

Denne rapport
tilhører

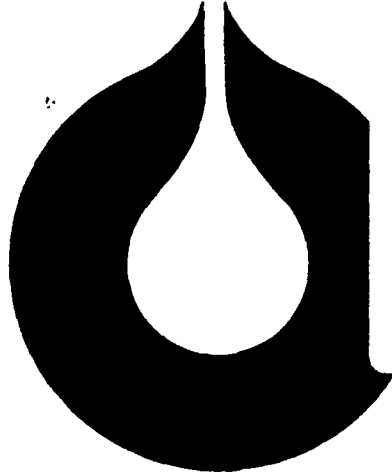
 **STATOIL**

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KODE Well 31/3-1 nr. 62

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statoil

TROLL Completion Fluids

Well 31/3-1

STATOIL

EXPLORATION & PRODUCTION

LABORATORY

by

Torgeir Lund

Jan.-84

LAB 8

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Den norske stats oljeselskap a.s



Classification

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Title

TROLL Completion Fluids
Well 31/3-1
STATOIL
EXPLORATION & PRODUCTION
LABORATORY

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Torgeir Lund

Jan.-84

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2/2-84

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3. COMPATIBILITY OF FLUIDS

During the drilling and gravelpacking operations the following fluids have been in contact with each other and the formation:

- a) Formation water
- b) Drilling fluid filtrate
- c) CaCl_2 -brines ($d = 1.14 \text{ gcm}^{-3}$ and $d = 1.30 \text{ gcm}^{-3}$)
- d) CaCl_2 -HEC mixtures
- e) HCl

The composition of the formation water can only be estimated from FMT-samples. The concentrations of major ions are close to what is found in water from well 31/2-11. In the laboratory experiments we used filtered DST-water from well 31/2-11, or synthetic water with the same composition (Table 1).

The KCl/polymer mud used in the upper sections was diluted to a dispersed lignosulphonate mud during drilling through the reservoir. The composition of the mud-filtrate from mud/samples taken by drilling at 1365 m is shown in Table 2. Mud filtrate prepared from fresh mud on the rig had a pH in the range 10.5 - 11. Mudfiltrates prepared later in the laboratory have pH 7-8. We anticipate that this change is due to precipitation by ageing of the mud.

The laboratory prepared mudfiltrate (MF) was mixed with formation water (FW) in ratios (5:1, 4:2, 3:3, 2:4, 1:5) The pH was increased slowly by adding KOH dropwise. In all mixtures precipitation occurred immediately at pH 11. The higher FW concentration gave more massive precipitation. To check the ageing effect a new series of mixtures was brought to pH < 8.5. No precipitate could be seen. After one week precipitate was found in all glasses.

We tested in the same way mixtures of $d = 1.14$ and 1.28 gcm^{-3} CaCl_2 -brine/mudfiltrate and $d = 1.28 \text{ gcm}^{-3}$ CaCl_2 -brine/formation water. Precipitates are formed in all mixtures at pH 11. Only the 1.28 gcm^{-3} CaCl_2 -brine/formation water system is unstable and form precipitates by ageing at pH <9.

We note that the CaCl_2 -brine (1.14 gcm^{-3}) obtained from the contractor and used in all experiments had pH = 10.3.

The laboratory prepared mudfiltrate was unstable and on standing produced a cloudy suspension. On filtering, again a further precipitate dropped out after some days. Alternatives are bacterial growth or a precipitated polymer previously in solution.

3.1 Comments:

The formation is open for mudfiltrate invasion during drilling. Because of the low pH of the fresh mudfiltrate and the long time contact with formation water some scaling may have occurred during drilling through the reservoir.

4. PERMEABILITY MEASUREMENTS

Samples were mounted in a Hassler cell at room temperature. A sealing pressure of 70 bar was applied to the samples. A screen of stainless steel was applied to the inlet and outlet faces of the plugs. This was of large mesh and designed to restrain larger rock grains.

After measuring the permeability to gas, vacuum was applied to the cores. The sample was saturated with liquid, pumping the fluid against vacuum, and then against a back pressure of 7 bar. This procedure assured that the sample was fully saturated with liquid.

In some cases the samples were not evacuated after the gas permeability measurement, and the permeability to fluids with trapped gas was measured.

The fluids used were as follows:

- 1) Formation water
- 2) CaCl_2 -brine, $d = 1.14 \text{ gcm}^{-3}$
- 3) Mud filtrate
- 4) Non-aromatic lamp oil
- 5) 15% HCl.

The formation water, CaCl_2 -brine, mudfiltrate and oil were filtered through 0.45μ filters before use.

4.1 Comments to Results

There was a marked difference in the rock quality between the two zones. This was reflected in the quality of the samples. Samples from the upper zone were irregular and difficult to measure for length and diameter. Samples from the lower zone were of good quality.

5. RESULTS OF PERMEABILITY MEASUREMENTS

Results from the permeability measurements have been summarised in Table 3 (1521 m, testzone 1) and Table 4 (1380 m, testzone 2).

For all cores; except number one in testzone 1, gas permeability was measured on washed and dried cores. The cores were then evacuated and the permeability to fluids at saturation was measured. The ratio between fluid and gas permeability for both zones is given in Table 5, and give information about the short time influence of fluids on core material.

5.1 1521 m, testzone 1

Results in Table 5 show that none of the fluids has a dramatic effect on the formation permeability.

The nonacidic fluids; formation water, CaCl_2 -brine and oil have a moderately lower permeability than the gas. The lowest ratio is found for mud filtrate. This value should, however, be considered as a lower limit because the plug was partly saturated with fluid (MF) before evacuation.

The CaCl_2 -brine was exposed to the sample overnight and a moderate production of fines was observed.

The HCl-permeability relative to gas permeability is somewhat lower than for the nonacidic fluids (Table 5). The acid was left to react overnight (static), and a bright green solution was produced (Table 3). The permeability was essentially unchanged. The acid was displaced by distilled water, and the permeability was again virtually unchanged.

Gas permeability was measured after mud filtrate, oil and HCl/H₂O at partial fluid saturation. Care should be taken to compare these numbers with initially measured gas permeabilities, as these were measured on washed and dried cores.

By displacing the fluids with gas fines material was produced.

5.2 1380 m, testzone 2

As for testzone 1, none of the nonacidic fluids gave a significant reduction in permeability compared to gas (Table 5).

Also for this zone, CaCl₂-brine was tested for longtime interaction. After leaving the brine in the core for one weekend the permeability was essentially unchanged (Table 4). However, a brown coloured "solution" was produced. This solution later settled, revealing that the colouration was due to fines produced from the core.

The 15% HCl interaction gave a prompt reduction in permeability (Table 4 and 5), and recovered to 30% of initial dry core gas permeability. ?

The permeability rose to 63% of initial gas permeability by flowing distilled water.

Also in this series of experiments care should be taken to compare gas permeabilities measured at partial fluid saturation to initially measured gas permeabilities on washed and dried cores. Also for this zone fines were produced by displacing the fluids with gas.

6. FORMATION STRUCTURE AND REACTION WITH FLUIDS

We have made the following tests:

- a) Screen analysis
- b) XRD-analysis of grain fractions and total
- c) Acid (15% HCl) solubility of grain fractions
- d) Solubility vs. time of formation in 15% HCl and 20% HAc.

6.1 The formation at 1521 m, testzone 1

The formation is well consolidated, fine sand (Table 6). The most striking observation is the high kaolinite content (Table 7). Analysis of the grain fractions shows that a large part of the kaolinite occurs as aggregates. The mechanical stress the material has been exposed to by sieve analysis is probably higher than the formation will be exposed to by fluid interaction in the well. Thus, the amount of kaolinite aggregates might be still higher.

The acid soluble minerals are mainly (Ca, Fe, Mg)-carbonates and pyrite, and comprise about 6% of the formation.

