The Effect of Pressure and Organic Constituents on the Cesium Ion Exchange Performance of IONSIV® IE-911

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SUMMARY

This study examined cesium (^{137}Cs) ion exchange of crystalline silicotitanate (CST) in simulated waste solution. In particular, the study focused on the effect of CST pretreatment on the kinetics and extent of cesium adsorption. The test used IONSIV®IE-911 (UOP LLC, Molecular Sieves Division, Des Plaines, IL), the engineered form of CST. Pretreatment steps examined include: soaking CST in 2M NaOH solution for three days, exposing CST to 50% relative humidity for one week, flowing organic-containing (saturated) salt solution through a CST packed bed (at 5 cm/min. superficial velocity), or drying CST in air at 100° C for three days. Some tests occurred under 50 and 25 psig of argon. The following conclusions summarize the results.

- Pretreatment of IE-911 in organic-containing (e.g., tri-n-butyl phosphate, dibutylphosphate, butanol, paraffin and Dow Corning H-10 defoamer) simulated waste or simulated waste yielded a 83% slower rate of cesium adsorption and 56% lower cesium capacity after one week.

- Pretreatment of IE-911 in 2M caustic solution for 48 hours yielded a slower approach to equilibrium cesium distribution in batch contact tests -- 7.7 mL/(g*h) during the first 48 hours and 2.4 mL/(g*h) thereafter. Carboxylates and adsorbed carbonates inside the pores likely affect the cesium transport by either increasing the path-length or reducing mass transfer rate.

- Heating IE-911 as received from the vendor at 100 °C for 24 hours significantly degraded its cesium removal performance by a 40.7% reduction in capacity and 43% reduction in sorption rate over one week of testing.

- Testing determined nearly identical distribution coefficients (K_d) between lot # 9990-9681-0004 and 9990-9881-0005 (i.e., difference of only 5.6%).

- Tests measuring water insertion rates into IE 911 show that hydration of the IE-911 does not appear to limit the rate of cesium sorption.

- Increasing the atmospheric pressure from 0 to 50 psig had no effect on cesium sorption.

Note that lower apparent capacity or slower cesium sorption rate in these limited-duration batch contact tests as a result of pretreatment do not necessarily imply reduced dynamic performance in a flowing ion-exchange application. The experiments that provided the bases for the currently proposed facility design

used caustic-pretreated IE-911. Another report will assess whether the presence of the organic compounds in the waste solution impeded column performance.

INTRODUCTION

Current engineering studies examine several processes for removing cesium from Savannah River Site (SRS) radioactive waste. One such process uses non-elutable ion exchange with crystalline –silicotitanate (CST) particles as the ion exchange media.[1] Column design requires knowledge of the equilibrium amount of cesium that adsorbs on CST. The typical measure of sorption involves the distribution coefficient (K_d), or the weighted (i.e., volume of salt solution per unit weight of solid) amount of adsorbed cesium relative to initial salt solution cesium concentration.

$$K_d = \left(\frac{Concentration_{Initial}}{Concentration_{Final}} - 1 \right) \times \frac{Volume\ of\ Salt\ solution}{Weight\ of\ CST}$$

The distribution coefficients measured previously varied significantly with method of CST pretreatment.[2] The initial water content as well as soaking CST in caustic solution significantly affected cesium adsorption. Further review of the data suggested the need to study other variables. In particular, the proposed application suggested the need to understand the effect of pressure and the presence of organic in salt solutions on the cesium distribution coefficient.[3-6] Since issuance of the work by McCabe, UOP developed a manufacturing process for an engineered form of CST, designated as IONSIV® IE-911. Engineering personnel asked Savannah River Technology Center to investigate the influence of various pretreatment steps on the performance of IONSIV® IE-911 (Technical Task Request # HLW-SSSDT-TTR-99-08.1, "K_d as Function of Pressure and Organics" R. A. Jacobs, February 1, 1999). The work follows the plan defined in:

> "Task Technical Plan For Pressure, Vacuum and Organic Fouling Effects on Cesium K_d (Decontamination) Values," F. F. Fondeur, WSRC-RP-99-00202, Revision 0, February 26, 1999.

EXPERIMENTAL

Water Insertion Test

The author placed about 0.3 g of IE-911 in a basket shaped 100 mesh carbon steel wire and placed the basket in de-ionized and distilled water for a specified amount of time. The author then removed the IE-911-wire mesh from the water and gravity drained to remove any water. The remaining free water was further dried with slow flowing air. The author judged the sample as dry when the IE-911 particles no longer stuck to each other and then weighted the sample at this point. From the weight the author determined the amount of water gained during the experiment.

IONSIV® IE-911 Pretreatments

Personnel prepared and filtered 2 liters of simulated waste with composition listed in Table 1.

Table 1. Simulated waste composition.

Component	Concentration (M)
Na^+	5.6
K^+	0.015
Cs^+	0.00014
OH^-	1.91
NO_3^-	2.14
NO_2^-	0.52
AlO_2^-	0.31
CO_3^{2-}	0.16
SO_4^{2-}	0.15
Cl^-	0.025
F^-	0.032
PO_4^{3-}	0.010
$C_2O_4^{2-}$	0.008
SiO_3^{2-}	0.004
MoO_4^-	0.0002

Testing examined lots # 9990-9681-0004 and 9990-9881-0005. Table 2 defines the different IE-911 pretreatment steps examined. Personnel made no effort to remove fines from IE-911 since previous study showed the presence of fines as insignificant to the adsorption performance.[2]

Table 2. IE 911 pre-treatment steps examined.

Treatment	Duration time
NaOH (2M)	3 days
50% Relative Humidity	7 days
100 °C	3 days
Organic saturated, simulated waste	9 hours
0, 25 and 50 psig during test	72 hours

Organic consists of tri-n-butyl phosphate (TBP), dibutylphosphate (DBP), n-paraffin, n-butanol and defoamer H-10 (Dow Corning).

High Pressure K_d Test

High pressure K_d testing used the device shown in Figure 3. The author added 25 mL of salt solution containing cesium and 0.1 g of IE-911 to the vessel. The sealed compartment shook under pressure (0, 25 and 50 Psig) for one week. At the end of the test, I separated the solution from the solids and analyzed for cesium.

Organic Pretreatment

The author set aside a finite amount of solution for organic addition. Table 3 lists the quantity of each organic added to this solution. I added more than a soluble quantity of each organic to the salt solution, mixed for about half a hour and filtered through a 0.5 micron glass-frit filter. The preparation occurred at room temperature (25 °C).

At the end of each pretreatment, I drained the solution and replaced with 25 mL of cesium containing salt solution. Each test used 0.1 g of wet IE-911 and a salt solution containing 19 mg/L cesium. The tests shook the

Table 3. Organic species for fouling test.

Material	Concentration
Tri-n-butyl phosphate	Saturation level (<10 ppm)
DBP and n-paraffin	Saturation level for each (2.0 g/L DBP, 10 ppm n-paraffin)
Butanol	2 g/L
Evaporator defoamer (Dow Corning Antifoam H-10)	100 ppm

resulting slurry using an orbital shaker at a frequency of 51 rpm for 24, 48, and 72 hours and for one week. At the end of each test, personnel separated the liquid from the solids and placed in glass bottles for analysis of cesium concentration in solution via Gamma Counting Spectroscopy

K_d Computation and Error Estimates

Computation of the K_d value requires the ratio of the initial to final cesium concentration. Instead, the author substituted the ratio of the corresponding gamma counts. This substitution remains valid as long as a linear relationship exists between cesium concentration and gamma counts. Figure 1 provides a calibration curve for the spectrometer showing the validity of the assumption. Calibration occurred only twice during these experiments: once before measurements and a second time after data collection. In addition to the check for linearity in performance, the author verified the performance of the gamma counter through independent analysis of a set of samples by the Analytical Development Section. Table 4 and Figure 2 shows the measured concentrations as determined by the ADS equipment and by the equipment used for this testing.

Table 4. Disintegration per minute per mL comparison between In-House Counter and the Analytical Division Services (ADS) equipment.

ADS Equipment	In-House Counter	% Difference*
4643.2	4902	5.6
5045.4	4678	7.3
4174.2	4320	3.3
3617.2	3424.5	5.3
23127	23740.5	2.7

* Percent difference relative to ADS

This table lists the highest percent difference between these five samples as 7.3%. Hence the combined error from the variation of the in-house instrument and its inaccuracy approaches 8%. The analysis included measurement of a standard and blank after every third sample.

Error determinations use the following propagation formula.

$$\left(\frac{\Delta K_d}{K_d}\right)^2 = \left(\frac{\Delta C_{initial}}{C_{initial}}\right)^2 + \left(\frac{\Delta C_{final}}{C_{final}}\right)^2 + \left(\frac{\Delta V}{V}\right)^2$$

Errors due to determination of weight of sample (~0.5%) remain insignificant in comparison to errors from gamma counting. Error determination in gamma counting is the square root of the total gamma count. The gamma count percent error ranged from 0.2 (for 6000 counts) to 0.8 (for 600 counts) percent. This error is insignificant compared to sampling errors considered in the propagation of error formula listed above. The difference in gamma counting from duplicate samples ranged from 20 counts for consecutive analyses to 450 counts when time between analyses measured two hours. These differences represent errors of 1.2 to 9% in the calculated K_d values.

