C-C_{\infty}}{C_0-C_{\infty}}$ and time, as predicted by the modified first-order kinetic equation, was obtained. Since the tangent to the curve $\ln \frac{C-C_{\infty}}{C_0-C_{\infty}}$ vrs time, according to Eq. (6), is numerically equal to the flotation rate coefficient (K), the value of K would vary with time.

A start-up error was involved in all the flotation tests because the delay in transportation of the froth over the weir cannot be eliminated. A small time correction (time difference between the start of the air flow into the cell and the flow of concentrate over the weir) had to be applied to the test data. This was taken into account graphically by extrapolating the data points and shifting the time axis so that zero time coincides with $F = 1$ (where F =Fraction of M_0 remaining in the cell). This required that a small time ($t=6$ secs) be subtracted from the experimental time to give an effective flotation time t .

For the modified first order kinetic theory, the slope of the curve gives the value of K . Figs. 13 through 15 show that the slope of the curve decreases with time. The decrease in K with time is negligible for the first 30 secs (i.e. the slope remains constant). The decrease in K is pronounced in the range from 30 secs to a few minutes and then again becomes constant. The recovery for the first half minute is generally greater than 50% and slowly builds up to R_∞ with time.

Hence it would be reasonable to consider the Molybdenite system to contain particles which are very fast floating (i.e. K is high) and particles which are slow floating (i.e. K is low), each group behaving according to a first

order equation. This concept was first introduced by Kelsall⁽²⁴⁾ in terms of high and low probabilities of flotation of any mineral constituent. The graphical method suggested was to draw a straight line which was asymptotic to the curve wt. % remaining in tails vrs time. A second straight line was obtained by reading the values from the first straight line for different times and subtracting them from the corresponding values of the curve (Fig. 16). The fraction of the material having a low probability of flotation is given by the intersection of the line for the slow floating material and the time $t = 0$.

Kelsall⁽²⁴⁾ has shown that the probability can be considered equivalent to the rate coefficient in the case of batch flotation. Hence the batch flotation data may be interpreted in terms of two rate coefficients - K_s for slow floating particles and K_f for fast floating particles. The weight proportions of the slow and fast floating particles are given by the intersection of their lines with the y-axis (or at $t=0$).

The two-spike (two rate coefficients) approach was used in the analyses of the data obtained in the present investigation. It was difficult to obtain the straight line for fast floating particles as suggested by Kelsall.

However a straight line was drawn parallel to the tangent drawn to the curve at $t=0$ and passing through r_f (where r_f is the fraction of fast floating particles). Figs. 17 and 18 show the theoretical and experimental curves for tests 1 and 4 respectively. Curves of the same general form were obtained for other tests. The values of $\frac{C_0 - C_\infty}{C - C_\infty}$ for different values of time for theoretical and experimental curves were used to obtain cumulative recovery vrs time curves. Fig. 19 shows the theoretical (as predicted by 2-spike model) and the experimental recovery vrs time curves. Table 6 gives the values of Cum. Recovery as obtained experimentally and as predicted by the 2-spike model. As can be seen from fig 19 and table 6, the 2-spike model predicts the results as obtained experimentally with reasonable accuracy.

In summary then, it is observed that a modified first order kinetic equation does not characterize the molybdenite flotation system, but the system could be described, with reasonable accuracy, by the "2-spike" approach of rate coefficients.

Second Order Rate Equation

In order to test the second order kinetic theory for batch flotation, eq. (12) can be used. The values of recoveries for different times can be obtained from Appendix D.

Table 6 (a): Experimental and predicted values of
recovery for test 1 using "two-spike" model

Time (mts)	$R_{\text{Experimental}}$	$R_{\text{predicted}}$
0	0	0
0.5	59	59.2
1.0	67	68.8
2.0	70.5	73.0
4.0	72.9	73.7

Table 6 (b): Experimental and predicted values of
recovery for test 4 using "two-spike" model

Time (mts)	$R_{\text{Experimental}}$	$R_{\text{predicted}}$
0	0	0
0.5	62.2	62.2
1.0	69.0	70.8
2.0	74.1	74.2
4.0	74.2	74.9
8.0	75.7	75.7

Typical curves for t/R vrs t are shown in fig. 20 for tests 1 and 4, but the curves of the same general form could be drawn for the other tests from the data in Appendix D. As can be seen from fig. 20, and as was the case for all the tests, a straight line relationship between t/R and t , as predicted by the second-order kinetic theory, was obtained. Similar results were obtained by Arbiter⁽⁵⁾ for low grade molybdenite ore from Climax, Colorado.

The slope of the straight line gives the value of $1/R_{\infty}$ and the intercept at time $t = 0$ gives the value of $1/R_{\infty}^2 k_2'$. The values of intercept, slope and the calculated values of R_{∞} and k_2' are reported in Table 7.

This method of plotting (t/R vrs t) becomes insensitive at longer flotation times, since R is large and changes very slowly, and effectively time is plotted against time. The agreement of the earlier points with the straight line was obtained in all the tests and hence it was concluded that the system can be characterised by a second order equation.

Fig. 21 shows the plots for tests 1 to 5 obtained using a more sensitive method of plotting discussed by Hukki⁽⁷⁾. The values of R_{∞} obtained using this method are reported in Table 4. The values are in reasonably good agreement with the values obtained by other methods.

Table 7: R_{∞} and k_2' values obtained using Arbiter's second order equation

Test no.	Slope	Intercept	R_{∞}	k_2'
1	0.0133	0.10	75.0	0.00178
2	0.0131	0.20	76.3	0.00086
3	0.0133	0.15	75.0	0.00119
4	0.0129	0.08	77.3	0.00167
5	0.0120	0.15	83.3	0.00096
6	0.0127	0.12	78.6	0.00135
7	0.0125	0.15	80.0	0.00104
8	0.0120	0.15	83.3	0.00096
9	0.0133	0.10	75.0	0.00178
10	0.0129	0.10	77.3	0.00167
11	0.0145	0.15	68.8	0.00141
12	0.0140	0.20	71.4	0.00098
13	0.0140	0.20	71.4	0.00098
14	0.0138	0.20	72.2	0.00096

For the tests of 4 and 7 minute grinds (i.e. test 1 and 2 respectively) straight-line relationships between $1/t$ and $1/R$ were obtained. For tests 3 to 11, there was a slight deviation from the straight line for short times ($t < 1/2$ min.) of flotation. For tests done at very slow air flow rates (tests 12 and 13) and without collector (test 14), the plots deviate from a straight line. The deviations for flotation time less than one minute and the distinct curvature (fig. 22) suggest that the order of the reaction may be non-integral or may change from first order at the start of the experiment to second order at the end of the experiment.

Non-integral Order

An attempt was made to define the system using Holland-Balt's General Approach. Since this approach depends upon the slope of recovery vrs time curves at different values of recovery, it is very sensitive to the shape of the curve. For a very fast floating system, as in the present investigation, it is difficult, if not impossible, to get accurate values of slopes at different values of recoveries.

Models Using Distributed Rate Constants

While discussing the modified first order kinetic equation, it was seen that a single rate coefficient "K" was not sufficient to describe the flotation of the molybdenum, but that a model including two rate coefficients appeared to describe the overall system with reasonable accuracy.

At the expense of greater mathematical complexity the treatment of the flotation process as a first order rate equation with a distributed rate coefficient may lead to better accuracy, and should provide a model that more realistically describes the actual system.

The simplest method of kinetic interpretation of batch flotation data in terms of species distribution is the curve-fitting technique discussed by Harris and Chakravarti⁽²²⁾. The flotation system is characterized by two parameters, namely n , the argument of a Gamma function species distribution and K_m , the maximum value of "K" in a truncated species distribution. Fig. 23 shows the theoretical and experimental curves for test 9. The curves of the same general form could be drawn for the other tests from the data in Appendix F.

As seen from fig. 23, and as was the case for all other tests, the curve fitting technique failed to give an acceptable fit. The failure of the two parameter model

suggests that the species distribution may be bimodal; this is a reasonable development from the argument that the "2-spike" method appeared to be a reasonable initial interpretation.

Harris' 3-parameter model takes into account the bimodal nature of the species distribution. The flotation process is described in terms of slow and fast floating particles with different species distributions. The 3 parameters n , m and r are obtained by a simple graphical procedure discussed by Harris and Chakravarti⁽²²⁾. Fig. 24 shows the tangent technique used in obtaining the parameters for test 1. Similar curves could be drawn for the other tests from the data in Appendix F and the values of the parameters could be obtained. The values of m , n and r are reported in Table 8.

In order to show the effect of the 3-parameter model, the species distribution was plotted as $\Psi(K, 0)$ vrs K using the equation

$$\Psi(K, 0) = \frac{r}{\Gamma(n)} K^{n-1} e^{-K} + \frac{1-r}{\Gamma(m)} K^{m-1} e^{-K}$$

where

r is the fraction of slow floating material and n and m are arguments of the Gamma function species distribution.

Typical curves for $\Psi(K, 0)$ vrs K are shown in Fig. 25 for tests 6, 9, 11 and 12. Curves of the same general form could be drawn for the other tests from the data in Appendix F.

Harris reported that the species distribution, or its parameters, may be used as performance indices in studying the flotation process and the effects of operating variables. The parameters of the model depend upon the accuracy with which the tangents are drawn to the $f(t)$ vrs t curves. A slight inaccuracy in drawing these tangents could lead to different values of the parameters n , m and r . Fig. 26 shows the effect of different values of m on the $\Psi(K, 0)$ vrs K curve for test 12. It is seen that systematic changes in m do not produce systematic changes in the $\Psi(K, 0)$ curve. Since the changes produced in the curve are not systematic, it is not possible to correlate the change in shape of the curve to the change in air flow rate or grind time. Also it has been shown by previous investigators⁽²²⁾ that widely different species distributions can generate similar $f(t)$ vrs time curves (or R vrs t). Hence it is reasonable to say that the use of species distribution as a performance index in studying the flotation process and the effect of operating variables on it is not justifiable in the present case.

Table 8: The values of r , n and m of the 3-parameter
or "2-spike" model

Test no.	r	n	m
1	0.145	1.08	6.0
2	0.096	0.35	5.11
3	0.135	0.88	4.29
4	0.11	0.78	5.13
5	0.136	0.78	5.11
6	0.118	0.76	4.18
7	0.088	0.63	4.19
8	0.120	0.91	5.09
9	0.075	0.60	5.06
10	0.135	1.07	7.13
11	0.166	1.15	5.75
12	0.17	0.98	5.53
13	0.175	1.12	3.93
14	0.155	0.70	3.77

Effect of Air Flow Rate and Grind on the Rate of Flotation

The screen analysis of the feed and the tailings for tests 1 to 8 are given in Appendix E.

An attempt was made to correlate R_{∞} , grind time, air flow rate and recovery. Table 9 and 10 report the values of r , m and n of the 3-parameter or the 2-spike model for different grinds (at air flow rate of 5.61 l/min.) and for different air flow rates (and for 7 minute grind) respectively. No general trends in relationships were found between air flow rate and recovery, grind time and recovery, and R_{∞} and grind time. It was not possible to find a general trend of the effects of grinding time and air flow rates upon the kinetics of the flotation of the molybdenite ore.