The solubility in 15% HCl of the fractions has been measured and is shown in Table 8. (Fig. 1-3). The solutions were heated to boiling three times and allowed to cool in between. The total reaction time was one hour. The solubility is given in weight %, and the numbers should be interpreted as relative. There is good relative agreement between the acid solubility and the amount of presumably acid soluble minerals in the different fractions from the XRD-analysis.

The solubility at 50°C of the total formation in 15% HCl and 20% HAc was examined as a function of time. The results are given as weight % of the formation of dissolved ions (Tables 9 and 10, Fig. 4-6). The major difference is that HCl attacks the formation much more aggressively and has a higher reaction rate than HAc. After one day treatment most of the iron containing minerals are dissolved. For calcium minerals the difference in efficiency of the two acids is much lower.

Calcite is effectively dissolved by both acids, i.e. any CaCO_3 used as filter control agent will be efficiently dissolved.

6.2 The formation at 1380 m, testzone 2

The formation is described as an unconsolidated, badly sorted sandstone with a large pore size. The pores are partly to totally filled with fines (Kaolinite).

The formation has a rather high content of the larger sized particles $> 150 \mu$, which amounts to about 70% (Table 11).

The XRF-analysis shows that the main components are quartz and feldspar. (Table 12). Kaolinite has its highest concentrations in the finer fractions, in agreement with the observation than one gets a rather stable suspension of fines by mixing sand and any fluid.

The larger grain fractions contain a significant part of the carbonates. Pyrite is also found in relative high concentrations in the cemented grains, but also as fines.

Acid solubility tests of grain fractions (Table 13, Fig. 7 and 8) show a rather good relative agreement with the content of presumably acid soluble minerals. The fractions $> 150 \mu$ contain about 80% of dissolved Ca. This might influence the stability of the formation by acid treatment.

The solubility at 40°C of the total formation in 15% HCl and 20% HAc was measured as a function of time. Also for this formation type, HCl is much more efficient in dissolving minerals than HAc. (Tables 14 and 15, Fig. 9 and 10).

The reaction rate and the fraction of dissolved carbonates and pyrite are lower than observed for material from testzone 1. This might partly be due to the lower temperature than in the other experiment (40°C vs. 50°C), but probably also because the minerals are somewhat more resistant to acid treatment.

7. CONCLUSIONS

Nonacidic fluids (as tested with CaCl_2 -brine) can have a detrimental effect on mobility of fines in the cores. This effect is much stronger in the upper zone, than in the lower zone. This can be understood by the larger pores in the upper zone formation.

The existence of kaolinite as aggregates in the lower zone also reduces movable fines. In the upper zone the relative amount of kaolinite increases with decreasing grain size.

A moving gas/liquid interface may more easily move fines than either gas or liquid alone.

Nonacidic fluids (as tested with mud filtrate) trap gas and reduce permeability to fluid when imbibed into gas saturated plugs. This effect is considerably higher in the lower zone.

15% HCl causes a prompt reduction in permeability in both upper and lower zones. This can clearly be correlated with the observation that HCl immediately attacks and dissolves carbonates and pyrite present in the rocks.

The prompt reduction in permeability is much higher in the upper zone than in the lower.

In the lower zone the acid was left in the plug to react overnight (at 20°C). The permeability was essentially the same. At higher temperature the reaction rate is much higher. After one day at 50°C most of the acid soluble minerals are dissolved. This will increase the pore volume and might improve permeability. On the other hand, prolonged acid treatment might activate fines, this having the reversed effect. 20% acetic acid has a lower reaction rate and dissolves less of the present carbonates and pyrite than HCl.

In the upper zone the prompt reduction in permeability to HCl was substantial. The permeability to fluid recovered well by flowing distilled H₂O after acid treatment.

Carbonates and pyrite in the formation are immediately attacked by HCl at 40°C. Both the reaction rate and fraction of material dissolved is lower than in the lower zone. The relative enrichment of acid soluble minerals in the larger grain fractions may have negative effects on formation stability after prolonged acid washing.

Again 20% HAc has a more moderate influence on the sand, and may have positive effects on formation by acid treatment.

APPENDIX I

Concentrations of Ca^{++} , Fe^{3+} and Mg^{++} ions were measured in the different fluids before and after flowing through cores.

The idea was to look for any chemical interaction between core material and fluid, f.ex. reduction of ion concentration due to deposition in the core. Due to experimental difficulties in sampling single core volumes of fluids after flow no detailed results could be obtained.

For none of the nonacidic fluids ion concentrations changed. For the HCl we measured the expected increase of ion concentration due to dissolved core material.

The fines produced in the flow experiment was filtered off and dissolved in acid. The amount of material was too small to make detailed analysis, but Fe^{3+} was found in the acidic solution, indicating that at least a part of the fines was pyrite.

APPENDIX II

Critical point drying is a method of plug preparation designed to preserve delicate clay structures. The method consists of saturating a rock sample with methanol and allowing to soak for 2-3 days. This removes salt and water.

Ethanol is then used to replace the methanol by diffusion over a further 2 to 3 days.

The ethanol is then removed by applying a CO₂ pressure of 80 bar, which is above the critical pressure of ethanol, methanol, and CO₂. At this pressure, all components are miscible. Thus CO₂ will enter the pores and dilute the ethanol & methanol present. The temperature is now raised to 38°C. This temperature is above the critical temperature for CO₂, which is now the major component of the system. Pressure is released isothermally until atmospheric pressure is reached. The temperature is allowed to come to ambient conditions. The samples are now dry and by following the above procedure, no liquid/vapor interface has been allowed to pass through the sample. Delicate clay structures have therefore not been subjected to mechanical stress, and so will be closer to their original reservoir state.

APPENDIX III

Grain size analysis of gravel after use in well 31/3-1

Fraction	W%
> 850	0.03
500 - 850	85.78
425 - 500	13.65
< 425	0.54

No significant reduction in gravel size is observed.

Table 1

Vannprøve brønn 31/2-11
DST 1, 17.3.83, 1681 - 85 m.

	Prøve 15	Prøve 16	Syntetisk vann
Ion	kons (mg/l)	kons (mg/l)	kons (mg/l)
Na	14200	18100	14000
K	200	465	200
Mg	480	470	500
Ca	2000	1945	2000
Ba	215		
Cl	29450	29590	30000
SO ₄	<5	<5	0
HCO ₃	220	220	200
CO ₃	0	0	0
pH	7.66	7.66	
Tetthet 20°C (g/cm ³)	1.034	1.034	
Resistivitet 20°C (Ωm)	0.168	0.168	
Kin.viskositet (mm ² /s)	1.083		1.075

Table 2

Mudfiltrate, well 31/3-1

Dissolved solids:

<u>Cations</u>	<u>mg/l</u>	<u>me/l</u>
Sodium, Na (meas)	5.250	228
Calcium, Ca	1.210	61
Magnesium, Mg	88	7
Barium, Ba	3.2	-
Iron, Fe	1.4	-
Strontium	23	1
Potassium	27.750	710
<u>ANIONS</u>		
Chloride, Cl	45.440	1.280
Sulfate, SO ₄	700	15
Carbonate, CO ₃	0	-
Bicarbonate HCO ₃	525	9
Hydroxide	0	-
<hr/>		
<hr/>		
Total Dissolved Solids (calc.)	80.991	
	<hr/>	mg/l
	<hr/>	
pH	7.82	20°C
Specific Gravity.	1.056	
Resistivity (ohm-meters)	0.089	20°C

Table 3. Permeability (md) to N₂ and different fluids for plugs from 31/3-1, 1521 m - testzone 1. Permeabilities for gas are Klinkenberg corrected. All plugs, except in Expt. 1 were washed and dried prior to the first gas permeability measurements.