Microscopy and Spectroscopy Equipment

The Analytical Development Section performed surface microscopy (SEM) and Energy Dispersed X-ray Fluorescence on samples using an ISI DS130 dual stage microscope. The author analyzed selected samples by molecular infrared spectroscopy (IR) using a Nicolet 220 spectrometer.

RESULTS AND DISCUSSION

The Rate of Water Insertion into IE-911

Figure 4 shows the approximate amount of de-ionized, distilled water that enters the pore volume after immersion for a specified time. The test examines both "as received" IE-911 and material after heating to 100 °C for 24 hours.

The two sets of data show comparable rates of water insertion and approach the same approximate water content after 60 minutes immersion. Note that the manufacturer claims a relative pore volume of between 37% to 43% for the IE-911. The close agreement between the measured water content after 60 minutes and this theoretical value suggest the presence of relatively large pore volumes.

The Effect of Heating and Relative Humidity

Figure 5 shows the effect of heating on the cesium removal capacity of IE-911. Inspection of the figure suggests most experiments showed a region where the rate of cesium exchange appeared relatively constant (i.e., zero order kinetics). One can approximate the rates from the slopes of the lines in the figure. Table 5 shows the cesium ion exchange rate of pretreated IE-911.

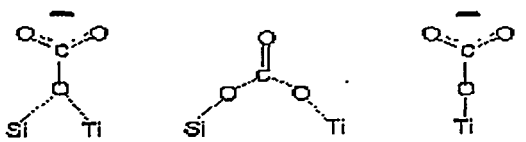
Table 5. The effect of pretreatment on the initial cesium ion exchange rate.

Pretreatment	Initial Rate (mL/(g*h))	K_d (mL/g) at 168 hours
"as received"	18.8	1949
50% Relative Humidity	22.3	2094
100 °C heated	12.7	1241
2 M NaOH	7.7	1440
Organic in Salt Solution	3.8	883

The treatment significantly reduce cesium ion exchange rate (12.7 mL/(g*h)) and extent (1241 mL/g) relative to that for the 'as received' material. The reduced capacity may reflect irreversible pore size reduction during dehydration[6] that significantly reduces the number of available absorption sites.

The Effect of Caustic Treatment

Soaking IE-911 in 2M NaOH largely converts the material to mostly the sodium form. According to literature, the cesium capacity should change slightly after soaking in 2M caustic solution.[1] However, the observed cesium ion exchange rate in these tests after caustic pretreatment proved slower than the "as received" IE-911 (see Figure 5 and Table 5). The difference may result from presence of foreign material inside the pores. Molecular spectroscopy (IR difference spectrum) identified carboxylate and carbonates inside the pores (see Fig. 6). The IR difference spectrum results from the subtraction of the IR spectrum of the "as received" IE-911 from the IR spectrum of the pre-treated IE-911. The IR difference spectrum (Figure 6) shows absorption near 1550 cm^{-1}, 1365 cm^{-1}, 877 cm^{-1}, 768 cm^{-1} and 679 cm^{-1}. The first two bands represent the asymmetric and symmetric stretch of the $-CO_2^-$ molecule and the rest of the bands associated with bending motions. The behavior at 905 cm^{-1} represents the surface Si-O-Ti bands perturbed by the sorption of carbonates. The loss of absorption near 3600 cm^{-1} arises from the loss of surface hydroxyls due to carbonate absorption as described in the caustic pretreatment section. The following sketch shows the possible carbonate configurations.



The third configuration best explains the absorption bands at 1532 and 1362 cm^{-1} seen in Figure 10. The second configuration requires an absorption band near 1620 cm^{-1} and 1265 cm^{-1}. The first configuration implies very limited stability due to the tri-coordinated oxygen atom. Figure 10 implies relative high stability of the adsorbed species. After prolonged exposure (i.e., 168 hours) of IE-911 to simulated waste, alkaline earth carbonate and bi-dentate carbonate form.

These compounds can form by exposure to carbon dioxide or carbonic acid as follows:

$$Tl - OH + NaOH + CO_2 \longrightarrow Tl - O - CO_2^- Na^+ + H_2O$$

The absorbed carbonates may sterically hinder cesium transport. Pretreating the particles may accumulate carbonates and carboxylates to a level that influences kinetics as well as capacity. Infrared analysis of the "as received" particles did not have any foreign substance on its surface or interior implying no carbonate present before the pre-treatments.

Another potential explanation involves an increase in film diffusion resistance. According to the literature, the effect of increased film diffusion resistance manifests itself as a linear relationship between K_d and time.[6] The pseudo-linear relationship seen in Figure 5 may indicate an increase in film diffusion resistance

as a consequence of caustic pretreatment. The limited data available, however, does not allow the author to quantify or verify that hypothesis.

Personnel investigated the material for physical and chemical changes. Microscopic observation (Figure 7) of the surface showed pits and craters after the exposure. Elemental analyses of these areas – by Energy Dispersed X-ray Fluorescence -- also showed depletion of binder as well as CST. The pretreatment results in larger surface area and loss of mass per particle. This finding indicates some degree of chemical instability in SRS waste solution. These effects could translate into weaker particles and loss of capacity and thus merit more extensive study.

The experiments also investigated the effect of drying after pre-treating IE-911 on the resulting cesium ion exchange ability. I placed IE-911 in caustic solution for two days. At the end of two days, personnel decanted the solution and maintained the solids at room temperature for a day. Table 6 and Figure 8 shows the result from a subsequent K_d test conducted on the dried material.

Table 6. K_d values of IE-911 dried and wet after caustic treatment.

Time (hours)	Dried after NaOH treatment (mL/g)	Wet after NaOH treatment (mL/g)	Percent difference*
24	610	834	26.8
48	774.5	973	20.4
72	995.5	1206	17.4

* Percent difference relative to wet sample.

Although the percent difference decreased with contact time, the data indicates drying IE-911 after treatment reduces the ion exchange ability. Drying of pretreated material adversely impacts performance. Hence, the facility should avoid circumstances that could lead to drying during interim storage of resin.

The Effect of Simulated Salt Solution Exposure on IE-911 Performance

Researchers placed IE-911 in salt solution (with no cesium) for two days. The researchers removed the salt solution and replaced with cesium containing salt solution. The test yielded a K_d of 1340 mg/L (not shown in Figure 5) after one week. Although the testing did not include a measurement at other times, the low value indicates slow kinetics relative to the as-received material. Personnel investigated the material for physical and chemical changes. Microscopic observation of the dried salt solution treated IE-911 (see Figure 9) showed a two-layer film on the surface. The upper layer contains sodium carbonate and the lower layer contains aluminum sulfate. The washed IE-911 showed pits and craters after the exposure to salt solution (figure not shown). Elemental analyses of these areas – by Energy Dispersed X-ray Fluorescence -- also showed depletion of binder as well as CST. Molecular examination (Figure 10) of the inside of the particle showed the presence of absorbed carbonates and

carboxylates. Energy Dispersed X-ray Fluorescence detected no nitrogen ruling out nitrate or nitrite salts as the cause of the reduced performance.

The Effect of Organic Pretreatment

Researchers placed IE-911 in salt solution saturated with selected organic compounds for extended periods of time. Figure 5 shows the cesium K_d values as a function of time. The K_d values increased linearly within a 168 hours period. At the end of this period the K_d value of 883 mL/g (see Figure 5) fell well below the value obtained from the "as received" material during the same time period. Personnel made efforts to identify physical and chemical changes in the material that correlated with material performance. Microscopic observation of the washed particle showed pits and craters just as in the case of the IE-911 in salt solution. Infrared analysis of the inside of IE-911 again indicated the presence of carboxylates and carbonates. The analyses showed no hydrocarbons or phosphate on or in the particles.

Personnel tried to isolate the effect of the internal carbonate by washing the material several times with distilled, de-ionized water. The cesium ion exchange capacity of the washed IE-911 reached 1357 mL/g after one week (not shown). Since this value falls below the "as received" material and suggests the inorganic material situated in the inside of the particles does affect kinetics and capacity. The low cesium ion exchange capacity of the unwashed material (883 mL/g) results from the combined effect of internal fouling and exterior film formation. Again the linearity between K_d and time serves as the best evidence for exterior film formation suggesting mass transfer mechanisms as a rate-limiting step

The Effect of Pressure on K_d Values

Researchers contacted CST particles with cesium containing salt solution under argon at various pressures (0, 25 and 50 psig). Figure 11 shows that pressure has negligible effect on cesium K_d values. The figure suggests no correlation of distribution coefficient with applied pressure. In an earlier report, K_d values proved sensitivity to vessel geometry and shaking frequency. [4] Since this experiment used a different vessel one can not directly compare with data from the other tests in this study.