The estimates of the reliability of the flotation experiments normally are expressed in terms of confidence limits. The replicate tests 2, 6 and 7 were analysed in terms of means and standard deviations. Fig. 27 gives the 95% confidence limits for these tests. The smaller the range, the more reproducible and reliable the data. For the molybdenite system, the confidence limits are large and the reproducibility of the data is not very good.

The reasons for the low degree of reproducibility may be due to any or all of the following: -

Table 9: The values of r , m and n of the 3-parameter or "2-spike" model for different grinds at air rate of 5.61 l/min.

Test no.	Grind time (mts)	r	n	m
1	4	0.145	1.08	6.0
2	7	0.096	0.35	5.11
6	7	0.1175	0.76	4.18
7	7	0.088	0.63	4.19
3	10	0.135	0.88	4.29
4	13	0.11	0.78	5.13
8	13	0.12	0.91	5.09
5	16	0.136	0.78	5.11

Table 10: The values of r , m and n of the 3-parameter or "2-spike" model for different air flow rates (and for 7 minute grind)

Test no.	Air flow rate (l/min.)	r	n	m
12	4.62	0.17	0.98	5.53
13	4.62	0.175	1.12	3.93
2	5.61	0.096	0.35	5.11
6	5.61	0.1175	0.76	4.18
7	5.61	0.088	0.63	4.19
9	7.64	0.075	0.60	5.06
10	7.64	0.135	1.07	7.13
11	9.68	0.166	1.15	5.75

- (i) experimental techniques
- (ii) errors in chemical analysis
- (iii) non-homogeneity of feed material.

The use of automatic froth paddles, and the fact that pulp levels was held steady throughout the tests reduced experimental errors as far as possible. Timed samples were collected in the pans. There could have been a possibility of errors of a few seconds in taking the timed samples. However, the values of R_{∞} should be reproducible. This was not the case in the present investigation. Dell et al⁽³⁶⁾ and Climax Research Lab.⁽³⁷⁾ have also reported difficulty in reproducing R_{∞} , the former on other ores, using other equipment, the latter treating the same ore as used in this investigation. There are possibilities of slight error in the chemical analysis of the samples. But R_{∞} being different for different tests suggests that the feed solids were not homogeneous, or that the test procedure is intrinsically not reproducible.

In summary, due to the fast floating rate of molybdenite, the degree of reproducibility of the data was not good. Hence, it was not possible to arrive at any conclusive decisions regarding the effect of grind and air flow rate on the flotation of molybdenite within the range of experimental conditions studied.

CONCLUSIONS

The kinetic analysis of the flotation of molybdenite in this investigation has led to two different approaches for establishing a mathematical model of the process.

First, the system could be described using Arbiter's second order equation. The results could be interpreted in terms of k_2' , the rate coefficient. If systematic changes in flotation variables produce corresponding changes in k_2' , then the model can be used for practical purposes. The simpler the model, the more attractive it will be to plant operators as an aid to optimization of flotation circuits.

Second, the "two-spike" model has been proposed to describe the flotation process. The procedure for obtaining K_s and K_f (which are same as n and m respectively) is simple, and the recoveries predicted by this model are in reasonably good agreement with the experimental results.

The extension of the two-spike model is the distribution of the rate coefficients. This is incorporated in Harris'

3-parameter model which makes use of the species distribution curves to predict the performance of the flotation system. The procedure for obtaining $\Psi(K,0)$ curves using the Gamma functions require tedious calculations. It has been seen that systematic changes in the parameters do not produce systematic changes in $\Psi(K,0)$ curves. From the point of view of practical application, the method does not seem to offer any advantages over the "2-spike" model.

In summary, the second order equation or the "two-spike" model could be used for describing the molybdenite system. The two-spike model (with parameters K_s , K_f and r_s) also gives the fraction of fast and slow floating particles (r_f and r_s respectively).

Due to the poor reproducibility of the results, no conclusions could be drawn regarding the effect of variables on the flotation process. The changes in air flow rate and grind times could not be related to the parameters K_s , K_f and r_s within the present range of experimental conditions.

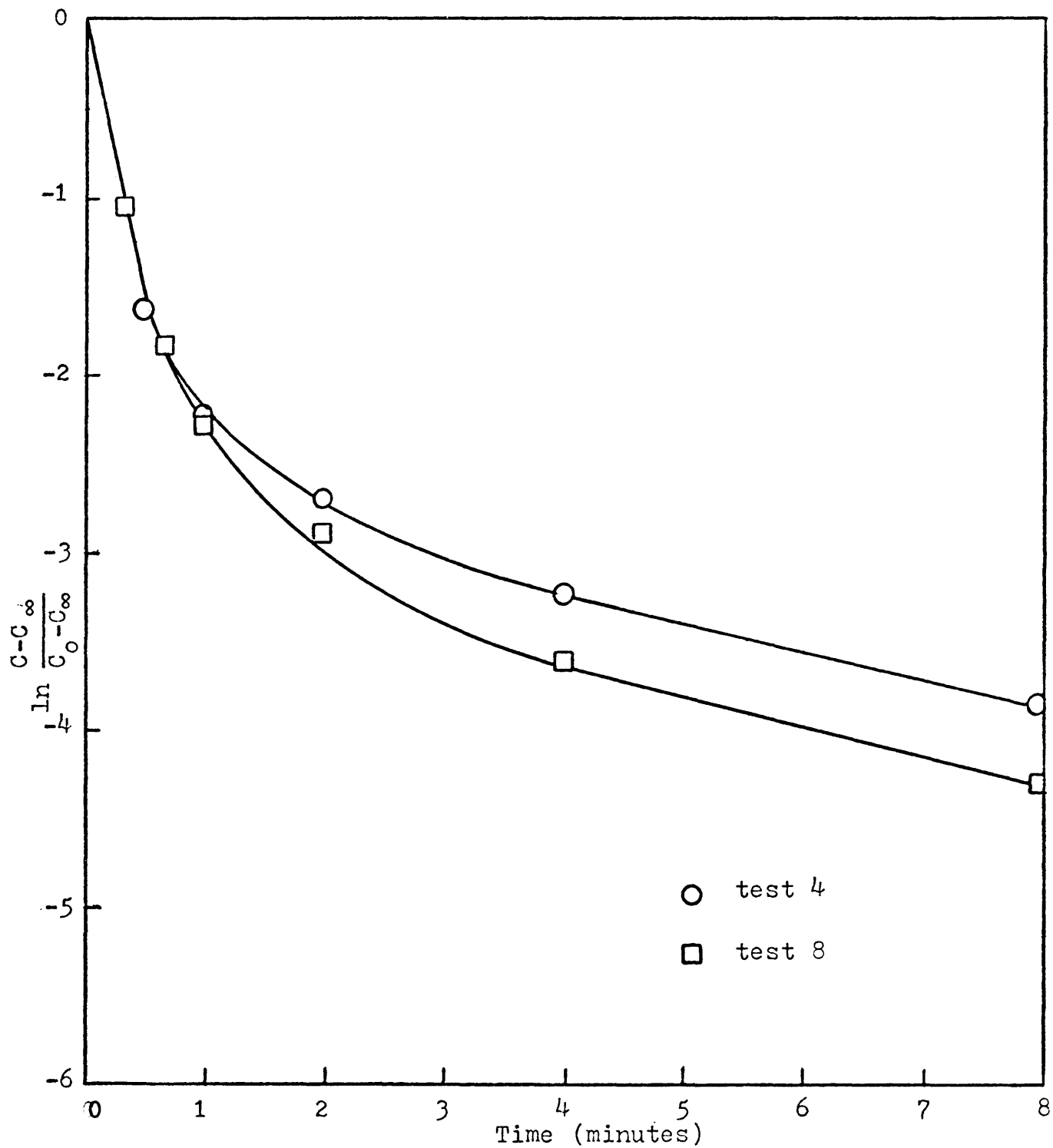


Figure 13. Plot of $\ln \frac{C - C_{\infty}}{C_0 - C_{\infty}}$ against time for tests 4 and 8 conducted under identical conditions (13 min grind and 5.61 l/min air flow rate).

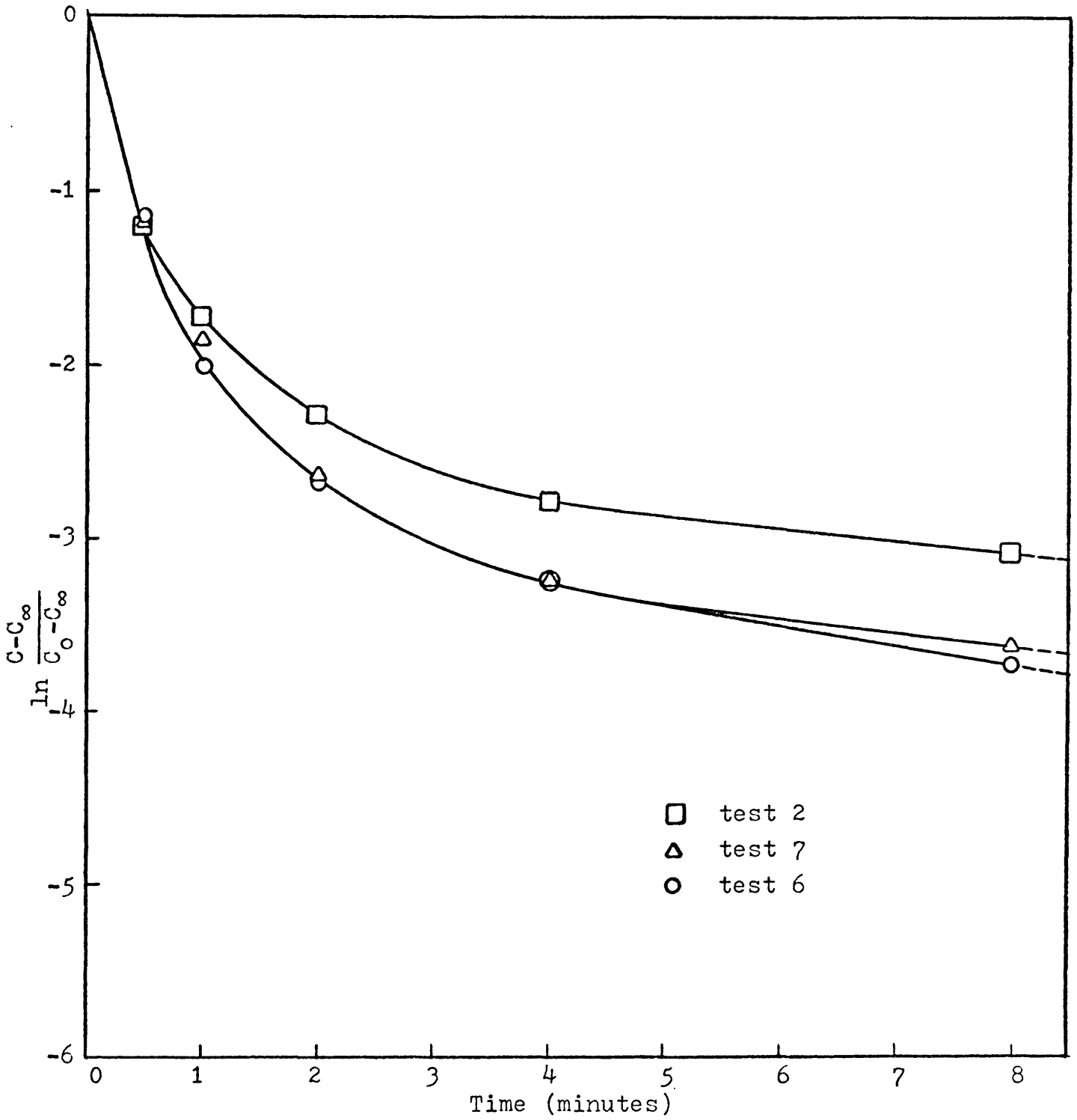


Figure 14. Plot of $\ln \frac{C-C_\infty}{C_0-C_\infty}$ against time for tests 2, 6 and 7 conducted under identical conditions (7 min grind and 5.61 l/min air flow rate).

