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Experimental Approach to Performance of Engineered Barriers under Repository Conditions

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2001



Experimental Approach to Performance of Engineered Barriers under Repository Conditions

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要 旨

本論文は、「処分環境下における人工バリア材の性能に関する実験的研究」と題して1987年8月24, 25日にスイスのローザンヌで開催された会議**で発表した内容の詳細を記述したものである。

処分環境下における人工バリア材（ガラス固化体，オーバーパック材及び緩衝材）の耐久性を評価する目的で実施した実験的研究は，次のようにまとめられる。

- (1) ガラス固化体浸出特性：1年以上の浸出試験の結果，高いSA/V比（試料表面積/浸出液量比）の条件で浸出が抑制された。これは，浸出液中のシリコン濃度の飽和により，他の元素の浸出が抑制されたためと考えられる。
- (2) オーバーパック候補材の腐食挙動：炭素鋼とチタンについて模擬地下水及びベントナイト共存下で試験をした結果，局部腐食は認められなかった。炭素鋼の腐食速度はベントナイト/水比に依存することがわかった。また，チタン系材料について，すきま腐食発生の臨界条件を求めた。
- (3) 緩衝材（圧縮ベントナイト）の止水性能：ベントナイト密度の増加とともに透水係数は小さくなり，膨潤圧は大きくなる。ベントナイトにクラックが存在しても著しい透水性能の劣化は起きなかった。また，処分開始時を想定し，乾燥ベントナイトへの水の浸入速度を評価した。

* 環境工学開発部

** Conference Seminar of 9th Structural Mechanics in Reactor Technology,

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EXPERIMENTAL APPROACH TO PERFORMANCE OF ENGINEERED BARRIERS UNDER REPOSITORY
CONDITIONS

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ABSTRACT

Experimental studies have been carried out in order to evaluate durability
of engineered barriers in geological disposal system. These are as follows;

(1) Leaching behavior of waste borosilicate glass

Leaching experiments of simulated waste glass were carried out for more than
one year in both scale of laboratory and of engineering. The leachability was
suppressed under the condition of high SA/V (the surface area of glass/
leachant volume). Saturation of leachant with silicon reduces leaching rates
of the other elements.

(2) Corrosion behavior of candidate overpack materials

Corrosion behavior of carbon steel and titanium was studied in synthetic
groundwater with or without bentonite. No localized corrosion was observed in
both the materials. Corrosion rate of carbon steel depends on bentonite/water
ratio. Crevice corrosion resistance of titanium and its alloy was also studied.

(3) Permeability of compacted bentonite as buffer material

Hydraulic conductivity of saturated compacted bentonite decreases and the
swelling pressure increases with the increase of density. Influence of cracks
on permeability was also studied, and significant effect was not observed.
Infiltration of water into dry compacted bentonite was also studied to
evaluate permeability of buffer material at the initial stage of disposal.

1. INTRODUCTION

For the sake of development of disposal system for high level radioactive waste in Japan, Japanese Atomic Energy Commission determined the following four phases:

Phase 1: Selection of effective geological formations

Phase 2: Selection of candidate sites for geological disposal

Phase 3: Demonstration of technology for disposal using simulated waste forms

Phase 4: Disposal of real wastes

Phase 1 was terminated and phase 2 is now underway since 1985. The Power Reactor and Nuclear Fuel Development Corporation (PNC) is prescribed as the central organization in the development of technology for treatment and disposal of radioactive waste in Japan.

The disposal system for high level radioactive waste consists of engineered barriers including glass (fuel), canister, overpack and buffer materials, and natural barriers such as host rock. Details of the disposal system of Japan is not yet designed.

Durability of engineered barriers is one of the governing factors for long-term performance assessment of the disposal system. Descriptions in this paper are focused on some preliminary studies of durability of engineered barriers in laboratory scale ; regarding

- 1) Leaching behavior of waste borosilicate glass,
- 2) Corrosion behavior of overpack materials, and
- 3) Permeability of buffer material.

Field tests and natural analogue investigation of the engineered barriers are already in our schedule.

2. LEACHING BEHAVIOR OF WASTE BOROSILICATE GLASS

Leaching studies under predicted disposal conditions are significant to evaluate a source term for the multiple waste package system and long-term stability of waste glasses. Experiments for more than 364 days (Table 1-1) have been made on and the long-term leaching behavior has been evaluated.

(1) Long-term Soxhlet Test

Figure 1-1 shows normalized mass losses as a function of time at 100 °C on 60 mL/hr flow rate. Mass losses increased linearly with time.

Specimen after leaching for 1095 days was studied by scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX). Transition elements (Fe, Cr, Ni) and rare-earth elements (La, Ce, Nd) were concentrated in the surface layer, while silicon and alkali elements were depleted.

(2) Long-term Static Test

Figure 1-2 shows the weight losses as a function of time in three kinds of leachant. Granite water was found to reduce the leach rate because of its relatively high silicon concentration [1].

On the other hand, acceleration of leaching was observed in the bentonite solution at low bentonite/water weight ratio (B/W=1/200). The enhanced leaching seems to be due to the sorption and ion exchange properties of the bentonite [1]. Figure 1-3 shows the weight losses as a function of time in various solutions. At high ratio of bentonite/water (B/W=1/2), the weight loss was one or two orders lower than those in distilled water and bentonite solution of B/W=1/200. Secondary ion mass spectrometry (SIMS) analyses of specimen (B/W=1/2, after 1 year leaching) showed that the thickness of boron depleted layer was approximately 0.3 μm (Figure 1-4).

Leaching behavior at high B/W ratio may be different from those in distilled water and bentonite solution of low B/W ratio.

(3) Solubility Test

The purposes of solubility test are to obtain the maximum concentration of elements dissolved in leachant, and to identify alteration products after leaching.

Most of solute elements seemed to reach the saturated concentration within one year in this condition. The saturated concentration of each element was not influenced by nature of leachants (Table 1-2).

X-ray diffraction pattern of the alteration products was of montmorillonite.

(4) Large Scale Test

Figure 1-5 illustrates concentration of the leachates from large scale and small leach tests. The relations between concentration and time of both the tests show similar tendency. Both the leaching behavior may be controlled by similar factors.

Large scale glass has many cracks, and the thickness of altered layer on a crack surface was $0.2 \sim 0.3 \mu\text{m}$. The thickness of altered layer on outer surface was about $100 \mu\text{m}$, so the mass loss per unit area of crack part was negligibly small in comparison with that of outer surface part. Presence of crack does not significantly affect leachability.

(5) Discussion

Composition of leachate solutions after one year leach tests is given in Table 1-3. The concentration of each element did not reach the saturated concentration that measured in solubility test. Silicon concentration reached about 19~63 % of the saturated concentration, while concentrations of more soluble elements such as boron and sodium reached only about 0.3 ~2 % of the saturated concentrations. Therefore, silicon has the leading role related to decrease of leaching rate with time in the closed system. The reduction of leaching rate in the granite water is also explained by the silicon saturation effect due to its initially high silicon concentration

[1~3] .

Comparison between the results of both large scale and small scale tests suggests that the leachability is suppressed under high SA/V ratio. Effect of SA/V ratio also explains the suppression of leachability under high B/W ratio, because high B/W ratio limits the amount of water in contact with the glass surface. On the conservative assumption that leaching rate is constant at this condition (B/W=1/2, 98 °C), the thickness of boron depletion layer would be no more than 300 μm for thousand years.

Thus the value of SA/V can be expected to have considerable effects on leach mechanism, and leach behavior is expected to be primarily controlled by silicon saturation.

3. CORROSION BEHAVIOR OF CANDIDATE OVERPACK MATERIALS

Two concepts concerning long-life overpacks are prevailing. One is to fabricate thin overpacks from highly "corrosion resistant" metals such as pure titanium, titanium alloy or nickel base alloy. The other is to utilize thick "corrosion allowance" metals, such as carbon steel and copper.

"Corrosion resistant" metals depend corrosion resistance on their passive films. Under some circumstances, however, these films can be breached, resulting in localized corrosions, such as pitting, crevice corrosion, and stress corrosion cracking. It is very difficult to evaluate how they generate and proceed under repository conditions.

General corrosion behavior has been studied for carbon steel and critical conditions of crevice corrosion for titanium and titanium alloy.

(1) Immersion Tests

Immersion tests were carried out in the presence of bentonite. The tests in aqueous solution without bentonite were also conducted for reference. Following materials and aqueous solutions were used in these tests.

- Sheet specimens : carbon steel
commercial purity titanium (Ti Grade 2)
- bentonite : sodium bentonite
calcium bentonite
- aqueous solutions : distilled water
3 % NaCl solution
(these are equilibrated with air before testing)

(i) Carbon Steel

Weight loss measurements using descaled specimens are given in Figure 2-1 to 2-3, and following results are obtained :

- (a) No significant differences in corrosion rate were observed between

in sodium bentonite and calcium bentonite.

- (b) Corrosion rate in bentonite depended on water content.
- (c) The significant influence of chlorine on the corrosion was not recognized.
- (d) X-ray diffraction revealed that corrosion products formed on the surface of specimens were mainly Fe_3O_4 .
- (e) Localized corrosion was not observed in all the specimens.

(ii) Titanium

Significant weight changes were not measured in both the bentonites. Localized corrosion was not observed.

(2) Electrochemical Studies

In order to investigate the crevice corrosion resistance of titanium (Ti Grade 2) and its alloy (Ti Grade 12), critical conditions [4] of crevice corrosion, such as the critical potential of metal, the lower limit temperature and the lower limit concentration of chlorine, were evaluated. These critical conditions were estimated with repassivation method. Since this method is the equilibrium approach to the corrosion, it may be possible to assess the long-term integrity of the overpack.

The experiments were undertaken in deaerated NaCl solution with metal/metal crevice specimens (Figure 2-4) and metal/bentonite crevice specimens (Figure 2-5).

(i) Metal/metal crevice

Critical potentials of crevice corrosion of Ti Grade 2 were about -460 mV (saturated calomel electrode, SCE) in 25 % NaCl solution in the temperature range 70 °C to 100 °C. In the case of Ti Grade 12, it was about -400 mV(SCE).

The crevice corrosion was not advanced at the temperature of 40 °C. Critical potentials in 25 % NaCl solution were constant in the pH range

5 to 11 at 100 °C. The study of critical potentials were also undertaken in various concentrations of NaCl solution at 100°C. The result is shown in Figure 2-6.

Effect of temperature and concentration of chlorine on the crevice corrosion were studied. The potential of specimen was constant (-200 mV) through all the experiments.