Variability of Cesium Sorption between Manufacturer Lots

Figure 12 shows cesium sorption data from experiments using two different lots of material: lot 9990-9681-0004 and lot 9990-9881-0005. The tests showed good agreement (5.6% difference) between the two lots both in the "as received" state and pretreated in flowing salt solution containing organic. On other hand, lot 9990-9681-0004 exhibit reduced cesium ion exchange capacity (28% difference) when treated in 2 M NaOH compared to the 'as received' state.

IE-911 Stability in Salt and Caustic (2M) Solution

The researcher placed IE-911 in salt and caustic solutions for two weeks. In one case, the test continuously flowed 4000 bed volumes of salt solution – over 2 weeks -- through a column packed with the CST (at a superficial velocity of 5 cm/min). After contacting the IE-911 with the solution, personnel washed the particles and analyzed for topography and chemical composition. Surface topography revealed the pits and craters. Chemical analysis of the same material showed titanium, as well as binder, depletion at the surface of the particles relative to its interior (see Figure 13). Similar surface changes occurred with materials exposed to caustic solution (2M). The pitting suggests the potential selective removal of titanium and attrition leading to fines containing cesium. Since titanium exists in tetrahedral and octahedral[7] coordination (with a minus charge), cesium attaches preferentially to the octahedrally coordinated titanium atoms. Microscopy analysis suggests the chemical attack and pitting appears limited to the surface, which could imply no significant reduction in column performance. However, formation of small fines may result in passage of radio-cesium from the column, thereby detracting from performance. Given the observed behavior, additional longer-term exposure with subsequent column testing will provide insight into process limitations.

Conclusions

Pretreatment of IE-911 affects the rate at which equilibrium is obtained. Depending on the treatment, the rate of cesium ion exchange may decline sharply. There may not be effect on column performance since current column models include the effects seen in this study. The current tests suggest fouling and film diffusion barriers as likely causes of poor performance. Infrared spectroscopy identified carbonate species that could cause internal fouling. These species developed as a result of the pretreatment. Linear time dependence of K_d provides evidence of high film diffusion resistance. In particular, IE-911 in salt solution with organic addition showed a linear temporal behavior and the most detrimental impact on performance. A second study, in progress, will examine whether the presence of the organic detracts from performance in a column test.

Manufacturer lot # 9990-9681-0004 and 9990-9881-0005 exhibited nearly identical cesium distribution coefficient (K_d) except when pretreated in 2M caustic solution. For the 2 M NaOH treatment, lot 9990-9681-0004 demonstrated lower capacity than lot 9990-9881-0005.

During two weeks of contact between IE-911 and simulated wastes, the particles developed pits and craters as well as showing losses of titanium and binder in

salt and caustic solution. These observations raise questions regarding the long-term stability of the CST in contact with waste solutions. Future tests should examine ion exchange column performance after prolong contact with waste solution.

Acknowledgement

The author thanks Jack Durden and Mike Summers for their help in obtaining the SEM photographs as well as their determination of elemental compositions.

References:

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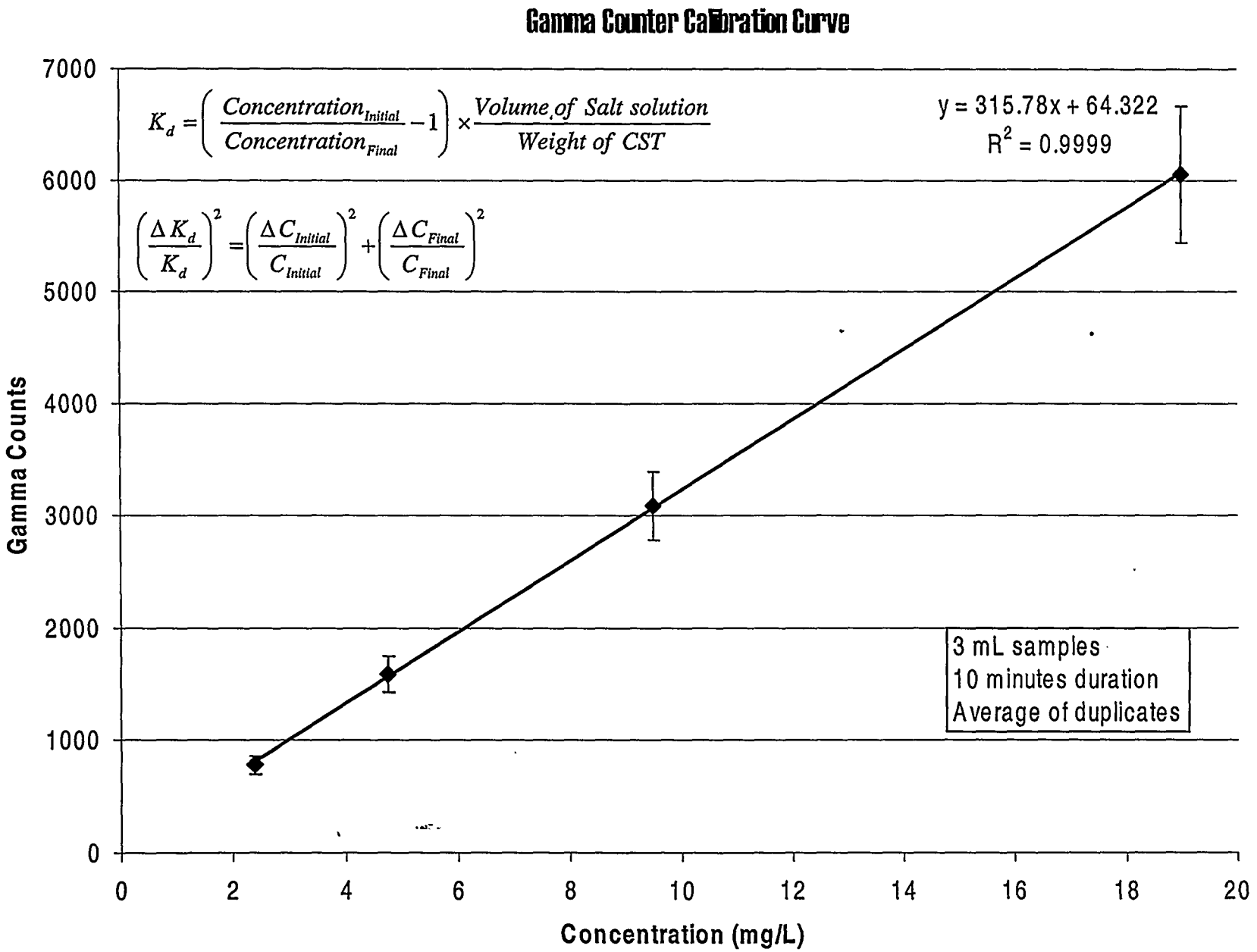


Figure 1. Calibration curve of in-house gamma counter.