SAMPLE FLUID/ SOLUTION	PERMEABILITY mD		PERMEABILITY mD		PERMEABILITY mD		PERMEABILITY mD	
	1	2	3	4	5	6	1	2
INITIAL GAS PERMEABILITY	—	15.9	26.4	27.9	217	210	—	—
FORMATION WATER								
(1) 100% SATD		13.2	—	—	—	—		
(2) TRAPPED GAS	20.3							
CaCl ₂ 100% SATD								
(1) INITIAL			23.0					
(2) OVERNIGHT			23.0*					
MUD FILTRATE								
(1) TRAPPED GAS				3.6				
(2) SATURATED				12.2				
NON-AROMATIC OIL								
(1) TRAPPED GAS					200			
(2) SATD					200			
HCl 15%								
(1) overnight						129		
(2) displaced with water						124*		
						127		
GAS FINAL			13.3	15.8	104	80.5°		

*green solution
°slight fines

*slight fines

Table 4. From 31/3-1, 1380 m, testzone 2. Permeabilities for gas are Klinkenberg - corrected. All samples were washed and dried prior to the first gas permeability measurement, and evacuated prior to the first fluid measurement.

SAMPLE FLUID/ SOLUTION	PERMEABILITY nm ²	1	2	3	4	5
		cleaned	cleaned	cleaned	cleaned	cleaned
INITIAL GAS						
PERM.Ker		1265	337	173	236	204
FORMATION WATER						
100% SATD		1297	—	—	—	—
CaCl ₂ 100% SATD		—		—	—	—
(1) initial			252			
(2) overnight			181			
(3) over weekend			*223			
MUD FILTRATE						
(1) Trapped Gas		—	—	51.2	—	—
(2) SATURATED				166		
NON-AROMATIC OIL (SATD)		—	—	—	225	—
HCl 15%		—	—	—	—	38.3 -> 61.6
(1) followed by water						112 -> 128
GAS FINAL (DISPLACING FLUID)		732	*76	53.4*	114	169*
			* fines produced			*fines much produced

Table 5. Ratio of permeability to fluids at saturation, R_{sf} (after evacuating core) and gas permeability R_{sf}/R_g

	1521 m, test-zone 1	1380 m, test-zone 2
Formation		
water (FW)	0.83	1.03
CaCl ₂ -brine (1.14 gcm ⁻³)	0.87	0.75
Mud-filtrate (MF)	0.44*)	0.96
Oil	0.92	0.95
15% HCl	0.61	0.19 - 0.30

*) Value may have been affected by evacuating the core after partial fluid (MF)-saturation, and should be treated as lower limit.

Table 6.

Grain size analysis, testzone 1, 1521 m

Grain size	W % of total
>600	0.20
300 - 600	11.00
150 - 300	17.32
75 - 150	30.90
45 - 75	16.66
<45	23.92

Table 7.

XRD-analysis of material from testzone 1, 1521 m

Mineral	Composition (% of fraction)				
	45- 75	75- 150	150- 300	30- 600	Total
Mixed layer	3.0	3.9	6.6	5.2	4.5
Mica	6.7	7.2	11.5	9.7	11.4
Kaolinite	30.6	30.7	47.6	49.7	45.8
Quartz	27.5	23.9	10.5	11.8	14.6
Feldspar	26.9	27.6	17.4	15.1	18.2
Calcite	trace	1.2	1.3	1.1	trace
Dolomite/Ankerite	1.5	2.6	1.0	1.3	1.9
Siderite	0.9	0.8	0.5	0.5	0.4
Pyrite	2.8	2.3	3.6	5.7	3.1

Table 8.

Solubility of fractions in 15% HCl, testzone 1, 1521 m.

Grain size	Fe		Ca		Mg	
	Weight % (relative units)					
	% of fraction	% of total	% of fraction	% of total	% of fraction	% of total
>600	0.84	0.00	0.13	0.00	0.11	0.00
300-600	2.01	0.22	0.35	0.039	0.15	0.017
150-300	2.20	0.38	0.46	0.079	0.21	0.037
75-150	2.09	0.65	0.21	0.065	0.37	0.113
45-75	1.33	0.22	0.18	0.031	0.13	0.022

Table 9.

Solubility vs. time in 15% HCl at 50°C
Material from testzone 1, 1521 m

Reaction time t	Fe W%	Ca W%	Mg W%
2 m	0.92	0.41	0.12
5 m	0.95	0.42	-
12 m	1.30	0.46	0.18
25 m	2.64	0.58	0.32
50 m	2.63	0.55	0.30
25.45 h	6.67	0.66	1.48
53.20 h	7.72	0.69	1.77

Table 10.

Solubility vs. time in 20% HAc at 50°C
Material from testzone 1, 1521 m

Reaction time t	Fe W%	Ca W%	Mg W%
2 m	0.36	0.16	0.06
5 m			
7 m	0.40	0.21	0.06
12 m	0.29	0.22	0.04
26 m	0.26	0.23	0.04
50 m	0.57	0.27	0.08
25.70 h	1.42	0.42	0.16
53.47 h	1.71	0.42	0.18

Table 11.

Grain size analysis, testzone 2, 1380 m

W % of total	
>600	13.30
300 - 600	26.35
150 - 300	26.42
75 - 150	15.38
45 - 75	6.66
<45	11.89

Table 12.

XRD-analysis of material from testzone 2, 1380 m

Composition (% of fraction)

Mineral	45- 75	75- 150	150- 300	300- 600	>600	Total
Mixed layer	3.0	2.6	1.7	-	-	trace
Mica	10.9	6.7	5.2	3.4	3.2	4.0
Kaolinite	27.5	23.0	10.3	3.8	4.3	12.5
Quartz	24.6	24.7	41.0	47.8	42.2	41.0
Feldspar	28.5	37.4	35.2	35.0	36.4	33.9
Calcite	0.5	0.4	1.4	2.4	6.1	2.2
Dolomite/ Ankerite	1.5	2.9	3.1	6.0	2.7	3.0
Linderite	0.4	-	trace	-	-	0.3
Pyrite	3.2	2.4	1.4	1.7	5.2	3.0

Table 13.

Solubility of fractions in 15 % HCl, testzone 2, 1380 m

Grain size	Fe		Ca	
	% av fraksjon	% av total	% av fraksjon	% av total
>600	0.36	0.048	0.284	0.038
300 - 600	0.23	0.060	0.067	0.018
150 - 300	0.22	0.058	0.073	0.019
75 - 150	0.74	0.114	0.029	0.005
45 - 75	0.96	0.064	0.112	0.007

Table 14.Solubility vs.time in 15 % HCl at 40°C
Material from testzone 2, 1380 m

Reaction time	Fe (W %)	Ca (W %)
2 m	0.43	0.58
12 m	0.47	0.53
20 m	0.56	0.49
37 m	0.62	0.54
4.92 h	0.73	0.49
23.25 h	0.94	0.51
24.78 h	0.96	0.51

Table 15.

Solubility vs.time in 20 % HAc at 40°C
Material from testzone 2, 1380 m

Reaction time	Fe (W %)	Ca (W %)
3 m	0.08	0.16
5 m	0.13	0.19
15 m	0.13	0.31
20 m	0.14	0.32
39 m	0.15	0.36
58 m	0.19	0.41
5.25 h	0.18	0.40
24.08 h	0.29	0.40

Figure Captions

- Fig. 1 Solubility of Fe-minerals in grain fractions of material from 1521 m.
- Fig. 2 Solubility of Ca-minerals in grain fractions of material from 1521 m.
- Fig. 3 Solubility of Mg-minerals in grain fractions of material from 1521 m.
- Fig. 4 Solubility of Fe-minerals in samples from 1521 m in 15% HCl and 20% HAc at 50°C vs. time.
- Fig. 5 Solubility of Ca-minerals in samples from 1521 m in 15% HCl and 20% HAc at 50°C vs. time.
- Fig. 6 Solubility of Mg-minerals in samples from 1521 m in 15% HCl and 20% HAc at 50°C vs. time.
- Fig. 7 Solubility of Fe-minerals in grain fractions of material from 1380 m.
- Fig. 8 Solubility of Ca-minerals in grain fractions of material from 1380 m.
- Fig. 9 Solubility of Fe-minerals in samples from 1380 m in 15% HCl and 20% HAc at 40°C vs. time.
- Fig.10 Solubility of Ca-minerals in samples from 1380 m in 15% HCl and 20% HAc at 40°C vs. time.