The result (Figure 2-7) suggests that Ti Grade 12 has more crevice corrosion resistance compared to Ti Grade 2, especially at high temperature and high concentration of chlorine.

(ii) Metal/bentonite crevice

Sodium bentonite mixed with 3 % NaCl solution was packed in the hole of metal/bentonite crevice specimen. The crevice corrosion did not occur even at the high potential of the specimen up to +2000 mV (SCE).

(3) Discussion

Although localized corrosion was not observed with carbon steel during this experiments, it may be subject to localized corrosion under a certain groundwater condition [5].

Future studies should be aimed at surveying the conditions in which localized corrosions may occur and the corrosion behavior under reducing condition.

As to the titanium and its alloy, it is considered that crevice corrosion is most susceptible of all the localized corrosions [6,7]. So it can be predicted that localized corrosion may not occur in the condition where crevice corrosion does not occur.

Electrochemical studies of crevice corrosion in this work indicate that metal/bentonite crevice is less susceptible to crevice corrosion compared to metal/metal crevice. It would be conservative to estimate the integrity of titanium (Ti Grade 2 and Ti Grade 12) for the overpack material with metal/

metal crevice.

As susceptibility of crevice corrosion for titanium and its alloy is greatly affected by the content of chlorine as shown in Figure 2-7, it is important to investigate the chemical composition of groundwater at the repository. Though there are unclarified points regarding the chemical compositions of groundwater in deep underground, it is said that groundwater with high chlorine content can exist in the deep underground.

Therefore careful analyses of crevice corrosion will be required provided titanium is used for overpack material under the environment of high chlorine content. Further studies, especially detail estimation on metal/ bentonite crevice, are still remained.

4. PERMEABILITY OF COMPACTED BENTONITE AS BUFFER MATERIAL

One of the most important property of buffer material is permeability. The function of buffer material is to limit the flow of the groundwater around overpack and to maintain the soundness of overpack, canister and glass, and in a later stage to limit the outward migration of leached radioactive substances.

Bentonite with high density is thought to be a candidate for buffer material mainly because of its very low hydraulic conductivity and diffusivity. The purpose of the present study is to clarify the permeability of compacted bentonite under various conditions.

(1) Physical Property of Test Pieces and Apparatus

The test pieces of compacted bentonite were made of bentonite powder by means of isostatic pressing in a cylinder. The relation between the compression pressure for forming test pieces and the dry density of the test pieces and the relation between compression pressure and compressive strength are shown in Figure 3-1 and Figure 3-2. The volume of the test piece was kept constant during the permeability test by the apparatus shown in Figure 3-3 and the swelling pressure was measured.

(2) Density and Permeability

The hydraulic conductivity of compacted bentonite of various densities was measured. The test piece was the disk of 40 mm in diameter and of 5 mm thick.

Hydraulic conductivity decreases and swelling pressure increases with the increase of density as shown in Figure 3-4. At a density of 2.0 g/cm³, hydraulic conductivity(k) is about 1.2×10^{-14} m/s and swelling pressure(Ps) is about 15 MPa. The result is similar to that of KBS [8].

(3) Influence of a Crack on Permeability

The compacted bentonite will be applied to the disposal in the form of

blocks. Cracks or gaps must be exist among the blocks and the surrounding rock and may be exist even in the blocks. In order to investigate the effect of these cracks, permeability tests on the compacted bentonite with a crack were performed.

The shape of a test piece is shown in Figure 3-5. The width of cracks were 0.5 mm and 2.0 mm. The density of the compacted bentonite was about 1.4 g/cm³. The water pressure was 2 MPa.

The result is shown in Table 3-1. Due to the swelling of bentonite surrounding the crack, the hydraulic conductivity of the whole test pieces with a crack are only 3 or 4 times as large as that without a crack.

(4) Initial Saturation of Bentonite with Water

Before the saturation of buffer mass with groundwater in the initial stage of disposal, the overpack surface will be kept dry and protected from corrosion. Infiltration rate of water into the dry compacted bentonite was studied in order to estimate the traveling time of groundwater from the peripheral rock mass to the overpack surface.

The migration speed of wetting front which means the clear boundary between the dry part and the wet one in compacted bentonite was measured under water pressure of 9 MPa. The test piece was the column of 40 mm in diameter and of 20 mm high and the density was 1.6 g/cm³. The hydraulic conductivity calculated from the migration speed of wetting front is about 1.6×10^{-13} m/s.

(5) Discussion

The hydraulic conductivity of compacted bentonite is very low ($1 \sim 9 \times 10^{-14}$ m/s). Therefore, diffusion will be the dominant mechanism for the transport of materials through the bentonite. According to the result of permeability test on the test pieces with a crack (crack width/block width ≤ 0.05), the hydraulic conductivity of the whole test piece is only four times as large

as that without a crack. This is the effect of the self-sealing property of compacted bentonite. Cracks or gaps have not so much influence on the permeability of buffer mass.

The experiment on the initial saturation of bentonite with water suggests that the hydraulic conductivity of unsaturated compacted bentonite is about five times as large as that of saturated one. This result indicates that groundwater will reach overpack within several decades. This term of infiltration is short in comparison with the expected life span of overpack, 1000 years.

5. SUMMARY

(1) Leaching behavior of waste borosilicate glass

Glass leaching rates was suppressed under the condition of high SA/V (the surface area of glass/leachant volume). Saturation of leachant with silicon reduces leaching rates of the other elements.

(2) Corrosion behavior of candidate overpack materials

Corrosion behavior of carbon steel and titanium was studied and localized corrosion was not observed in both the materials. Corrosion rate of carbon steel depends on bentonite/water ratio. Experiments on crevice corrosion lead to the preliminary conclusion that durability of Ti Grade 12 (Ti alloy) is more superior than that of Ti Grade 2 (pure Ti) under predicted repository environment.

(3) Permeability of compacted bentonite as buffer material

The traveling time of wetting front from the surrounding rock to overpack through dry compacted bentonite with 0.3 m thick is estimated within several decades. The hydraulic conductivity of saturated compacted bentonite is very low ($1\sim 9 \times 10^{-14}$ m/s). Cracks or gaps have no significant influence on the permeability of the bentonite.

ACKNOWLEDGMENT

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CAPTIONS FOR FIGURES

- Figure 1-1 Normalized mass losses as a function of time in soxhlet test.
- Figure 1-2 Total weight losses as a function of time in one year leaching experiment.
- Figure 1-3 Weight losses of PNC reference glasses leached in various media at 98°C.
- Figure 1-4 Boron depth profile, SIMS, P0500, 364days, 98 °C.
- Figure 1-5 Concentrations of the leachates from large-scale and small-scale tests.
- Figure 2-1 Effect of water content on general corrosion of carbon steel.
- Figure 2-2 Effect of kind of bentonite on general corrosion of carbon steel (under restricted oxygen content conditions).
- Figure 2-3 Weight loss as a function of exposure time up to 4 months in Na-bentonite for carbon steel.
- Figure 2-4 Specimen for crevice corrosion tests.
- Figure 2-5 Experimental apparatus for electrochemical measurements.
- Figure 2-6 Relationship between repassivation potential (E_R) and NaCl concentration for metal/metal-crevice of C.P.Ti and G12Ti at 100°C.
- Figure 2-7 Crevice corrosion map of C.P.Ti and G12Ti in terms of NaCl concentration and temperature.
- Figure 3-1 Relation between compression pressure and density.
- Figure 3-2 Relation between compression pressure and compressive strength.
- Figure 3-3 Permeability cell.
- Figure 3-4 Relation between hydraulic conductivity, swelling pressure and density.
- Figure 3-5 Test piece of compacted bentonite with a crack.

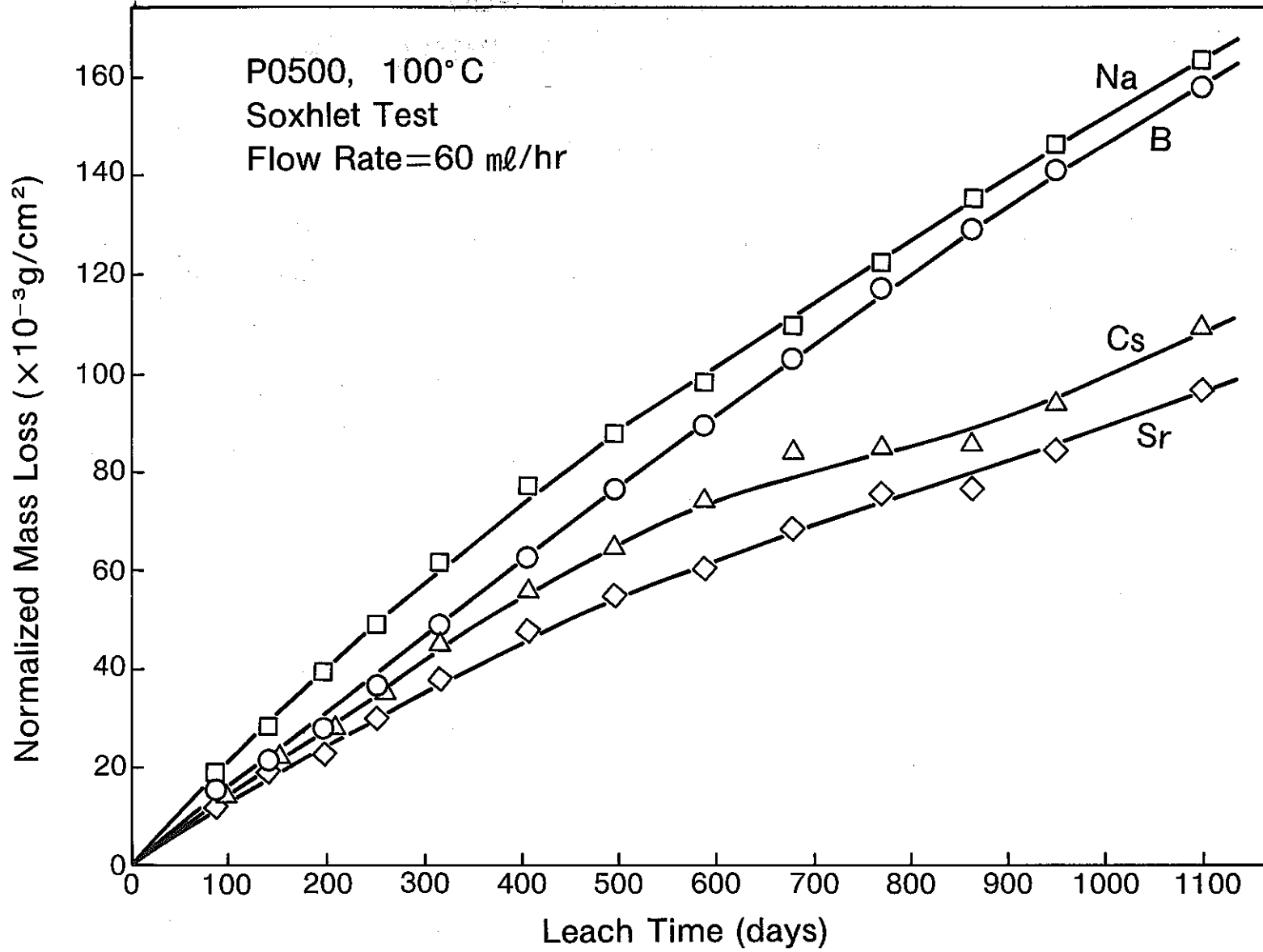


Figure 1-1 Normalized mass losses as a function of time in Soxhlet test.

