SKB TECHNICAL REPORT 89-25

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Volume and surface effects

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May 1989

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RADIONUCLIDE SORPTION ON CRUSHED AND INTACT GRANITIC ROCK
VOLUME AND SURFACE EFFECTS

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Information on SKB technical reports from 1977-1978 (TR 121), 1979 (TR 79-28), 1980 (TR 80-26), 1981 (TR 81-17), 1982 (TR 82-28), 1983 (TR 83-77), 1984 (TR 85-01), 1985 (TR 85-20), 1986 (TR 86-31), 1987 (TR 87-33) and 1988 (TR 88-32) is available through SKB.

Radionuclide sorption on crushed and intact granitic rock. Volume and surface effects.

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Abstract.

The specific surface areas and distribution ratios for sorption of ^{85}Sr, ^{137}Cs and ^{152}Eu were measured for crushed and intact granitic rock.

The experimental data can be accommodated by a sorption model encompassing sorption on outer and inner surfaces. It is clearly demonstrated that the time required to obtain reliable Kd-values for the sorption of strongly sorbing radionuclides like ^{252}Eu is very long due to solution depletion and slow diffusion into the rock.

A combination of surface area measurements and batch sorption with small particles may therefore be preferable when studying strongly sorbing nuclides.

Content.

1. INTRODUCTION.

The major driving force for migration of radionuclides from a repository for radioactive waste is advection by ground water. It is expected that the radionuclide will move with a velocity lower than the velocity of water

$$v = u/R$$

where v is the velocity of the radionuclide
u is the velocity of water
R is the retardation factor.

The retardation may be due to electrostatic or chemical bonding between the radionuclidic species and the solid in contact with the aqueous phase i.e. what is generally called sorption.
The sorption is empirically characterized by the distribution coefficient Kd defined by:

$$Kd = Cr/Cw \ (cm^3.g^{-1})$$

where Cr = nuclide concentration in rock
Cw = nuclide concentration in aqueous phase.

In a very simple approach the retardation for radionuclide transport in a single fracture may be described by the equation

$$R = 1 + Ka \cdot A/V$$

where A = wetted surface area of the fracture

V = void volume of fracture

Ka ($cm^3 \cdot cm^{-2}$) = surface area based distribution coefficient

The study of radionuclide sorption in the far field is thus an important part of the overall investigations needed for the assessment of potential sites for radioactive wastes disposal.

Measurements of sorption on geological material have been carried out on crushed as well as intact rock by the means of several techniques e.g batch sorption, through diffusion and high pressure convection (Berry el al., 1988).
Sorption is known to depend on the chemical conditions e.g temperature, pH, concentration of complexing agents and in batch experiments also on the water: rock ratio and the particle size range.
In our previous sorption work with crushed granite the particle size dependence was accommodated by a sorption model based on the assumption that the crushed rock consists of porous spheres with outer and inner surfaces available to interaction with the aqueous phase (Eriksen and Locklund, 1987). This raises the question whether the specific surface area of the rock as determined by e.g. a B.E.T. technique can be used as a measure or "index" for the sorption capacity of the rock as suggested by Jackson (1988).
This paper presents data from surface area and strontium, cesium and europium sorption measurements on crushed and intact granitic material. The aim of the study was to see if the

sorption capacity can be correlated to the specific surface areas.

2. EXPERIMENTAL.

2.1. Material.

The rock samples used in this study are granitic rocks with varying porosity and mineral content.

Radionuclides (^{85}Sr, ^{137}Cs , ^{152} Eu) in acidic solutions were obtained from Amersham International. The spiked solutions were prepared by diluting aliquots of the stock solutions with artificial groundwater synthesized to represent the natural groundwater in contact with granitic rock (Table 1). The radionuclide concentrations used were ^{85}Sr 10^{-10}, ^{137}Cs 10^{-9} and ^{152}Eu 10^{-8} $mol \cdot dm^{-3}$.

2.2 Sorption.

2.2.1 Crushed material: The rock was crushed using Agat mortar and pestle. The crushed material was ultrasonically disaggregated and size sorted by wet sieving into the required size fractions. 100 mg rock (dry weight) was equilibrated with 3 cm^3 solution in polypropylene tubes, the tubes being gently agitated at ambient temperature ($\sim 20^{o}C$). At chosen time intervals tubes containing each size fraction were removed and the solutions vacuumfiltered using a Millipore sampling manifold with 0.5 um polypropylene filters.

The activity of the solid phase and the filtered solution was measured by γ-spectrometry using a (2-x2 in) well type NaI detector connected to a multichannel analyzer. The amount of radionuclide sorbed on the tube walls was counted and corrected for.

2.2.2 Rock coupons: The granite was cut into 16x14x4 mm coupons. The coupons were washed with distilled water in a ultrasonic bath to remove fine particles, dried and evacuated before being saturated with groundwater. A typical experiment employed approximately 10 g rock (dry weight) and 50 cm^3 radionuclide solution. 100 ul samples of the solution were removed at chosen time intervals, diluted to 5 ml and counted.

2.3 Surface measurements.

The total surface areas of crushed rock and rock coupons were measured using a Micromeritics Flowsorb II 2300 apparatus, applying N_2 as the sorbing gas.

3. SORPTION MODEL.

It is assumed that crushed rock consists of spherical particles (Vandergraaf and Abry, 1982) with both outer and inner surfaces accessible for interaction with the solution. It is further assumed that the inner surface area is proportional to the particle volume.

The surface area to volume ratio of a spherical particle with diameter d is given by the relationship

$$A/V = 6/d \qquad (3.1)$$

and the specific surface area of crushed rock can thus be written

$$A(t) = A(v) + A(s) \cdot (6/\rho d) \quad (3.2)$$

where $A(t)$ = total surface area ($m^2 \cdot g^{-1}$)
$A(v)$ = inner surface area ($m^2 \cdot g^{-1}$)
$A(s)$ = outer surface area ($m^2 \cdot cm^{-2}$)
d = particle diameter (cm)
ρ = rock density (2.61 $g \cdot cm^{-3}$)

Correspondingly the distribution coefficient Kd is given by the equation

$$Kd = Kd(v) + Kd(s) \cdot (6/\rho d) \quad (3.3)$$

4. EXPERIMENTAL RESULTS.

4.1 Surface areas.

The specific surface areas measured on crushed rock are plotted versus 1/d in Figs 1-5. The diameter d characterizing each size fraction is taken to be the arithmetic mean of the apertures of the sieves bracketing the various size fractions.

The surface areas calculated by linear regression are given in Table 2. together with the surface areas of the coupons.

4.2 Sorption.

The sorption data are presented as measured distribution ratios (Rd) and not the more strictly defined Kd.

4.2.1 ^{85}Sr sorption: The results from the ^{85}Sr experiments with crushed rock are plotted in Figs 6-9 and the Rd(v) values obtained by linear regression are given in Table 3.

The experimental Rd values,obtained in sorption experiments with coupons, are plotted versus contact time in Figs 10-14 and the Rd values obtained after ~120 days are given in Table 3.

4.2.2 ^{137}Cs sorption: The Rd values mesured at differing times of contact between the radionuclide solution and coupons of Stripa granite are depicted in Fig 15. As can be seen equilibrium is not reached after 120 days.

4.2.3 ^{152}Eu sorption: Sorption experiments were only carried out with crushed rock. The experimental results are summarized in Table 4 and the Rd versus time and Rd versus 1/d plots are depicted in Figs 16-17.

5. DISCUSSION.

The surface related distribution coefficients for the inner and outer surfaces of the crushed rock are given by the ratios of the the intercepts and slopes respectively obtained by linear regression analysis of the Rd and A versus 1/d plots.

The calculated Ra(v), Ra(s) and Ra values for ^{85}Sr sorption on crushed and intact (coupons) rock respectively are summarized in Table 5. The experimental uncertainty is rather large, but the data clearly indicate that Ra(v) and Ra(s) are of the same magnitude. Moreover,there is no systematic variation between Ra obtained in the coupon experiments and Ra(v),Ra(s) obtained using crushed rock. It ought to be emphasized that the calculation of Ra is based on the specific surface of the coupon i.e. both outer and inner surfaces are included. The geometric surface to volume ratio is however small, corresponding to a 1/d value of 0.43 in the plots for crushed rock.
As can be gleaned from the ^{137}Cs plot in Fig 15 sorption equilibrium was not obtained during 120 days of contact between the solution and the rock coupons. The stronger sorption of ^{137}Cs as compared to ^{85}Sr causes more radionuclide depletion in the solution. Also more radionuclide must diffuse into the rock and the time required is therefore longer.

The attainment of equilibrium in sorption experiment with ^{152}Eu requires very long time as can be seen from Figs 16, 17. This is to be expected as ^{152}Eu is strongly sorbed and the depletion of the solution results in a very slow diffusion into the rock and thereby redistribution of ^{152}Eu on all surfaces. Extrapolating the Rd versus time plot for the 125-250 µm fraction, an equilibrium value of approximately 280 $cm^3 \cdot g^{-1}$ seems reasonable. The calculated Rd(v) and Rd(s)$\cdot$(6/ρd) values for the 125-250, 250-500 and 1000-2000 µm fractions are given in Table 6. It should be noted that fitting the Rd values obtained after 240 hours by linear regression, which may seem reasonable in view of the experimental uncertainties, assuming sorption on the outer surface only result in an overestimate of Kd(s).

The distance l diffused into rock in time t is approximately given by $l = (D_i t/\varepsilon + Rd)^{1/2}$, where D_i is the intrinsic diffusion coefficient and ε is the porosity of the rock.

If it is assumed that equilibrium is reached within the 125-250 µm fraction after 15 days we estimate that at least 165 and 960 days are required to reach equilibrium for the 500-750 and 1000-2000 µm fractions respectively.

6. CONCLUSIONS.

1) Measurements of the specific surface area of crushed granitic rock indicate that the assumption of spherical particles with an inner surface area A(v) proportional to the particle volume is reasonable.

The A(v) obtained from plots of total surface area versus 1/d, where d is diameter of particle, is in agreement with A(v) values obtained by measurements on granite coupons.

2) The measured distribution ratios Rd for ^{85}Sr and ^{137}Cs sorption on crushed rock can accommodated by a sorption model based on the assumption of porous particles.

3) The time required to determine reliable Kd-values for the strongly sorbing ^{132}Eu is very long, even for small particles.