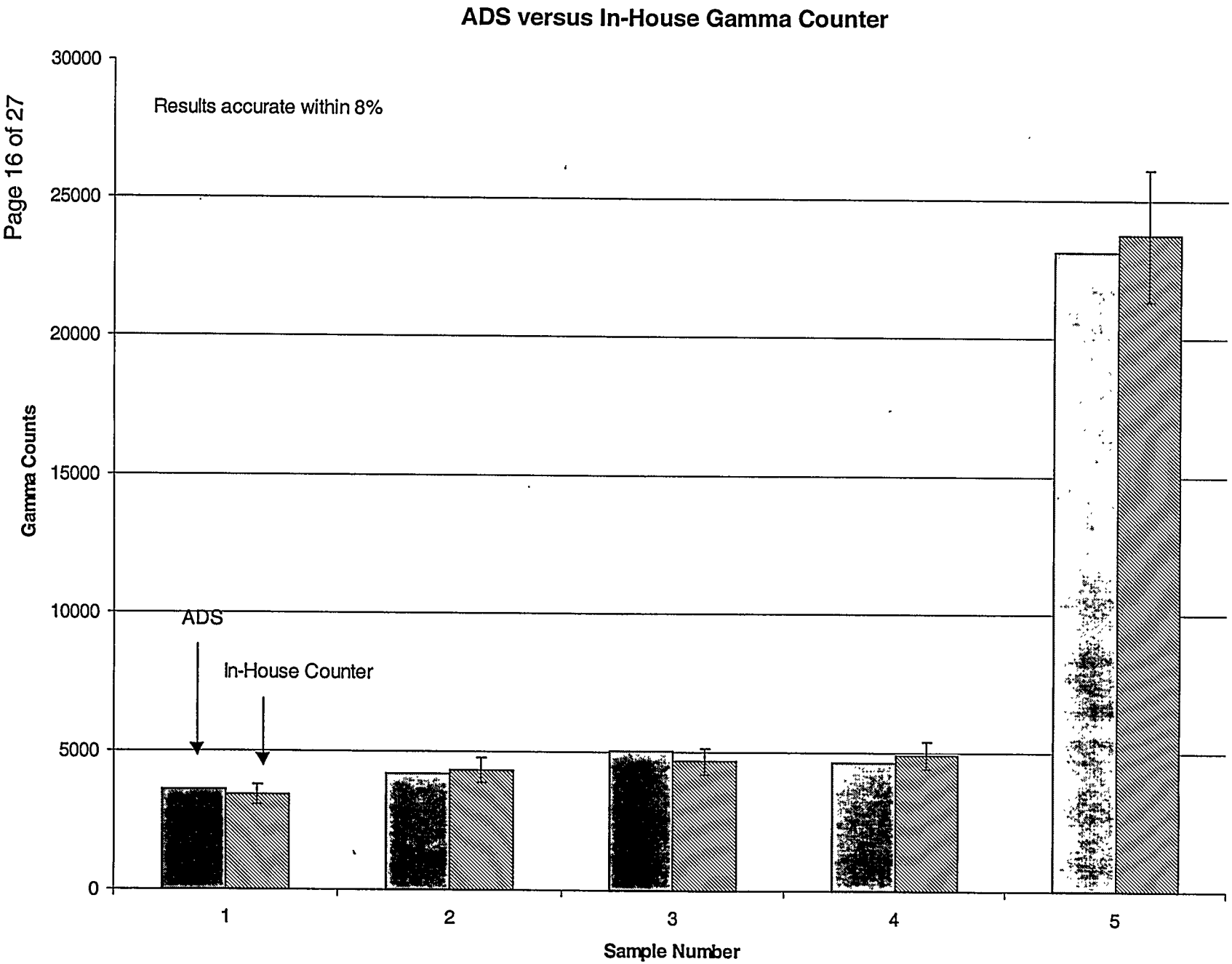


Figure 2. Gamma counts comparison between in-house and ADS gamma counter

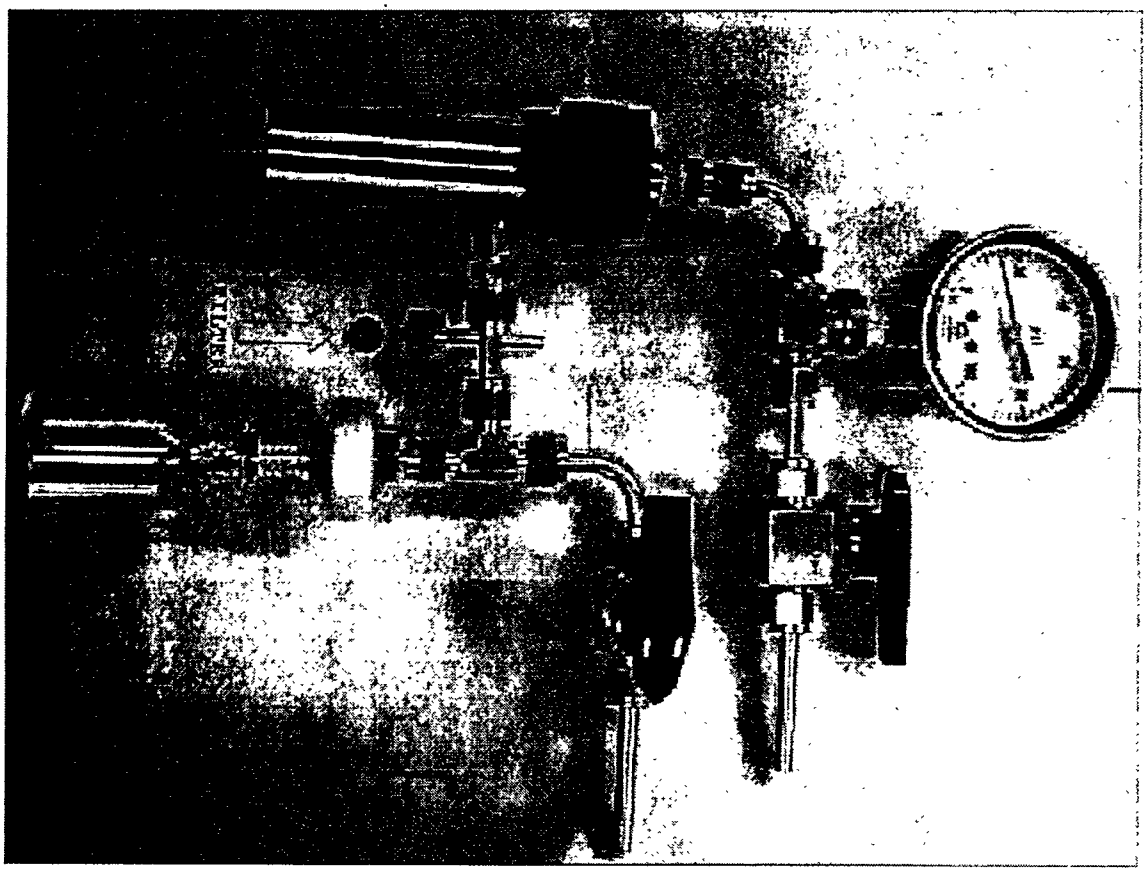


Figure 3. Device used for testing pressure effects on K_d

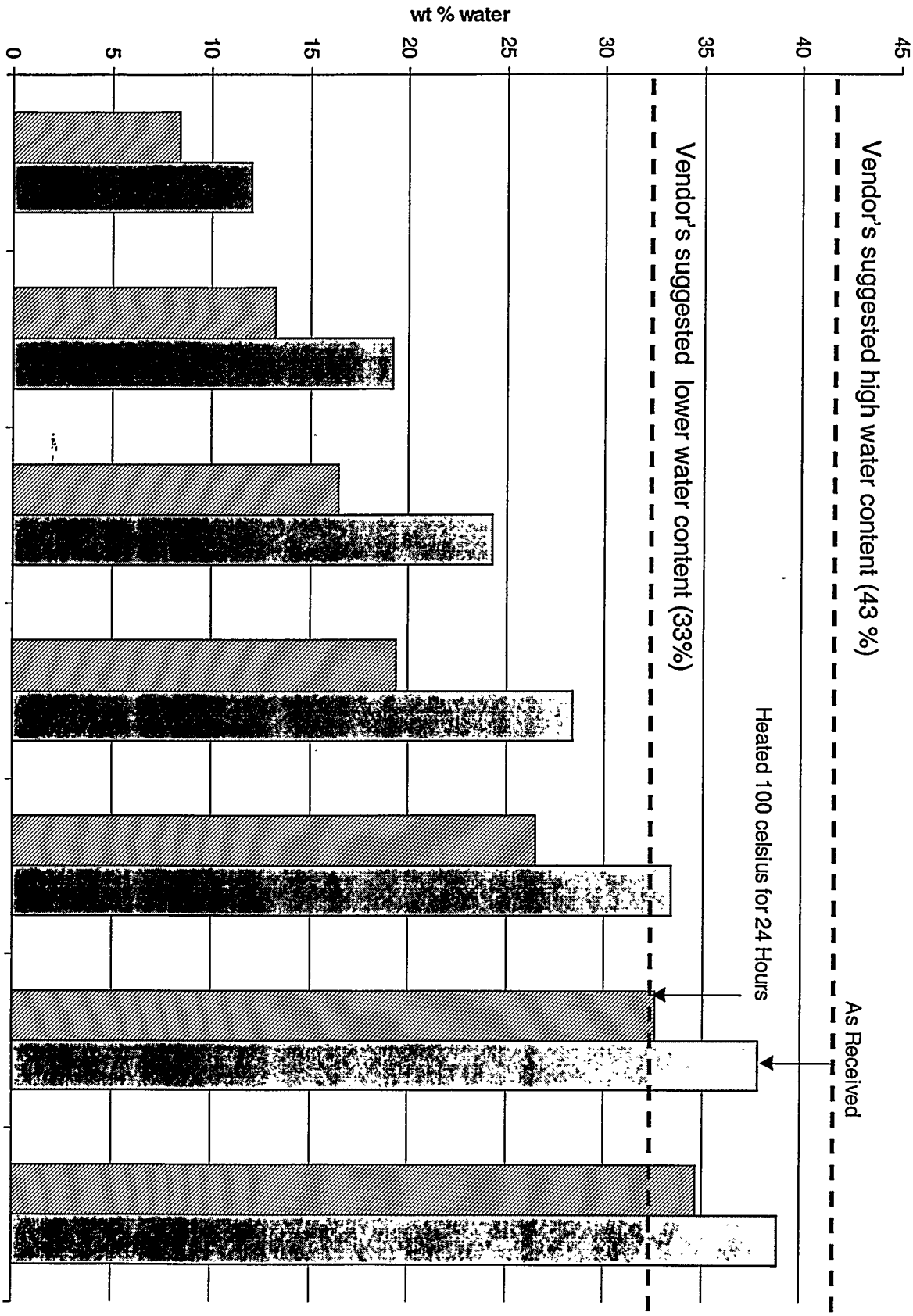


Figure 4. The amount of water uptake of IE-911 as a function of time.

