

The Use of Chloral Hydrate as a Neutral Current Additive  
in the SNO Experiment -

a Chemistry and Engineering Study

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## §1 Introduction

The requirements on a neutral current additive to the heavy water in the SNO project are as follows: water solubility, low inherent radioactivity or the availability of an easy path of purification from thorium and radium, the possibility for easy separation from the water phase and safety. The solution of such an additive is expected to be transparent in the visible range, stable for extended (e.g. several months-years) periods of time, of relatively neutral pH, and of low reactivity with respect to acrylic, stainless steel and possibly polypropylene. A method of assaying the radium and thorium in such a solution should be available.

Sodium chloride, which is the default choice for neutral additive, presents a high cross section for the neutral current reaction, is highly soluble and forms a transparent and stable solution of neutral pH. Yet it still has to be demonstrated that it can be adequately purified, on a large scale, from contaminating radioactivity, in particular thorium. It is known that stainless steel may corrode in saline solutions, and that separation of NaCl from water is a multistage process and will involve heavy duty reverse osmosis systems, the membranes and pumps for which should all be free from contaminating and corrosion sensitive materials. Such a purification process will involve the mobilisation of large amounts of brine between the cavity and the surface. One other concern is the possibility of monitoring very small amounts of lead 212 (one of the final products in the thorium chain and a tracer of unsupported radon 224 in the system) when in saline solution.

It was suggested (B. Hollebhone, July 1991 collaboration meeting) that chloral

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hydrate may have several advantages over sodium chloride. Amongst these are the fact it is an organic molecule, and thus less likely to be incorporate radioactive impurities, its lower osmotic pressure (assumed, on basic principles, to be 1/6 of that for NaCl solution of the same weight concentration) would render it easier to be removed by a reverse osmosis system, and the organic nature of the molecule should insure lower degree of reactivity with metals in general and stainless steel in particular. Furthermore, lead assay in the presence of chloral hydrate will be relatively straight forward since both reverse osmosis and distillation will be applicable as separation techniques. It was thus deemed necessary to study the available information on chloral hydrate solutions and to assess their transparency, stability, interaction with acrylic, stainless steel and radium adsorbers (e.g.  $MnO_2$ ) and pH as well as the osmotic pressure and Reverse Osmosis (R.O.) performance in terms of chloral hydrate- water separation.

## §2 Literature study:

A comprehensive review on chloral hydrate (C.H.) was conducted by Fairbrother. The review includes the physical and chemical properties; degradation processes and their products, incompatible substances, analytical methods etc. Important data includes the molecular weight of C.H. which is 165.4 g, its boiling point is quoted to be  $96^\circ C$  ( $98^\circ C$  according to Merck 1976) and its solubility in water is extremely high: 6600 g/l at  $20^\circ C$ .

Synthesis is from chloral and water, where the chloral is synthesized from ethanol and chlorine, followed by treatment with sulfuric acid. This product may then be purified by recrystallization from an organic solvent, sublimation, or, at the chloral stage, by heating metachloral (a trimer of chloral) at  $180-200^\circ C$ .

Solid C.H. in the presence of excess oxygen degrades into phosgene,  $CO_2$  and HCl. The decomposition of chloral is faster than that of the hydrate. C.H. degrades in water to trichloroacetic acid (TCA), hydrochloric acid (HCl) and dichloroacetaldehyde. This process is accelerated by light and increased temperature, as well as by high- energy ultrasound. C.H. decomposition also takes place in basic solutions.

C.H. measurement is commonly achieved by use of the Fujiwara reaction (Ross, 1923) which is not entirely specific and identifies also other halocarbons such as TCA and chloroform. A more specific colour reaction is the one described by Archer and Haugas (1960, see next section).

The separation of water from the chloral hydrate molecule (to form chloral and water) may be achieved by azeotropic distillation in benzene (B.P. of constant boiling mixture-  $69-70^\circ C$ ) or hexane (B.P. of constant boiling mixture-  $63^\circ C$ , Mahoney, Linden and Pierson, 1946, U.S. patent no.2584036).

Regarding biological activity, aqueous solutions of chloral hydrate which are weakly acidic are prone to development of mold growth (Compendium of Pharma-

ceuticals and Specialties). Studies of the degradation of chloral hydrate in soils suggest the degradation is microorganism assisted, in conjunction with chemical and photochemical processes (Fairbrother).

