

# Effect of Fe<sub>2</sub>O<sub>3</sub>/ZnO on two glass compositions for solidification on Swedish nuclear wastes

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EFFECT OF  $\text{Fe}_2^{O_3}/\text{ZnO}$  ON TWO GLASS COMPOSITIONS FOR SOLIDIFICATION ON SWEDISH NUCLEAR WASTES

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This report concerns a study which was conducted for the KBS project. The conclusions and viewpoints presented in the report are those of the author(s) and do not necessarily coincide with those of the client.

A list of other reports published in this series during 1981, is attached at the end of this report. Information on KBS technical reports from 1977-1978 (TR 121), 1979 (TR 79-28) and 1980 (TR 80-26) is available through SKBF/KBS. Effect of Fe<sub>2</sub>O<sub>3</sub>/ZnO on Two Glass Compositions

for Solidification on Swedish Nuclear Wastes

by

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#### INTRODUCTION:

Low melting alkaliborosilicate glasses have been considered for use in the immobilization of high level radioactive wastes for years (1-2). A recent study comparing the surface behavior of two nuclea waste glasses concluded that "Addition of  $Fe_2O_3$  to a soda borosilicate nuclear waste glass significantly reduces damage by water attack because of a Fe-rich film that forms on the glass surface".(3) However, in the previous study there were significant differences in the concentration of  $SiO_2$ ,  $B_2O_3$ , CaOand simulated fission products in the glasses which made it impossible to ascribe the improved leach resistance solely to  $Fe_2O_3$  content. Thus, the objective of the present investigation is to compare the leaching and surface behavior of two nuclear waste glasses which differ only by the substitution of  $Fe_2O_3$  for some of the ZnO in the glass. By this comparison we hope to establish whether  $Fe_2O_3$  provides a unique contribution to improvements in the leach resistance of these complex glasses.

Both glass compositions studied are compatible with the low melting temperature, <  $1150^{\circ}$ C, required for the French AVM Process. The quantity of simulated waste products is 9%, characteristic of the Swedish nuclear waste program.

#### MATERIALS PREPARATION:

The materials studied (Table 1) are two zinc borosilicate glasses. The only difference between the two compositions is= ABS 29 contains ten times more ZnO than  $Fe_2O_3$  and ABS 41 contains the same quantity of each oxide. Table 1 also includes the composition of the 9% (by <sup>Wt</sup>) of simulated waste products added to the glass compositions.

The glasses were prepared by T.Lakatos of the Swedish Glass Research Institute by melting at  $1200^{\circ}$ C in platinum crucibles for 2-3 hours followed by casting into rods 1 x 1 cm in steel molds. After annealing at 550°C for 15 minutes, followed by clow cooling, the blocks were sliced into approximately 2 mm thick samples using a diamond blaced watering saw operating saw operating at moderate speed with water as a coolant and lubricant.

The as-cut samples were dry polished with 320 and 600 grit SiC paper. After polishing, the samples were cleaned twice in an ethanol ultra-sonic bath for three minutes.

#### EXPERIMENTAL PROCEDURE:

Leachings were conducted by suspending the samples on Teflon(R) thread inside Teflon or polypropylene containers filled with distilled and deionized water of initial pH= 5.5. (Figure 1). Two different ratios of glass surface area (SA) to solution volume (V)were used in the study, SA/V=1cm<sup>-1</sup> and SA/V=0.1 cm<sup>-1</sup> to determine the influence of this parameter on the chemical durability. Other studies have indicated that the dependence of leaching behavior of glasses on SA/V can be related to the mechanisms of leaching and the long term performance of glasses (4).

All procedures followed were as far as possible equivalent (5) to that described in the MCC-1 Standard Test for Static Leaching. Leaching times were 1, 3, 7, 14 and 28 days for each glass. An oven was used to maintain the temperature of 90-0.25°C.