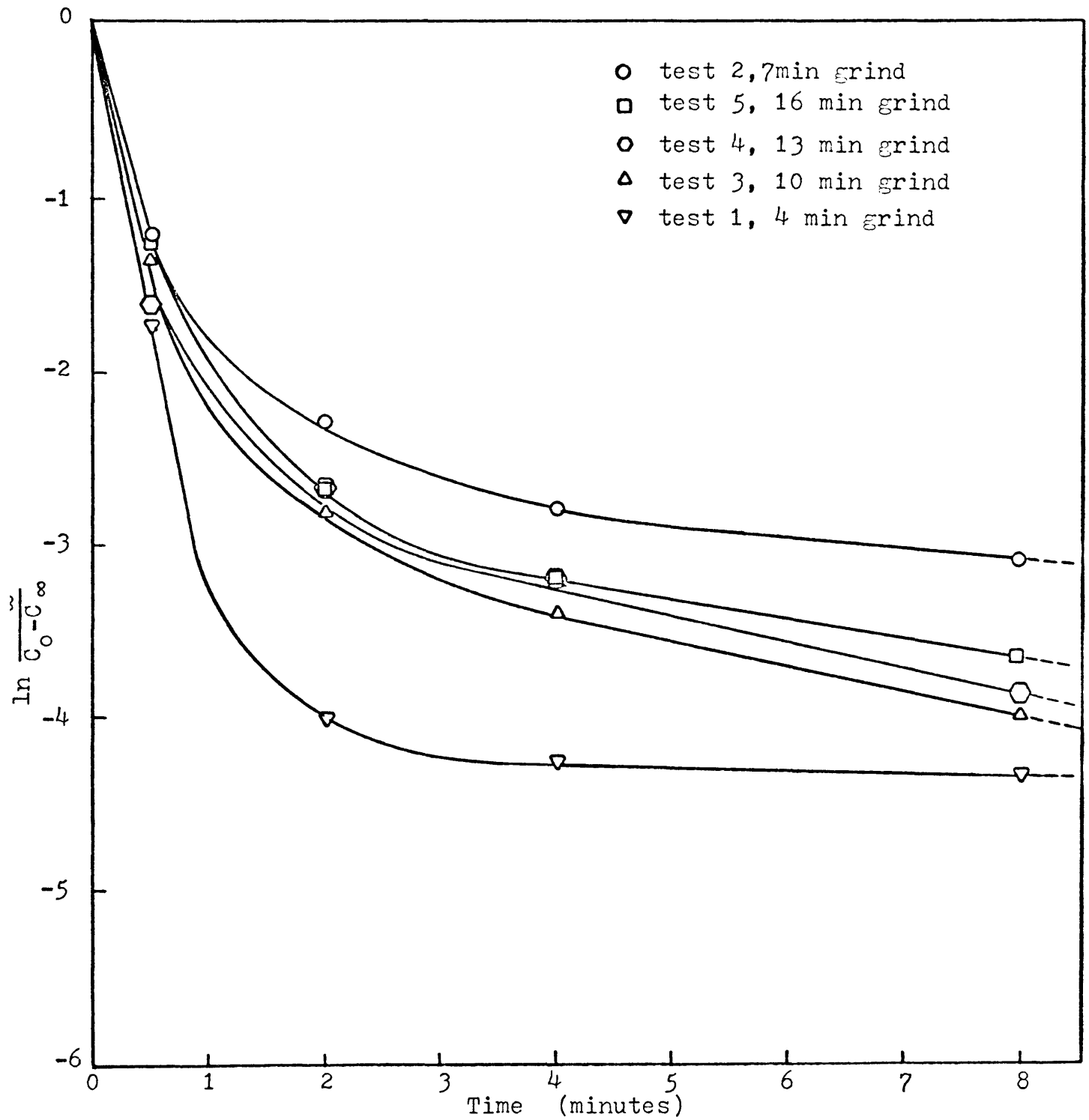


Figure 15. Plot of $\ln \frac{C-C_{\infty}}{C_0-C_{\infty}}$ against time for tests 1 through 5
at 5.61 l/min of air flow rate

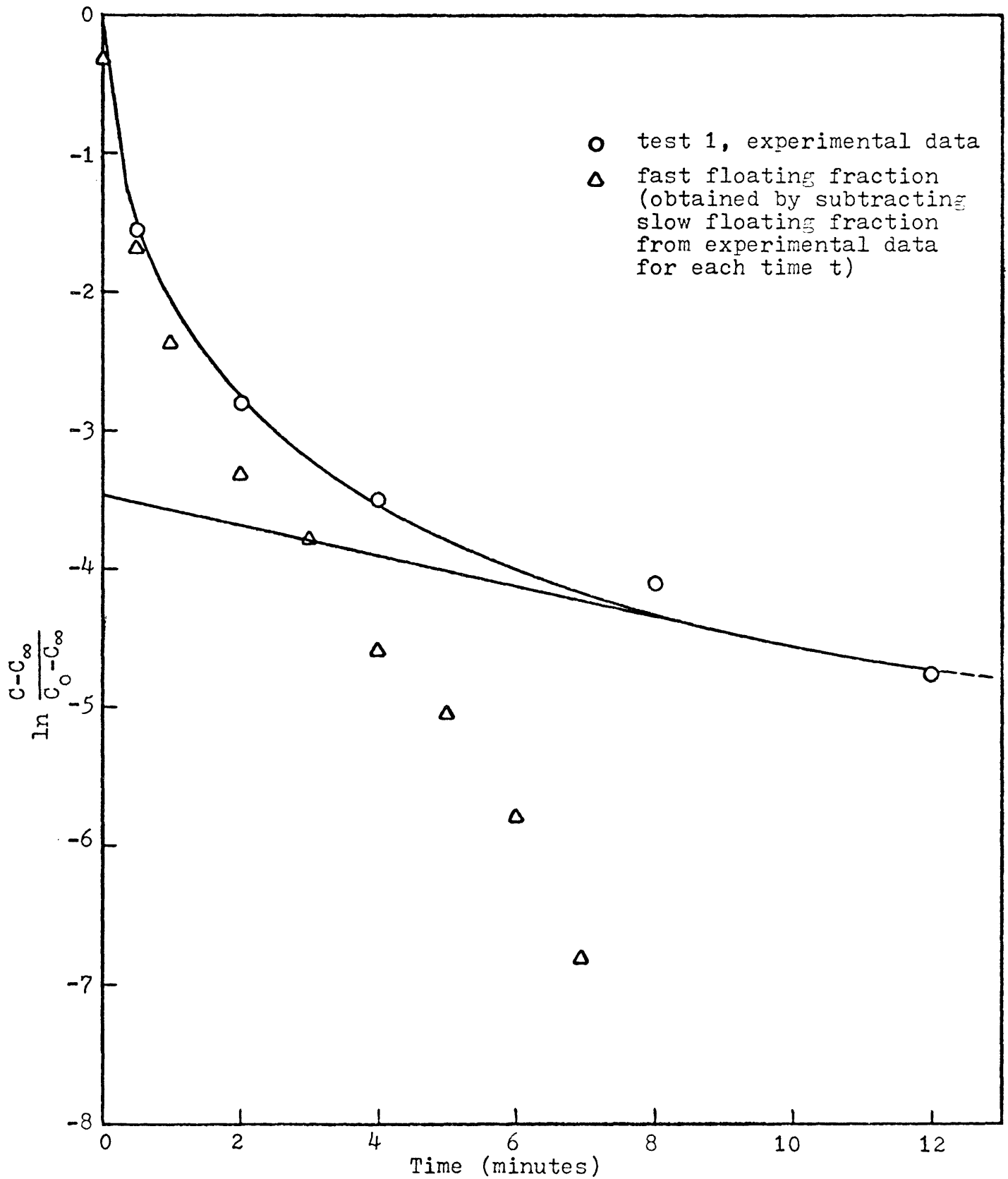


Figure 16. Kelsall's graphical method for obtaining slow and fast floating fractions for any minerals.