#### Safety information:

According to the Compendium of Pharmaceuticals and Specialties C.H. affects the central nervous system and thus may be used, at low levels, as a sedative. The common adult dosage is 250 mg, 3 times a day. Children are administered 50mg/Kg body weight. It is reduced in the body into the apparently active form of trichloroethanol and is distributed throughout the body, so that it can be found in breast milk and across the placental barrier. High concentrations may result in stupor, low blood pressure, fall in body temperature, gastric irritation, collapse, delirium or death. Concentrated solutions applied to the skin may cause pain and tissue damage. According to the Sigma Safety Data Sheet (1991), acute effects will result if C.H. is swallowed, inhaled or absorbed through the skin. The material is destructive to tissue of the mucus membrane and upper respiratory tract as well as to the eyes and skin. It is a possible mutagen, where the target organ is the central nerve system.

#### Material compatibility:

C.H. is incompatible with the substances with which it/chloral form a complex (e.g. benzene), as well as with bases and hydroxyl group bearing compounds. It is also incompatible with permanganates (Fairbrother). According to Bosich (1970) use of C.H. with polyethylene and/or polypropylene is not recommended. Compatibility charts from George Fischer Signet Inc. (1992) similarly recommend not using C.H. with polyethylene or polypropylene, and neither with fluoroelastomers viton A.

### §3 Identification and measurement of C.H. and some of its degradation products:

For the measurement of C.H. we have employed a colorimetric method which allows the measurement of C.H. in the presence of its decomposition products. The method (Archer and Haugas, 1960) is based on the reaction between quinaldin ethiodide and C.H. in an alkaline solution to form a blue cyanine dye when heated to approximately 60°C. A blank run consisted of ultra pure water treated with the above reagents. Standards were prepared from freshly purchased Sigma reagent. All solutions and reagents were prepared and stored in glass vessels and the C.H. solutions were further protected from light by aluminum foil wrappings. The optical density at 606 nm was measured by a UV-Vis Perkin Elmer Lambda-48 spectrophotometer, operated at the range 550-650 nm. The color developed was found to be stable over a period of about 3 days. The light absorption at 606 nm was found to vary linearly with increasing C.H. concentration at the range of 0.3-10 mg/l. The error associated with this measurement was found to be about

4%.

C.H. degradation was assessed both by measuring changes in C.H. concentrations and by monitoring the  $\text{Cl}^-$  concentrations and the pH of the studied solutions.  $\text{Cl}^-$  was measured by an argentimetric titration (Vogel, 1981), where the chloride ions were titrated with silver nitrate, using a potassium chromate and potassium dichromate solution as an indicator. The error in this procedure is smaller than 5%, when employing 25 cc of sample and a 0.1M silver nitrate solution. In some of the measurements where only 5 cc of sample were used the error was 10%. The standard used in these measurements was a 0.1M sodium chloride solution.

pH values were determined using a Barnant 20 pH electrode.

#### §4 Transparency studies:

These studies employed the He-Cd laser and 2.5 meter trough used by Noel and Mes (SNO-STR-91-002), and the method of measurement was identical. Five solutions of different chloral hydrate concentrations were tested: 0.2, 0.5, 1, 2 and 3 weight %. All solutions were prepared using fresh SIGMA reagent and ultra pure water. A solution of a third of the desired volume and three times the desired concentration was prepared, and then mixed, in the trough, with the remaining 2/3 of ultrapure water. Final mixing was achieved by moving the reflecting mirror several times along the trough, a process which was repeated after several hours. At this stage the solution appeared to be homogenous. A reference run of ultrapure water was also performed as well as a blank run (empty trough). The results did not differ significantly from those for ultra pure water.

#### §5 $\text{MnO}_2$ compatibility studies:

Since  $\text{MnO}_2$  is the default adsorber for radium in the SNO monitoring and purifying setups, its compatibility with C.H. had to be tested. The material used was SeaStar  $\text{MnO}_2$  which is  $\text{MnO}_2$  on XAD-7 beads. Three modes of investigation were employed:

- 1) Evaluating the  $K_D$  for a  $\text{MnO}_2$  column leached by C.H. (high C.H.: $\text{MnO}_2$  ratio).
- 2) Comparing the level of C.H. before and after passing through a  $\text{MnO}_2$  column.
- 3) Analyzing the change in C.H. concentrations and in pH in  $\text{MnO}_2$  and C.H. mixtures after 24 hours shaking (low C.H.: $\text{MnO}_2$  ratio).