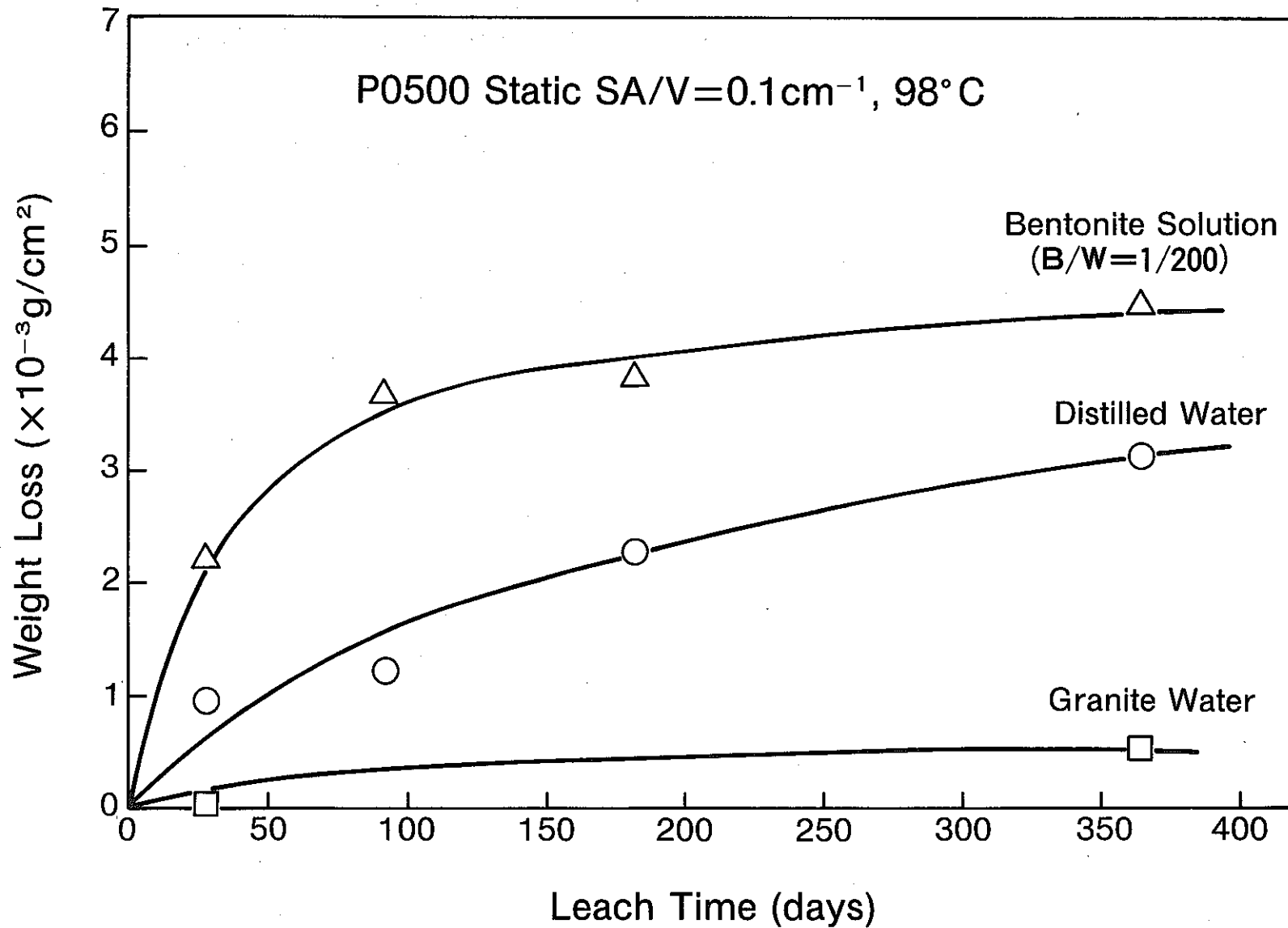


Figure 1-2 Total weight losses as a function of time in one year leaching experiment.

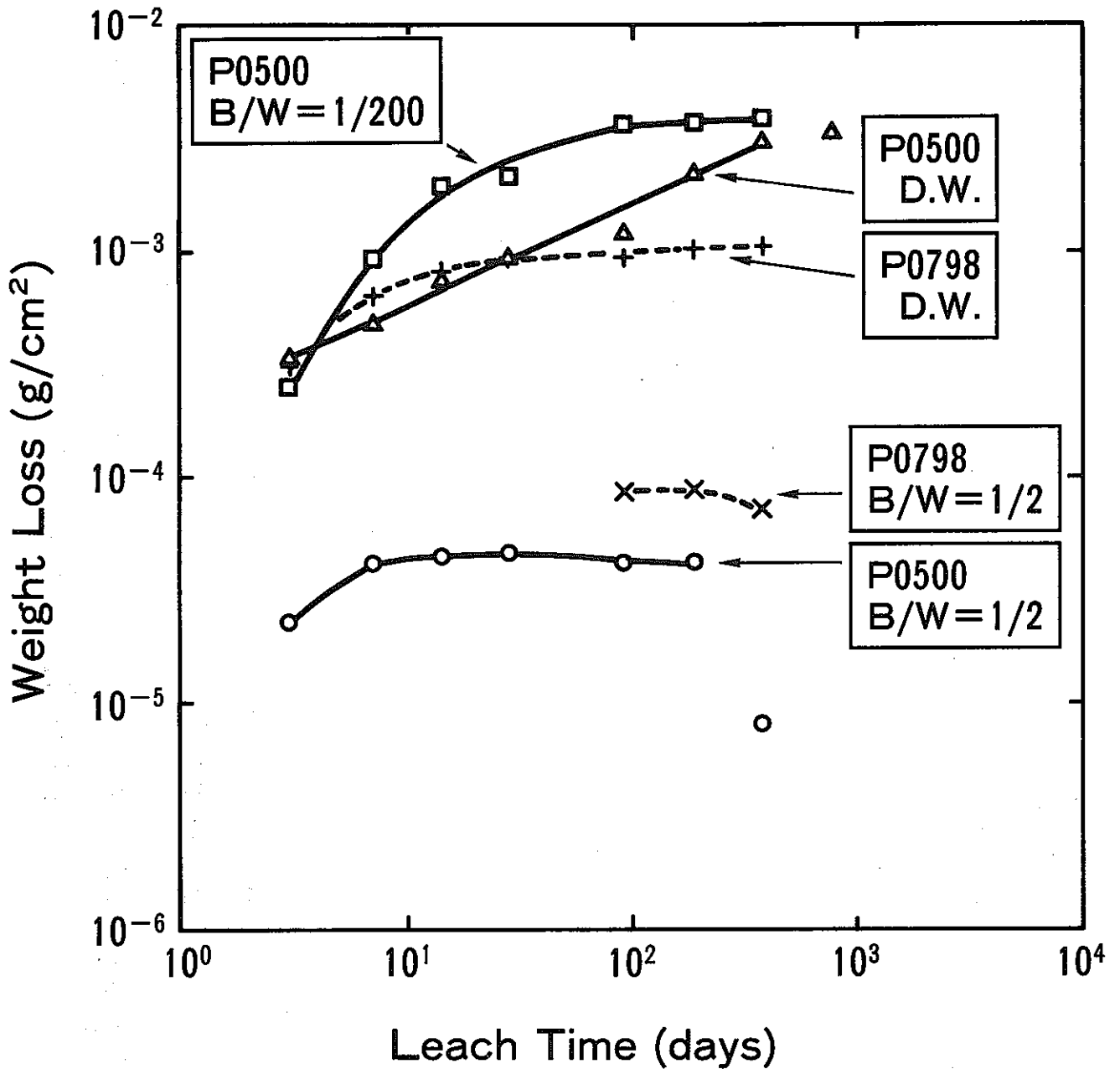


Figure 1-3 Weight losses of PNC reference glasses leached in various media at 98°C.