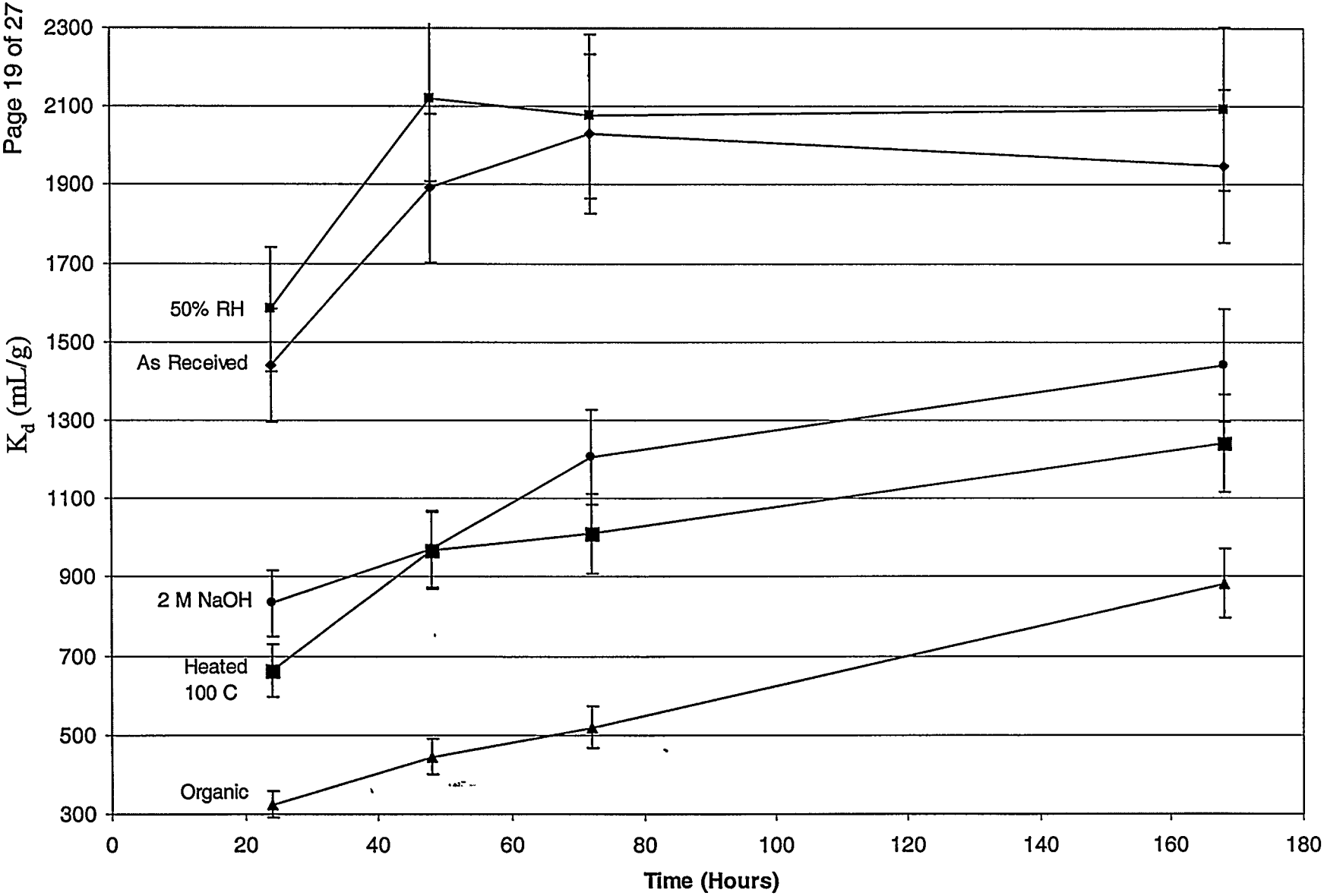


Figure 5. The effect of IE-911 pretreatment on cesium K_d values.

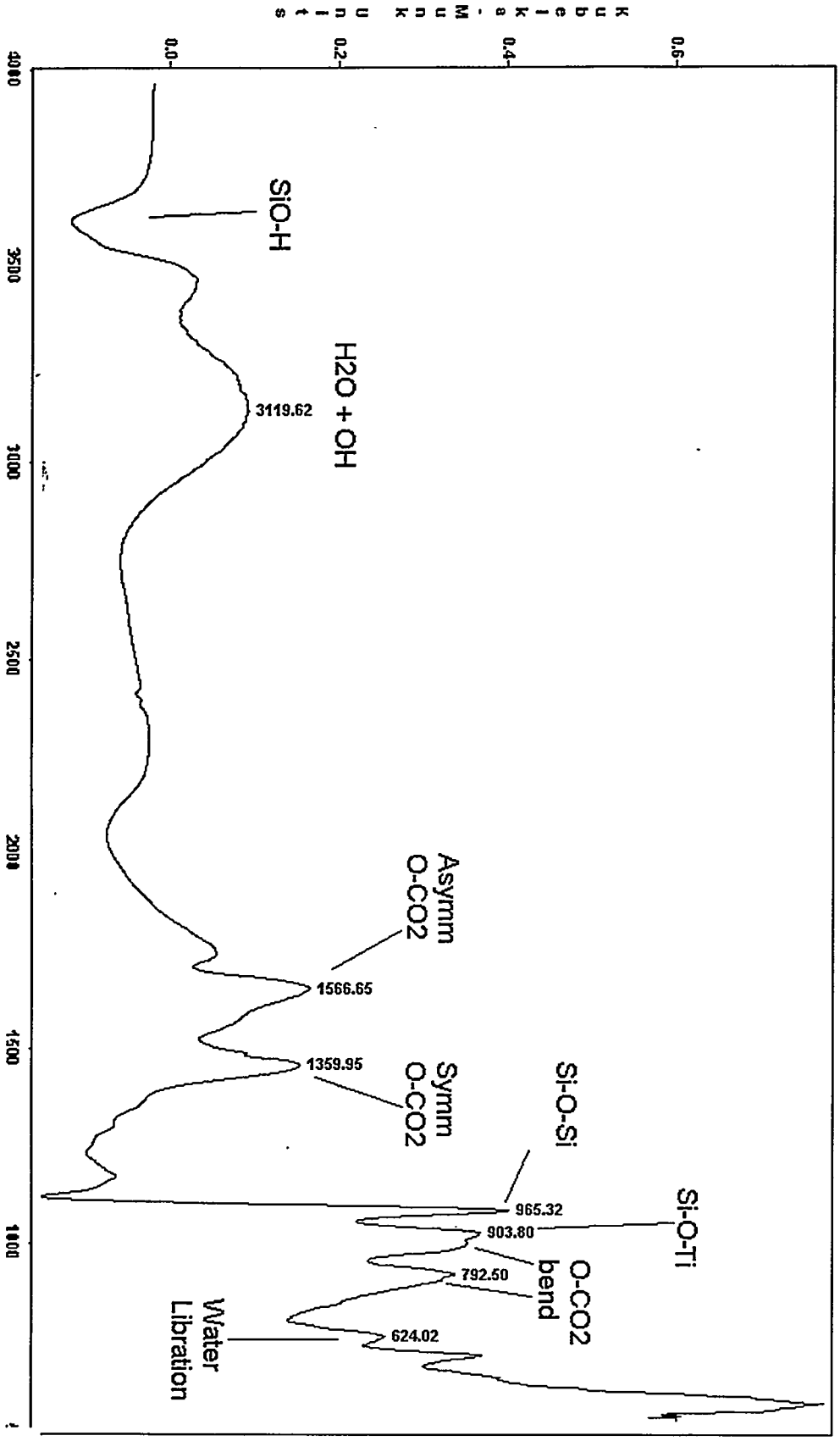
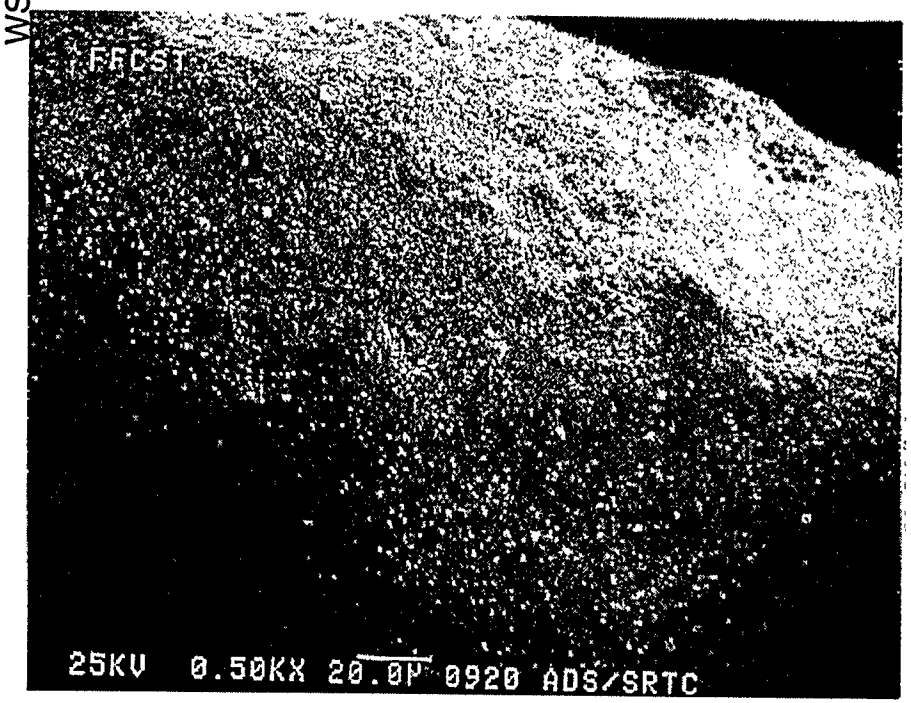