An estimate of Rd-values for intact rock may therefore preferably be based on combining measurements of specific surface areas of crushed and intact rock with batch sorption experiments using size fractions with small diameter.

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Table 1

Composition of artificial ground water[a].

Species	Concentration $mg\cdot dm^{-3}$	Species	Concentration $mg\cdot dm^{-3}$
HCO_3^-	123	Ca^{2+}	18
SO_4^{2-}	9.6	Mg^{2+}	4.3
Cl^-	70	K^+	3.9
SiO_2	12	Na^+	65

a) pH 8 - 8.2, Eh 260 mV (aerated)

Table 2

Surface areas of crushed granite and granite coupons, measured by N_2-sorption. The particle size is defined by the particle diameter d(cm), i.e. assuming spherical particles.

Granite	Surface area $m^2 \cdot g^{-1}$ (crushed rock)	Surface area $m^2 \cdot g^{-1}$ (rock coupons)
Stripa	$(0.25 \pm 0.02) + (2.4 \pm 0.6) \cdot 10^{-3} \cdot 1/d$	0.205 ± 0.005
Pingstaberg	$(0.3 \pm 0.04) + (2.1 \pm 1.1) \cdot 10^{-3} \cdot 1/d$	0.28 ± 0.01
M-73002	$(0.074 \pm 0.009) + (1.4 \pm 0.3) \cdot 10^{-3} \cdot 1/d$	0.1 ± 0.005
85004	$(0.061 \pm 0.0026) + (3.85 \pm 0.76) \cdot 10^{-3} \cdot 1/d$	0.08 ± 0.005
85015	$(0.21 \pm 0.0015) + (1.57 \pm 0.1) \cdot 10^{-3} \cdot 1/d$	

Table 3

Distribution ratios Rd for ^{85}Sr sorption on inner surfaces of crushed and intact (coupons) granitic rock.

Granite	$Rd(v)(cm^3 \cdot g^{-1})$ (crushed)	$Rd(cm^3 \cdot g^{-1})$ (coupons)
Sr-85		
Stripa	5.1±0.3	2.3±0.3
Pingstaberg	5.6±0.7	2.9±0.4
73002	2.13±0.3	2.0±0.1
85004	3.20±0.38	1.6±0.2
85015	10.7±2.6	
Cs-137		
Stripa	33±5	

Table 4

Distribution ratios for Eu-152 sorption on crushed Stripa granite after differing time of contact with spiked solution.

Fraction	$1/d(cm^{-1})$	$Rd(cm^3 \cdot g^{-1})$			
		1h*)	24h*)	96h*)	240h*)
63-90	130.7	16.95±0.3			
90-125	93.01	14.75±0.3			
125-250	53.33	11.2±0.3	88.56	194.86	264.5
250-500	26.67	8.9 ±0.2			
500-750	16.0	7.9±0.3	53.92	82.11	107.9
750-1000	11.43	6.7±0.15			
1000-2000	6.67		23.2	40.9	47.4

*) time of contact with spiked solution.

Table 5

Surface-related distribution ratios Ra(s), Ra(v) for sorption in outer and inner surfaces respectively of crushed and intact granitic rock.

Granite	crushed $Ra(v)\cdot 10^4$ $cm^3 \cdot cm^{-2}$	crushed $Ra(s)\cdot 10^4$ $cm^3 \cdot cm^{-2}$	coupons $Ra \cdot 10^4$ $cm^3 \cdot cm^{-2}$
Sr-85			
Stripa	20.4±2.7	30.8±12.5	11.2±1.5
Pingstaberg	18.7±3.4	13±9	10.4±1.5
M-73002	28.8±5.4	10.8±2.3	20±1.4
85004	52.5±23.2	10.1±2	20±2.8
85015	51±12.4	66±26	
Cs-137			
Stripa	132±27	259±104	>24*
Eu-152			
	740	740	

* Equilibrium not obtained

Table 6

Calculated distribution coefficient Rd(v), Rd(s) for sorption of Eu-152 on crushed Stripa granite, assuming equilibrium within the 63-125 μm fraction after 10 days sorption.

Fraction	1/d cm^{-1}	A(v) $m^2 \cdot g^{-1}$	A(s) (6/ρd) $m^2 \cdot g^{-1}$	Rd(v) $cm^3 \cdot g^{-1}$	Rd(s) (6/ρd) $cm^3 \cdot g^{-1}$
125-250	53.33	0.25±0.03	0.128±0.048	185	95
500-750	16	0.25±0.03	0.0384±0.014	185	28.45
1000-2000	6.67	0.25±0.03	0.016±0.0058	185	11.85

FIGURE LEGENDS.

Fig. 1

Specific surface area of crushed Stripa granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 2

Specific surface area of crushed Pingstaberg granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 3

Specific surface area of crushed 73002 granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 4

Specific surface area of crushed 85004 granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 5

Specific surface area of crushed 85015 granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 6

Distribution ratio Rd for ^{85}Sr sorption on crushed Pingstaberg granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 7

Distribution ratio Rd for ^{85}Sr sorption on crushed 73002 granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 8

Distribution ratio Rd for ^{85}Sr sorption on crushed 85004 granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 9

Distribution ratio Rd for ^{85}Sr sorption on crushed 85015 granite plotted versus 1/d, where d is a diameter characterizing the particle size fraction.

Fig. 10

Distribution ratio Rd for ^{85}Sr sorption on intact Stripa granite plotted versus time of contact with radionuclide solution (∿16x14x4 mm coupons).

Fig. 11

Distribution ratio Rd for ^{85}Sr sorption on intact Pingstaberg granite plotted versus time of contact with radionuclide solution (∿16x14x4 mm coupons).

Fig. 12
Distribution ratio Rd for ^{85}Sr sorption on intact 73002 granite plotted versus time of contact with radionuclide solution (~16x14x4 mm coupons).

Fig. 13
Distribution ratio Rd for ^{85}Sr sorption on intact 73002 granite plotted versus time of contact with radionuclide solution (~16x14x4 mm coupons).

Fig. 14
Distribution ratio Rd for ^{85}Sr sorption on intact 85004 granite plotted versus time of contact with radionuclide solution (~16x14x4 mm coupons).

Fig. 15
Distribution ratio Rd for ^{137}Cs sorption on intact Stripa granite plotted verus time of contact with radionuclide solution (~16x14x4 mm coupons).

Fig. 16
Distribution ratio Rd for ^{152}Eu sorption on crushed Stripa granite plotted versus time of contact with radionclide solution

○ 125 - 500 um, △ 500 - 700 um

□ 1000 - 2000 um

Fig. 17

Distribution ratio for Rd for ^{152}Eu sorption on crushed Stripa granite plotted versus 1/d, where d is a diameter characterizing the size fraction

contact time: O 1h, Δ 24h, ◘ 96h, X 240h.

Fig. 1

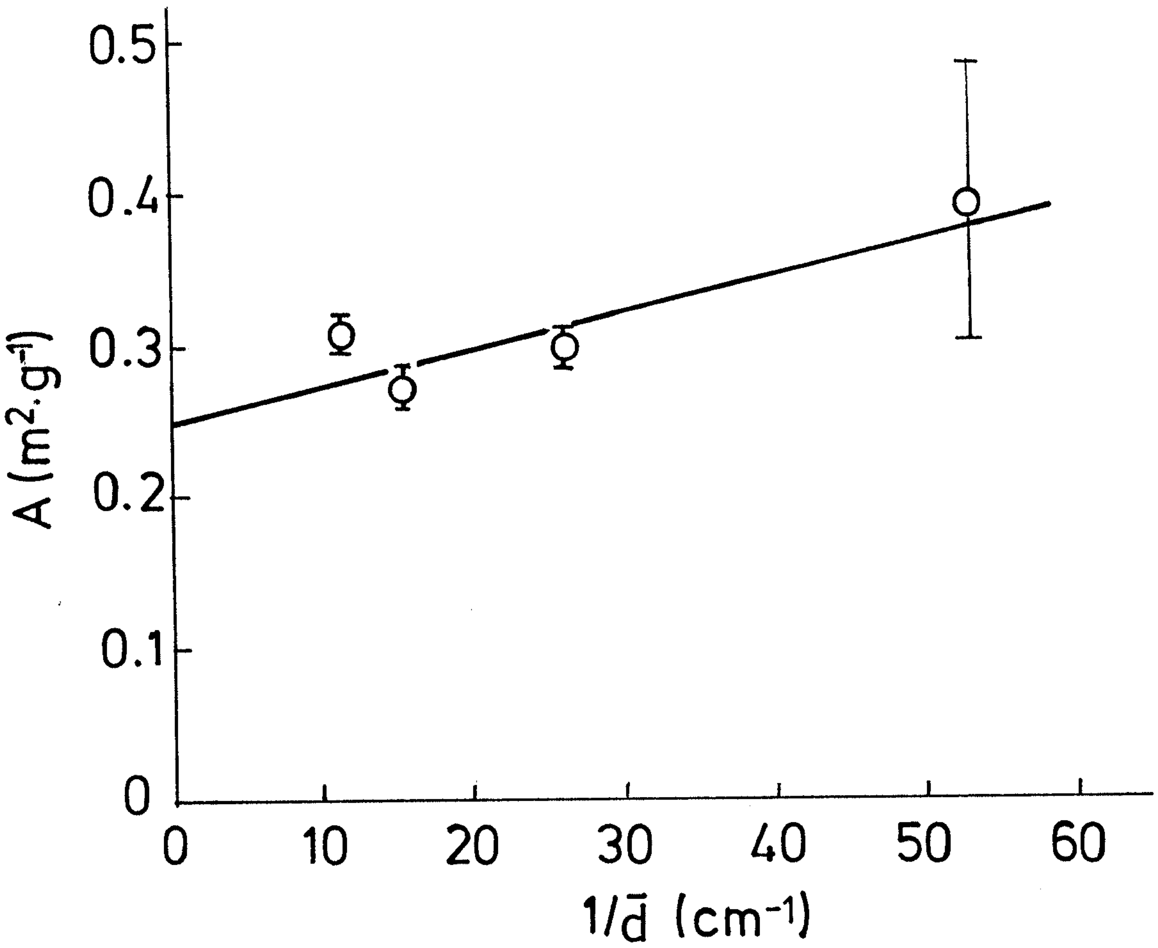
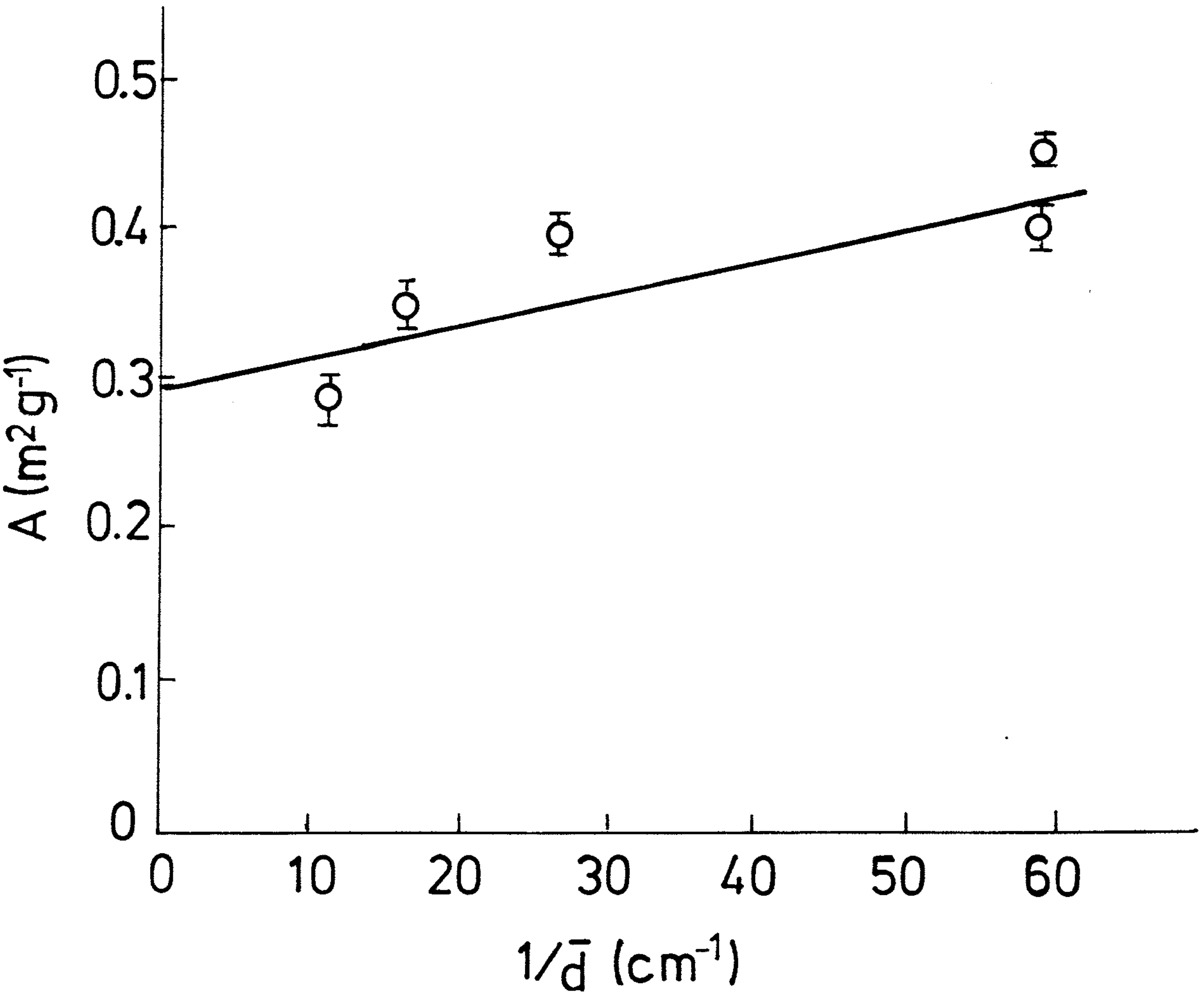