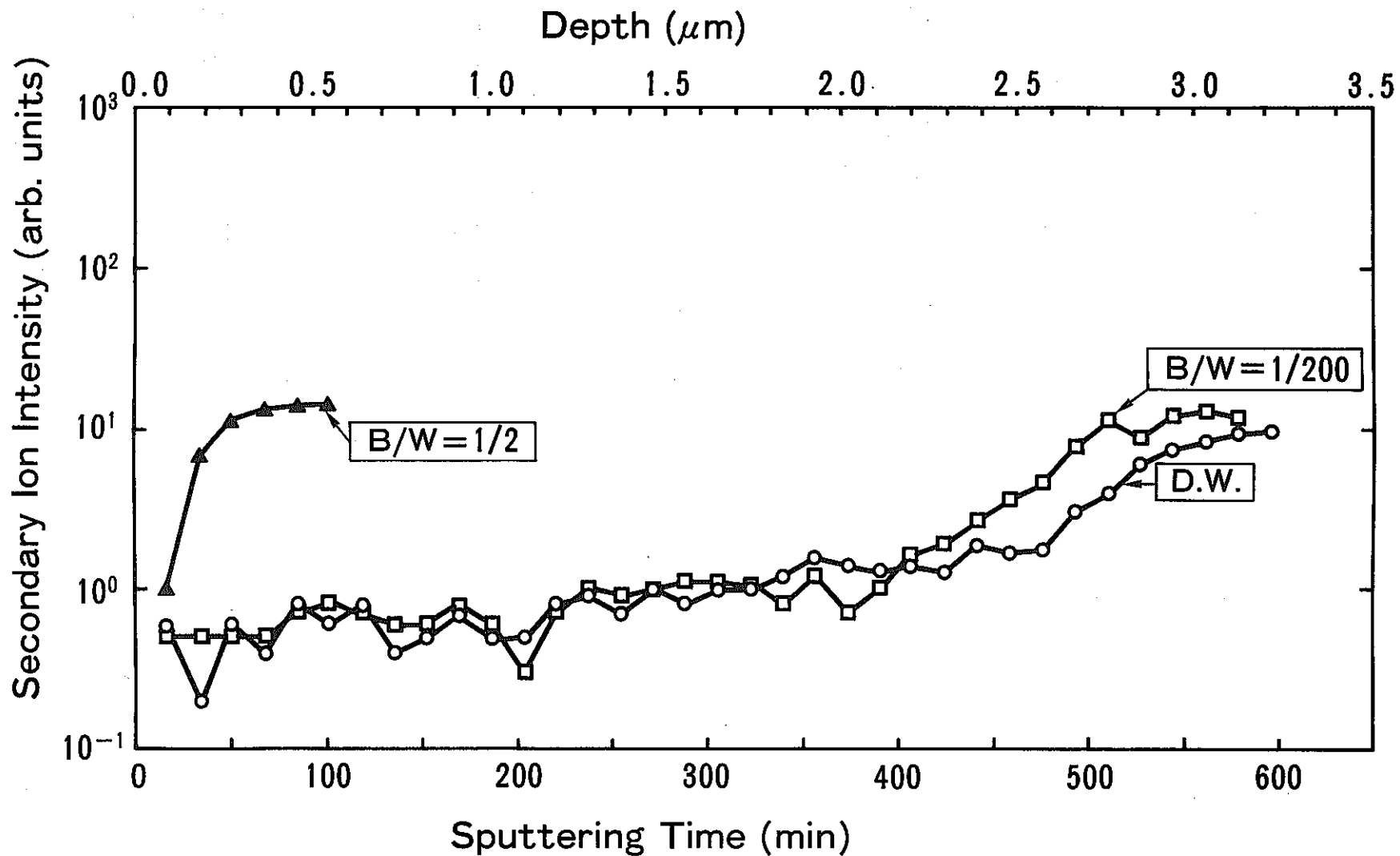


Figure 1-4 Boron depth profile, SIMS,
P 0500, 364 days, 98°C.

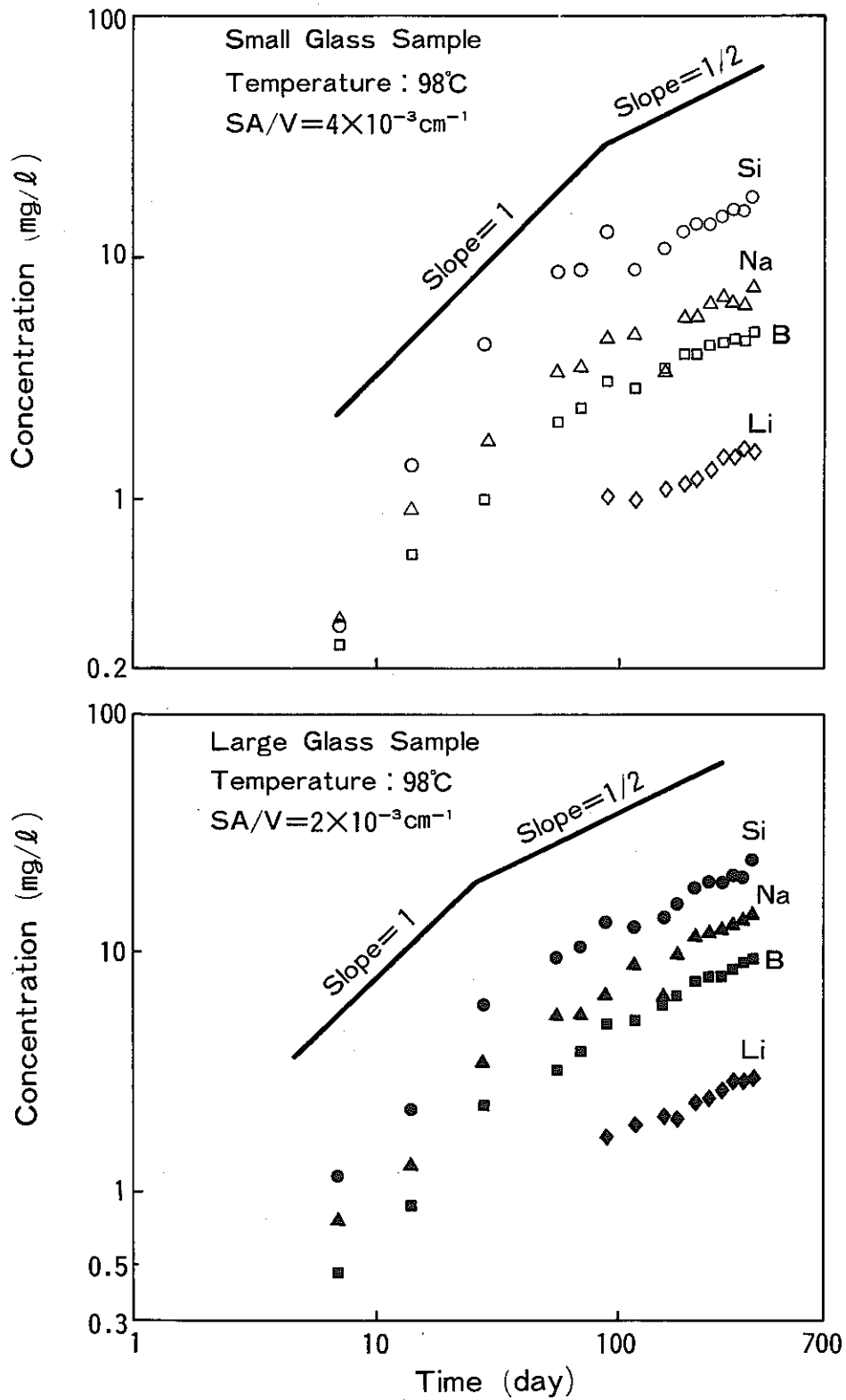


Figure 1-5 Concentrations of the leachates from large-scale and small-scale tests.

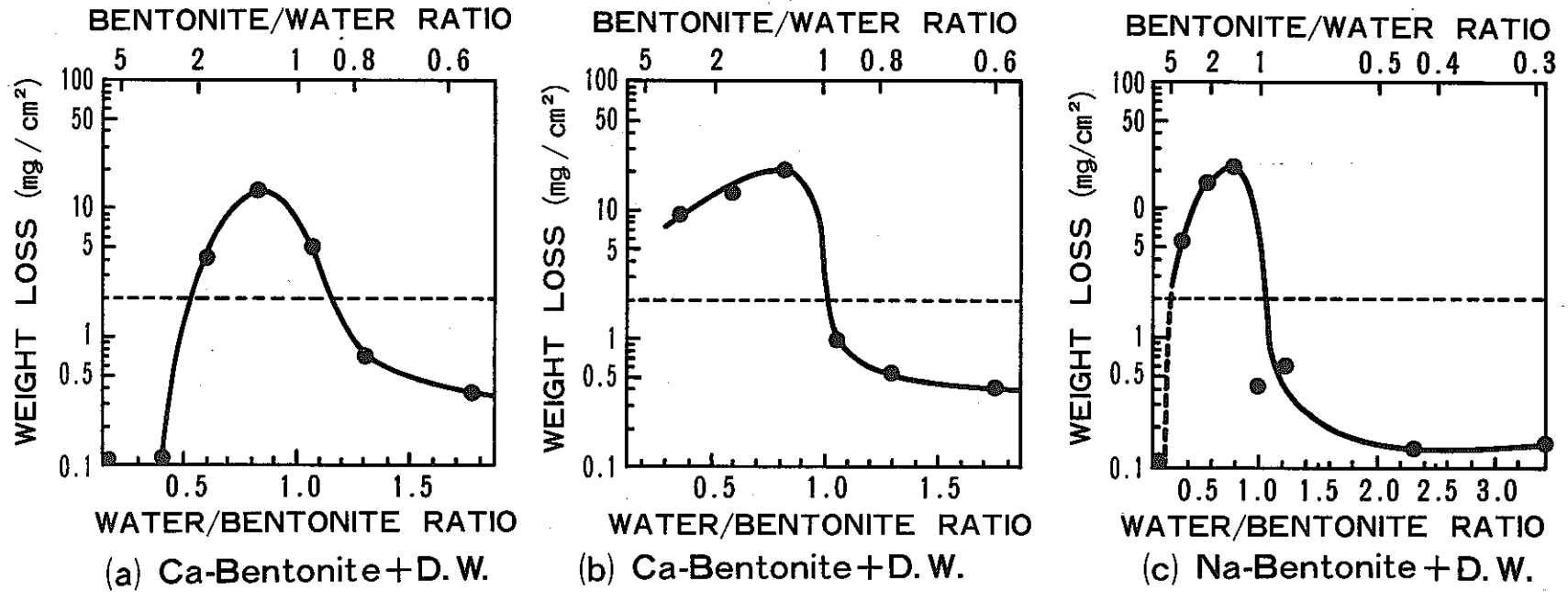
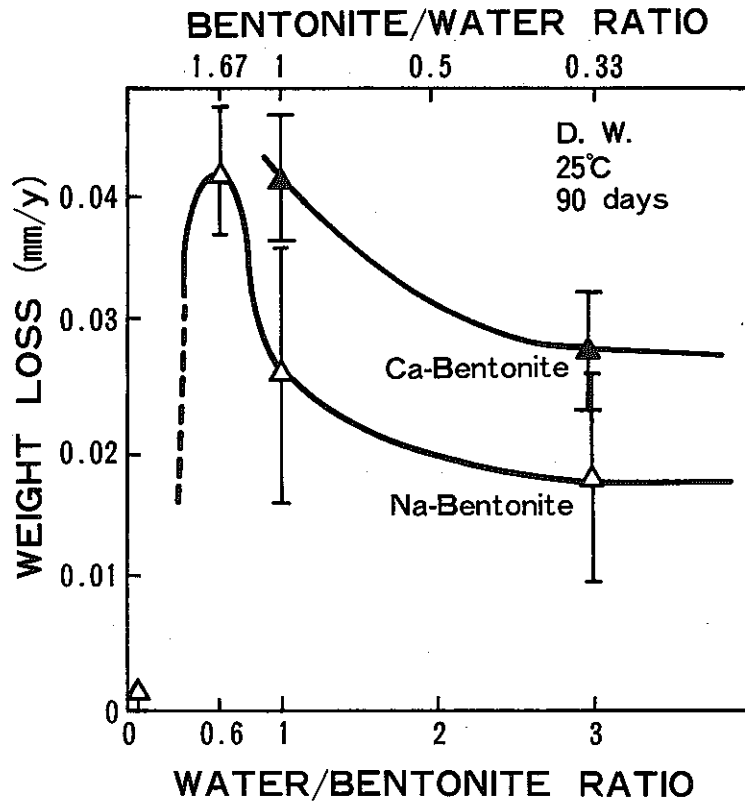
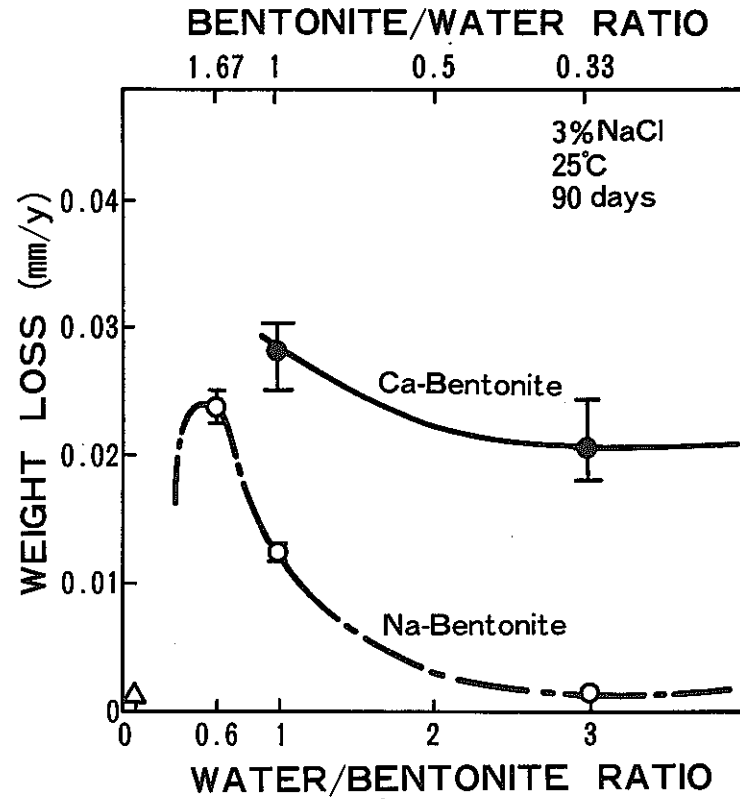