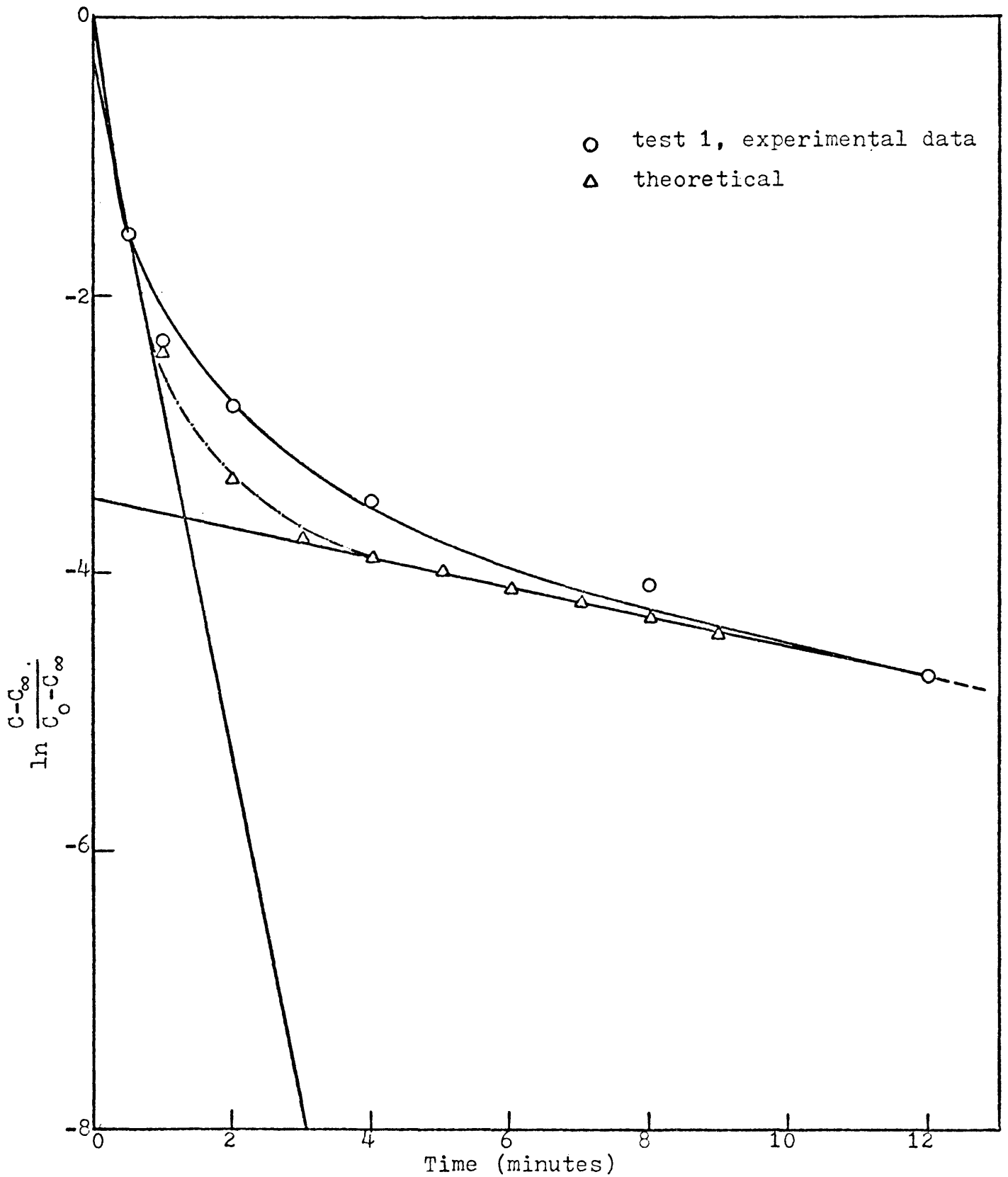


Figure 17. Experimental and theoretical curves using modified

Kelsall's graphical procedure for obtaining "2-spike

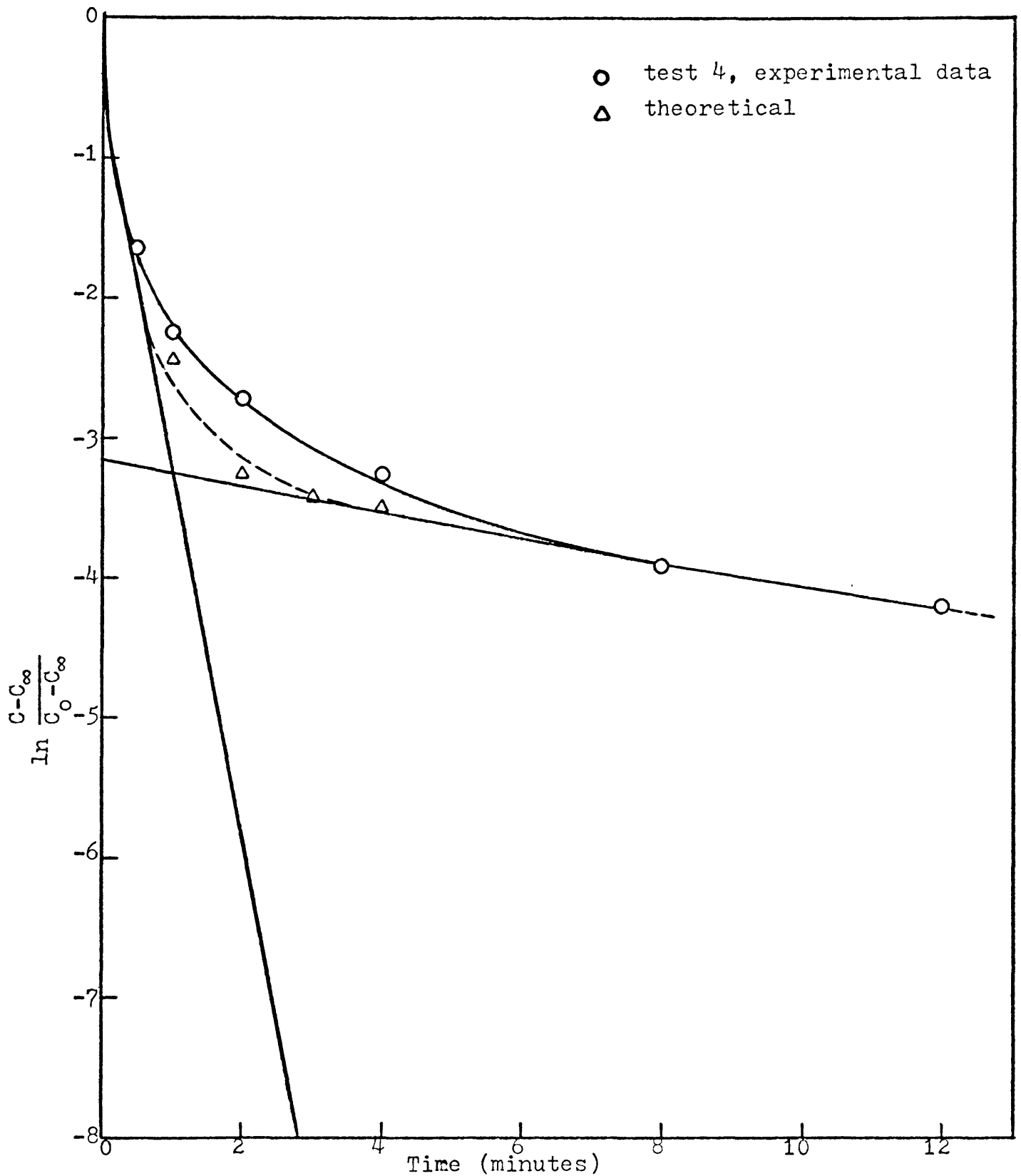


Figure 18. Experimental and theoretical curves using modified Kelsall's graphical procedure for obtaining "2-spike" model for test 4.

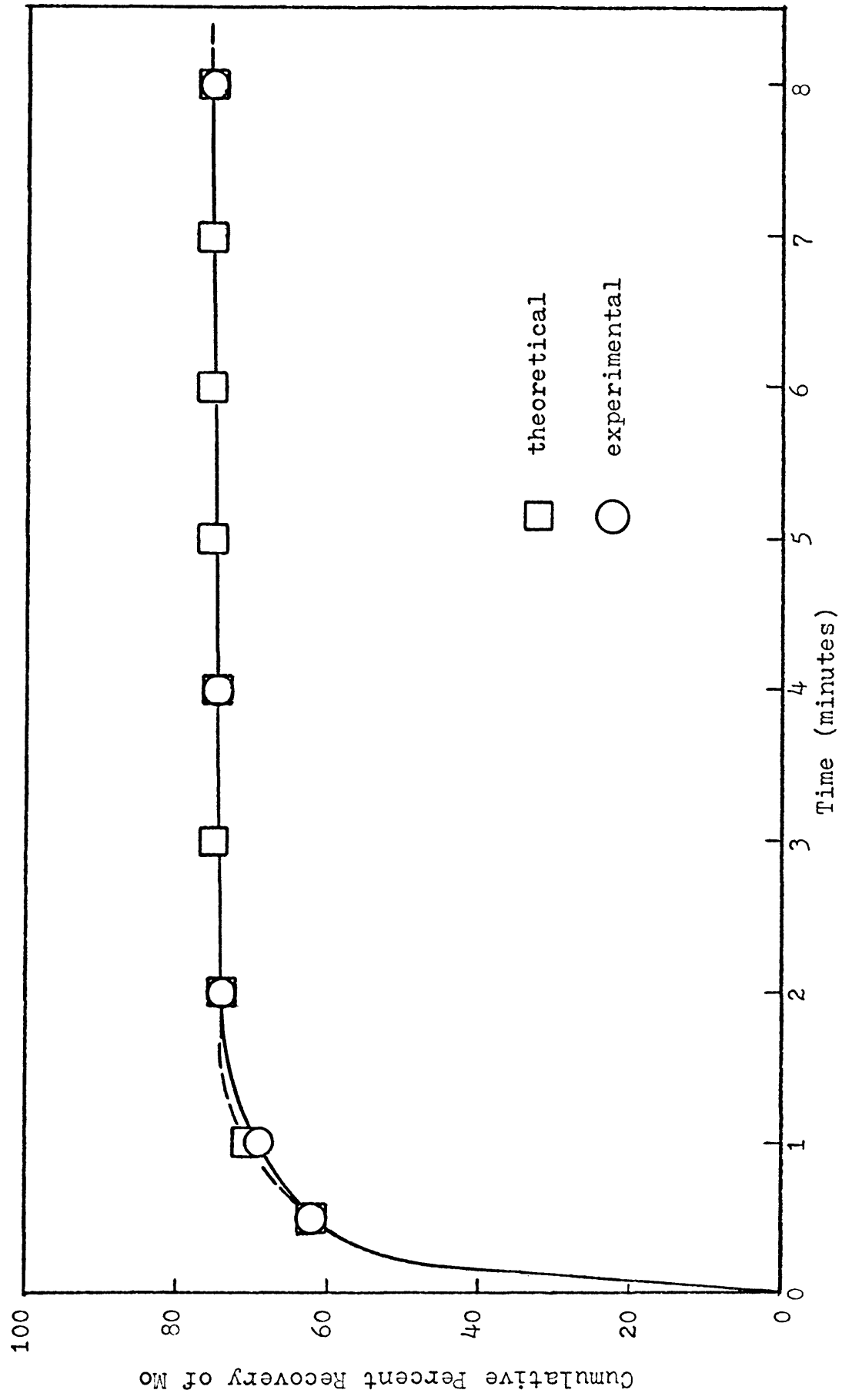


Figure 19. Theoretical (as predicted by 2-spike model) and experimental recovery-time curves for test 4.