Fig. 2



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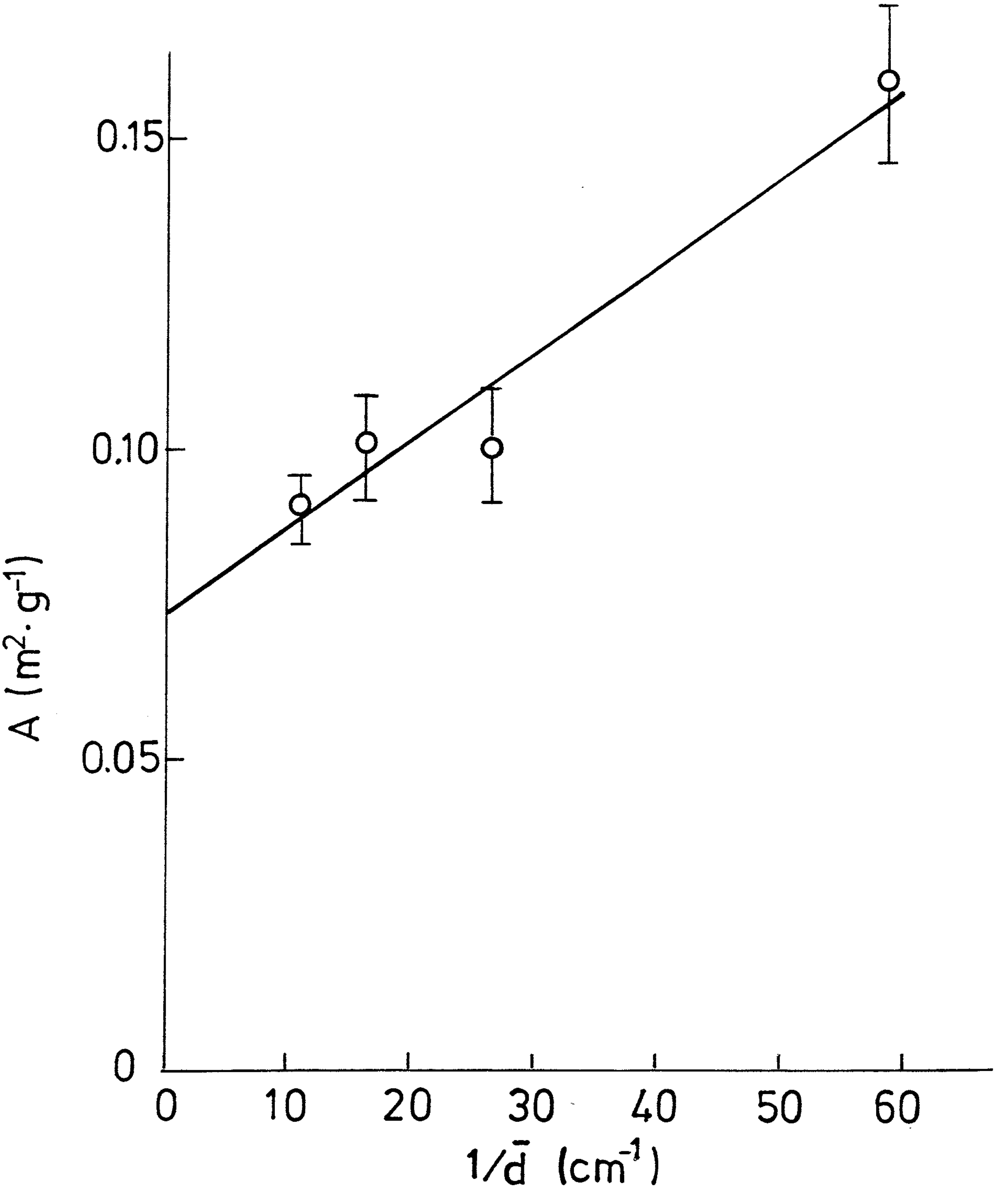

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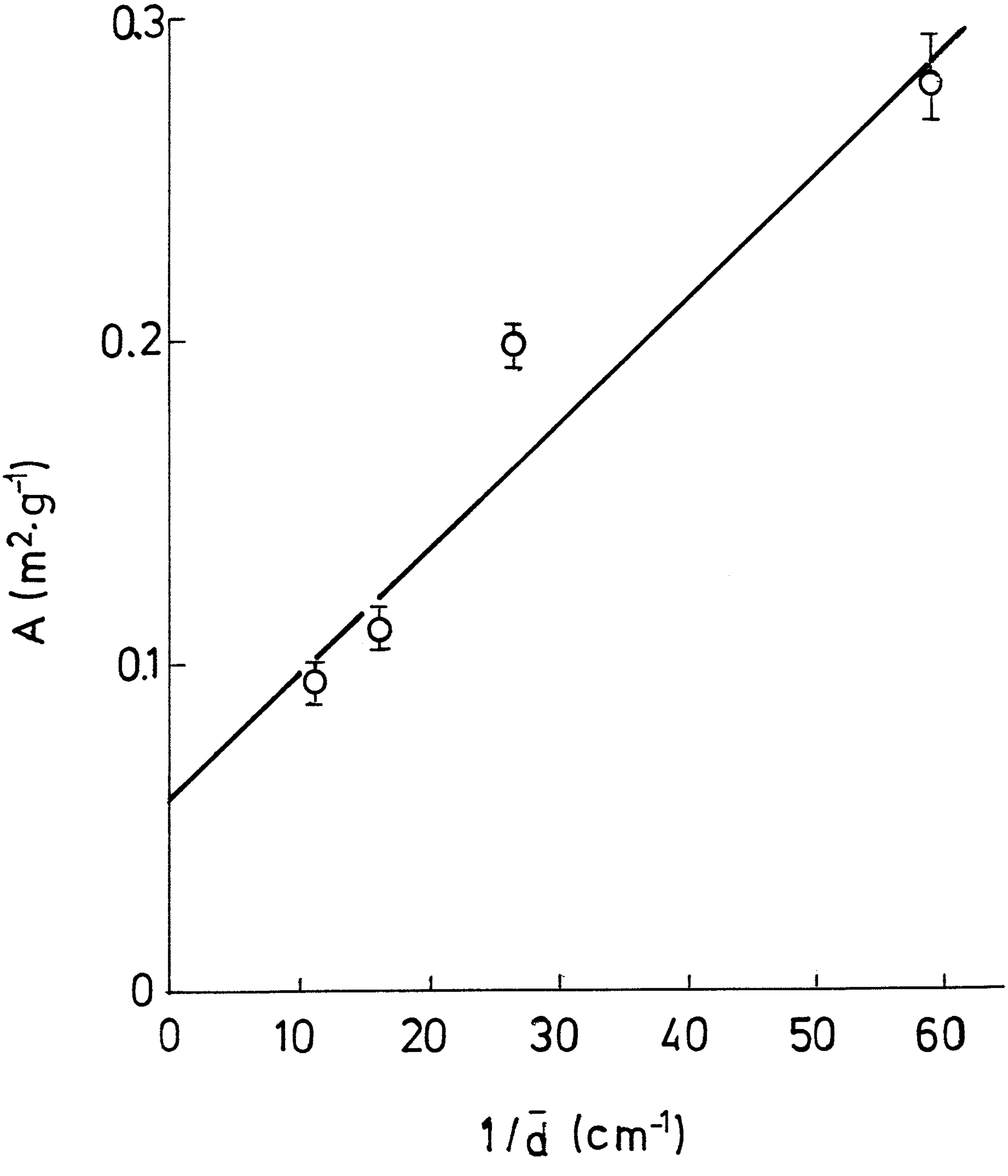