Figure 2-1 Effect of water content on general corrosion of carbon steel.



(a) Bentonite + Distilled Water



(b) Bentonite + 3% NaCl Solution

Figure 2-2 Effect of kind of bentonite on general corrosion of carbon steel (under restricted oxygen content conditions).

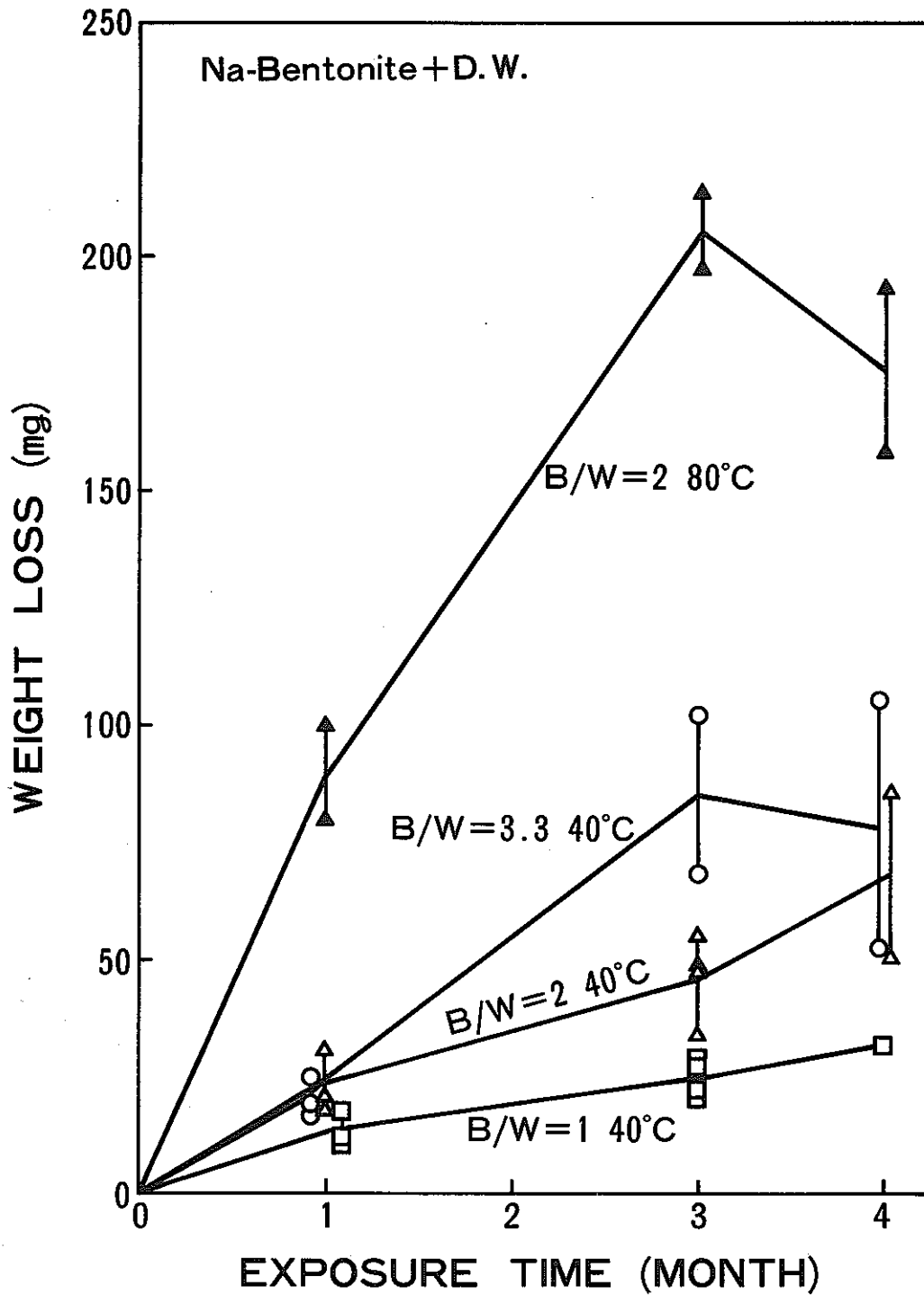
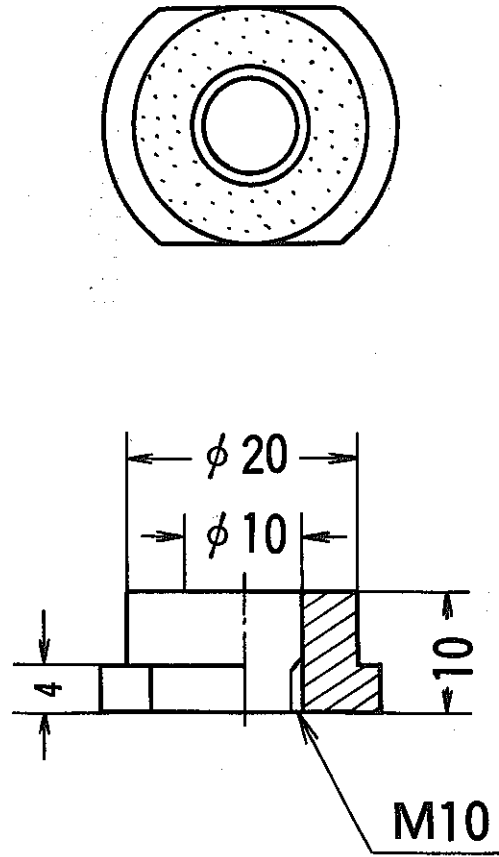
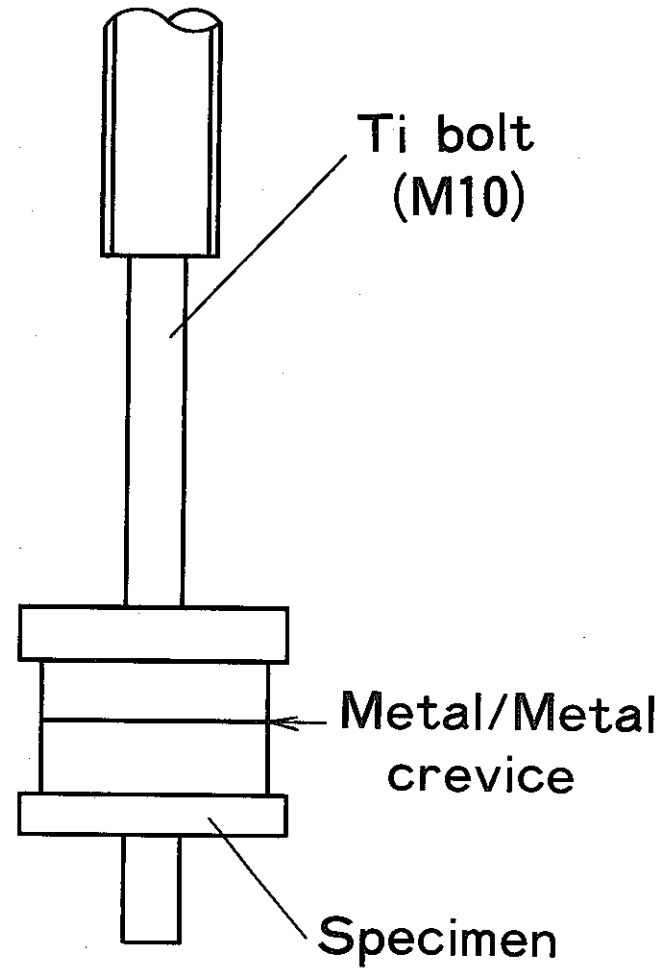


Figure 2-3 Weight loss as a function of exposure time up to 4 months in Na-bentonite for carbon steel.



(a) Crevice Corrosion Test Specimen



(b) Assembly of Test Specimen

Figure 2-4 Specimen for crevice corrosion tests.