After leaching, the glass samples were removed from the leaching cells, dried at room temperature and stored in a desiccator. Infrared reflection spectroscopy (IRRS) was used for surface analysis on all leached samples using the techniques previously described. (6-8). The pH of the leachant solutions was measured using a pH microelectrode. The concentrations of Si<sup>4+</sup>, B<sup>3+</sup>, Al<sup>3+</sup> and Mo<sup>6+</sup> in the leachant solutions were determined with ICP spectroscopy. The concentrations of Na<sup>+</sup> were determined with Atomic Emission spectroscopy and the concentrations of Fe<sup>3+</sup> with Atomic Absorption spectroscopy.\*\*

Average leach rates are calculated from the leachant data and with the different parameters as:

 $L_{i}(0,t) = \Delta \frac{m_{i}(0,t)}{m_{i},0} \cdot \frac{m_{i},0}{s.t}$ (1)

 $-\Delta m_i(0,t) = mass of element i in the solution water t days of leaching (g).$ 

 $m_0 = initial total mass of the sample (g).$ 

(R) Trademark EI Dupont de Nemours, Inc.

- \* Micro combination pH probe MI-410. Microelectrodes, Inc. Model 201 digital pH meter. ORION RESEARCH
- \*\* ICP Plasmatherm (1.5KW). AA/AE spectrometer IL 451 Videos Instrumentation Laboratory.

	mi,0	=	initial	mass	of	eleme	nt	i i	n	the	samp	le	(g).
-	S	=	surface	area	exp	posed	to	the	e 1	each	ing	(cm	2).

- t = leaching time (day).

This formula of average leach rate is the formula used in the Marcoule Research Program (Commissariat à l'Energie Atomique, FRANCE) and proposed in the MCC-1 Static Leach Test of the Materials Characterization Center (5).

To permit the comparison of this work with the researches of other laboratories (Swedish Nuclear Waste Program KBS), a second formula was used as well:

Loss rate= 
$$\frac{\text{ppm of element}}{10^6}$$
 .  $\frac{1}{\text{day}}$  .  $\frac{1}{SA/V}$  (2)

- loss rate in  $g.cm^{-2}$ .  $d^{-1}$
- SA= surface area exposed to the leaching  $(cm^2)$ .
- V = volume of leachant (cm<sup>3</sup>).
- day= leaching time (day).

The weight losses were too little to give good results and therefore are not reported.

**RESULTS:** 

#### Solution data

Table 2 presents the pH of the leaching solutions for the two ratios of SA/V. Figure 2 shows the curves of pH=f(t) for each glass and each SA/V. All curves have the same behavior, the pH increasing continously with time. The values of the pH for SA/V=1 cm<sup>-1</sup> are higher (9.45 after 28 days) than the pH for SA/V= =0.1 cm<sup>-1</sup> (8.8 and 9.2 after 28 days). The shape of the pH curves indicates that the pH attains a limiting value under static leaching conditions of > 8.5 after 28 days.

After 28 days the solution pH for both ABS 29 and ABS 41 are the same (9.45) at  $SA/V= 1 \text{ cm}^{-1}$ , but at  $SA/V= 0.1 \text{ cm}^{-1}$  the pH of the leachant for ABS 41 is higher (9.20) than the pH of the leachant for ABS 29 (8.80).

Leaching results for the different experiments are given in parts per million (ppm) of ions in the leachant (table 3). Tables 4 and 5 present the average leach rates and the loss rates calculated from the concentration data with formulas (1) and (2). The solution analysis were made only for SA/V= = 0.1 cm<sup>-1</sup>; for SA/V= 1 cm<sup>-1</sup> the quantity of the leachant was too little for accurate measurements. Figures 3 a, 3 b and 4 a, 4 1 show the time dapende be of the average leach

rates and the loss rates for the two glasses.

The values of the solution concentrations (ppm), the average leach rates, the loss rates and the solution pH are all higher for the mixed  $ZnO-Fe_2O_3$  composition (ABS 41) than for the high ZnO composition glass (ABS 29). The differences between the two compositions become important and increase after 7 days of leaching showing that ABS 29 is more resistant to attack of 90°C water than glass ABS 41.