31/3-1

1521 m Testzone 1

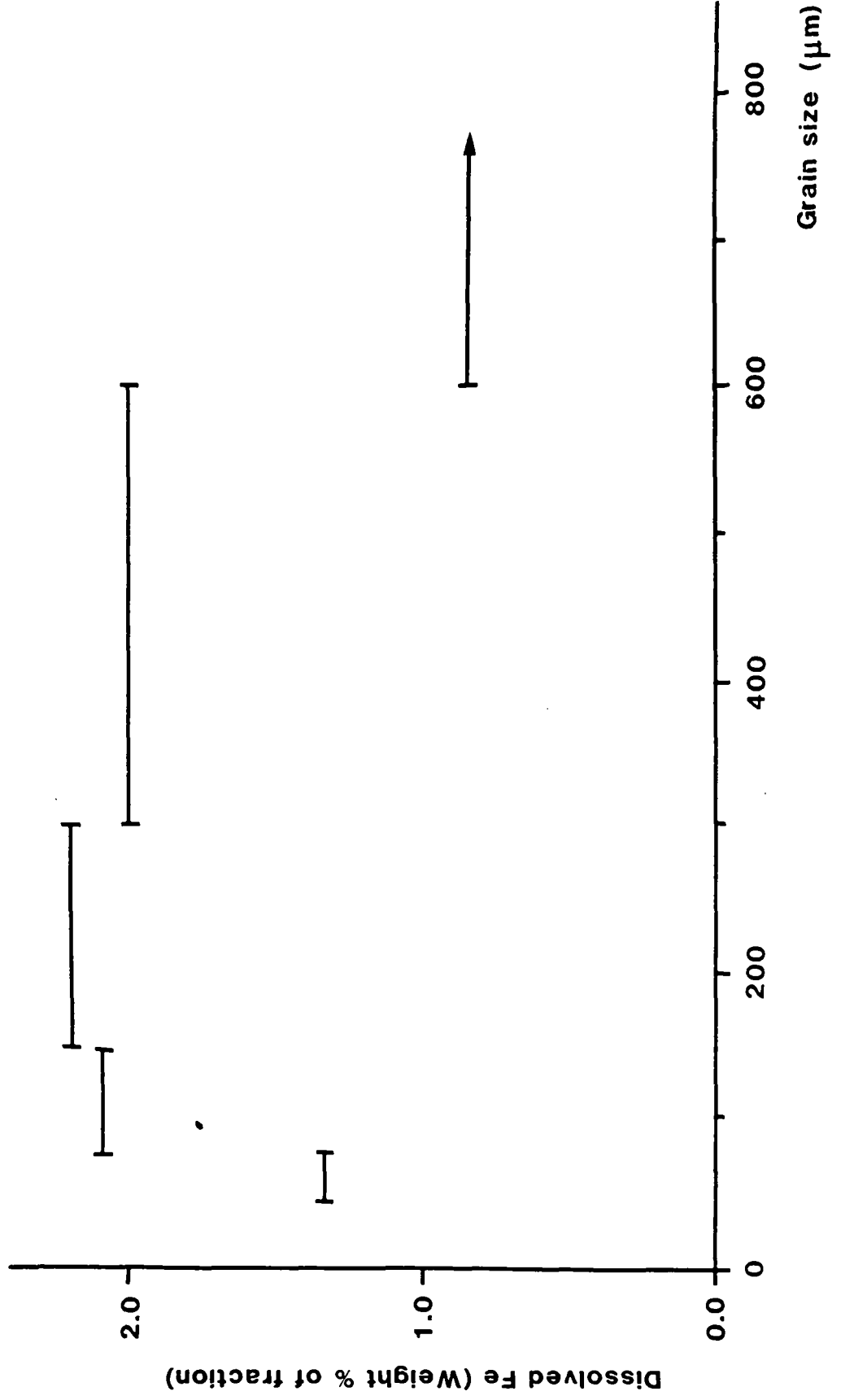


Fig. 1

31/3-1

1521m Testzone 1

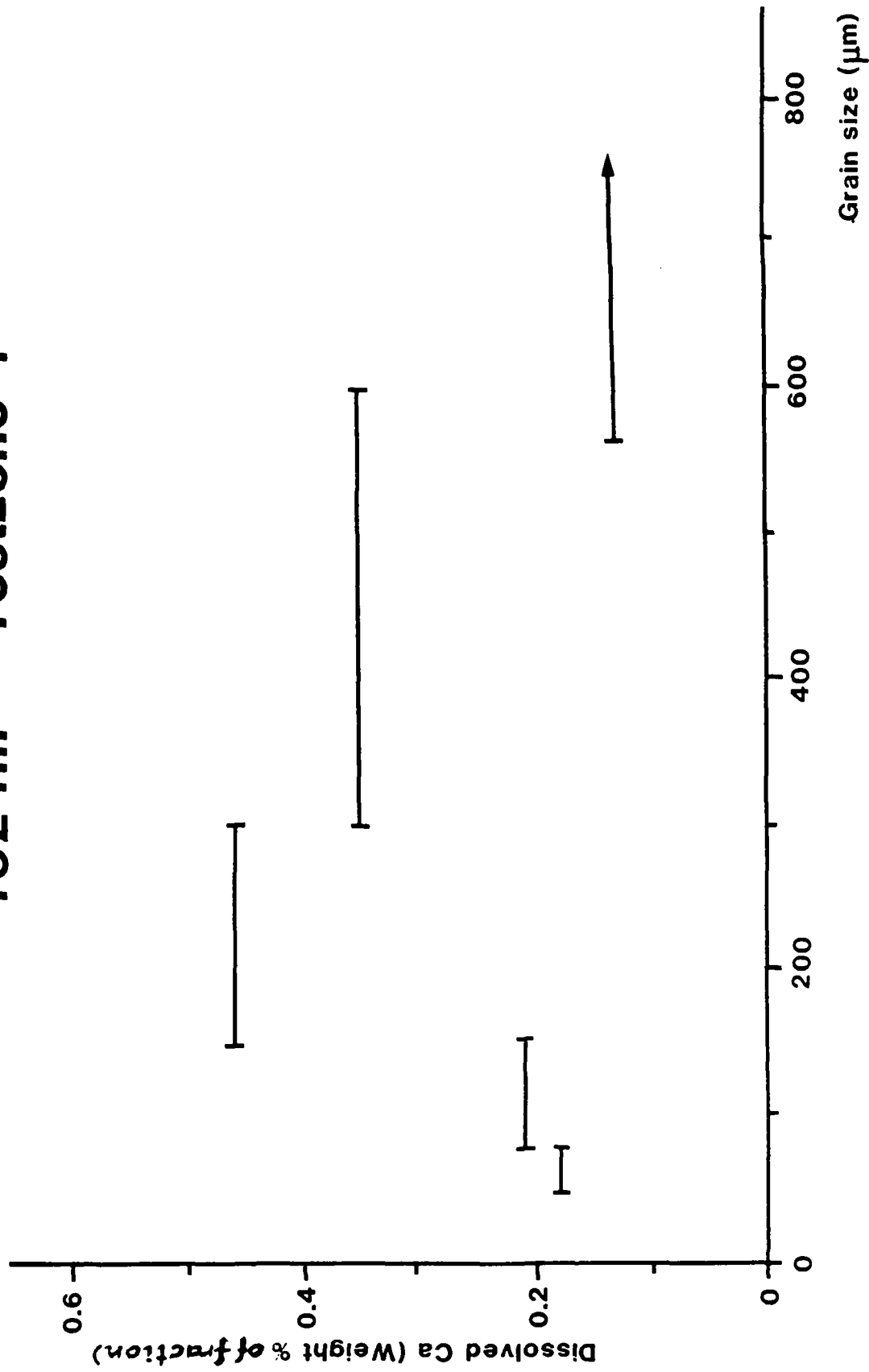


Fig. 2

31/3-1

1521 m Testzone 1