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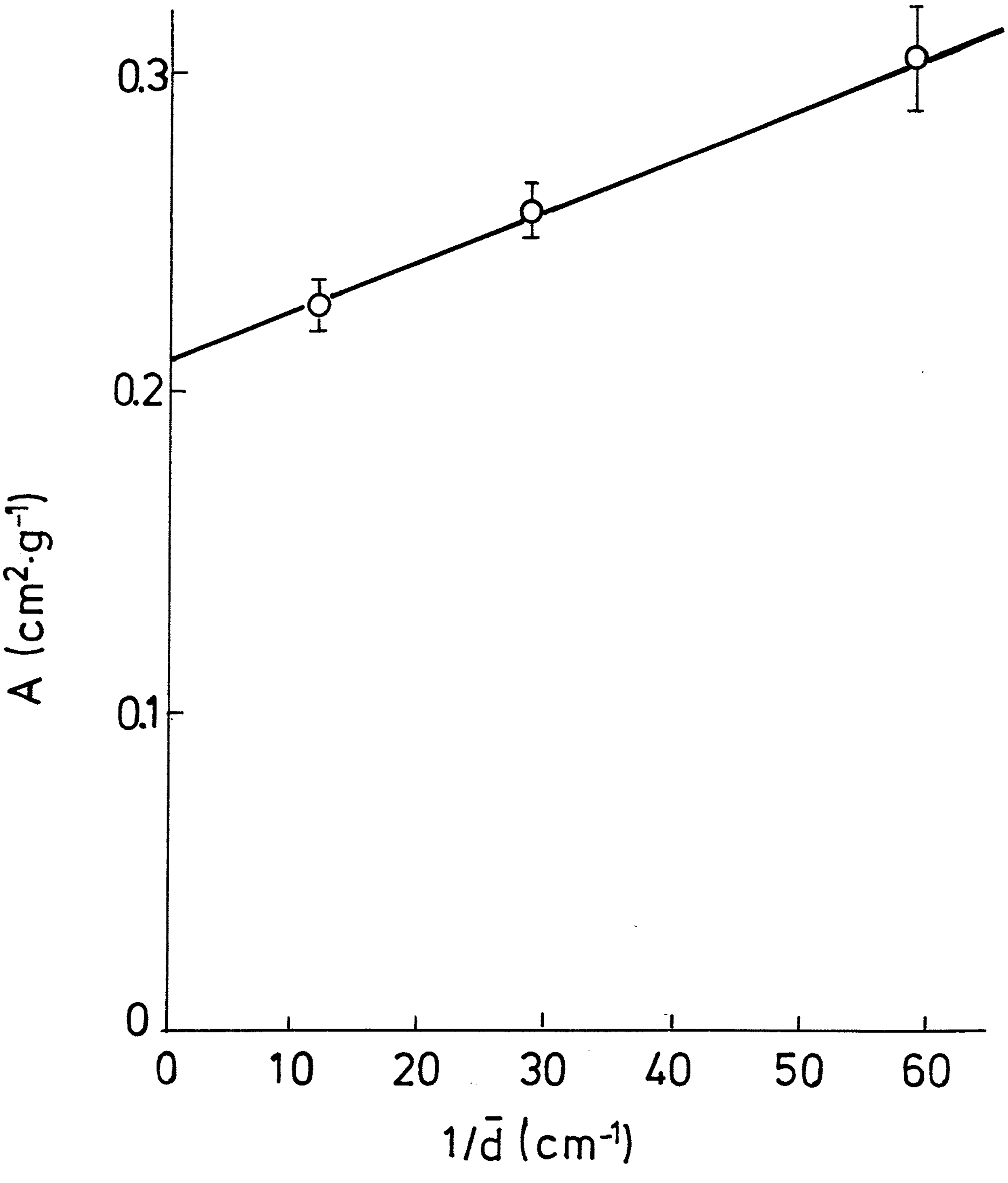

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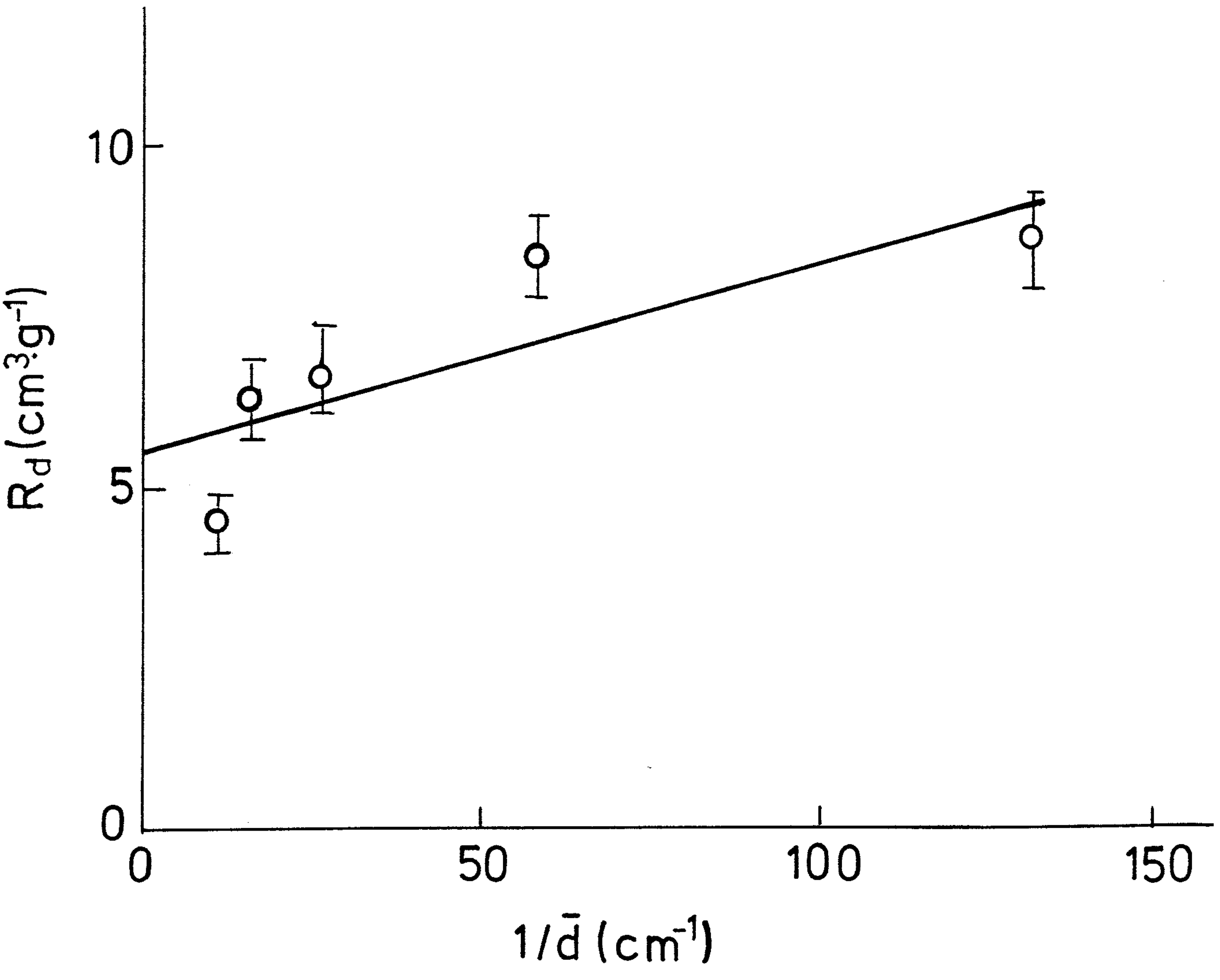

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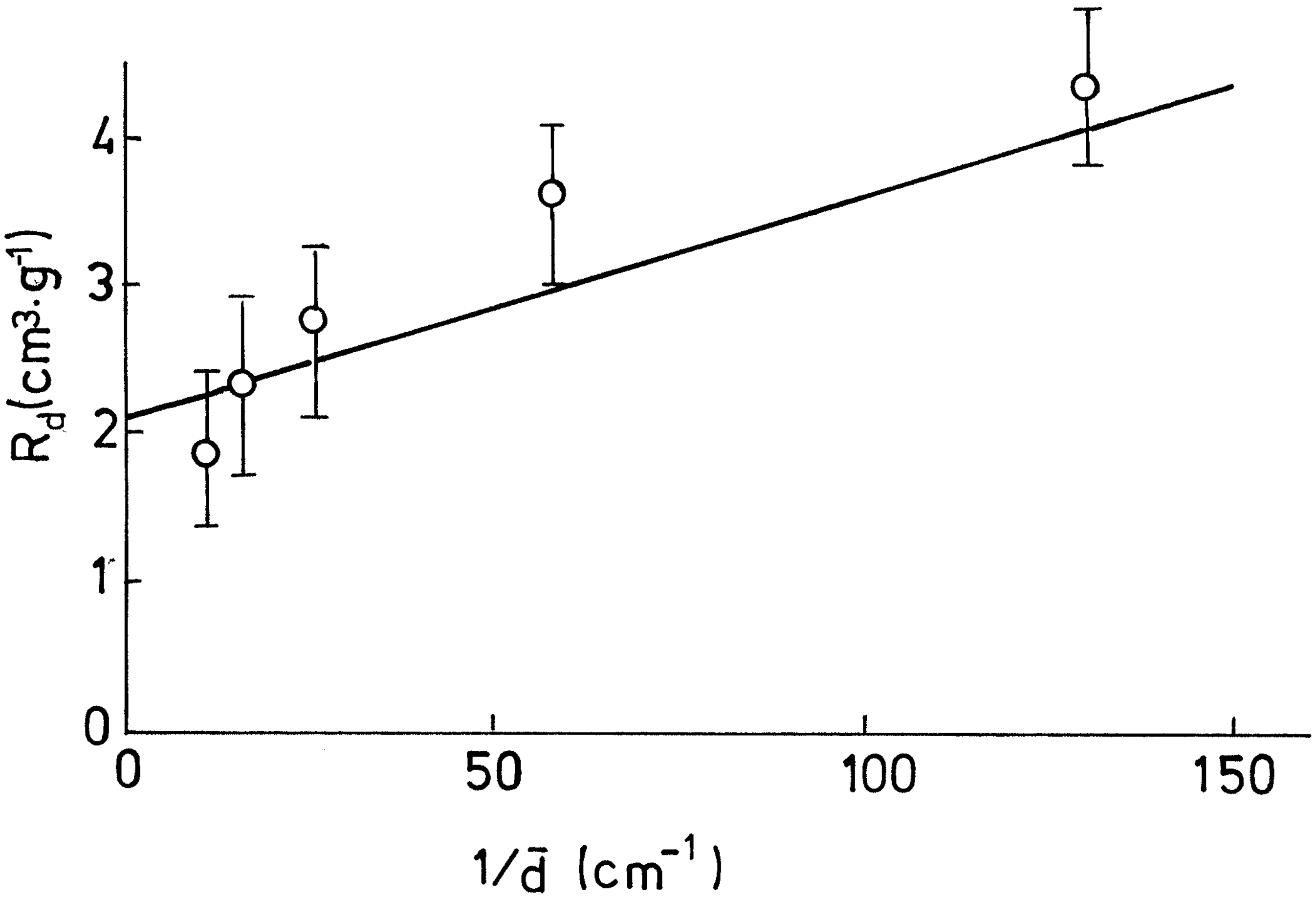