For ABS 29, the average leach rates for all the elements decrease with duration of exposure. For example,

 $L_{Si}$  (0,1) = 5.3 10<sup>-5</sup> >  $L_{Si}$  (0,28) = 1.9 10<sup>-5</sup> g. cm<sup>-2</sup>.d<sup>-1</sup>  $L_{B}$  (0,1) = 1.6 10<sup>-4</sup> >  $L_{B}$  (0,28) = 5.8 10<sup>-5</sup> g. cm<sup>-2</sup>.d<sup>-1</sup>  $L_{Na}$  (0,1) = 7.0 10<sup>-5</sup> >  $L_{Na}$  (0,28) = 2.4 10<sup>-5</sup> g. cm<sup>-2</sup>.d<sup>-1</sup>

Thus the quantity of element passing each day into solution decreases with duration of the leaching. This result suggests a continuing change in the surface character of the glass ABS 29 and that equilibrium has not been reached.

For contrast, for ABS 41 the average leach rates are relatively constant with duration of exposure. For example,

 $L_{Si}$  (0,1) = 5.1 10<sup>-5</sup>  $\simeq$   $L_{Si}$  (0,28) = 5.3 10<sup>-5</sup> g. cm<sup>-2</sup>.d<sup>-1</sup>  $L_{B}$  (0,1) = 2.1 10<sup>-4</sup>  $\simeq$   $L_{B}$  (0,28) = 1.4 10<sup>-4</sup> g. cm<sup>-2</sup>.d<sup>-1</sup>

For the other elements Al, Na, Fe, Mo, Zn the average leach rates decrease a little with the number of days. However, for this glass equilibrium is almost attained and the quantities of Si<sup>4+</sup> and B<sup>++</sup> passing each day into solution are nearly the same. Thus ABS 41 seems to attain an equilibrium behavior in 90°C water more rapidly than ABS 29.

The average leach rates for Al<sup>3+</sup> are rather important and are comparable with the average leach rates for B<sup>3+</sup> and Na<sup>+</sup> for these conditions. Leaching results for Zn are somewhat lower but the leaching rates for Fe<sup>3+</sup> are nearly 30 times less than the other elements, even for ABS 41 which contains significant quantities of Fe<sub>2</sub>O<sub>3</sub> in the glass.

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### Surface Analysis

IRRS spectra were obtained to compare changes in the sample surface due to leaching. The interpretation techniques of the IRRS spectra were previously described (6-8). The spectrometer is calibrated with a vitreous silica sample. The primary peak of vitreous silica is located at 1120 cm<sup>-1</sup> and corresponds to silicon - bridging oxygen - silicon stretching vibrations (8). A second major peak of vitreous silica is located around 475 cm<sup>-1</sup> and is due to the silicon - bridging oxygen - silicon bending vibrations. The little peak around 800 cm<sup>-1</sup> is not used for the interpretation. Most interpretations of surface corrosion of glasses is based upon changes in the stretching vibration peak (6,9). The spectrometer is calibrated to have 80% of reflectance for the primary peak (1120 cm<sup>-1</sup>) and 50% of reflectance for the second major peak (475 cm<sup>-1</sup>) of pure vitreous silica.

Figure 5 shows the vitreous silica spectrum with its two characteristic peaks and the spectra of glasses ABS 29 and ABS 41 before corrosion. The spectral intensity in the region between 800 and 900 cm<sup>-1</sup> for the nuclear waste glasses is due to the presence of stretching vibrations of the silicon - non bridging oxygen - alkali and alkaline earth ions in the glass surfaces.

When corrosion of the surface occurs, the exchange of alkali and alkaline earth cations with hydrogen ions from the solution reduces the intensity of that portion of the spectral region. The same reactions cause the maximum of the peak around 1000 cm<sup>-1</sup> to increase and move to 1120 cm<sup>-1</sup>. These changes in spectra are characteristic of formation of a silica-rich film on the surface of the sample. This phenomenon can be seen by comparing the spectra before corrosion (Figure 5) and the spectra after 1 day of corrosion (figure 6).