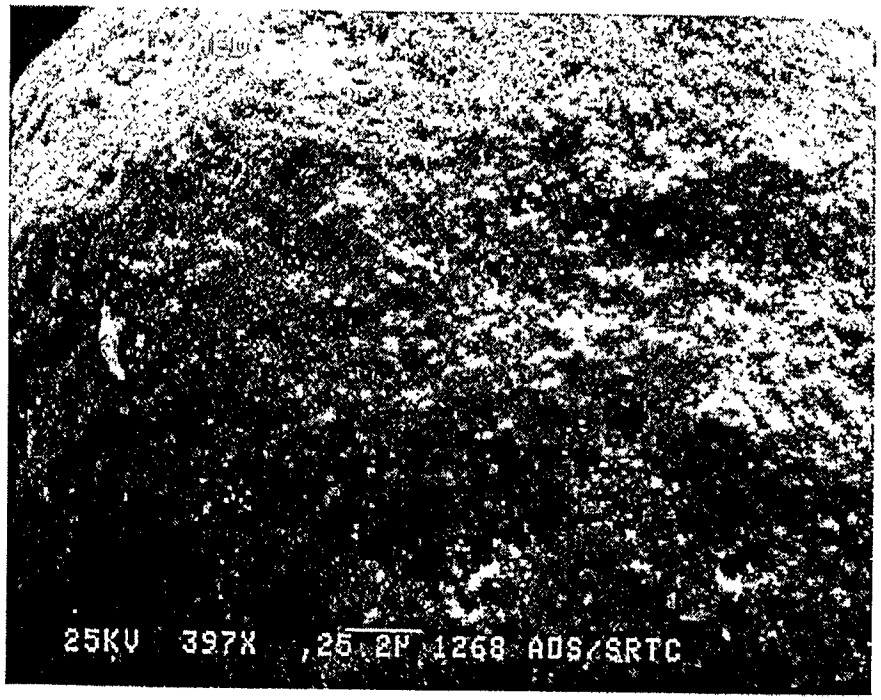
Figure 6. Infrared difference spectrum of IE-911 after 48 hours in 2M caustic solution

Before Exposure



* White spots shows regions enriched with binder

After 48 hours in 2 M NaOH



* This pictures shows the formation of pits and craters after caustic solution treatment.

Figure 7. SEM picture of the surface of IE-911before treatment (left) and after 48 hours in 2M caustic solution (right).

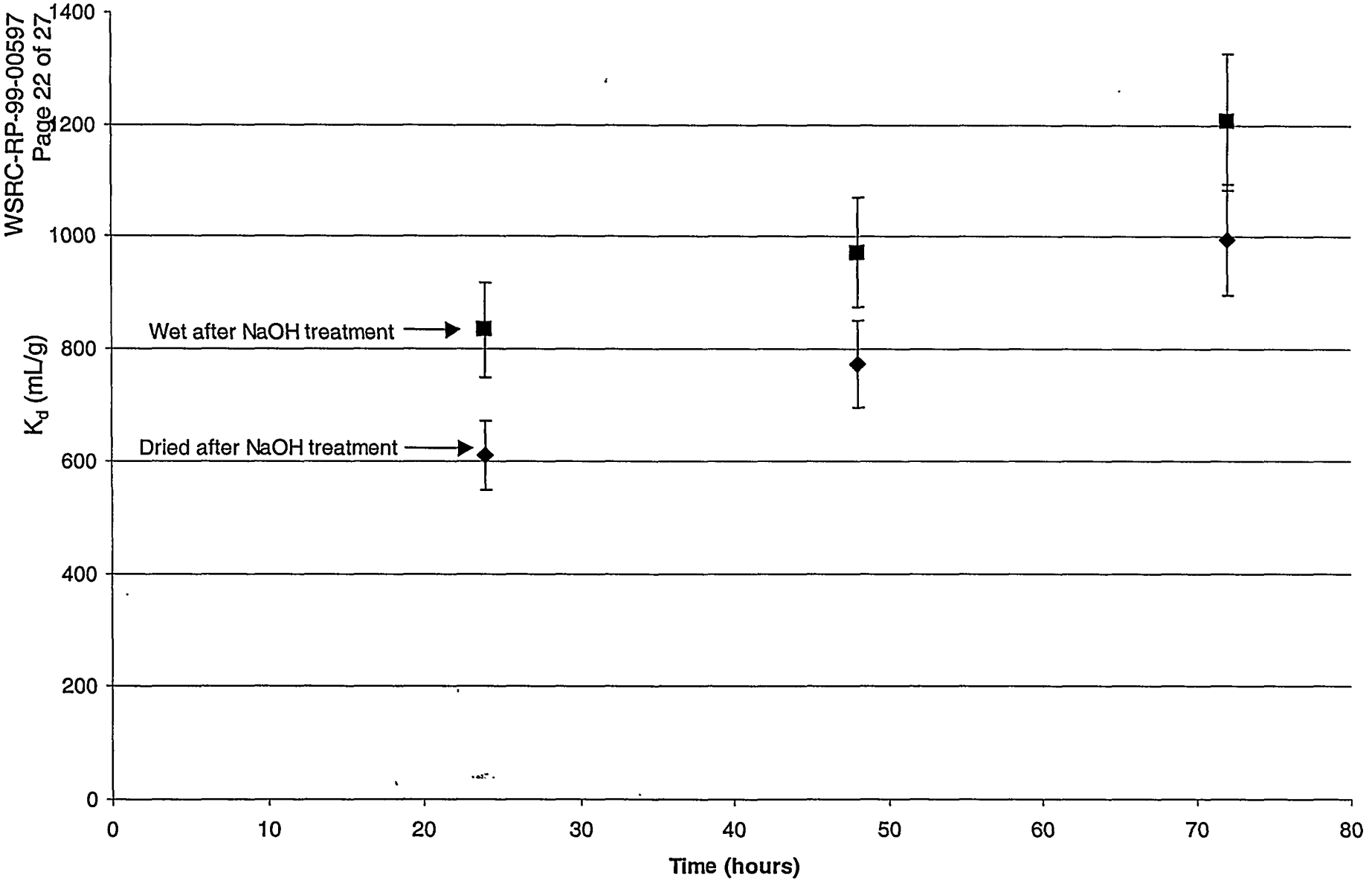


Figure 8. The effect of drying after pretreating IE-911 on cesium K_d value.

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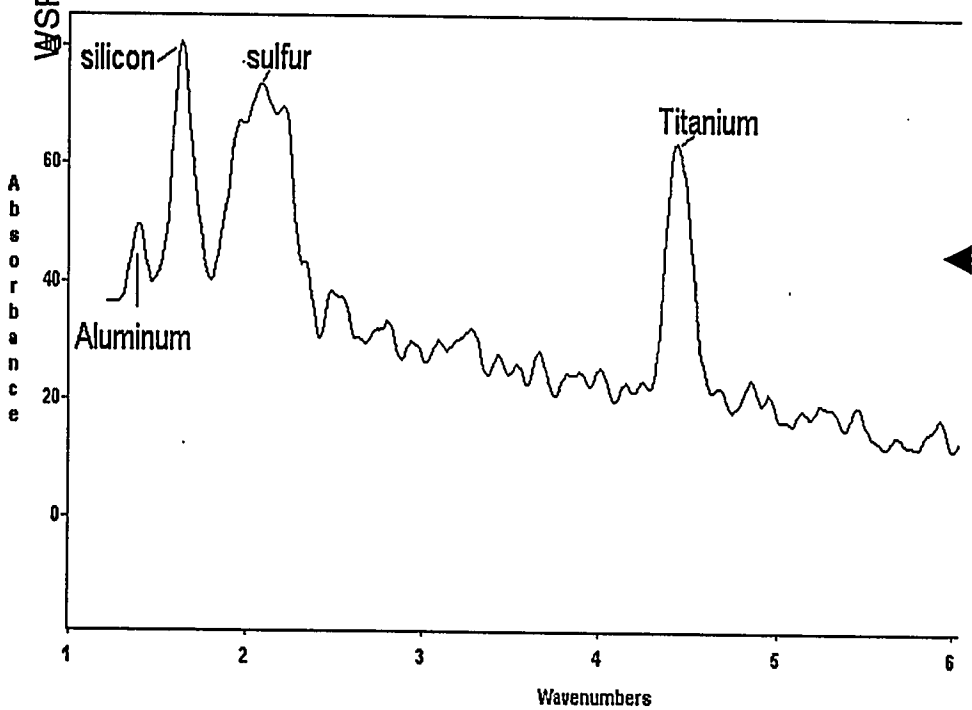