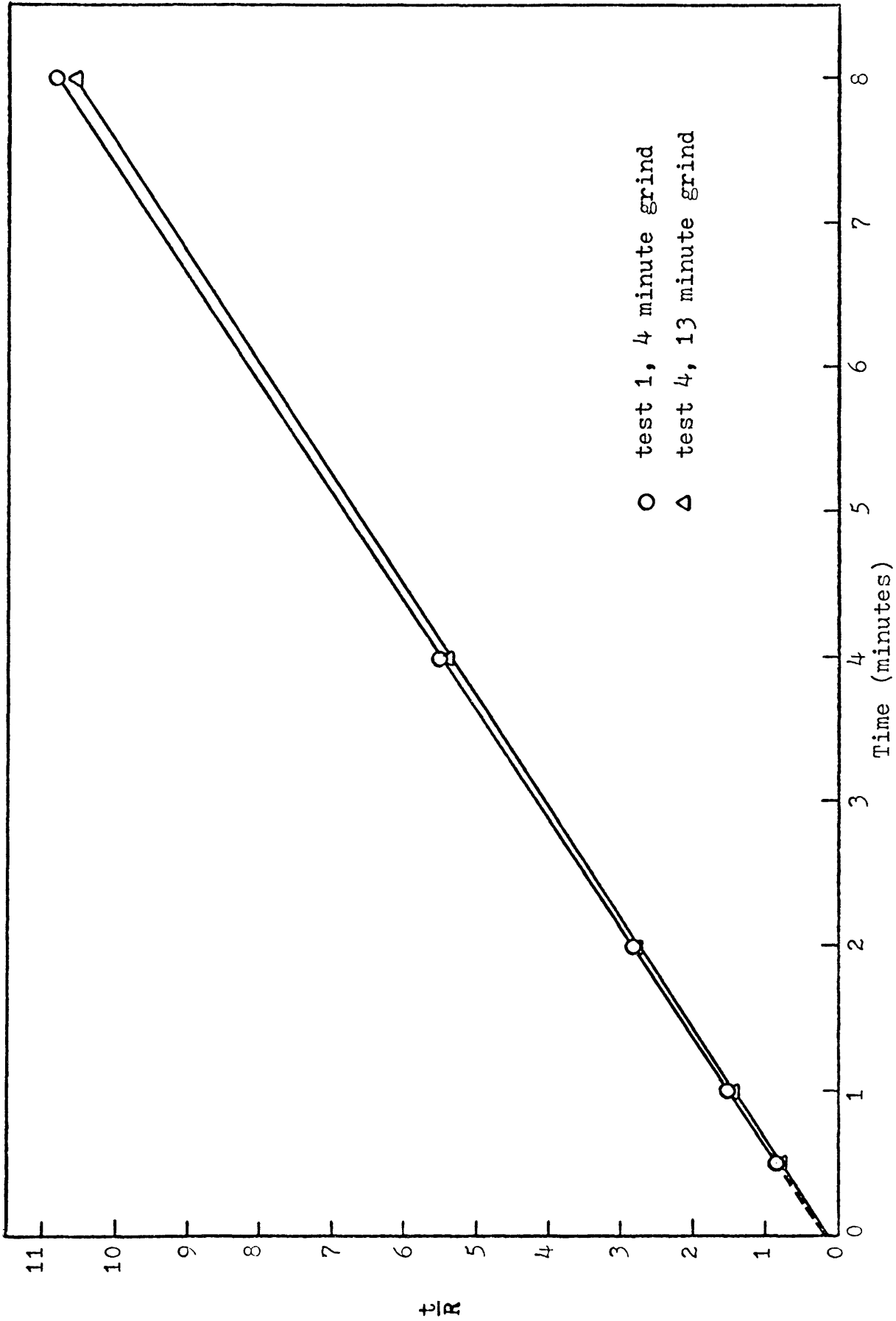


Figure 20. $\frac{t}{R}$ vs t curves for test 1 and 4.

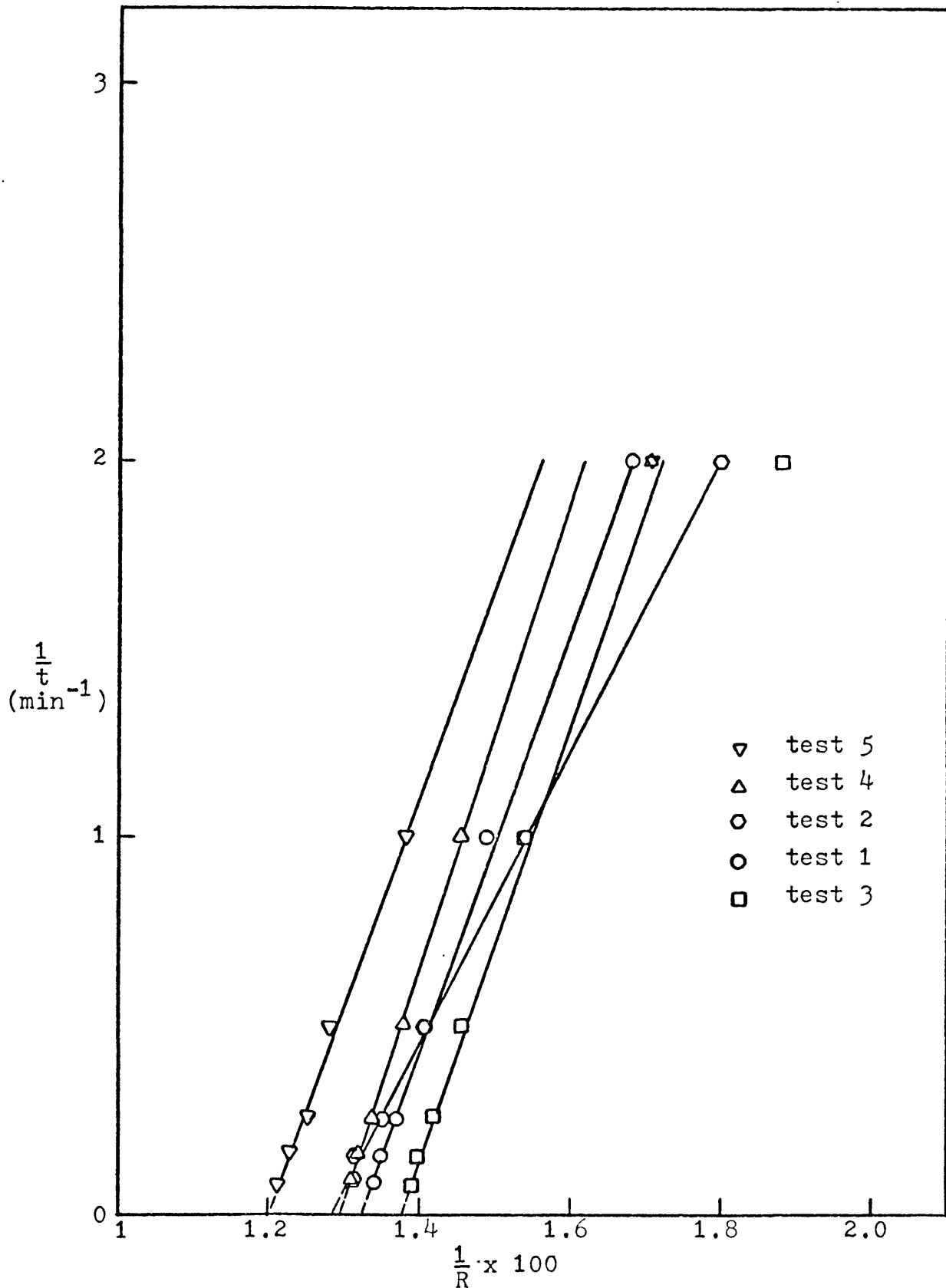


Figure 21. $\frac{1}{R}$ vrs $\frac{1}{R}$ plots for tests 1 to 5 using Hukki's⁽⁷⁾ modified second order equation.

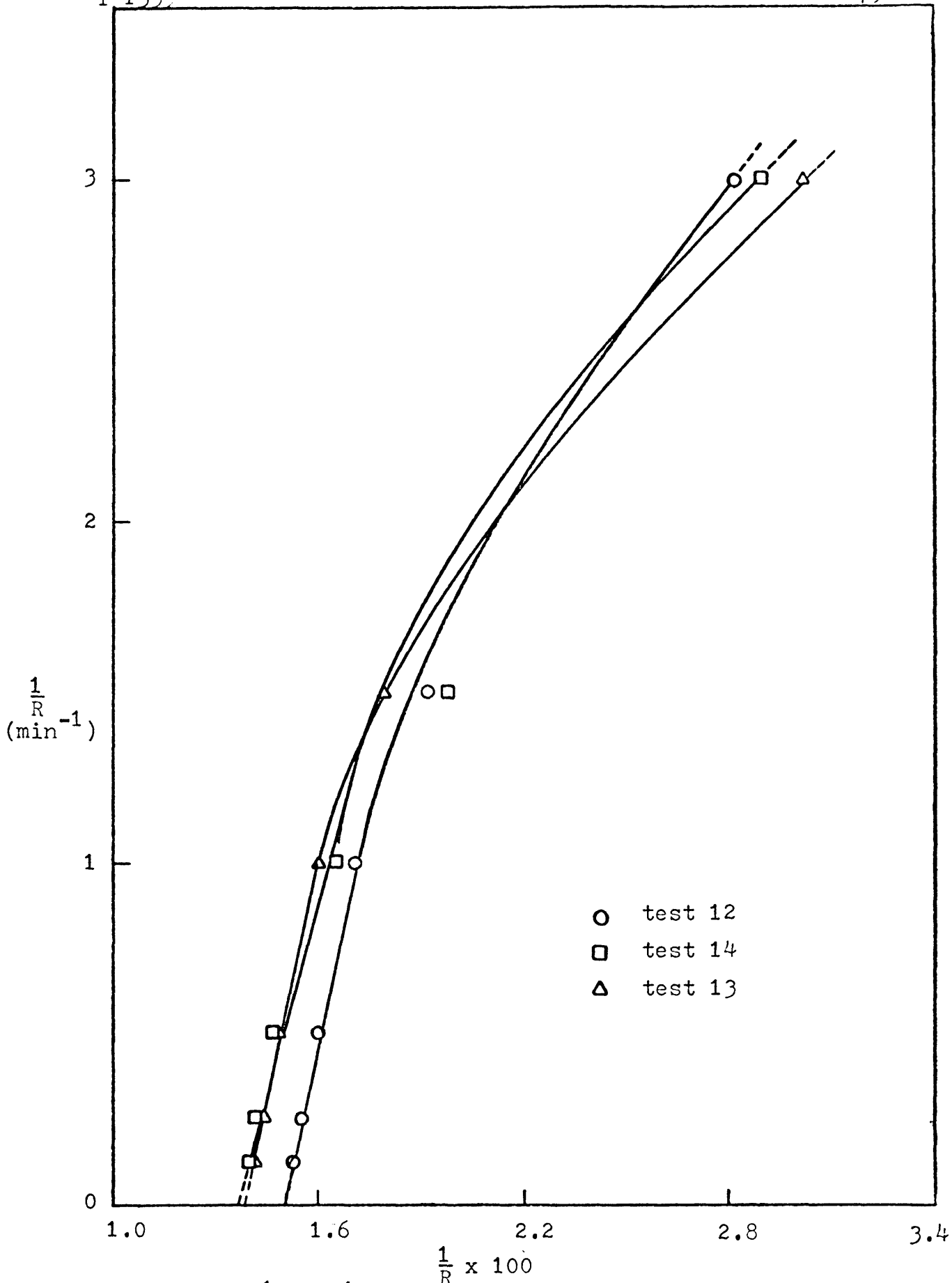


Figure 22. $\frac{1}{R}$ vrs $\frac{1}{R}$ plots for tests 12,13 and 14 using Hukki's⁽⁸⁾ modified second order equation.