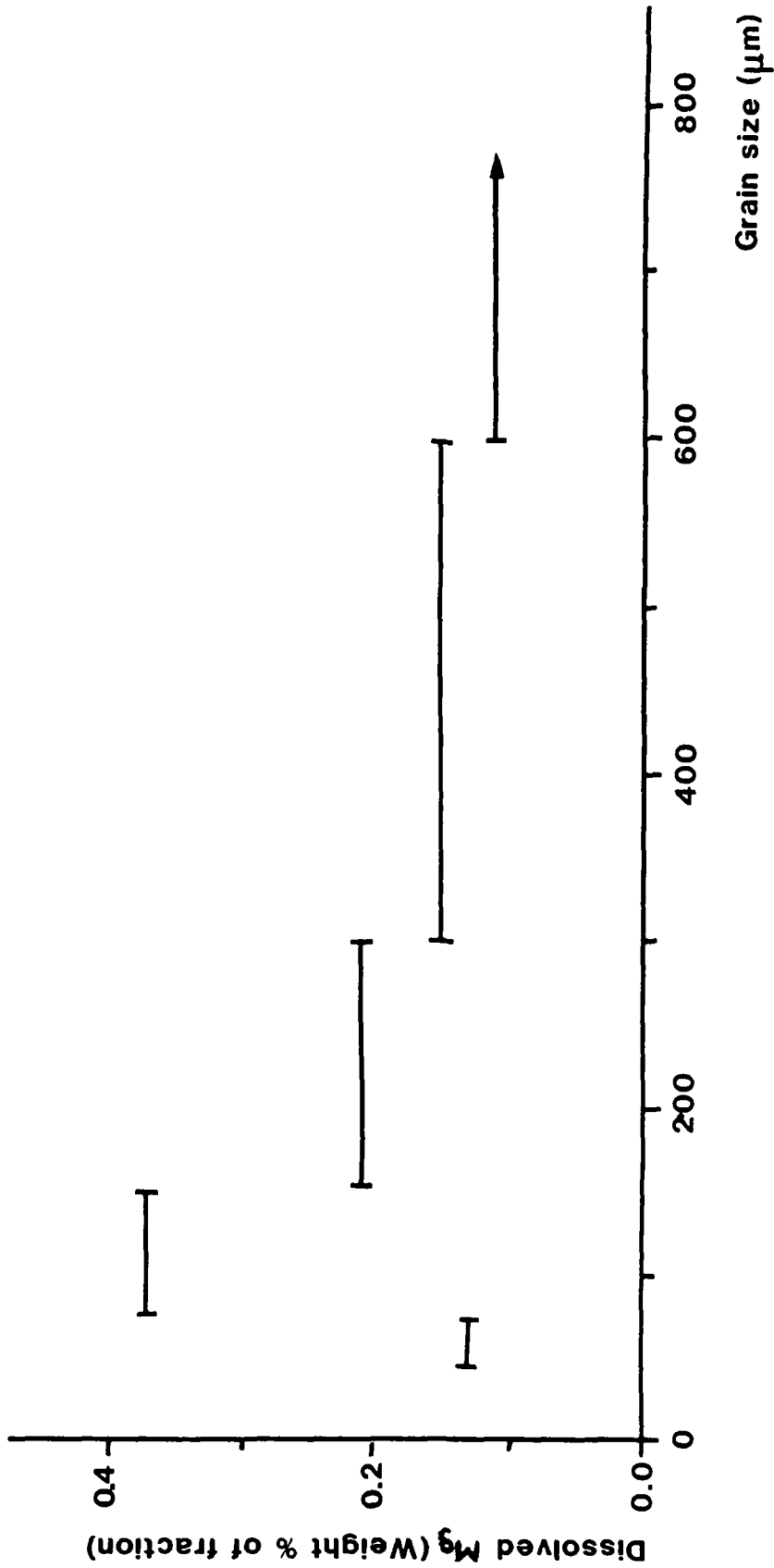


Fig.3

31/3-1

1521 m Testzone 1

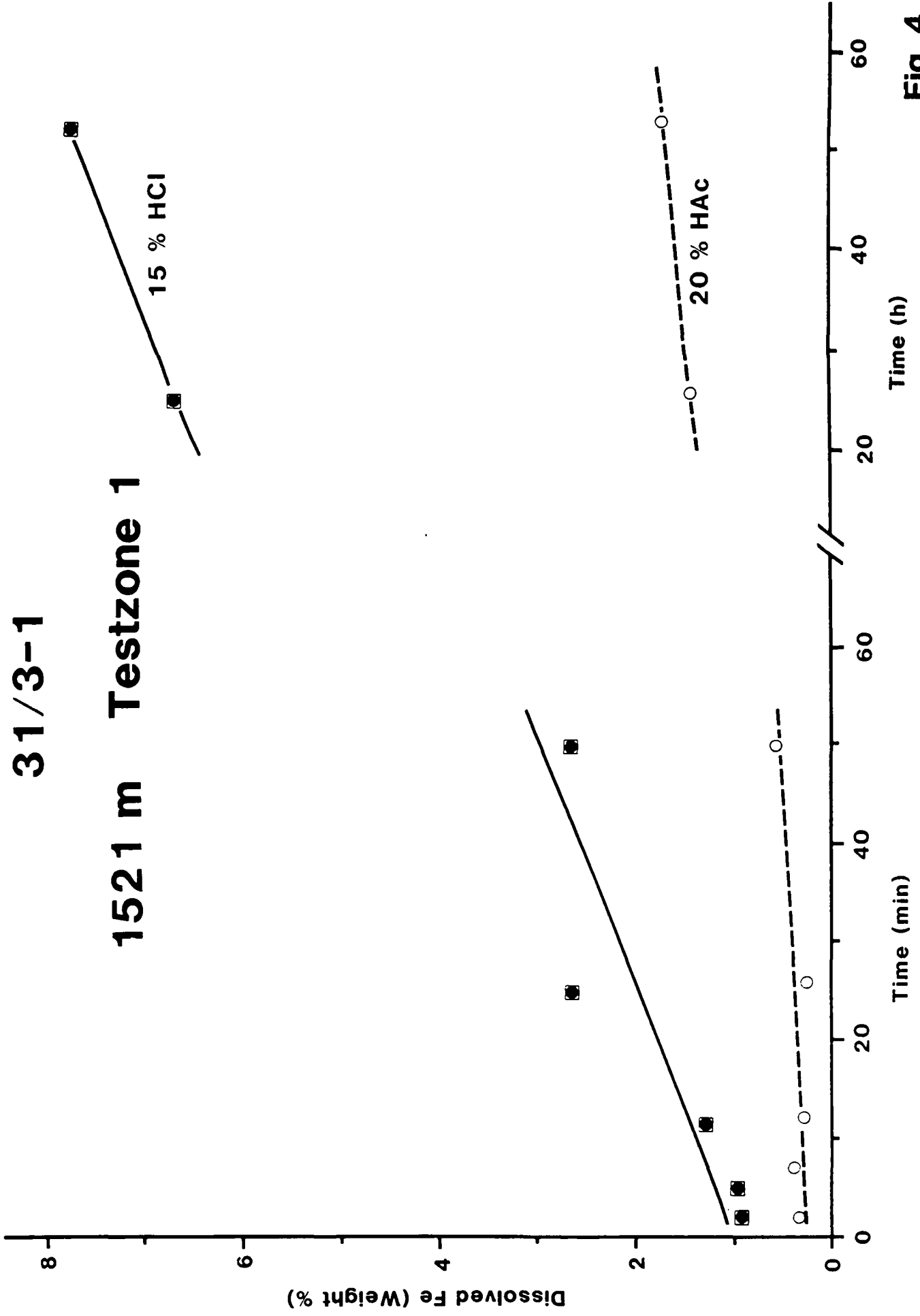


Fig. 4

31/3-1

1521 m Testzone 1