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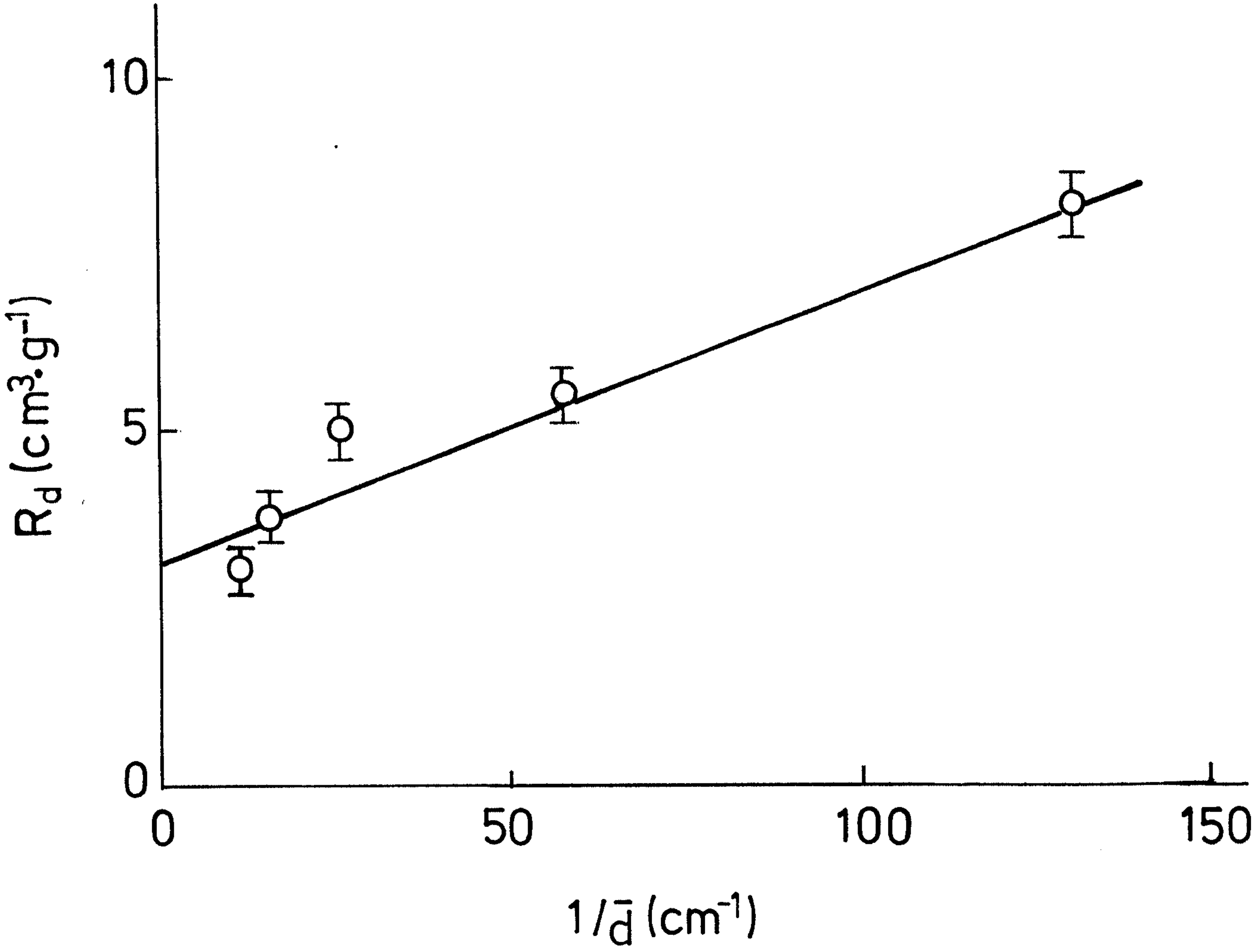


Fig. 9

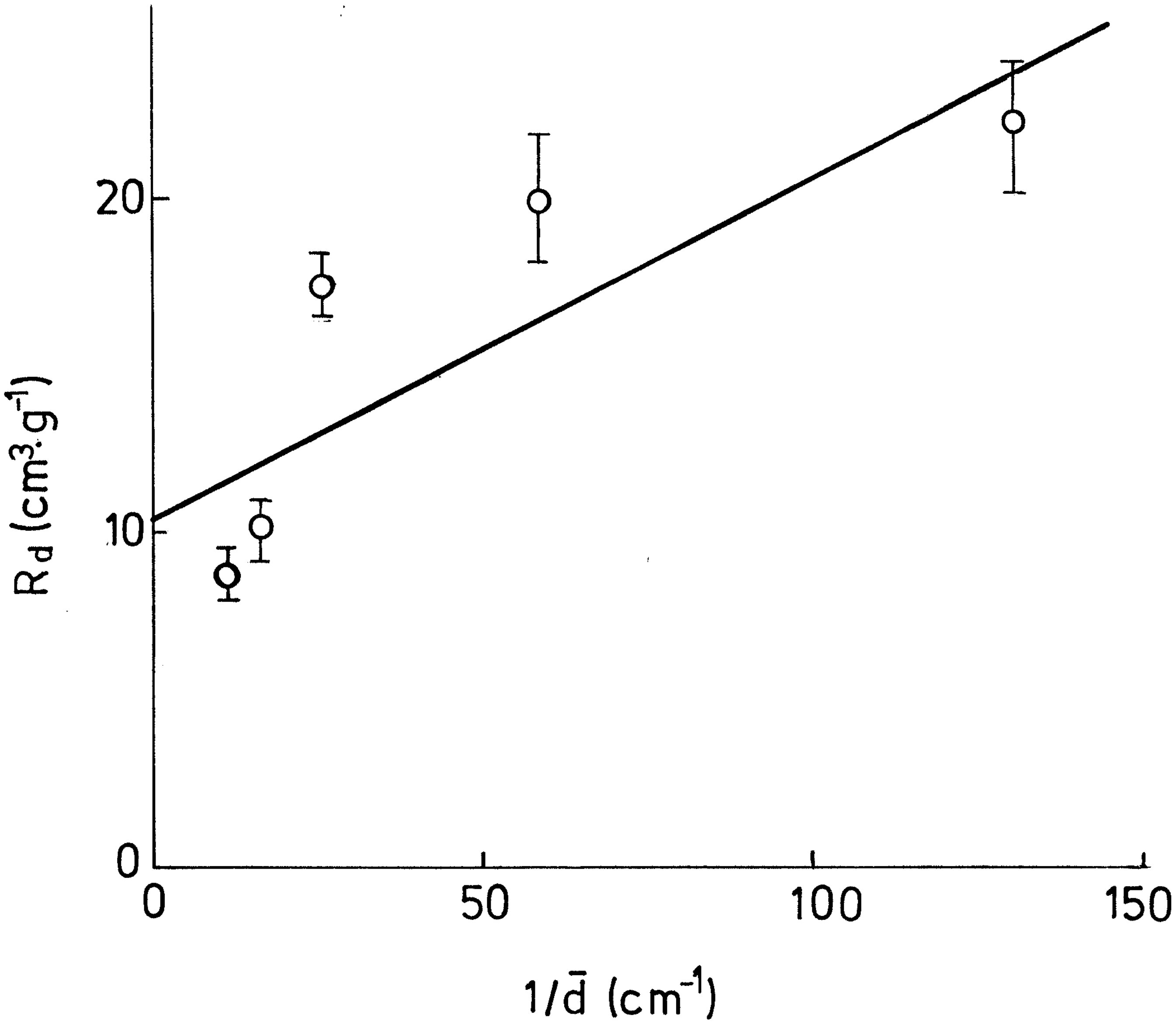
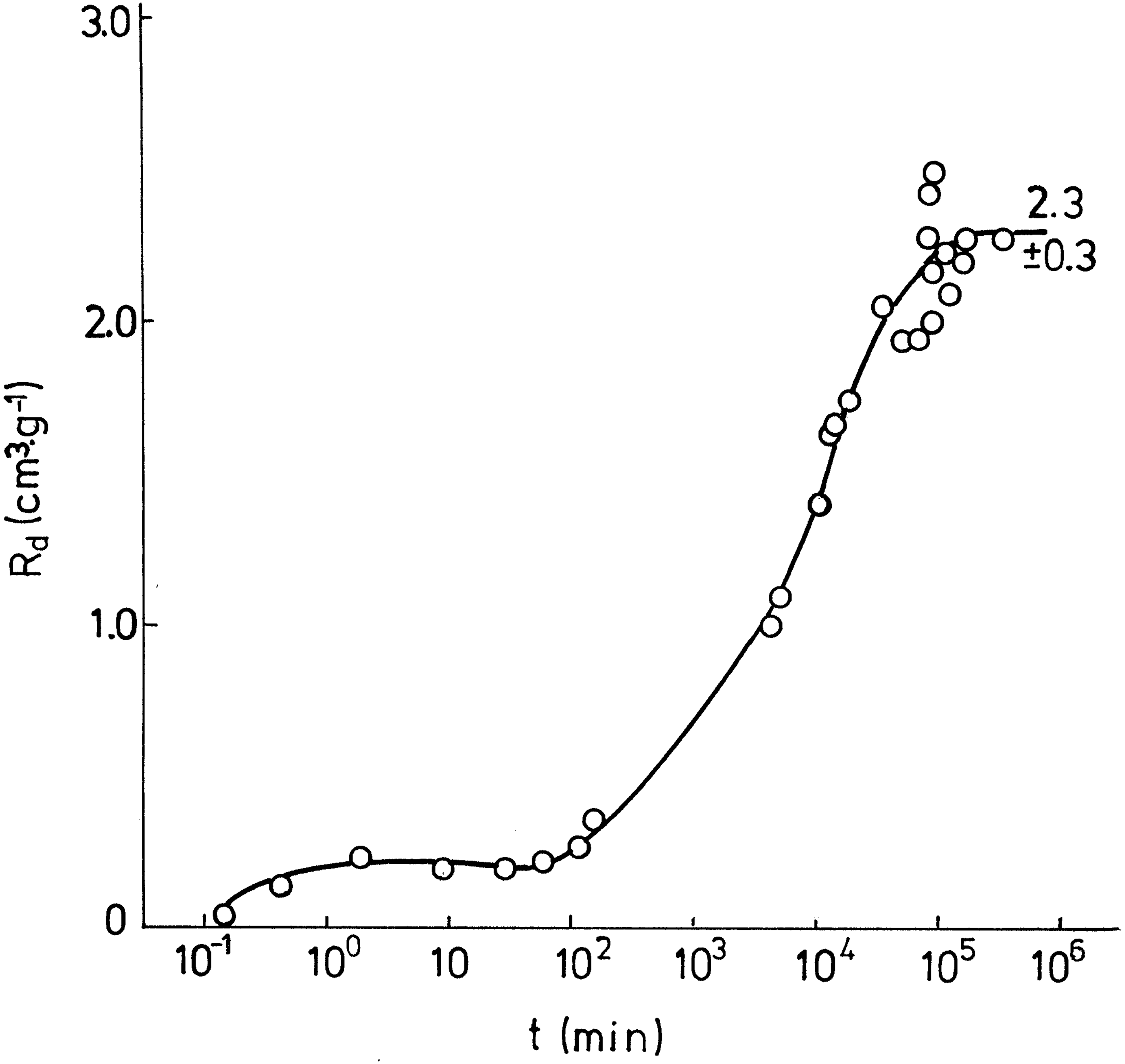