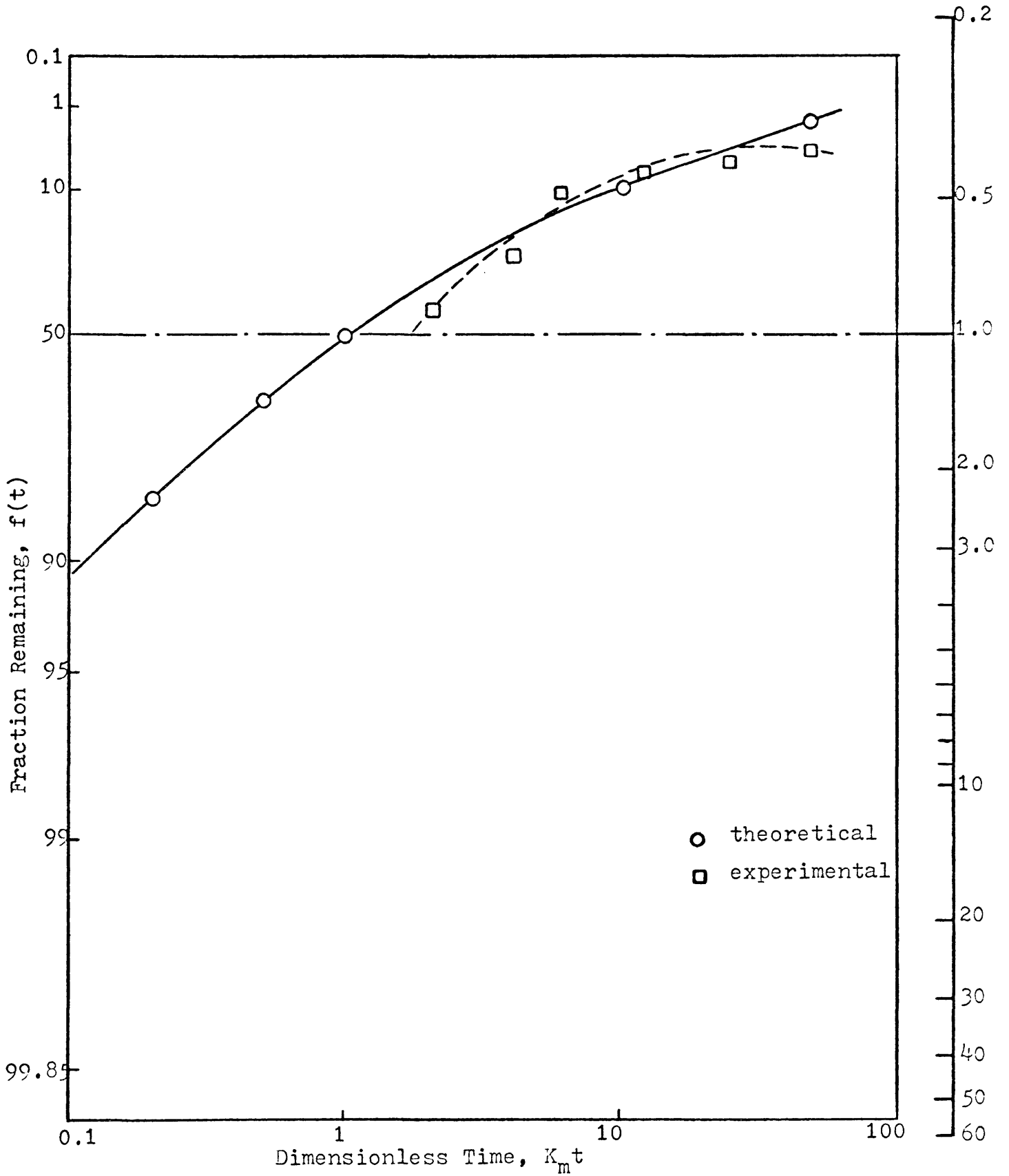


Figure 23. Curve-fitting technique of Harri's and Chakravarti⁽²³⁾.

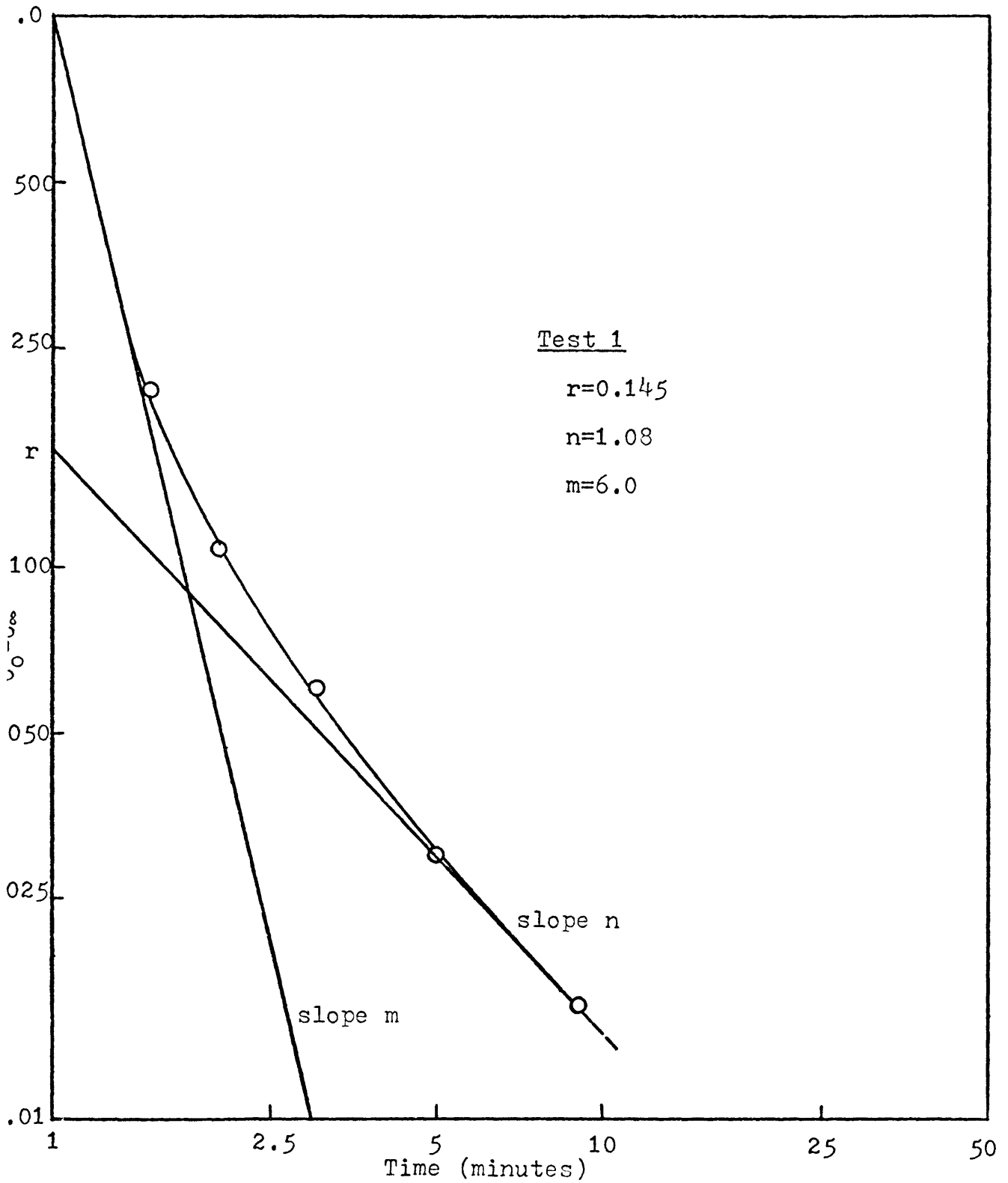


Figure 24. Graphical technique for obtaining parameters r , m and n for Harris' 3-parameter model.

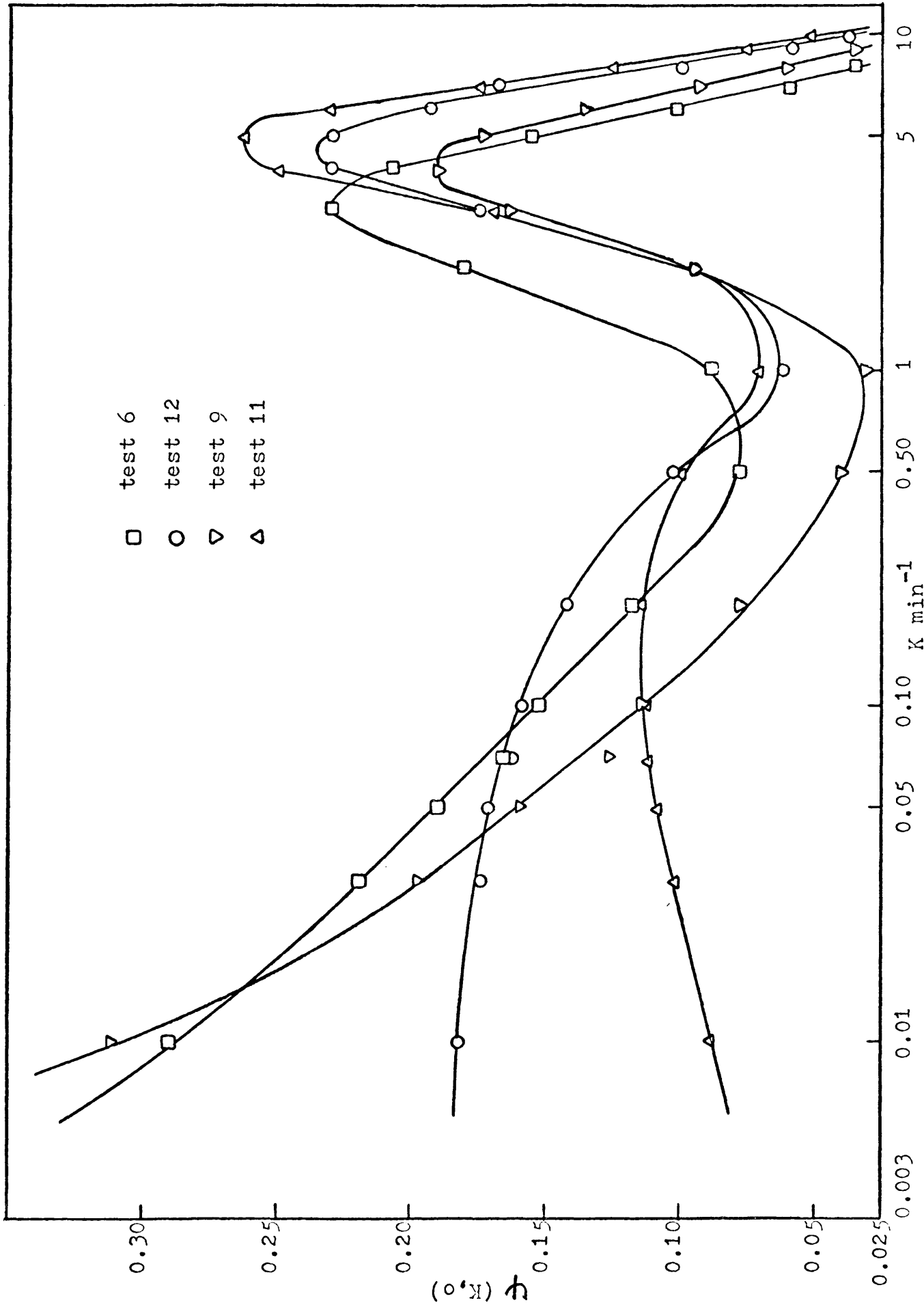


Figure 25. Species distribution plot of $\Psi(K,o)$ vs K for tests 6, 9, 11 and 12.