For the 7 first days, the evolution of the spectra (Figures 6, 7 and 8) shows only the formation of a silicarich film on the glass surface and no difference between the two glasses. After 14 and 28 days (Figures 9 and 10) the reflectance intensity of the spectra is reduced by a gradual destruction of the surface film that had formed on the glass. This produces a roughening of the surface and scattering of the infrared beam incident on the sample. The attack of the surface film is due to the high solution pH obtained after 14 days which results in an attack of the Si-O-Si bonds on the silica-rich film and a breakdown of the film structural network.

Thus the IRRS analysis confirms the solution results; i e ABS 29 is a more leach resistant composition than ABS 41 for  $SA/V = 0.1 \text{ cm}^{-1}$ . However, when solution conditions correspond to  $SA/V = 1 \text{ cm}^{-1}$  the two glasses give nearly identical results.

<sup>\*</sup> Spectrometer model 577 PERKIN-ELMER

#### Discussion of Results:

The substitution of  $Fe_2O_3$  for ZnO in this pair of glasses results in important differences in leaching and surface characteristics. ABS 41 (3.0%  $Fe_2O_3$ , 3.0% ZnO) yields average leach rates and elemental loss rates that are nearly 3 times higher for Si<sup>4+</sup>, B<sup>3+</sup>, Na<sup>+</sup> and Mo<sup>6+</sup> than ABS 29 (0.6%  $Fe_2O_3$ , 6.0% ZnO).

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Although the leach rates and loss rates for  $Al^{3+}$  are the same for the two compositions, the rates for  $Fe^{3+}$  release are three times higher for the low  $Fe_2O_3$  glass (ABS 29) than for high  $Fe_2O_3$  glass (ABS 41). However, the values of leaching and elemental loss rates of  $Fe^{3+}$  are more than 30 times lower for both glasse than observed for the other elements.

The difference in behavior of the two glasses cannot be attributed to variations in solution pH since little difference was observed between the glasses. Higher solution pH values were observed for SA/V = 1 cm<sup>-1</sup> than for SA/V = 0.1 cm<sup>-1</sup> (Figure 2). However, there was only little evidence of a difference in rate of attack of the glass surface as shown by IRRS analysis (Figures 8-10). Glass ABS 29 which shows the lowest solution pH value after 14 and 28 days (Figure 2) at SA/V = 0.1 cm<sup>-1</sup> also shows the least change in the IRRS spectra for this condition (Figures 9b, 10b). However, at the higher value, SA/V = 1.0<sup>-1</sup>, the solution pH for both glasses is the same, and higher, and the IRRS spectra for both glasses shows little difference between them (Figures 9a, 10a).

The decrease in intensity of the IRRS spectra of ABS 41 after 14 and 28 days at  $SA/V = 0.1^{-1}$  shows that the surface is undergoing network breakdown and destruction of protective films on the surface. In contrast, the IRRS spectra of glass ABS 29 shows little change under the same conditions. This difference in extent of surface damage is consistent with the leach behavior which shows glass ABS 29 having the lower leach rates for most species.

Thus, a mixture of ZnO and Fe<sub>2</sub>O<sub>3</sub> in the glass is less effective in controlling release of ions into 90°C water than ZnO by itself. The previous study that reported enhanced leach resistance due to Fe<sub>2</sub>O<sub>3</sub><sup>(3)</sup> was comparing a glass containing 6.4 weight percent Fe<sub>2</sub>O<sub>3</sub>. In the present study, ABS 29 contains 6 weight percent ZnO and exhibits good leach resistance. This suggests that a critical concentration of multivalence ions may be necessary for the surface to develop a protective second film on top of the SiO<sub>2</sub>-rich layer that results from rapid dealkalization. This type of dual protective film is termed a Type III glass surface (10), and was characteristic of the previously reported nuclear waste glass containing 6.4 per cent Fe<sub>2</sub>O<sub>3</sub>. Apparently if a mixture of multivalent ions are present, the concentration required to stabilize the protective secondary film is higher than if one species is concentrated in the surface film.