Fig. 10



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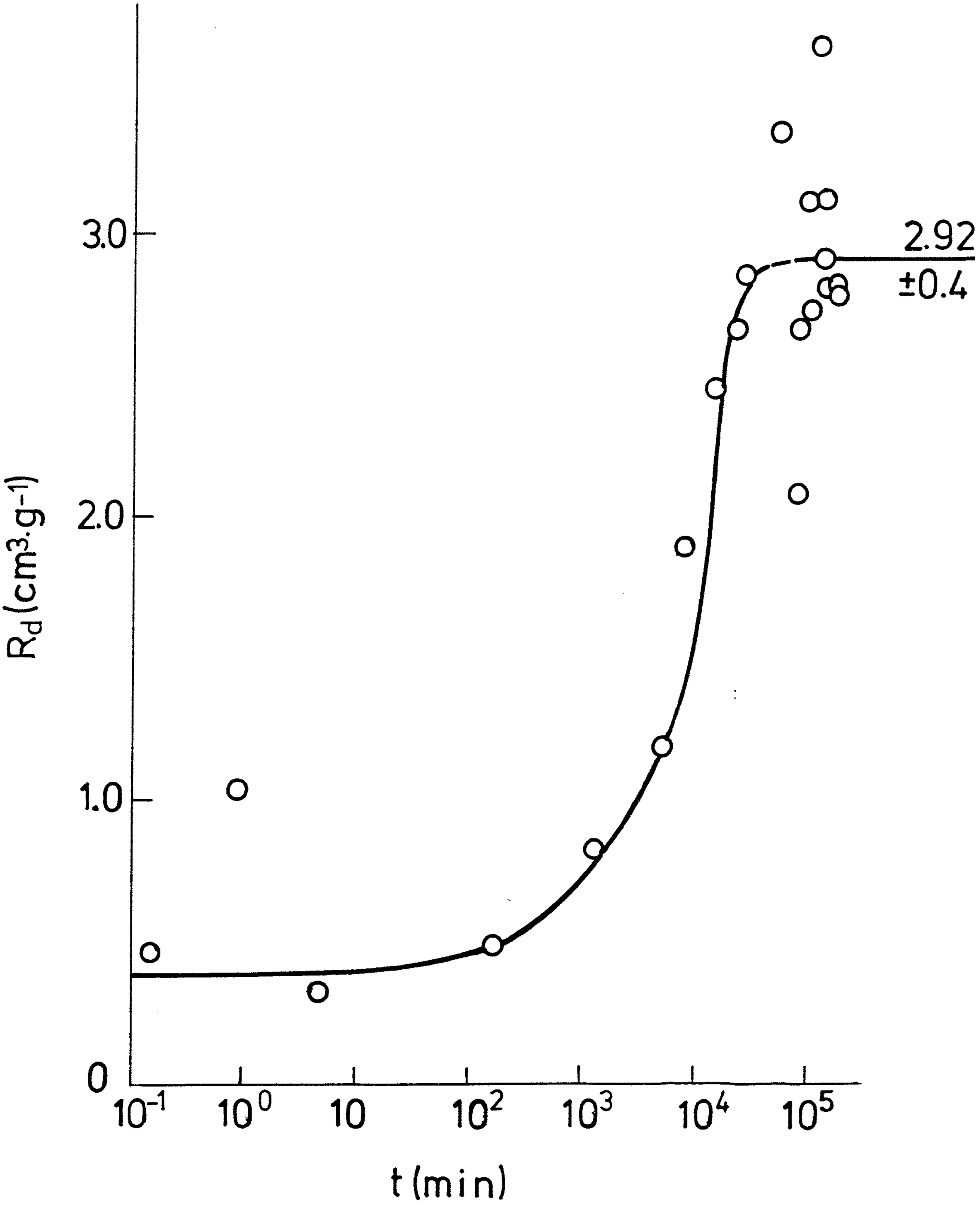