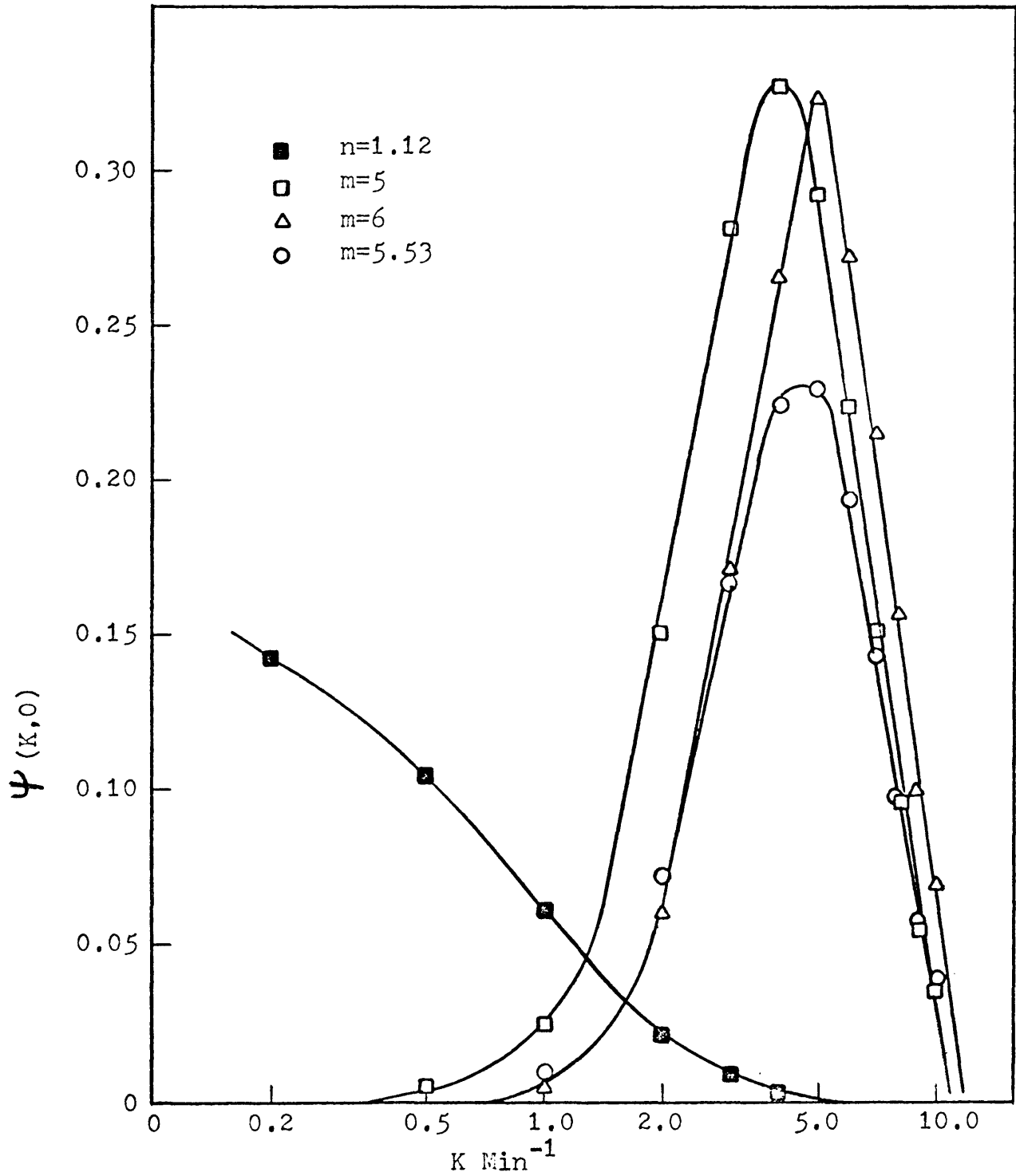


Figure 26. Species distribution: plot of $\Psi(K,0)$ vs K for different values of m for test 12 ($r=0.17$, $n=1.12$, $m=5.53$).

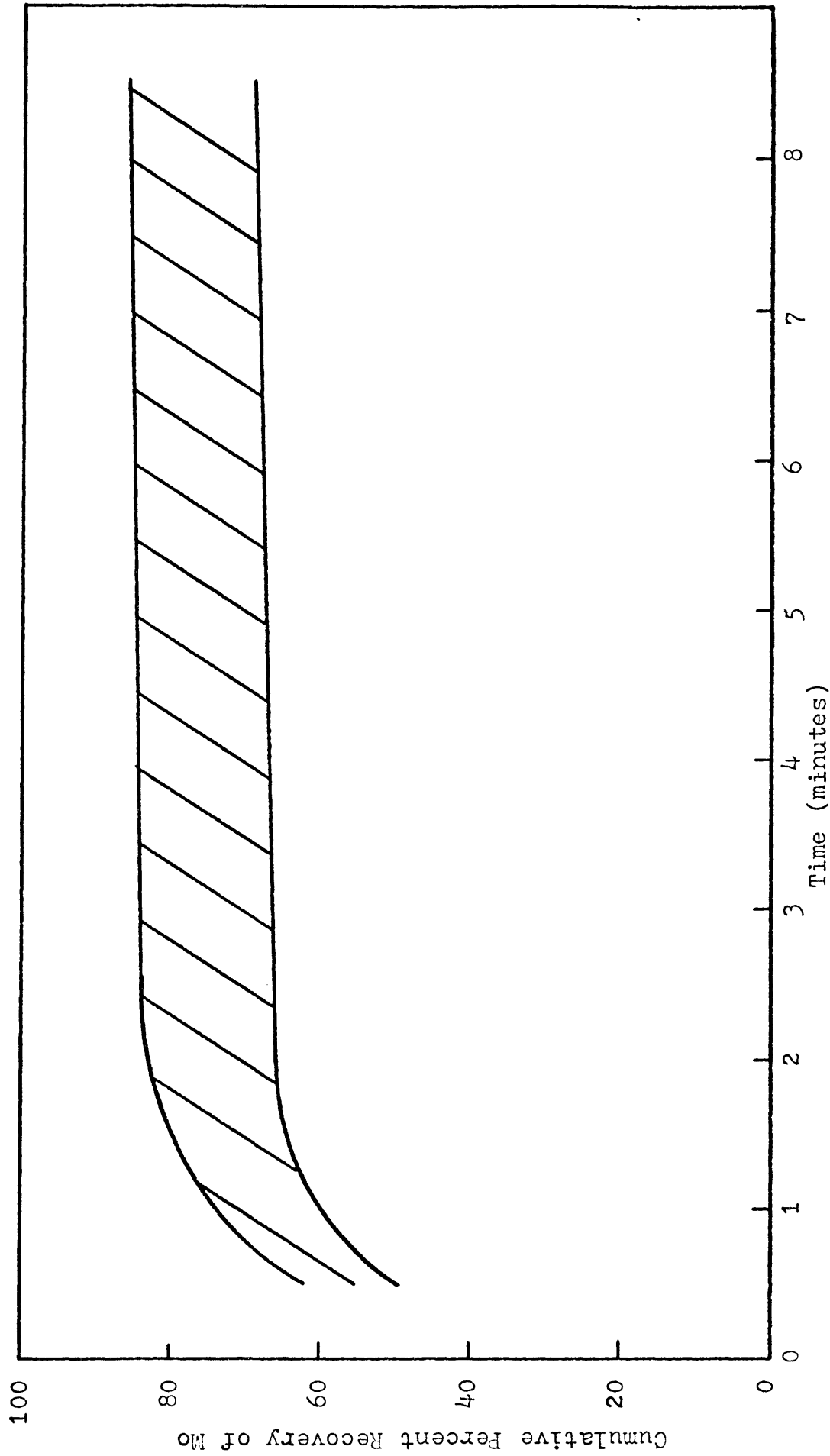


Figure 27. Hatched region showing the 95% confidence limits for tests 2, 6, and 7 conducted under identical conditions (7min grind and 5.61 l/min air flow rate).

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APPENDIX ADerivation of the Equation Used by Hukki (8)

Equation 11 gives

$$R = \frac{R_{\infty}^2 k_2' t}{1 + R_{\infty} k_2' t}$$

$$R + RR_{\infty} k_2' t = R_{\infty}^2 k_2' t$$

$$t(R_{\infty}^2 k_2' - R_{\infty} k_2' R) = R$$

$$\frac{1}{t} = \frac{1}{R} (R_{\infty}^2 k_2' - R_{\infty} k_2' R)$$

$$\frac{1}{t} = \frac{R_{\infty}^2 k_2'}{R} - R_{\infty} k_2'$$

This is of the form

$$y = mx + c$$

where

$$y = \frac{1}{t}$$

$$x = \frac{1}{R}$$

$$m = R_{\infty}^2 k_2'$$

$$c = -R_{\infty} k_2'$$

APPENDIX BUse of Reagents in the Flotation of Molybdenite

The following reagents are added to the mill prior to grinding:

H₂O - 1/2 weight of ore so resulting % solids is 67%

Ca(OH)₂ - 0.5 lbs/Ton

"N" Sodium silicate - 0.5 lb/T (dry basis)

P₂S₅ - 0.06 lb/T

NaOH - 0.12 lb/T

NaCN - 0.005 lb/T

Vapor Oil - 0.5 lb/T

Syntex - 0.03 lb/T

2 drops of pine oil were added to the pulp in the flotation cell.

The flotation of molybdenite is done with the aid of a unique reagent combination. The amounts of these reagents used were discussed above. The major collector is the Pale Neutral Oil vis. 100, locally called 'Vapor Oil.' Improved

dispersion of this oil is obtained by the use of Syntex L, a sulfated coconut oil. Sodium silicate and soda ash are employed for slime deflocculation as well as pH control and sulfide gangue minerals are depressed with cyanide and P_2S_5 -NaOH combination. Pine oil is used as a frother.

APPENDIX C

Determination of Molybdenum in Ores and Mill Products⁽⁴⁰⁾

1. Scope

1.1 This method covers the determination of molybdenum in concentrations of 0.005% to 15%.

2. Reagents

- 2.1 Saturated Ammonium acetate solution: Dissolve 2268 grams of $\text{NH}_4\text{C}_2\text{H}_3\text{OH}$ in two litres of water.
- 2.2 Lead Nitrate Solution (2%): Dissolve 20.0 grams of lead nitrate in water and dilute to one litre.
- 2.3 Methyl Orange Indicator (0.1%): Dissolve 0.1 gram of methyl orange in water and dilute to 100 ml.
- 2.4 Tannic Acid Indicator: Dissolve 1.0 gram of tannic acid in 300 ml. of water.

3. Procedure

3.1 Depending upon the molybdenum concentration, weigh from 0.5 to 5.0 grams of sample and transfer to a 250 ml. beaker. Add 10 ml. of nitric acid and 10 ml. of perchloric acid to the sample,

cover and heat at boiling temperature on the hot plate for 20 minutes. Then heat rapidly to fumes of perchloric acid (Note 1). Cool for 3 minutes, add 5 ml. of HCl acid and heat to boiling. Wash cover and sides of the beaker and dilute the solution to about 50 ml. with distilled water.

Note 1: A multiple unit shaking hot plate is a valuable time saver for this step, but if not available, hold the beaker with tongs, and with a swirling motion to prevent spitting, heat the solution over a bunsen burner to perchloric acid fumes.

- 3.2 Precipitate the iron with 20 ml. of ammonium hydroxide, heat to boiling and filter through an S and S 597 paper, or equivalent, containing paper pulp. Collect the filtrate in a 600 ml. beaker and wash the precipitate four times with hot water (Note 2).

Note 2: Careful washing techniques should be observed at this point to avoid the danger of washing some colloidal iron and silica through the paper thus causing high results.

- 3.3 With a stream of water wash the hydroxide precipitate from the filter paper into the

original beaker and add 5 ml. of hydrochloric acid to dissolve the hydroxide precipitate. Stir and reprecipitate the iron with 15 ml. of ammonium hydroxide, again stir and, without boiling, refilter the solution through the same filter paper as used in the first filtration. Wash the precipitate four times with hot water. Discard the precipitate.