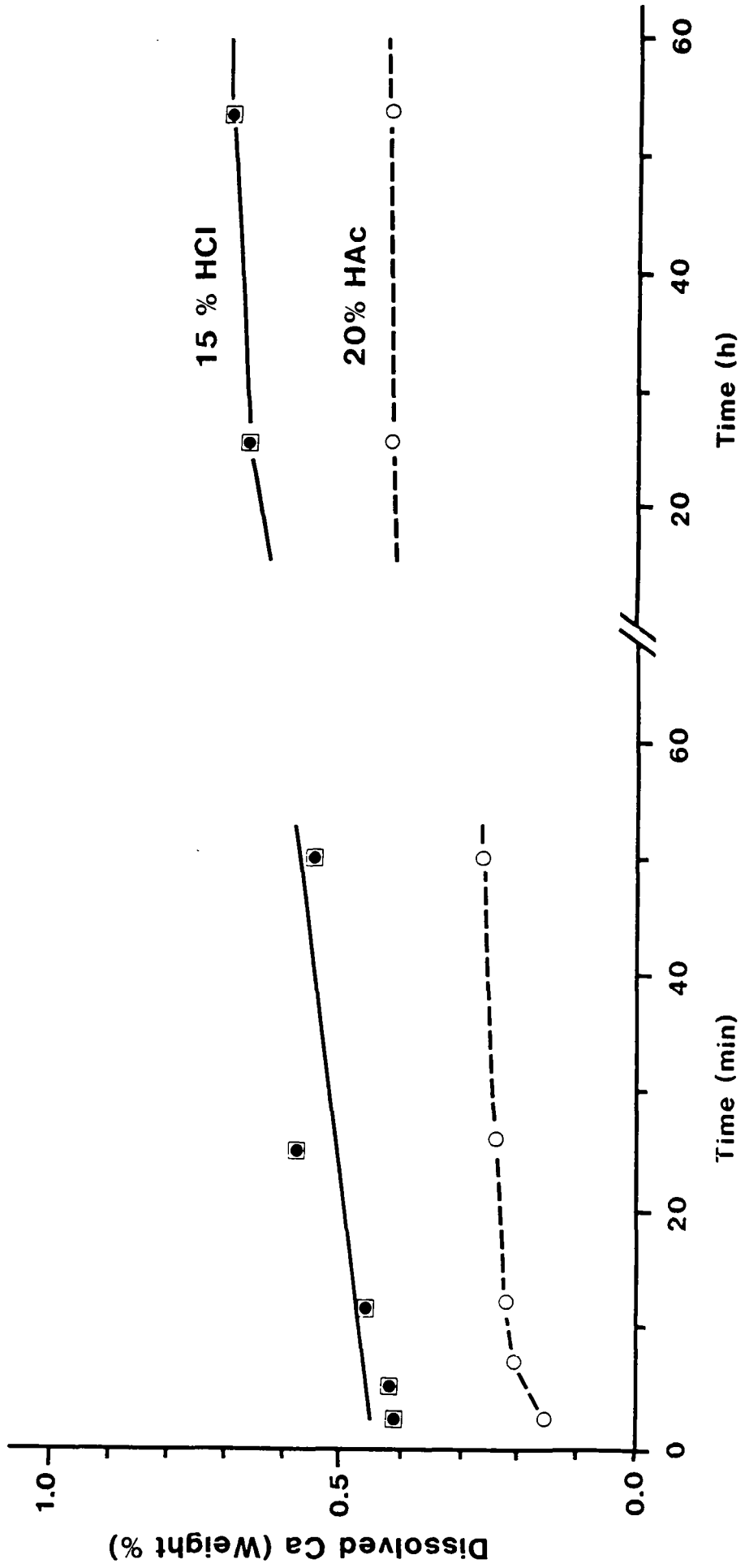


Fig. 5

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1521 m Testzone 1

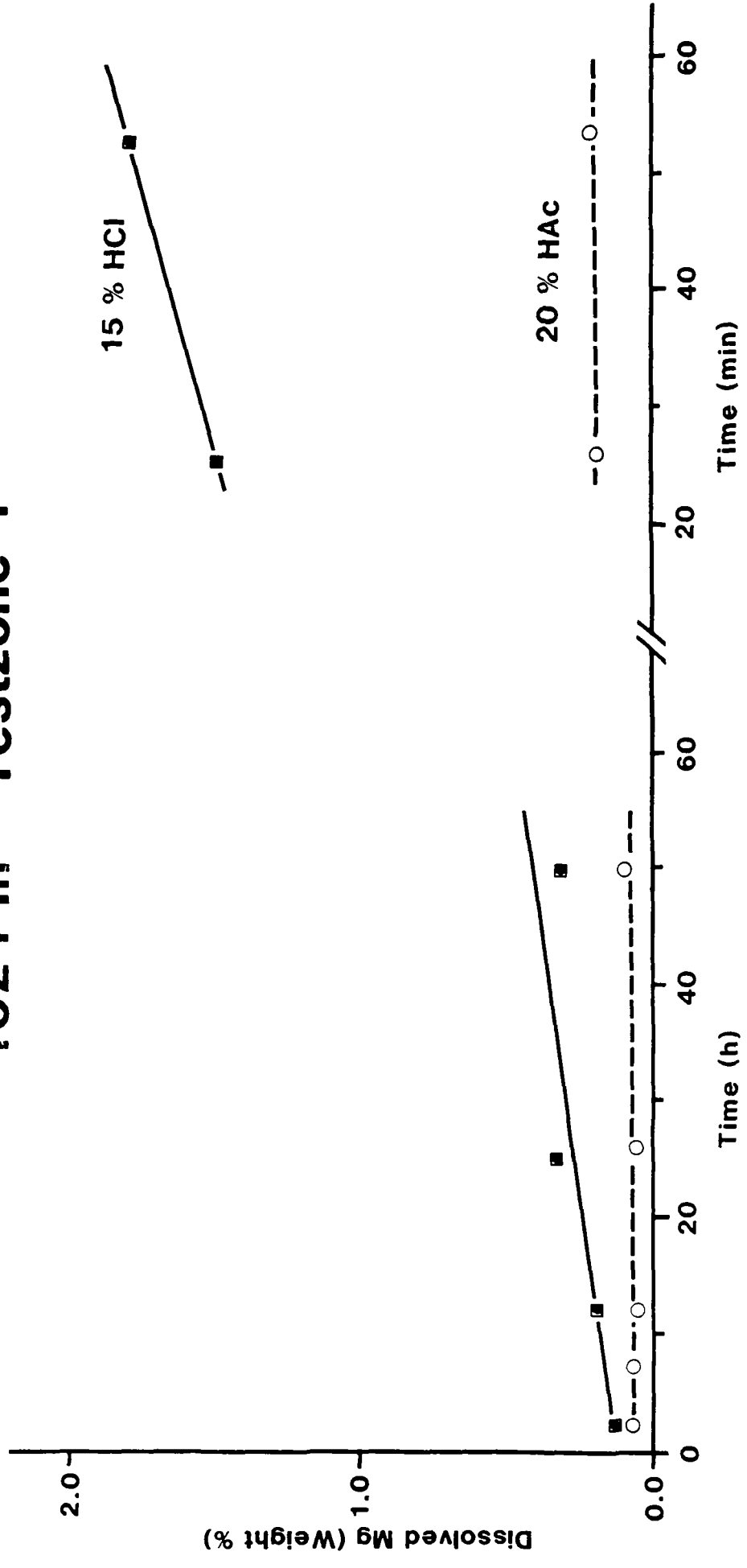


Fig. 6