At the higher  $SA/V = 1.0 \text{ cm}^{-1}$  value the concentration of the mixed Fe + Zn film is able to be reached and the surface is protected more effectively than in the more dilute solution of  $SA/V = 0.1 \text{ cm}^{-1}$ . These results show the importance of understanding both the effects of glass composition and solution concentrations on the behavior of nuclear waste glasses.

#### Conclusions:

Replacement of half of the ZnO in an alkali-zinc-borosilicate nuclear waste glass with Fe<sub>2</sub>O<sub>3</sub> degrades leach resistance by approximately a factor of 3. Leach rates for the higher ZnO containing glass after 28 days are generally in the range of  $5.10^{-5}$  g.cm<sup>-2</sup>. d<sup>-1</sup> for B<sup>3+</sup>, Al<sup>3+</sup>, Mo<sup>6+</sup>;  $2.10^{-5}$  g. cm<sup>-2</sup>.d<sup>-1</sup> for Na<sup>+</sup>, Si<sup>4+</sup>; and 2 to  $7.10^{-6}$  g.cm<sup>-2</sup>.d<sup>-1</sup> for Fe<sup>3+</sup> and Zn<sup>2+</sup>. The surface of both types of glass appears to be protected by dual protective layers, one rich in SiO<sub>2</sub> and a second very thin film rich in multivalent species. The second film that contains a mixture of Zn and Fe is less effective as a diffusion barrier and is less resistant to network breakdown than the film without the Fe<sup>-+</sup>. This apparently is because a critical concentration of multivalence species is necessary to stabilize the second protective film. Differences in SA/V ratios affect the formation of the protective films and the rates of surface damage of the glass.

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GLASS OX IDE	ABS 29	ABS 41
SiO2	52.0	52.0
<sup>B</sup> 2 <sup>O</sup> 3	15.9	15.9
A1203	2.5	2.5
Na <sub>2</sub> 0	9.4	9.4
<sup>Fe</sup> 2 <sup>0</sup> 3	0.6	3.0
ZnO	6.0	3.0
Li <sub>2</sub> 0	3.0	3.0
UO2	1.66	1.66
S.W.P*	9	9

NUCLEAR WASTE GLASS COMPOSITIONS (WEIGHT %)

\* Simulated waste products

COMPOSITION OF SIMULATED NUCLEAR WASTE (WEIGHT %)

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OXI DE	<sup>Cs</sup> 2 <sup>0</sup>	Sr0	BaO	<sup>Y</sup> 2 <sup>O</sup> 3	Zr0 <sub>2</sub>	M00 <sub>3</sub>	MnO <sub>2</sub>	Ag <sub>2</sub> 0
WEIGHT %	9.78	2.89	5.11	1.67	14.22	18.11	8.56	0.12

OX IDE	SnO	Sb_0 2 3	La2 <sup>0</sup> 3	Nd203	Pr203	Ce203	NiO	CdO
WEIGHT %	0.19	0.04	7.89	13.44	4.22	8.33	4.11	0.29

LEACHANT pH

$-\frac{SA}{V} = 1 \text{ cm}^{-1}$	<b>.</b>				
DAYS GLASS	1	3	7	14	28
ABS 29	6.35	7.95	7.85	9.2	9.45
ABS 41	6.30	7.55	8.55	9.05	9.45