- 3.4 To the filtrate add ash free analytical filter pulp and two drops of methyl orange indicator solution and neutralize (orange to pink) with hydrochloric acid. Add 10 ml. of ammonium acetate solution, 3 ml. of acetic acid and heat it for some time. Then precipitate the molybdenum with lead nitrate solution. Add the solution from a buret using tannic acid solution as an outside indicator. The end point is reached when a drop of the indicator fails to show a brown coloration. Then add 3 ml. of lead nitrate solution in excess. Cover the beaker, heat nearly to boiling on the hot plate and allow the precipitate to settle from the hot solution for one hour.
- 3.5 Decant the clear solution through an S and S 589 White Ribbon Filter paper, or equivalent, containing ashless paper pulp and finally wash the precipitate into the

filter paper. With a rubber "policeman" carefully scrub off any precipitate adhering to the sides of the beaker and wash into the filter. Wash the paper and precipitate four times with hot water.

- 3.6 Fold the paper around the precipitate, place it in a No. 0 for clay crucible and ignite the paper in a muffle furnace at a temperature not to exceed 550°C, for 40 minutes (Note 3). Remove from the furnace, cool to room temperature and brush the precipitate onto a weighing pan and weigh.

Note 3: Lead molybdate has been found to volatilize to an appreciable degree at temperatures higher than 600°C. To prevent loss, the precipitate must be ignited at a temperature no higher than is necessary to ash the filter paper.

- 3.7 Calculations:

$$\text{Mo (\%)} = \frac{A \times 0.2613}{B} \times 100$$

where:

A = weight of lead molybdate

B = weight of sample in grams .

APPENDIX D

Cumulative Recovery and Grade for Tests 1 through 14

Tables 1-D through 14-D show the relevant kinetic data for each flotation test.

Table 1-D: Cumulative Recovery and Grade for Test 1 (Head pH=9.10, tail pH 8.5)

Time (secs)	Wt.of Concentrate (gms)	Grade (% Mo)	Recovery %Mo	Cumulative Recovery (% Mo)
30	17.9	10.320	59.26	59.26
60	9.0	2.705	7.81	67.07
120	8.4	1.305	3.52	70.59
240	15.5	0.465	2.31	72.90
480	10.8	0.310	1.07	73.97
720	6.9	0.260	0.57	74.54
tailings	927.4	0.092	-	-

Table 2-D: Cumulative Recovery and Grade for Test 2 (Head pH=9.15; tail pH=9.0)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
30	11.5	15.06	55.29	55.29
60	6.7	4.51	9.64	64.93
120	6.6	2.82	5.94	70.87
240	12.6	0.77	3.08	73.95
480	12.8	0.31	1.27	75.22
720	9.1	0.16	0.45	75.67
tailings	941.3	0.077	-	-

Table 3-D: Cumulative Recovery and Grade for Test 3 (Head pH=9.0; tail pH=8.9)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
30	13.5	12.2	53.04	53.04
60	10.4	3.48	11.65	64.69
120	8.7	1.32	3.70	68.39
240	14.1	0.43	1.93	70.32
480	17.8	0.19	1.09	71.41
720	6.9	0.17	0.38	71.79
tailings	920.7	0.037	-	-

Table 4-D: Cumulative Recovery and Grade for Test 4 (Head pH=9.1; tail pH=8.9)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
30	15.4	12.60	62.20	62.20
60	7.8	2.70	6.75	68.95
120	8.0	1.22	3.12	72.07
240	16.0	0.42	2.15	74.22
480	22.3	0.20	1.43	76.65
720	7.0	0.19	0.41	77.06
tailings	920.2	0.0165	-	-

Table 5-D: Cumulative Recovery and Grade for Test 5 (Head pH=9.3; tail pH=9.0)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
30	12.1	15.97	61.77	61.77
60	7.0	4.73	10.58	72.35
120	5.5	3.03	5.33	77.68
240	13.1	0.55	2.30	79.98
480	14.2	0.28	1.25	81.23
720	12.5	0.23	1.04	82.27
tailings	935.1	0.01	-	-

Table 6-D: Cumulative Recovery and Grade for Test 6 (Head pH=9.2; tail pH=9.0)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
30	11.3	14.87	53.24	53.24
60	9.7	4.50	13.82	67.06
120	7.8	2.03	5.01	72.07
240	9.0	0.78	2.22	74.29
480	9.3	0.39	1.14	75.43
720	7.3	0.24	0.56	75.99
tailings	955.3	0.038	-	-

Table 7-D: Cumulative Recovery and Grade for Test 7 (Head pH=9.05; tail pH=8.8)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
30	13.8	13.29	58.06	58.06
60	7.6	5.20	12.50	70.56
120	9.7	2.25	6.90	77.46
240	10.8	0.78	2.67	80.13
480	6.9	0.48	1.04	81.17
720	6.2	0.34	0.66	81.83
tailings	954.0	0.0634	-	-

Table 8-D: Cumulative Recovery and Grade for Test 8 (Head pH=9.05; tail pH=8.8)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
30	14.7	12.61	59.73	59.73
60	10.6	3.44	11.71	71.44
120	8.8	1.59	4.50	75.94
240	10.2	0.51	1.68	77.62
480	11.3	0.48	1.74	79.36
720	9.2	0.32	0.95	80.31
tailings	926.7	0.04	-	-

Table 9-D: Cumulative Recovery and Grade for Test 9 (Head pH=9.2; tail pH=9.1)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
20	12.9	12.79	52.65	52.65
40	10.2	3.69	12.01	64.66
60	8.1	1.73	4.47	69.13
120	13.7	0.97	4.23	73.36
240	14.1	0.38	1.71	75.07
480	5.8	0.31	0.57	75.64
720	6.5	0.27	0.55	76.19
tailings	930.0	0.055	-	-

Table 10-D: Cumulative Recovery and Grade for Test 10 (Head pH=9.2; tail pH=9.1)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
20	14.5	13.17	60.87	60.87
40	9.6	3.41	10.42	71.29
60	6.3	1.74	3.50	74.79
120	7.1	1.14	2.58	77.37
240	10.2	0.54	1.76	79.13
480	7.5	0.41	0.99	80.12
720	6.1	0.21	0.39	80.51
tailings	941.0	0.042	-	-

Table 11-D: Cumulative Recovery and Grade for Test 11 (Head pH=9.3; tail pH=9.2)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
20	11.4	12.32	44.10	44.10
40	9.1	4.41	12.60	56.70
60	6.3	1.96	3.88	60.58
120	7.7	1.28	3.09	63.67
240	7.8	0.78	1.90	65.57
480	5.7	0.50	0.90	66.47
tailings	969.7	0.027	-	-

Table 12-D: Cumulative Recovery and Grade for Test 12 (Head pH=9.2; tail pH=9.1)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
20	9.3	19.65	35.42	35.42
40	10.8	8.61	16.57	51.99
60	9.0	4.06	6.33	58.32
120	7.5	2.13	3.81	62.13
240	4.9	0.83	2.04	64.17
480	6.3	1.00	1.07	65.24
tailings	953.4	0.04	-	-

Table 13-D: Cumulative Recovery and Grade for Test 13 (Head pH=9.3; tail pH=9.2)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
20	5.1	11.94	31.79	31.79
40	8.7	4.81	23.76	55.55
60	5.4	2.21	6.95	62.50
120	8.2	1.59	5.53	68.03
240	6.2	1.30	1.63	69.66
480	3.9	0.53	1.24	71.90
tailings	969.6	0.03	-	-

Table 14-D: Cumulative Recovery and Grade for Test 14 (Head pH=9.3; tail pH=9.2)

Time (secs)	Wt. of Concentrate (gms)	Grade (% Mo)	Recovery (% Mo)	Cumulative Recovery (% Mo)
20	10.5	10.28	34.47	34.47
40	9.1	5.45	15.85	50.32
60	10.4	3.00	9.96	60.28
120	10.6	2.18	7.38	67.66
240	8.0	0.89	2.28	69.94
480	10.8	0.39	1.36	71.30
tailings	941.0	0.063	-	-

APPENDIX E: - Wet and Dry Screening Analysis of Tailings from Tests 1 to 8

Screen Size (meshes)	Test #	lot % retained							
		1	2	3	4	5	6	7	8
+ 65	22.38	26.23	4.35	1.05	0.17	25.30	24.95	1.25	
65/100	18.00	17.20	21.24	14.93	5.54	17.36	16.31	15.68	
100/150	13.49	13.03	18.90	21.95	21.65	13.04	13.41	20.10	
150/200	8.23	7.69	10.80	12.86	14.81	7.82	8.10	12.01	
- 200	33.78	35.82	44.68	49.18	57.80	36.47	37.21	50.93	

APPENDIX F

Values of $\frac{C-C_{\infty}}{C_0-C_{\infty}}$ for Tests 1 through 14

The values of R_{∞} and C_0 are reported in Table 1 for tests 1 through 14. Tables 1-F and 2-F show the values of $\frac{C-C_{\infty}}{C_0-C_{\infty}}$ for different time intervals for tests 1 through 14.

Table 1: Initial concentration and R_{∞} values for tests
1 through 14

Test no.	R_{∞} (%)	C_0 (gms)
1	75.19	3.1172
2	77.22	3.1572
3	72.73	3.1053
4	77.22	3.1197
5	83.33	3.1284
6	77.22	3.1572
7	83.33	3.1582
8	80.65	3.1032
9	77.22	3.1341
10	81.14	3.1372
11	67.34	3.1854
12	65.20	3.1338
13	71.94	3.1522
14	73.80	3.1313

Table 1-F: Values of $\frac{C-C_{\infty}}{C_0-C_{\infty}}$ for tests 1 to 8

Time (secs)	$\frac{C-C_{\infty}}{C_0-C_{\infty}}$							
	1	2	3	4	5	6	7	8
30	0.21186	0.29742	0.27072	0.19453	0.2588	0.31058	0.25932	0.31814
60	0.10800	0.17494	0.11048	0.10711	0.1318	0.13158	0.11411	0.16266
120	0.06151	0.09952	0.05960	0.06677	0.06789	0.06673	0.05837	0.10477
240	0.03053	0.06033	0.03312	0.03887	0.04028	0.03800	0.03751	0.04994
480	0.01628	0.04418	0.01815	0.02036	0.02532	0.02321	0.01598	0.02779
720	0.00864	0.03842	0.01297	0.0150	0.01281	0.01591	0.00419	0.02035

Table 2-F: Values of $\frac{C-C_{\infty}}{C_0-C_{\infty}}$ for tests 9 to 14

Time (secs)	$\frac{C-C_{\infty}}{C_0-C_{\infty}}$					
	9	10	11	12	13	14
20	0.31814	0.25000	0.34505	0.46891	0.55807	0.53292
40	0.16266	0.12136	0.15792	0.22056	0.22779	0.31820
60	0.10477	0.07822	0.10039	0.12559	0.13105	0.18319
120	0.04994	0.04644	0.05452	0.06847	0.05414	0.08314
240	0.02779	0.02479	0.02632	0.03796	0.03143	0.0522
480	0.02035	0.01257	0.01294	0.02193	0.01428	0.03381