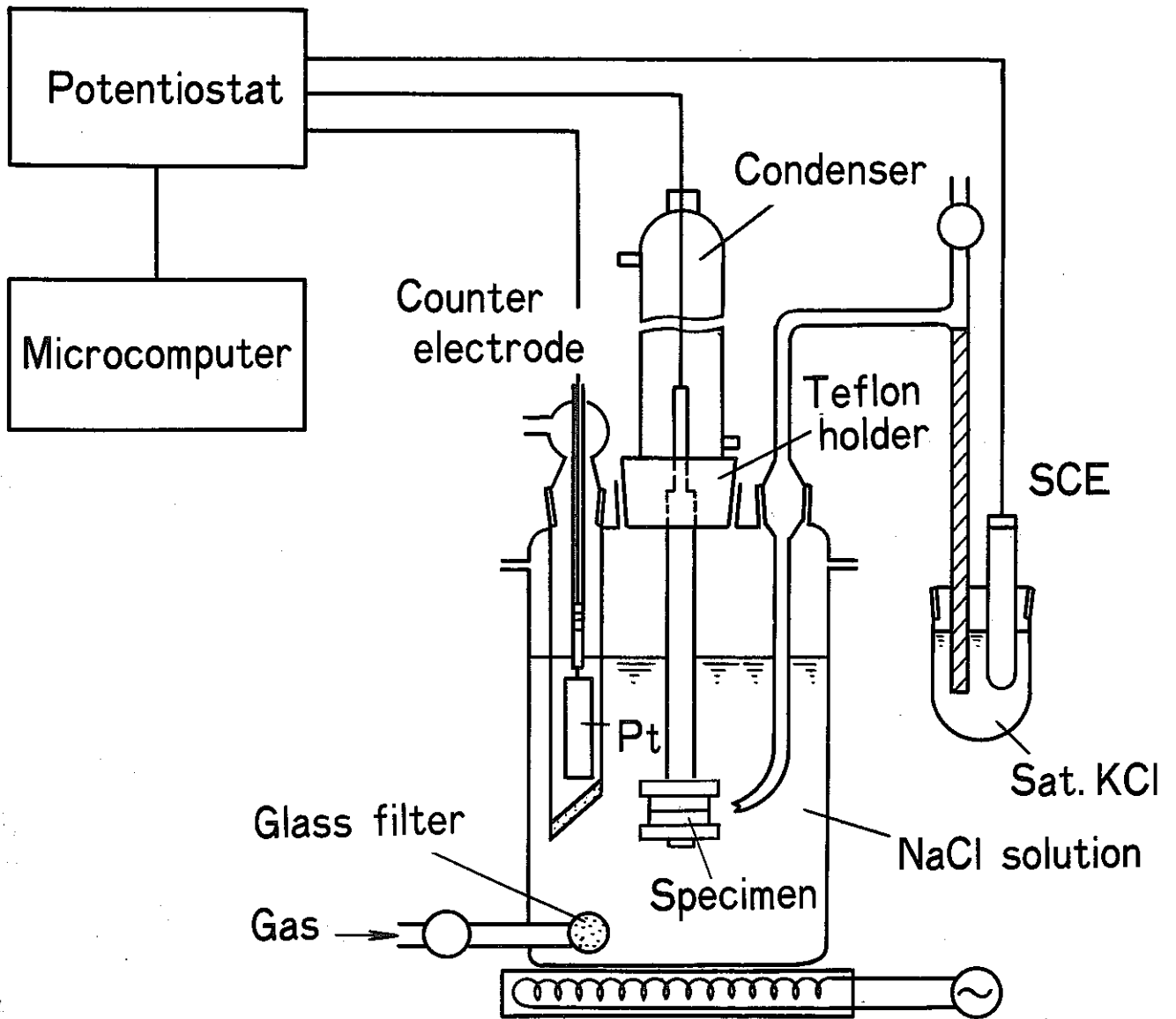


Figure 2-5 Experimental apparatus for electrochemical measurements.

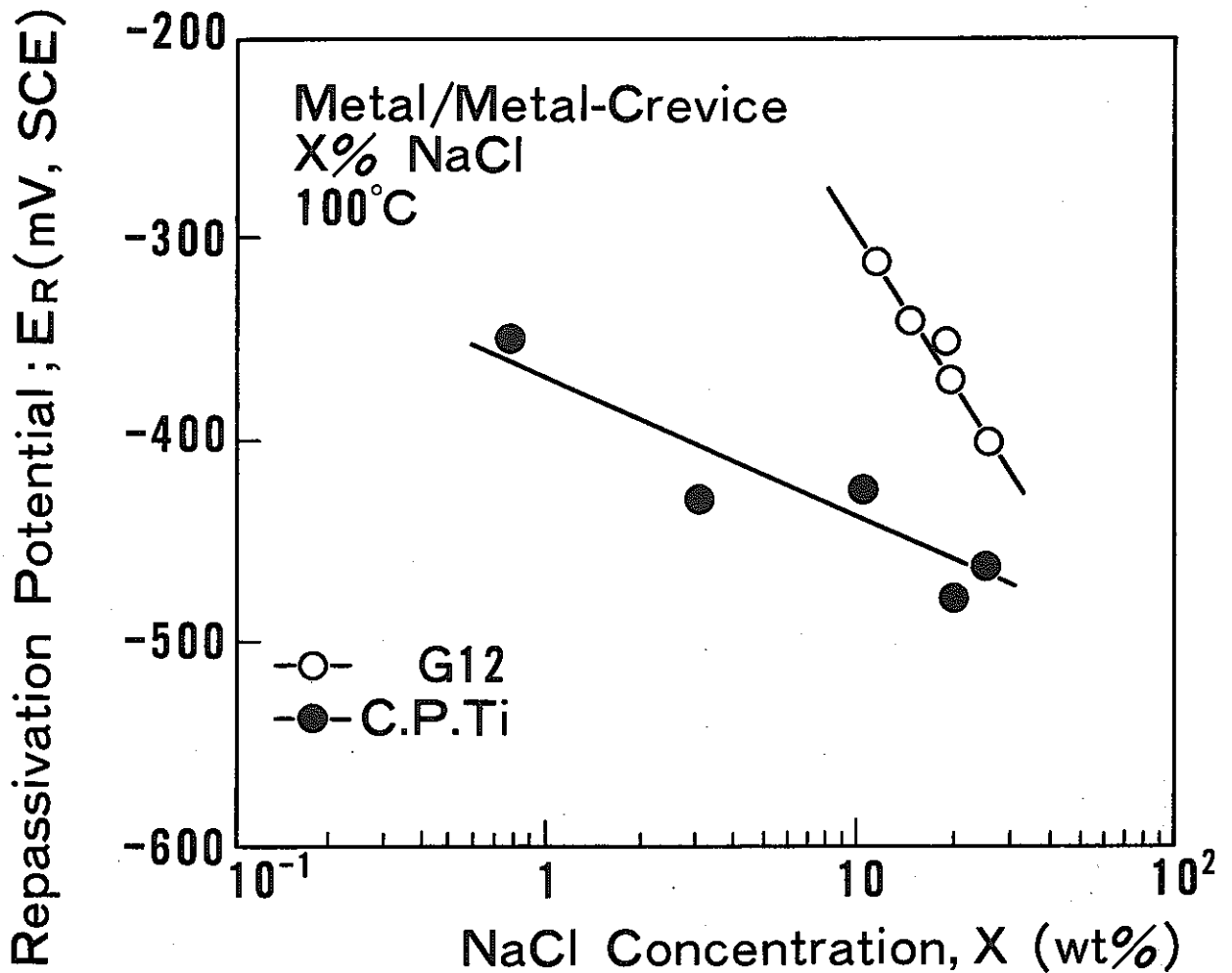


Figure 2-6 Relationship between repassivation potential (E_R) and NaCl concentration for metal/metal-crevice of C. P. Ti and G12Ti at 100°C.

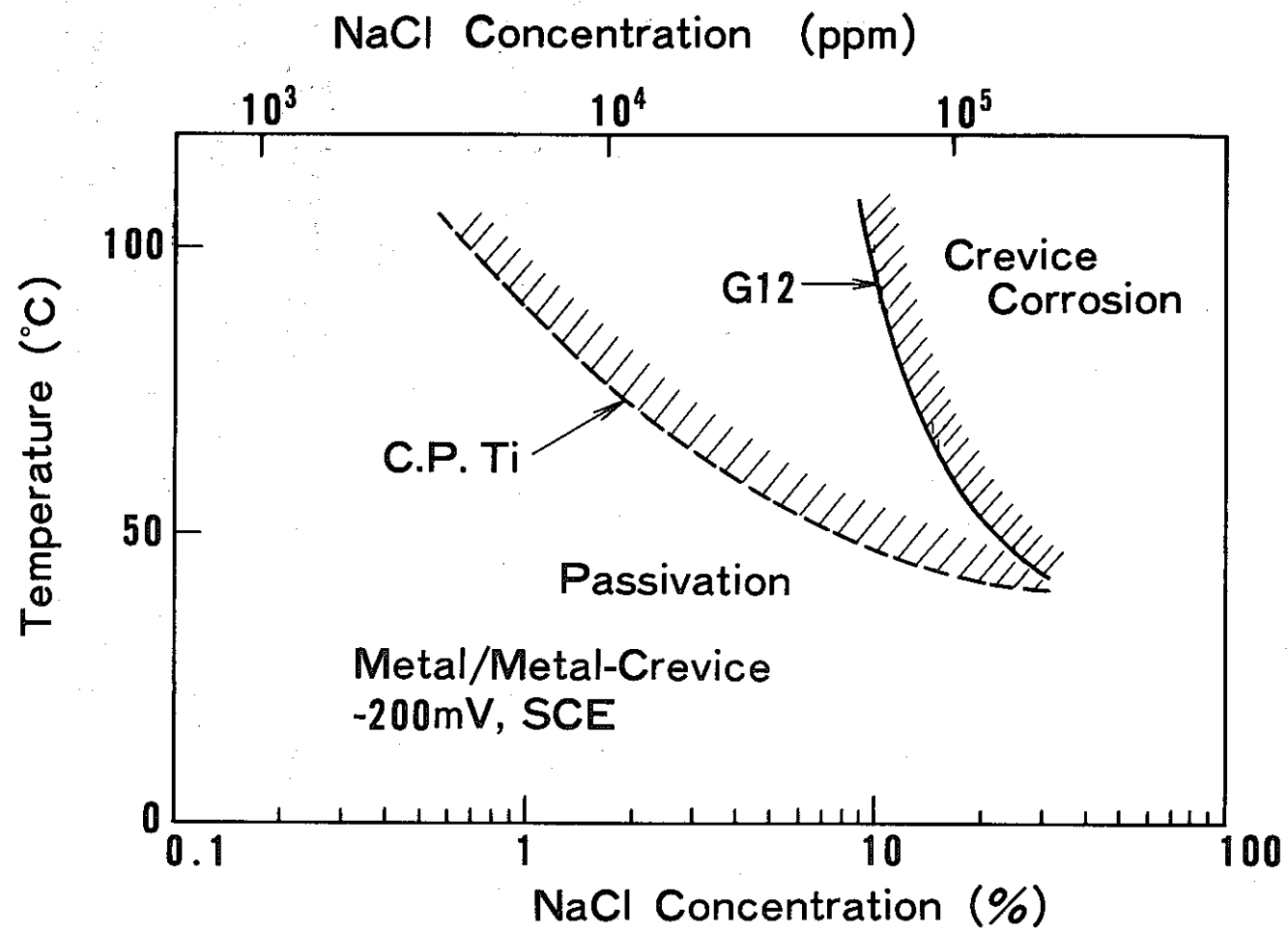


Figure 2-7 Crevice corrosion map of C. P. Ti and G12 Ti in terms of NaCl concentration and temperature.