After Exposure to Organic Containing Salt Solution

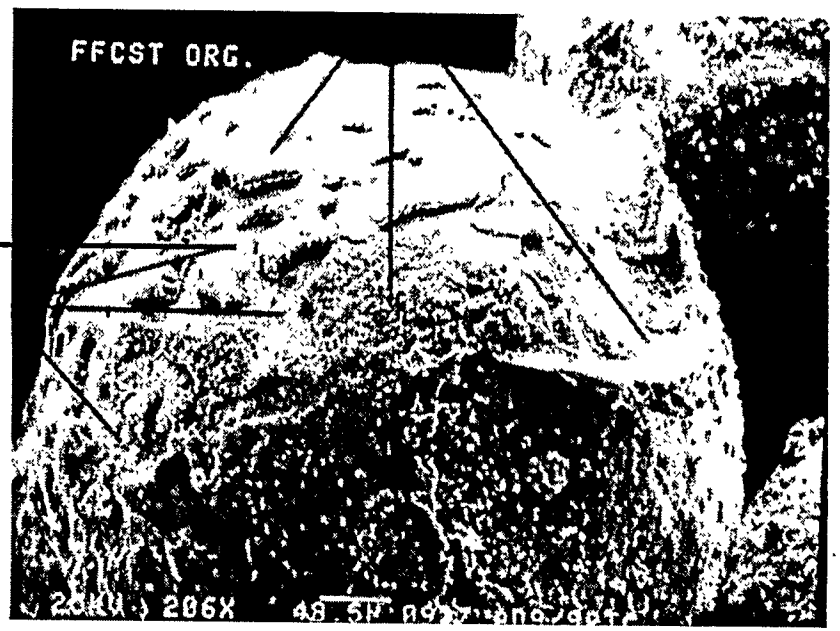


Figure 9. SEM picture of the surface of IE-911 after flowing 4000 bed volumes of salt solution and the corresponding elemental analysis showing aluminum sulfate precipiation.

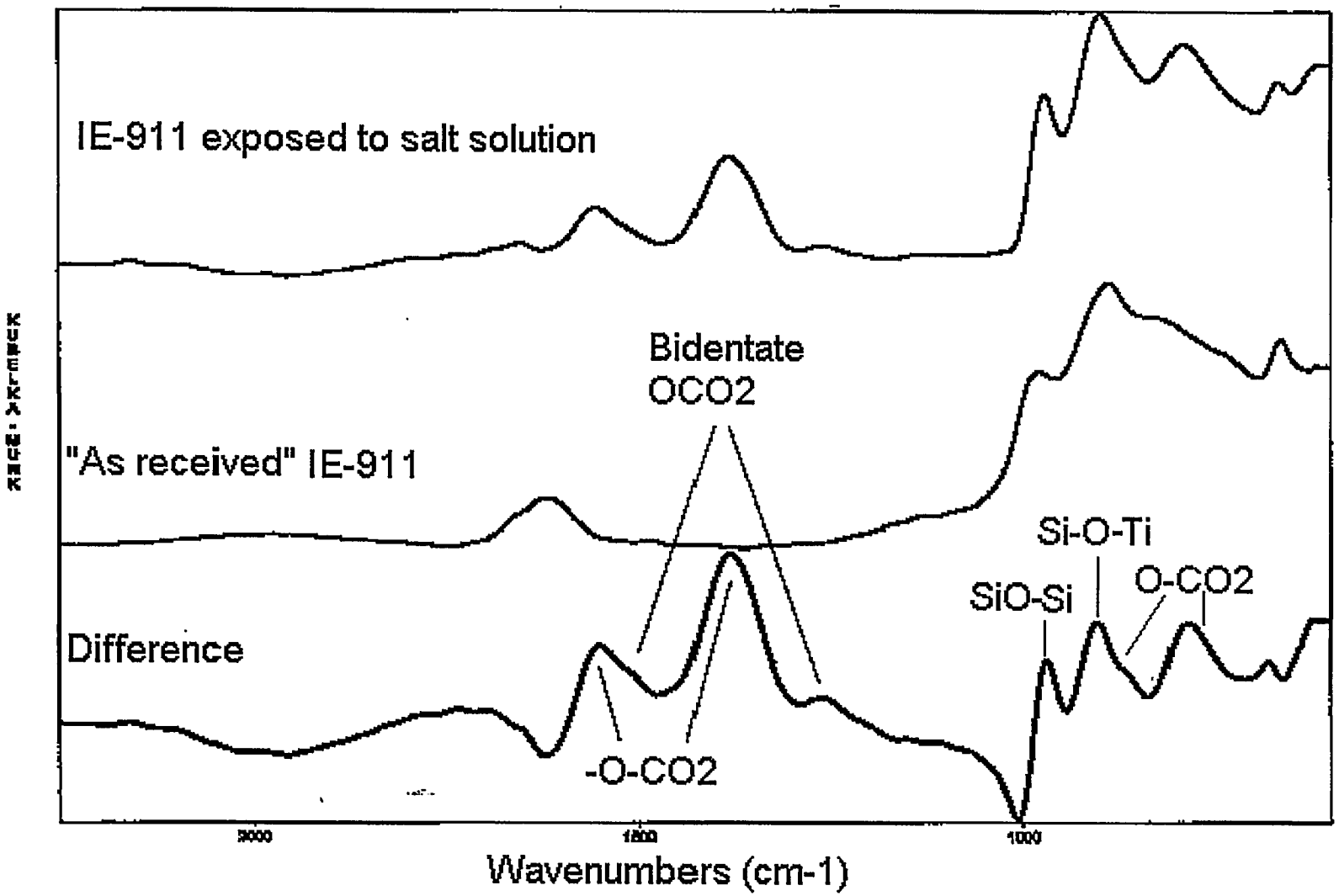


Figure 10. The infrared difference spectrum of IE-911 after flowing 4000 bed volumes of salt solution.