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1380 m Testzone 2

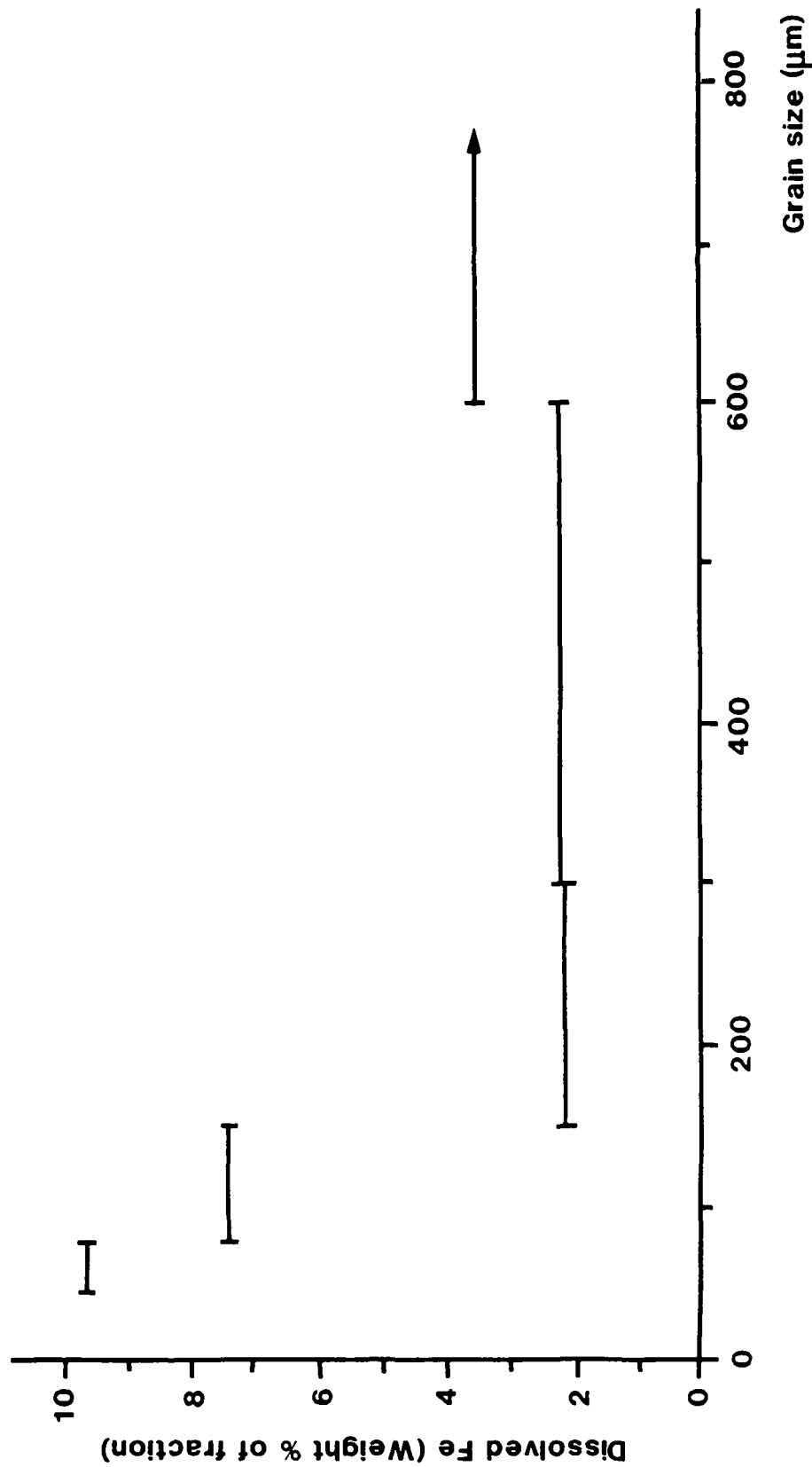


Fig. 7

31/3-1

1380 m Testzone 2

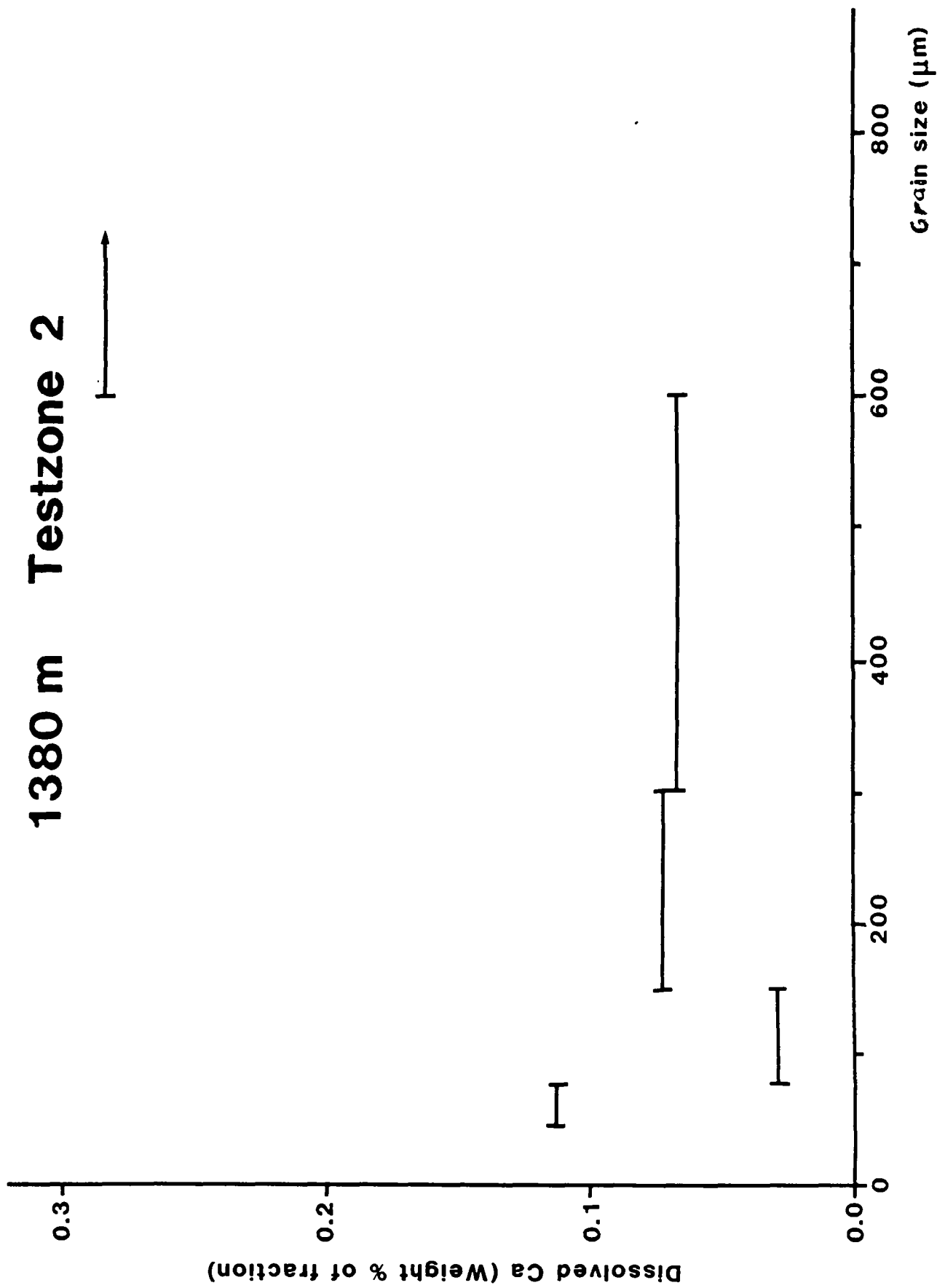