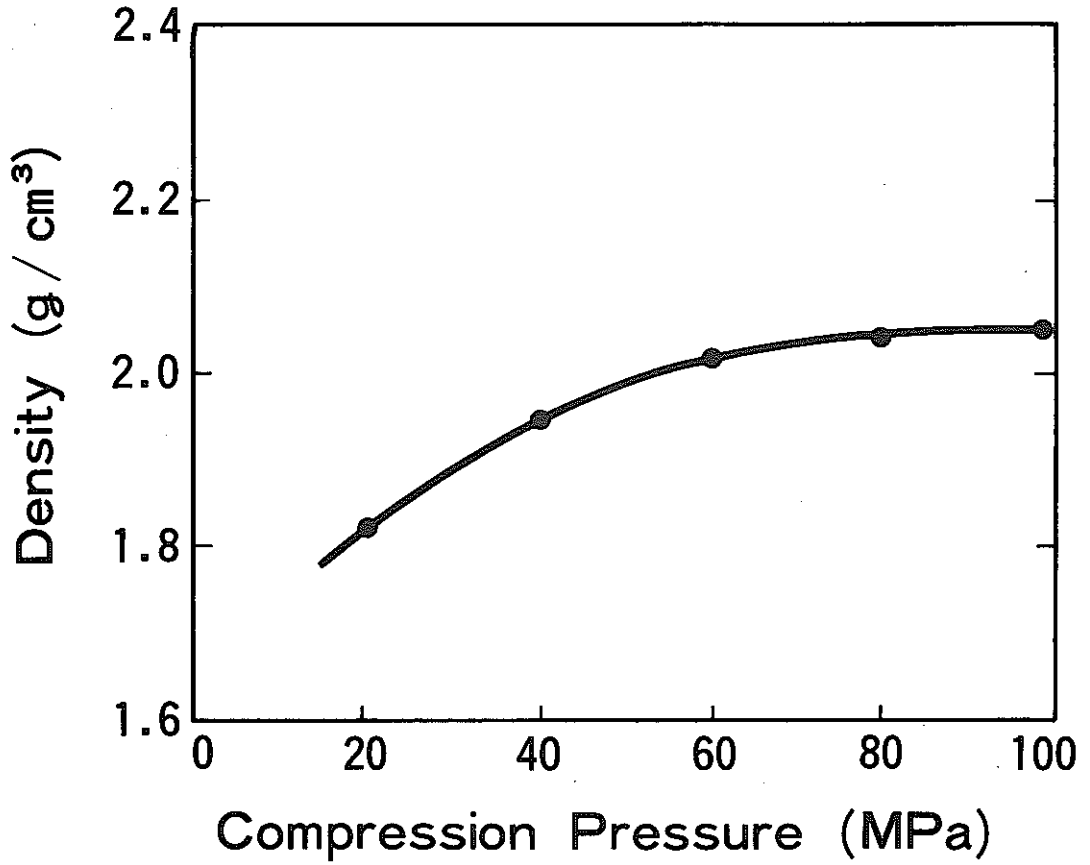


Figure 3-1 Relation between compression pressure and density.

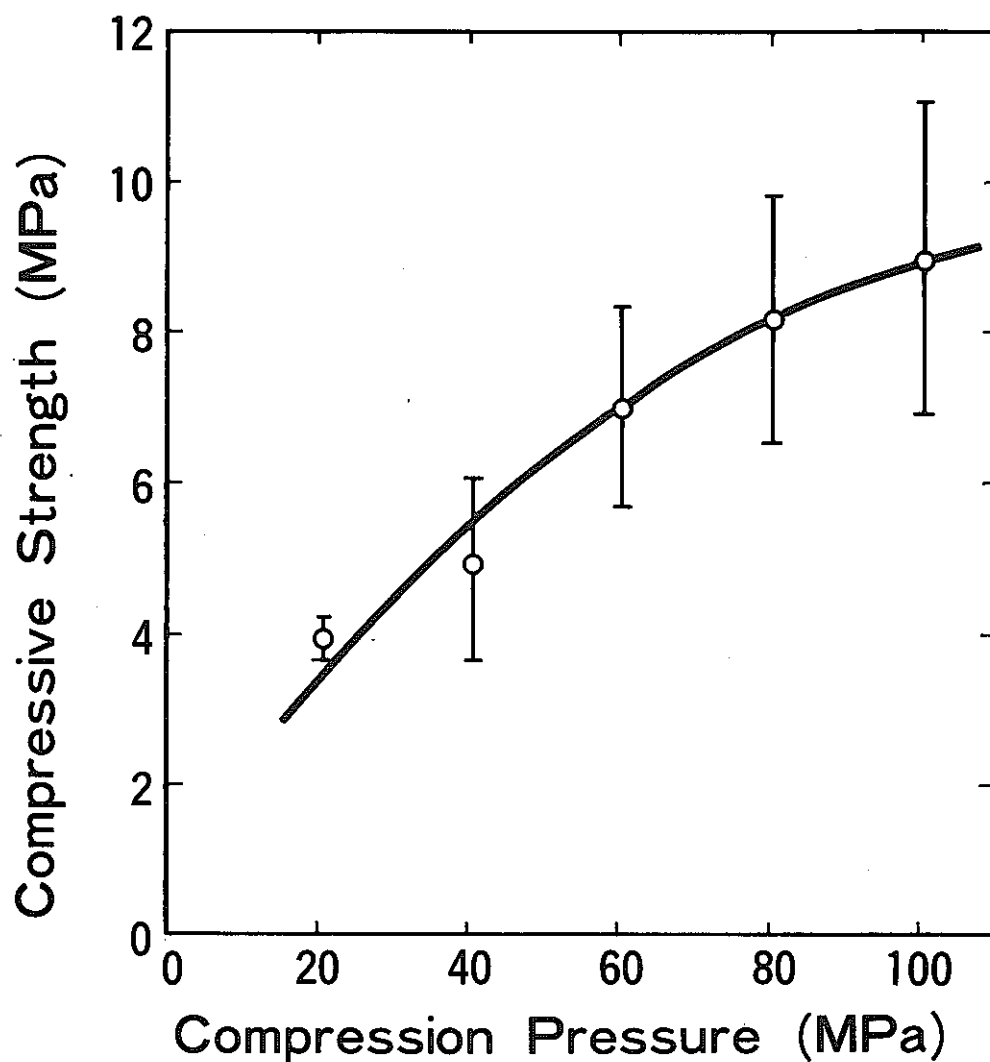


Figure 3-2 Relation between compression pressure and compressive strength.

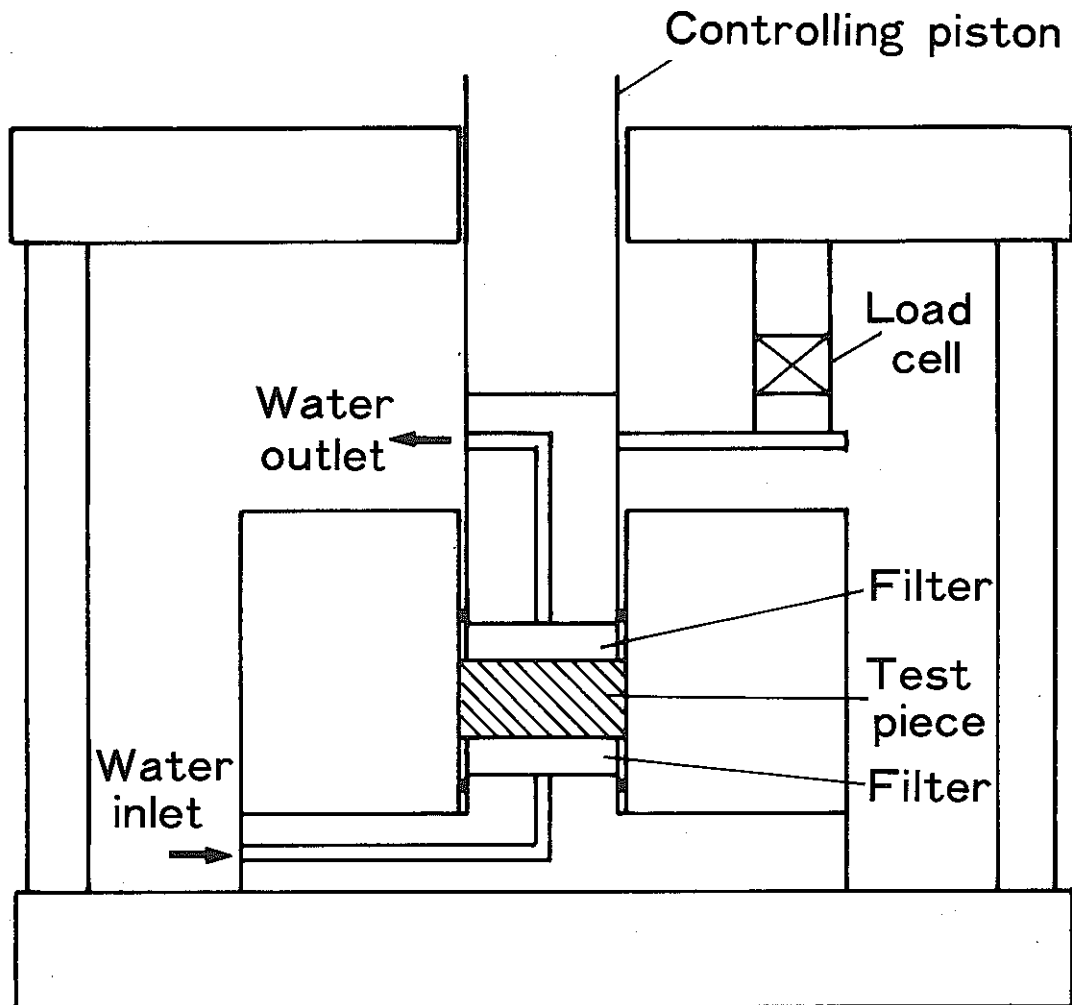


Figure 3-3 Permeability cell.