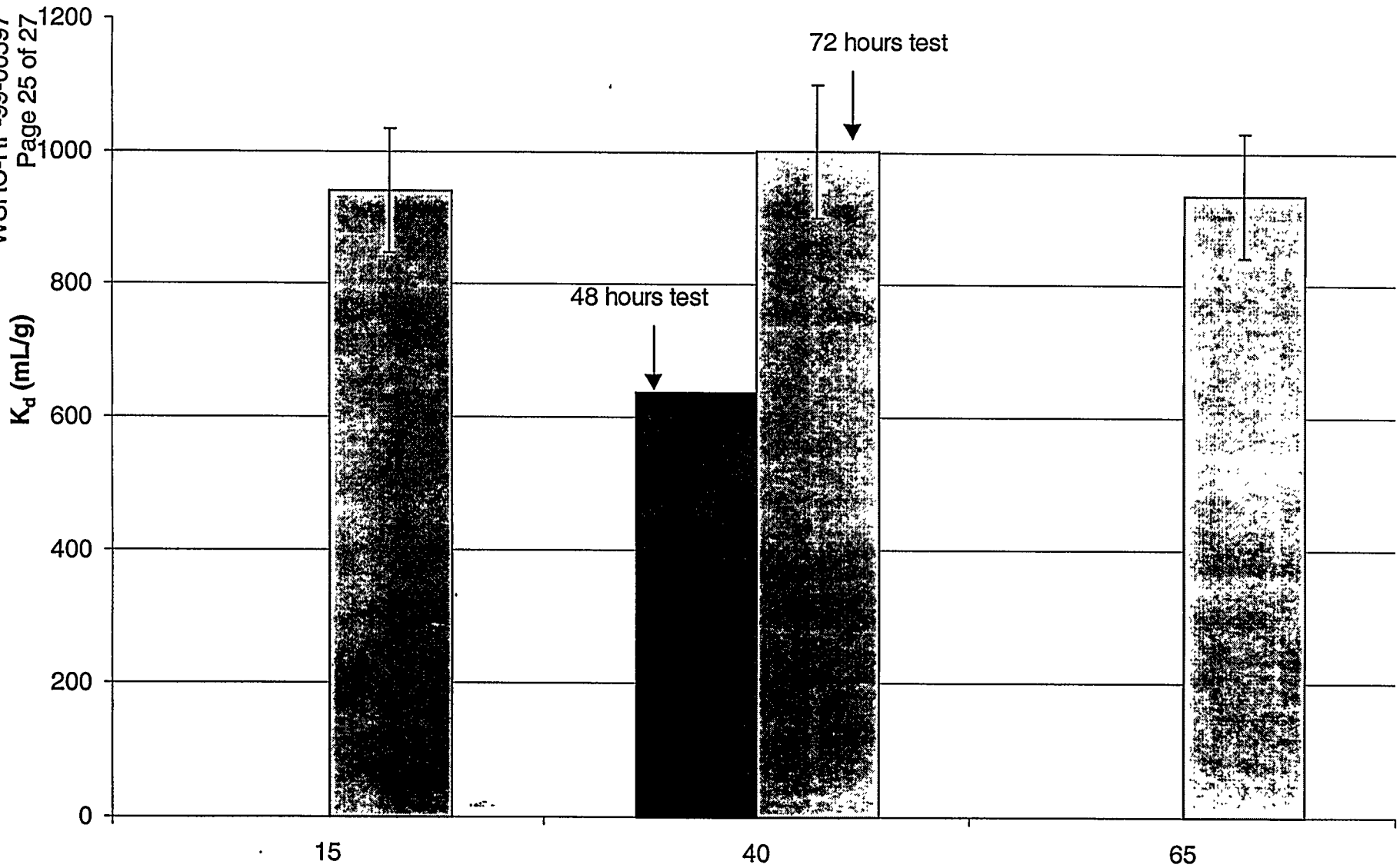


Figure 11. Pressure effects on cesium K_d values.

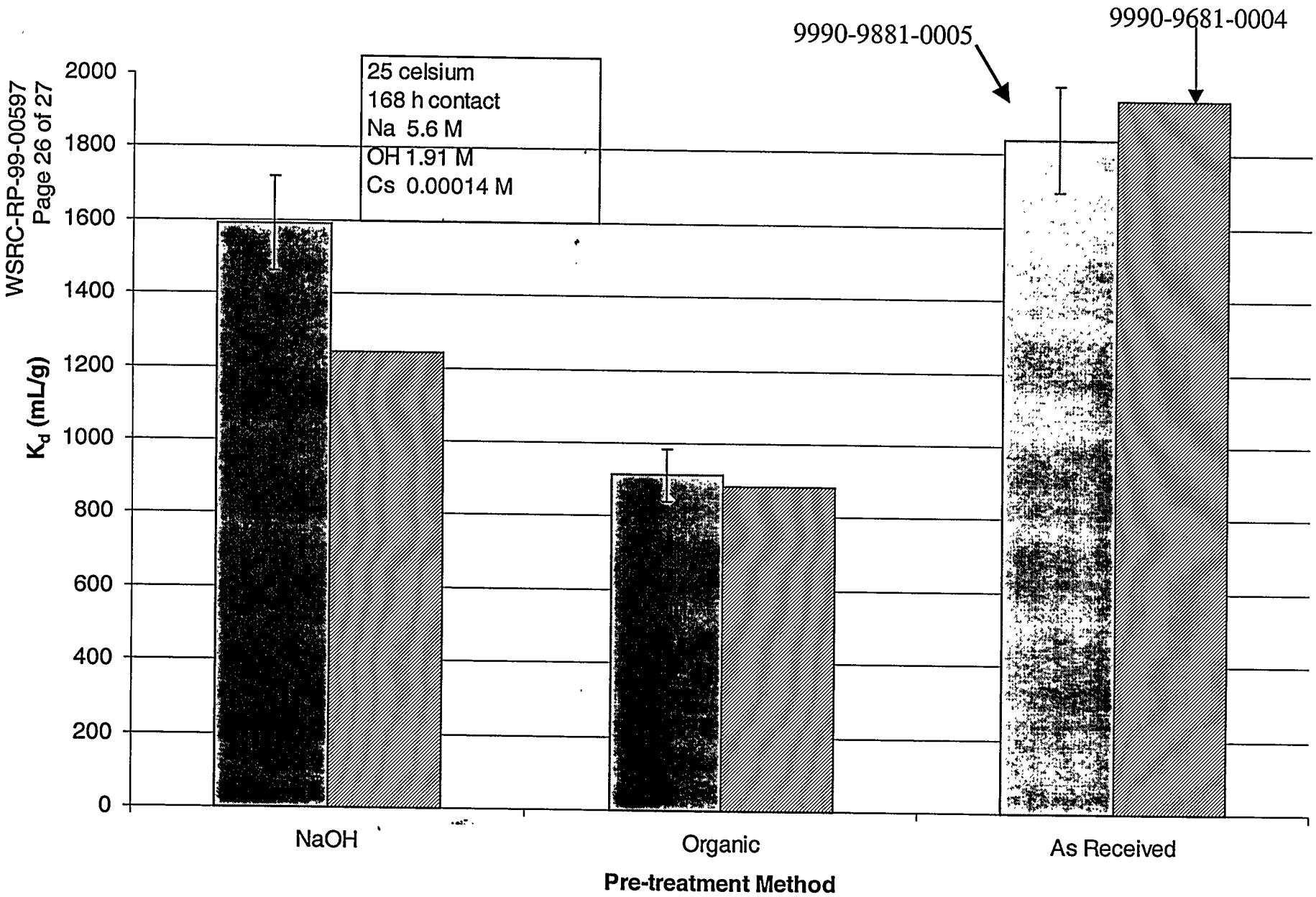
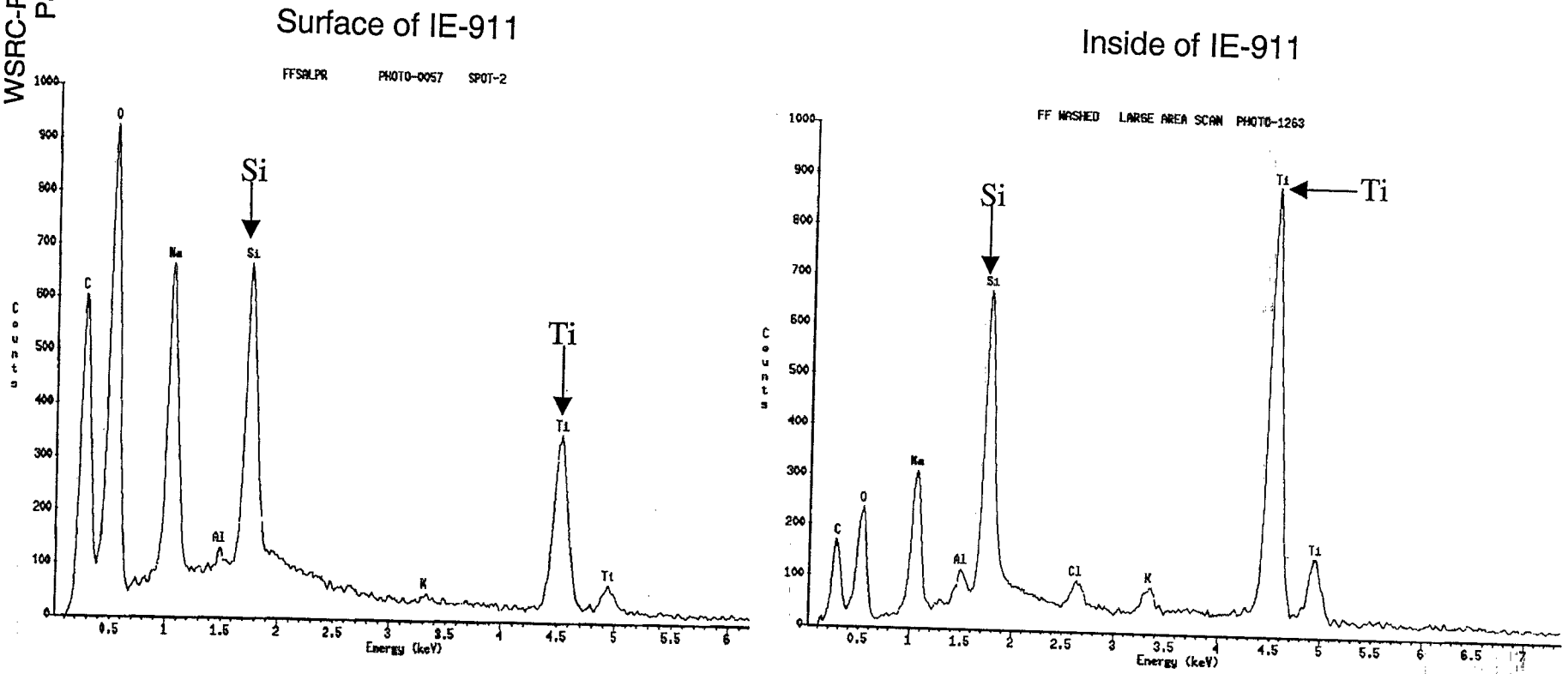


Figure 12. The combined effect of lot variability and pre-treatment on cesium K_d values.



* Original scan altered to protect identity of proprietary elements.

Figure 13. Elemental analysis of the surface (left) and inside (rigth) of IE-911 after pretreatment in salt solution.

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