Fig.8

31/3-1

1380m Testzone 2

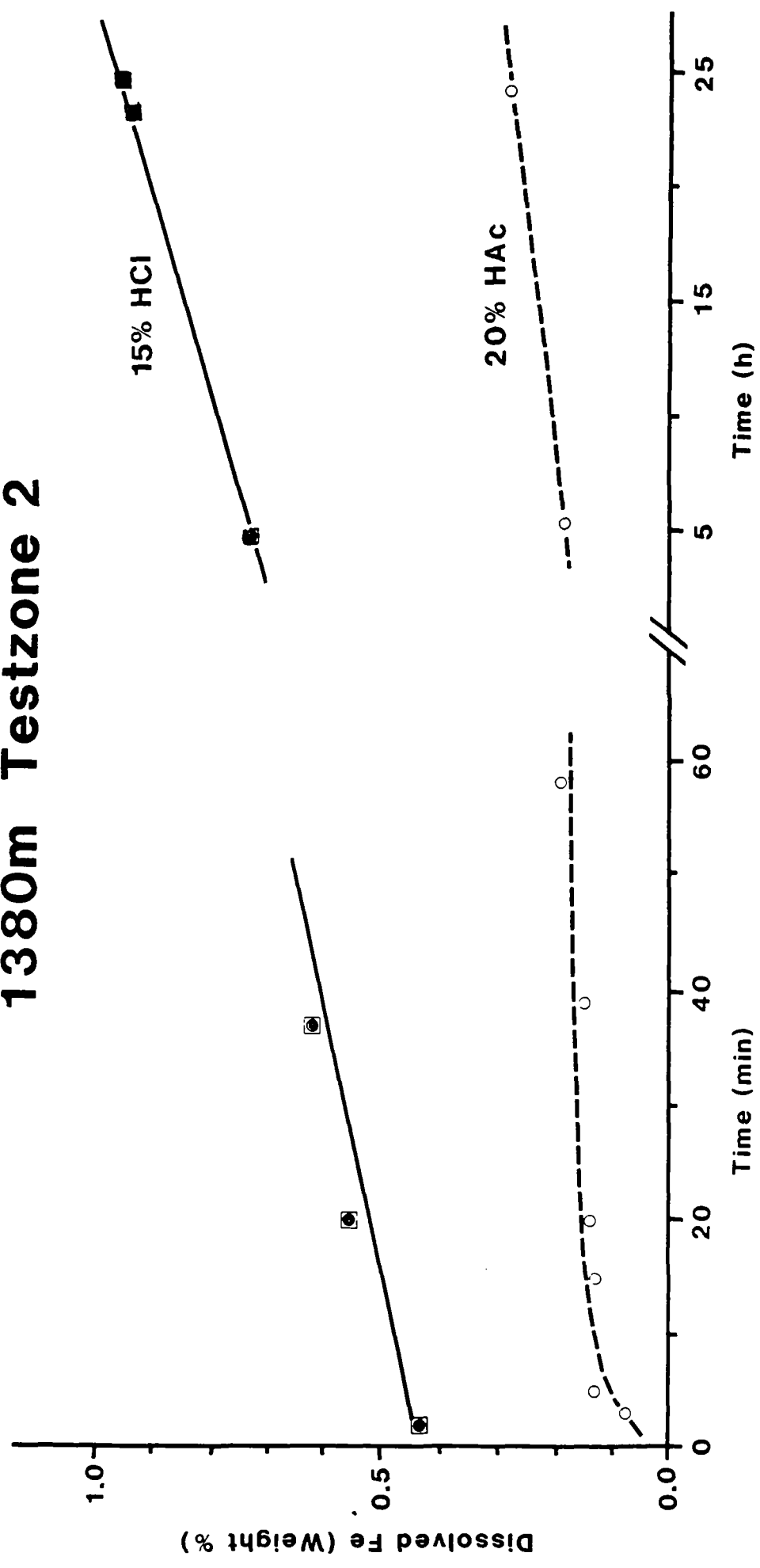


Fig.9

31/3-1

1380m Testzone 2

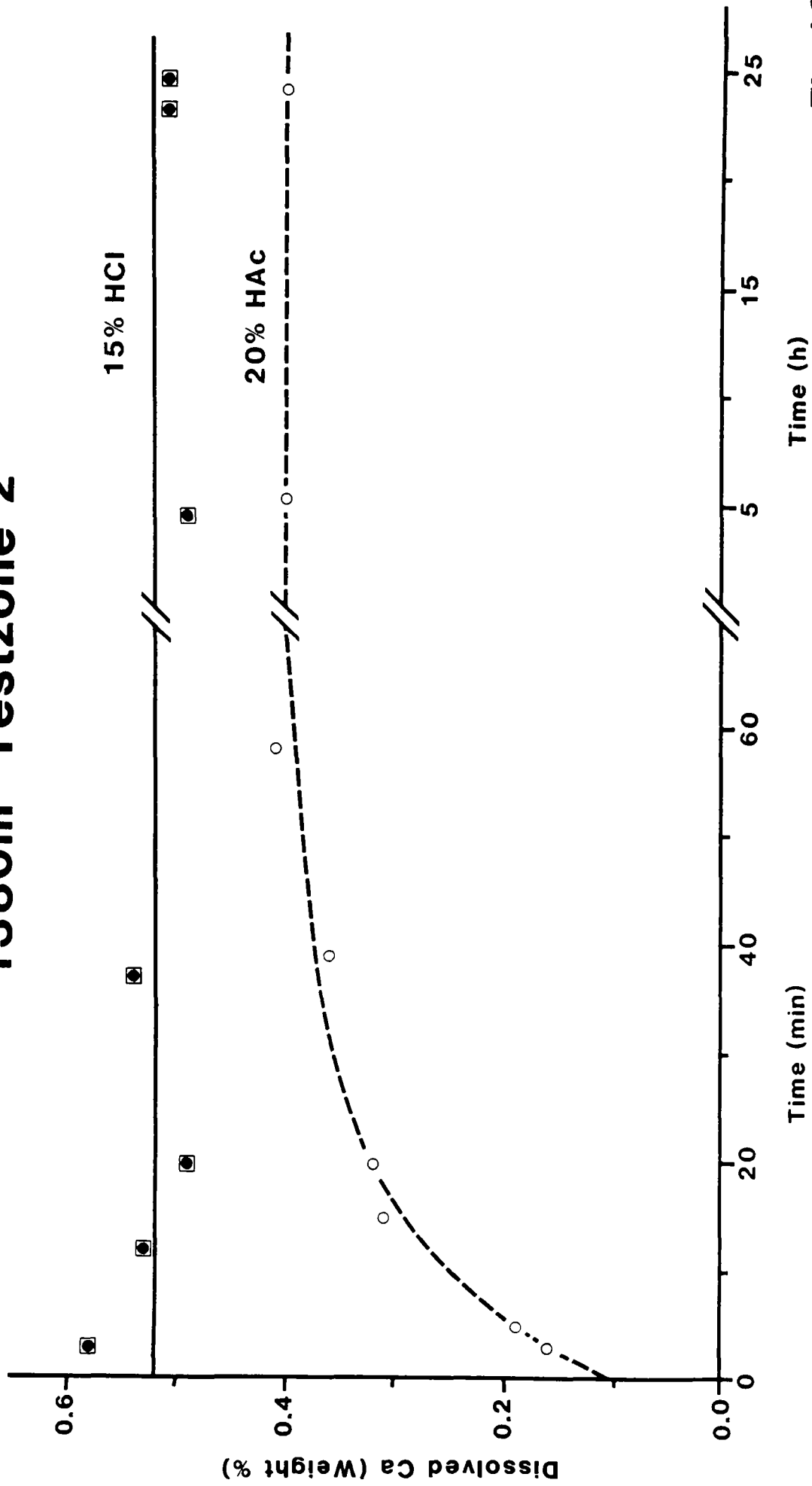


Fig. 10