 $-\frac{SA}{v} = 0.1 \text{ cm}^{-1}$ 

DAYS GLASS	1	3	7	14	28
ABS 29	6.40	7.10	8.25	7.75	8.80
ABS 41	6.35	7.25	8.35	8.70	9.20

CONCENTRATION (ppm) OF ELEMENTS

LEACHED INTO SOLUTION

\_\_\_\_\_

$$\frac{SA}{V} = 0,1 \text{ cm}^{-1}$$

<u>- ABS 29 =</u>

Days	рH	Si	В	Al	Na	Fe	Мо	Zn
1	6.40 7.10	1.3 3.48	0.79 1.98	0.25 0.72	0.49 1.19	<b>〈</b> 0.01 0.01	0.16 0.43	0.22 0.52
7	8.25	5.58	3,36	1,09	1,92	0,01	0,62	0,80
14	7.75	13.56	8,61	2.01	4.75	0,02	1.52	0,78
28	8.80	13.10	8.02	2.30	4.61	0.03	1.44	0.96

## - ABS 41 =

Days	рH	Si	В	Al	Na	Fe	Мо	Zn
1 3	6.35 7.25	1.25 4.82	0.78 3.12	0 <b>.19</b> 0 <b>.</b> 59	0.65 1.80	0.01 < 0.01	0.26 0.59	0.12 0.17
7	8.35	7.36	4.67	0.83	2.69	0.03	0.73	0.19
14	8.70	21.88	14.52	1.97	7.50	0.04	2.65	0.23
28	9.20	36.31	24,90	2,91	10.80	0,05	4.55	0.24

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AVERAGE LEACH RATES IN 90° C WATER

AT  $SA/V = 0.1 \text{ cm}^{-1}$ 

 $-ABS 29 = (g.cm^{-2}.d^{-1})$ 

DAYS	рH	Si	В	A1	Na	Fe	Мо	Zn
1 3 7 14 28	<ol> <li>6.40</li> <li>7.10</li> <li>8.25</li> <li>7.75</li> <li>8.80</li> </ol>	$5.3 10^{-5}$ $4.8 10^{-5}$ $3.3 10^{-5}$ $4.0 10^{-5}$ $1.9 10^{-5}$	1.6 $10^{-4}$ 1.3 $10^{-4}$ 9.7 $10^{-5}$ 1.2 $10^{-4}$ 5.8 $10^{-5}$	$1.9 10^{-4}$ $1.8 10^{-4}$ $1.2 10^{-4}$ $1.1 10^{-4}$ $6.2 10^{-5}$	7.0 $10^{-5}$ 5.7 $10^{-5}$ 3.9 $10^{-5}$ 4.9 $10^{-5}$ 2.4 $10^{-5}$	$2.4 \ 10^{-5}$ $7.9 \ 10^{-6}$ $3.4 \ 10^{-6}$ $3.4 \ 10^{-6}$ $2.6 \ 10^{-6}$	$1.5 10^{-4}$ $1.3 10^{-4}$ $8.2 10^{-5}$ $1.0 10^{-4}$ $6.7 10^{-5}$	$4.6 \ 10^{-5}$ $3.6 \ 10^{-5}$ $2.4 \ 10^{-5}$ $1.2 \ 10^{-5}$ $7 \ 10^{-5}$

- ABS 41 (g.cm<sup>-2</sup>.d<sup>-1</sup>)

		1		1	Y	T		
DAYS	PH	Si	В	A1	Na	Fe	Мо	Zn
1 3	6.35 7.25	5.1 10 <sup>-5</sup> 6.6 10 <sup>-5</sup>	1.6 $10^{-4}$	$1.4 \ 10^{-4}$	9.3 ro <sup>-5</sup>	4.8 10 <sup>-6</sup>	2.4 10 <sup>-4</sup>	5.0 10-5
7	8.35	4.3 10 <sup>-5</sup>	1.4 10 <sup>-4</sup>	9.0 10 <sup>-5</sup>	5.5 10 <sup>-5</sup>	$1.6 10^{-6}$ 2.0 10 <sup>-6</sup>	1.8 10 <sup>-4</sup> 9.6 10 <sup>-5</sup>	2.4 10 <sup>-5</sup>
14 28	8.70 9.20	6.4 10 <sup>-5</sup> 5.3 10 <sup>-5</sup>	2.1 10 <sup>-4</sup> 1.8 10 <sup>-4</sup>	1.1 10 <sup>-4</sup> 7.9 10 <sup>-5</sup>	7.7 10 <sup>-5</sup> 5.5 10 <sup>-5</sup>	1.4 10 <sup>-6</sup> 8.5 10 <sup>-7</sup>	1.7 10 <sup>-4</sup>	6.8 10 <sup>-6</sup> 3.6 10 <sup>-6</sup>
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LOSS RATES IN 90° C WATER