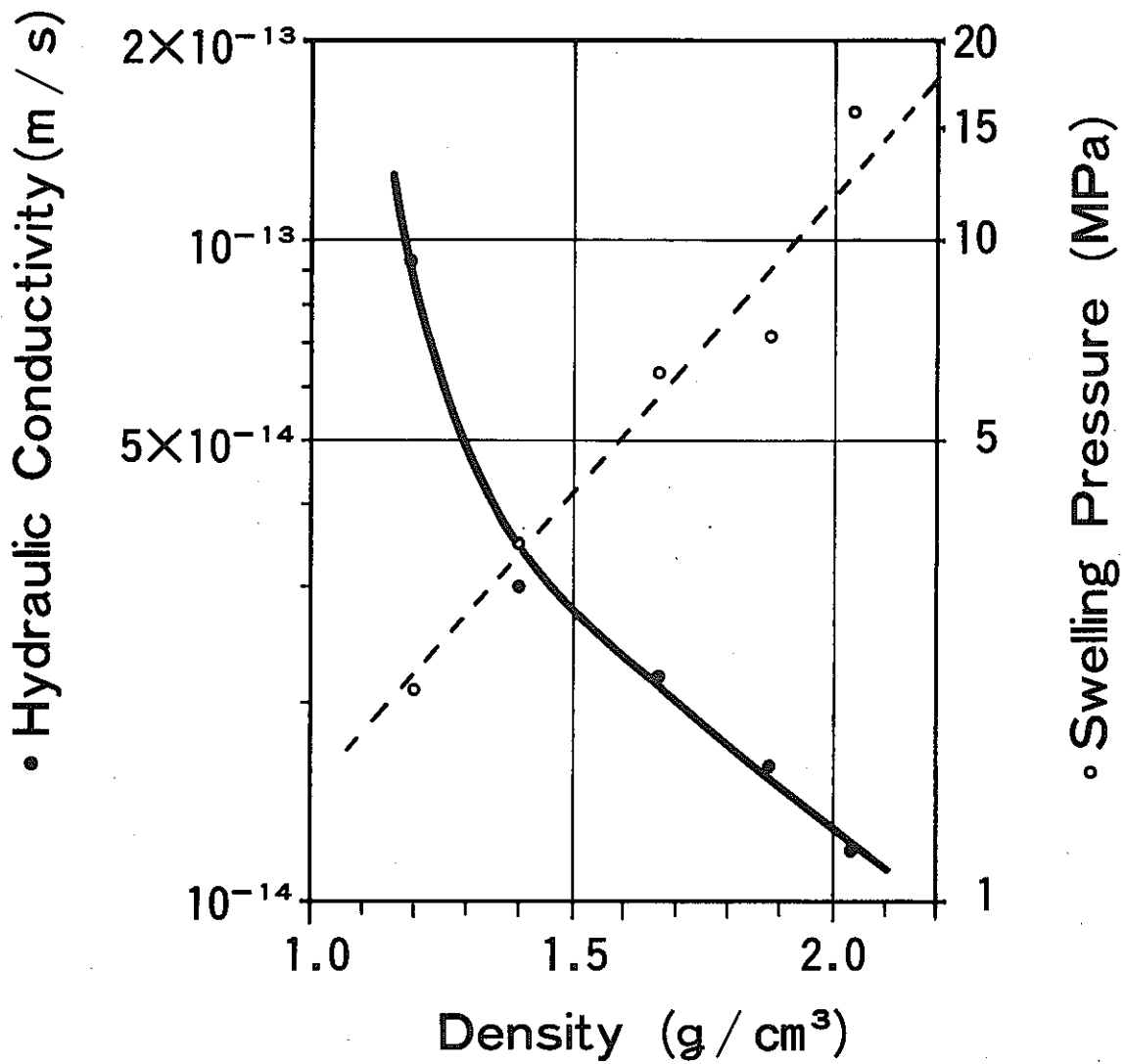


Figure 3-4 Relation between hydraulic conductivity, swelling pressure and density.

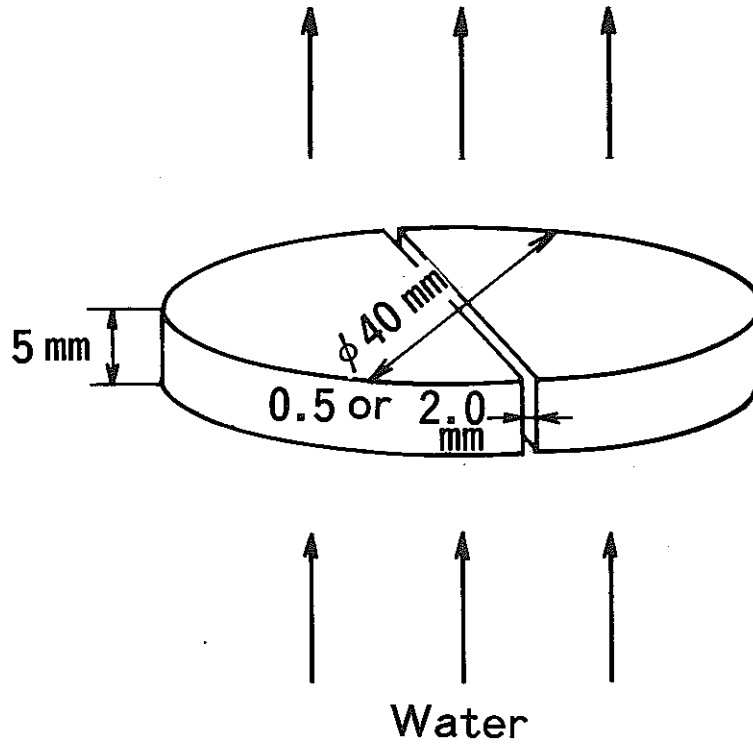


Figure 3-5 Test piece of compacted bentonite with a crack.

Table 1-1 Experimental conditions

Test Methods Conditions	Soxhlet Test	Static Tests		Solubility Test	Large Scale Test	Small Scale Test		
		Simulated Waste Glass						
(1) Specimens • Glass • Bentonite	PNC reference glass (P0500, 20×10×10mm, finely polished)	PNC reference glasses (PO 500, PO 798, 10×10×10 mm, finely polished)		PNC reference glass (PO 500, powder < 74μm)	Large scale glass (410mmφ × 620mm, 80 ℓ) with canister (stainless steel, SUS 304 L)	Small scale glass (23mmφ × 45mm, 0.02 ℓ) with canister (stainless steel, SUS 304 L)		
		Na - montmorillonite ("KUNIPIA")		Na-montmorillonite				
(2) Leaching Conditions • Leachate • Temperature • Surface Area / Leachate Volume ratio, Flow Rate, Agitation Condition • Leach Time	Distilled Water (D. W.)	• D. W. • Granite Water • Bentonite Solution (Bentonite/D. W. weight ratio (B/W) = 1/200)	Bentonite/D. W. weight ratio (B/W) = 1/2	• D. W. • Granite Water • Bentonite Solution (B/W = 1/200)	D. W.	D. W.		
		40 ~ 98 °C		98 °C			98 °C	98 °C
		0.1 cm ⁻¹	1 glass block / 50g gel bentonite	100 cycles/min. 100 g / ℓ			10 ⁻³ cm ⁻¹	10 ⁻³ cm ⁻¹
		364 days	364 days	728 days			364 days	364 days

Table 1-2 Analyses of leachate solution after solubility tests of P0500 glass at 98 °C for different leachants (mg/ℓ)

	1 year			2 years
	Granite	Bentonite	D. W.	D. W.
Si	128	116	134	110
B	3800	4000	3800	4000
Li	740	760	740	740
Na	4200	4600	4200	4300
K	288	328	294	280
Ca	11.0	14.8	11.0	15.0
Al	36	40	38	39
Zn	7.8	—	7.4	< 0.05
Fe	< 0.6	< 0.6	< 0.6	—
Ni	< 1.0	< 1.0	< 1.0	—
Cr	6.8	6.8	6.6	11.3
Rb	9.8	11.2	10.2	11.5
Cs	28	34	30	21
Sr	2.6	2.8	2.6	2.8
Ba	—	—	1.2	1.5
Zr	< 0.4	—	< 0.4	1.2
Mo	1300	1420	1320	1500
Ru	10.0	10.6	10.4	4.2
Rh	< 1.0	< 1.0	< 1.0	< 0.5
Pd	< 0.6	—	< 0.6	< 0.3
Te	0.56	0.52	0.44	—
Y	< 0.2	—	< 0.2	< 0.1
La	< 0.4	< 0.4	< 0.4	< 0.2
Ce	< 0.4	< 0.4	< 0.4	< 0.2
Nd	< 0.4	< 0.4	< 0.4	0.5
Sm	< 0.4	—	< 0.4	< 0.2

< Under detection limit

Table 1-3 Analysis of leachate solutions after one year leach test at 98°C

(mg/l)

	Distilled Water SA/V=0.1cm ⁻¹	Large Scale SA/V=1.9×10 ⁻³ cm ⁻¹	Granite SA/V=0.1cm ⁻¹	Bentonite SA/V=0.1cm ⁻¹	Solubility Test Distilled Water
Si	70	25	42	85	134
B	35	9.5	9.5	39	3800
Na	46	14.2	74	104	4200
Cs	2.8	0.65	2.0	0.24	30
Sr	0.01	0.15	0.41	0.02	2.6

Table 3-1 Summary of permeability tests
of compacted bentonite with a crack

Crack width (mm)	0	0.5	2.0
Hydraulic conductivity (m/s)	3.0×10^{-14}	8.7×10^{-14}	1.2×10^{-13}
Density (g / cm ³)	1.37	1.46	1.37
Swelling pressure (MPa)	2.7	2.7	2.3