Fig. 12

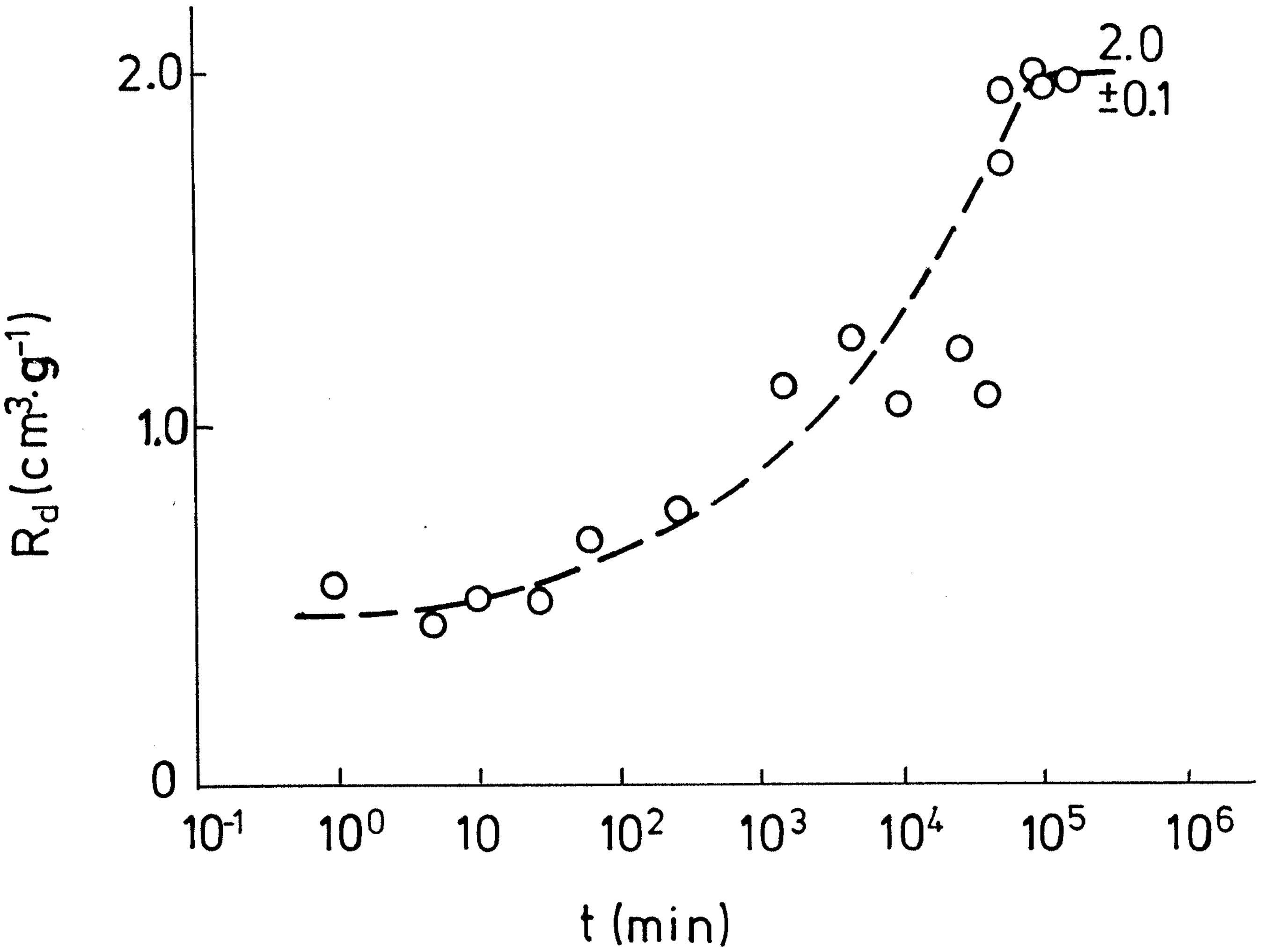


Fig. 13

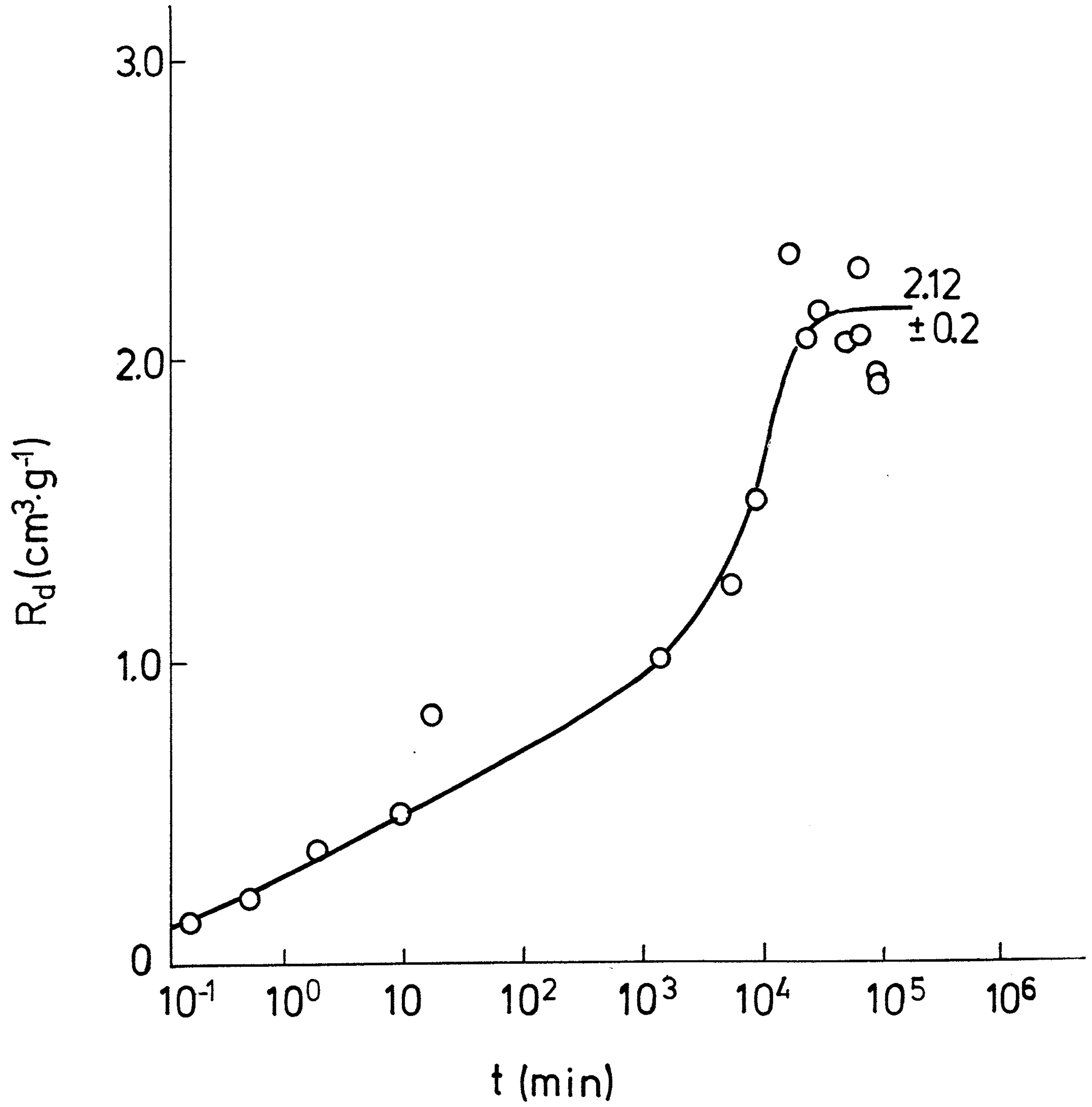


Fig. 14

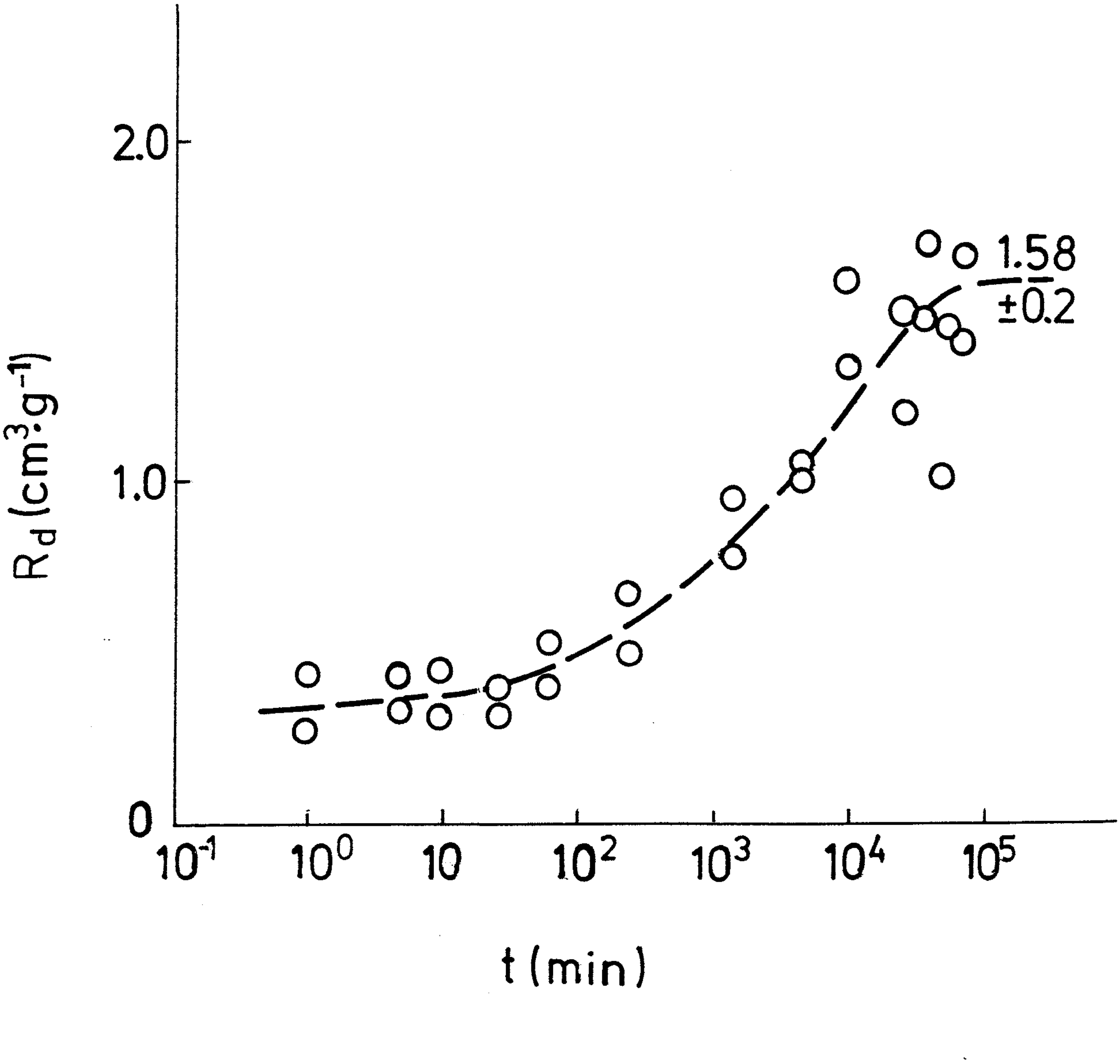
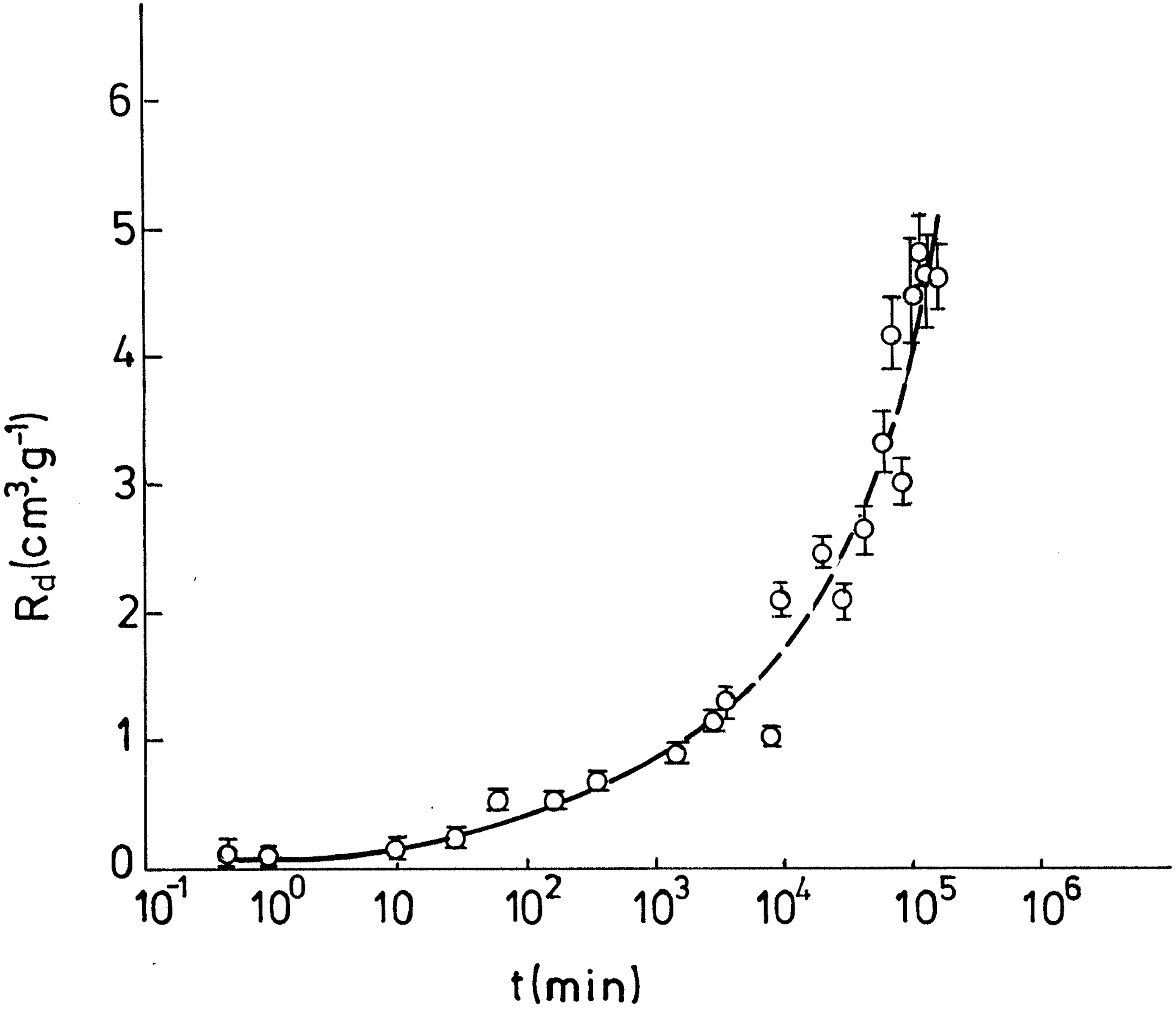