AT  $SA/V = 0.1 \text{ cm}^{-1}$ 

- ABS 29 =  $(g.cm^{-2}.d^{-1})$ 

DAYS	рН	Si	В	A1	Na	Fe	Мо	Zn
1	6.40	1.3 10 <sup>-5</sup>	7.9 10 <sup>-6</sup>	2.5 10-6	4.9 10 <sup>-6</sup>	<1.0 10 <sup>-7</sup>	1.6 10 <sup>-6</sup>	2.2 10 <sup>-6</sup>
3	7.10	1.2 10 <sup>-5</sup>	6.6 10 <sup>-6</sup>	2.4 10 <sup>-6</sup>	4.0 10 <sup>-6</sup>	3.3 10 <sup>-8</sup>	1.4 10 <sup>-6</sup>	1.7 10 <sup>-6</sup>
7	8.25	8.0 10 <sup>-6</sup>	4.8 10 <sup>-6</sup>	1.6 10 <sup>-6</sup>	2.7 10 <sup>-6</sup>	1.4 10 <sup>-8</sup>	8.9 10 <sup>-7</sup>	1.1 10-6
14	7.75	9.7 10 <sup>-6</sup>	6.2 10 <sup>-6</sup>	1.4 10 <sup>-6</sup>	3.4 10 <sup>-6</sup>	1.4 10 <sup>-8</sup>	1.1 10 <sup>-6</sup>	5.6 10 <sup>-7</sup>
28	8.80	4.7 10 <sup>-6</sup>	2.9 10 <sup>-6</sup>	8.2 10 <sup>-7</sup>	1.6 10 <sup>-6</sup>	1.1 10 <sup>-8</sup>	5.1 10 <sup>-7</sup>	3.4 10 <sup>-7</sup>

- ABS 41 =  $(g.cm^{-2}.d^{-1})$ 

DAYS	рН	Si	B	A1	Na	Fe	Мо	Zn
1	6.35	1.3 10 <sup>-5</sup>	7.8 10 <sup>-6</sup>	1.9 10 <sup>-6</sup>	6.5 10 <sup>-6</sup>	1.0 10 <sup>-7</sup>	2.6 10 <sup>-6</sup>	1.2 10 <sup>-6</sup>
3	7.25	1.6 10 <sup>-5</sup>	1.0 10 <sup>-5</sup>	2.0 10 <sup>-6</sup>	6.0 10 <sup>-6</sup>	<3.3 10 <sup>-8</sup>	2.0 10 <sup>-6</sup>	5.7 10 <sup>-7</sup>
7	8.35	1.1 10 <sup>-5</sup>	6.7 10 <sup>-6</sup>	1.2 10 <sup>-6</sup>	3.8 10 <sup>-6</sup>	4.3 10 <sup>-8</sup>	1.0 10 <sup>-6</sup>	2.7 10 <sup>-7</sup>
14	8.70	1.6 10 <sup>-5</sup>	1.0 10 <sup>-5</sup>	1.4 10 <sup>-6</sup>	5.4 10 <sup>-6</sup>	2.9 10 <sup>-8</sup>	1.9 10 <sup>-6</sup>	1.6 10 <sup>-7</sup>
28	9.20	1.3 10 <sup>-5</sup>	8.9 10 <sup>-6</sup>	1.0 10	3.9 10 <sup>-6</sup>	1.8 10 <sup>-8</sup>	1.6 10 <sup>-6</sup>	8.6 10 <sup>-8</sup>





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FIGURE 2





FIGURE 3b



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FIGURE 5











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