Fig. 15



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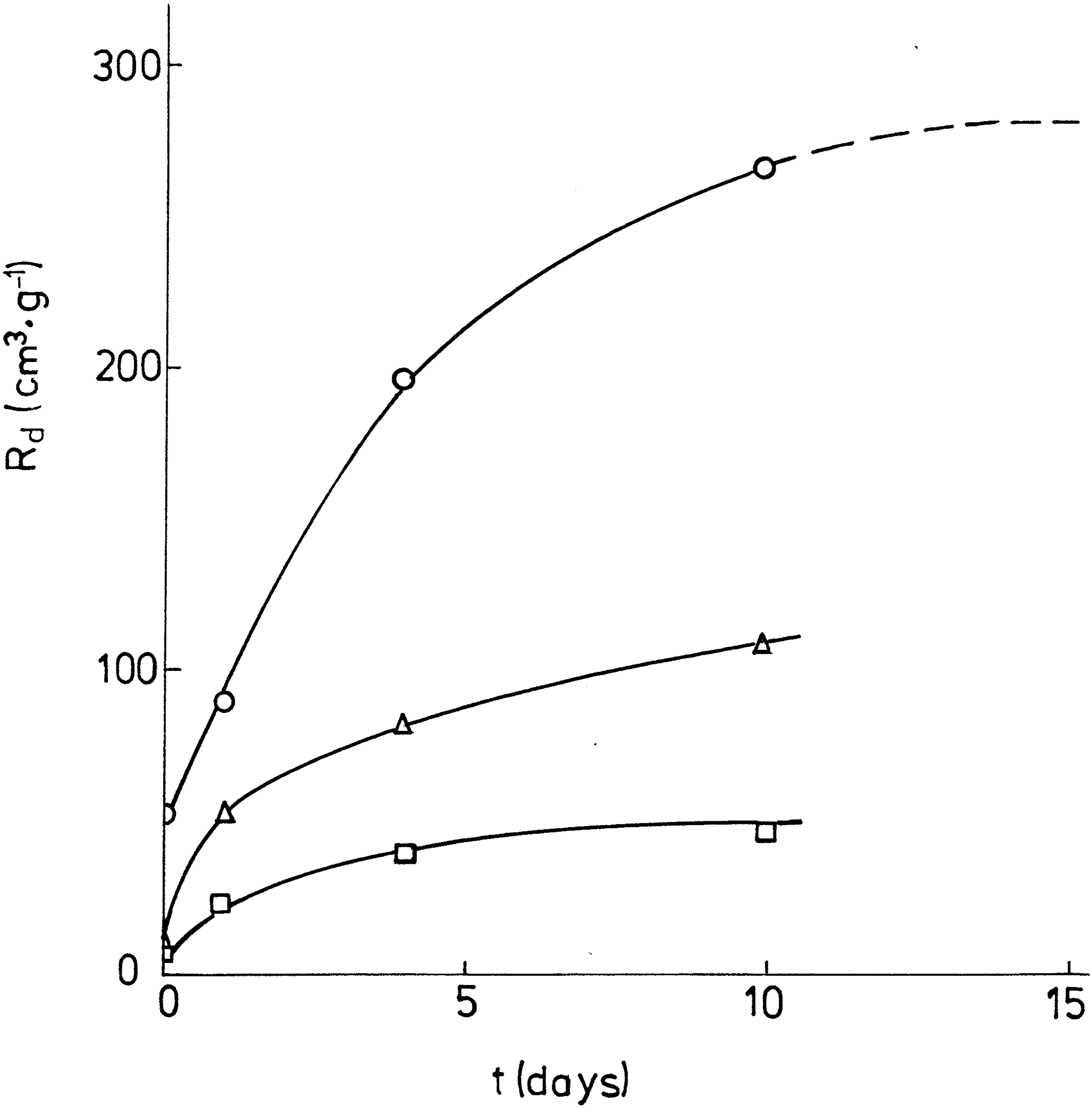

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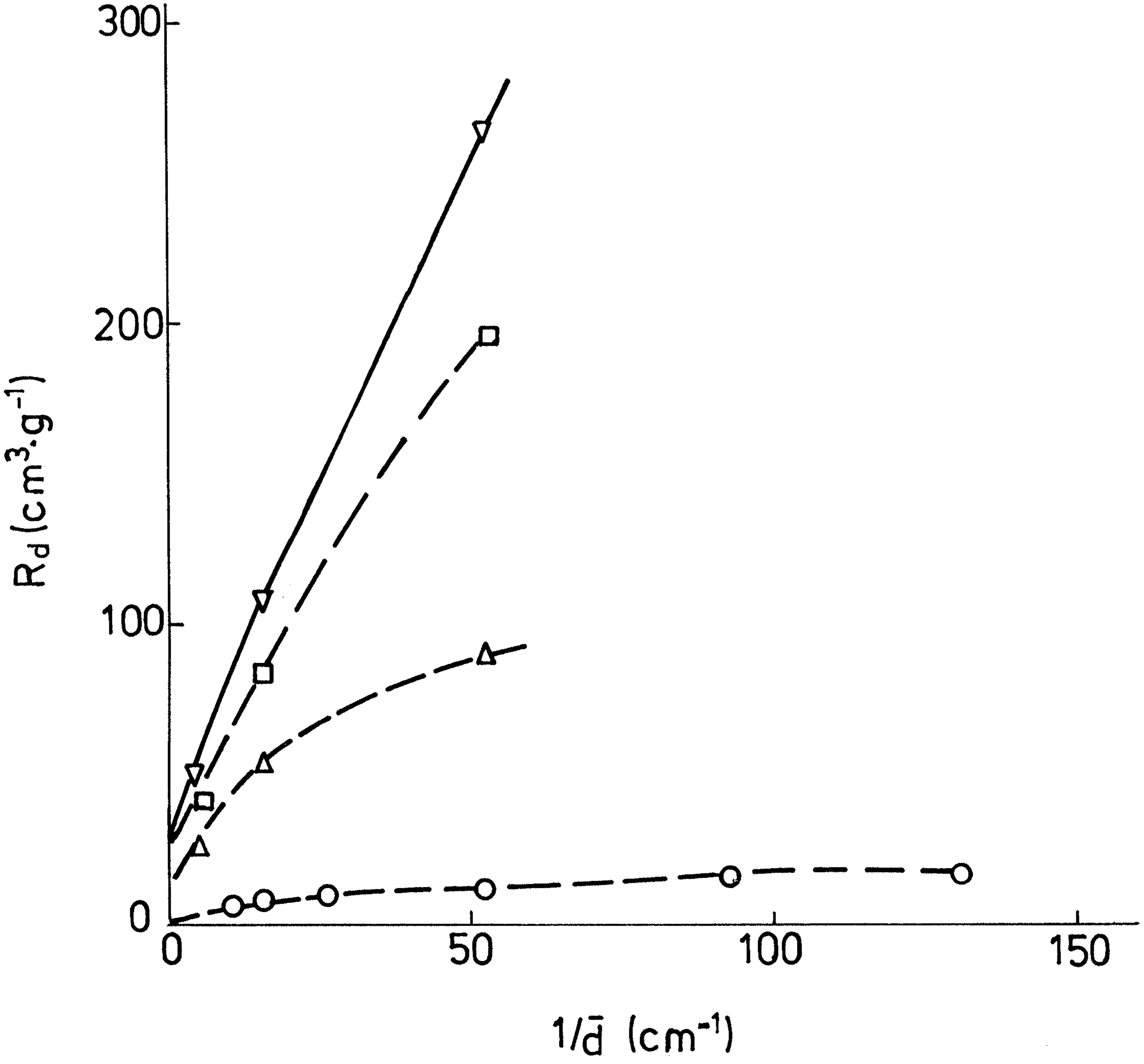

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TR 121
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Including Summaries of Technical Reports Issued during 1986
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TR 87-33
SKB Annual Report 1987
Including Summaries of Technical Reports Issued during 1987
Stockholm, May 1988

1988
TR 88-32
SKB Annual Report 1988
Including Summaries of Technical Reports Issued during 1988
Stockholm, May 1989

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1989

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Conny Holmqvist, Hans-Edy Mårtensson
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January 1989

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Ebbe Eriksson, Stefan Sehlstedt
SGAB, Luleå
February 1989

TR 89-03
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August 1988

TR 89-04
SKB WP-Cave Project
Radionuclide release from the near-field in a WP-Cave repository
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April 1989

TR 89-05
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April 1989

TR 89-06
SKB WP-Cave Project
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Sture Nordlinder, Ulla Bergström
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April 1989

TR 89-07
SKB WP-Cave Project
Some Notes on Technical Issues

- Part 1: Temperature distribution in WP-Cave: when shafts are filled with sand/water mixtures
  Stefan Björklund, Lennart Josefson
  Division of Solid Mechanics, Chalmers University of Technology, Gothenburg, Sweden
- Part 2: Gas and water transport from WP-Cave repository Luis Moreno, Ivars Neretnieks
  Department of Chemical Engineering, Royal Institute of Technology, Stockholm, Sweden
- Part 3: Transport of escaping nuclides from the WP-Cave repository to the biosphere. Influence of the hydraulic cage
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August 1989

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TR 89-10
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