The first stage, involving the  $K_D$  assessment was conducted with a polycarbonate column containing 1.5 cc  $\text{MnO}_2$ . The leaching C.H. solutions were generally 20 litres at 1% concentration, except for one leaching cycle using 20 litres at 7% (in which case these were considered to be equivalent to 140 litres at 1%). Prior to

leaching the MnO<sub>2</sub> column was spiked with approximately 2ng of <sup>226</sup>Ra, left for 10 days till secular equilibrium with the <sup>222</sup>Rn daughter was established and then the initial spiked activity was measured with a gamma counter (GeLi detector) at 351.9 KeV. Leaching was conducted at approximately 7 cc/min (calculated to match the recommended residence time as measured by Ferraris, 1992). After ten days from the end of the leaching cycle the activity was remeasured. The values measured after several leaching cycles were plotted as a function of leaching volume in order to calculate an average K<sub>D</sub>. The results are included in figure 1 and in table 1 (assuming an adsorbant volume of 1.5 cc). When analyzing the K<sub>D</sub> value for each leaching interval (table 1) rather than the average K<sub>D</sub> value, the values seem to decrease with increasing leaching, suggesting a degradation of the solid MnO<sub>2</sub> phase.

When running the 7% C.H. leaching cycle, approximately 18 cc of the leaching solution and the eluate solution, respectively, were collected in dark vials and analyzed for their C.H, concentrations, as well as for the respective pH values and densities. The results are depicted in table 2. Both the C.H. concentration and the density or pH of the leaching C.H. solution did not change as a result of passing through the MnO<sub>2</sub> column,

In order to increase the MnO<sub>2</sub>:C.H. amount ratio at the time of interaction, 1.5 cc MnO<sub>2</sub> resin were added to 10 cc of C.H. (1% and 10% solutions) in dark vials, and placed in a reciprocal shaker for 24 hours. A MnO<sub>2</sub>+water sample was placed as a control as well as C.H. solution where no MnO<sub>2</sub> resin was added. The supernatant solutions were then analyzed for C.H. concentrations and pH. It is evident that C.H. concentration was reduced by as much as 40 % due to the C.H.-MnO<sub>2</sub> interaction. The MnO<sub>2</sub> has thus oxidized the C.H., while buffering the pH against the acidifying effect of the degradation products (table 3).

## §6 Chloral hydrate aging:

The effect of aging on chloral hydrate was assessed in two ways:

1. pH and conductivity of solutions made by using chloral hydrate from different distributors and of different shelf lives, at room temperatures, were monitored (table 4). In one case the effect of ultrasonication was also checked. No clear difference between C.H. of different origin (supplier, shelf life) was evident. Sonication seemed to have resulted in a slight decrease of pH.
2. Two sets of chloral hydrate solutions (sigma 1991) of 1% and 10% were prepared. The solutions were all kept in the dark, but one set was placed in the oven at 60°C while the other was left at room temperature. After 30 days. These conditions were considered to simulate aging of a 0.2% solution during 10 yrs of SNO experiment. All solutions were analyzed for pH, C.H. and Cl<sup>-</sup> concentrations (table 5). The heated samples seem to have the same concentration of C.H. as the non heated ones, but chloride and pH

analysis reveals signs of degradation.

It should be born in mind that C.H. degradation yields equal molar amounts of HCl, dichloroacetaldehyde and TCA, the last of which is highly corrosive, so that the mole amount of chloride measured should be multiplied by 3 to estimate the total molar amount of degradation products.

A similar experiment was conducted where the heated solutions contained acrylic samples. In this case only changes in pH were monitored (table 4).

### §7 Effect of C.H. on radium entrapment by 18-6 Crown Ether:

The  $K_D$  of C.E. 18-6, fused to silica powder, was assessed previously to be in the order of  $10^5$  (Shatkey and Shewchuk, 1992). In the present study a C.E. column with about 1.5 ng Ra 226 ( $9397 \pm 685$  counts/3 hrs) was subjected to leaching by 20 litres of a 1 % C.H. solution. The leftover Ra was measured to be  $2800 \pm 571$ , suggesting a  $K_D$  of  $5 \times 10^3$ . Thus C.H. affects in a negative manner the entrapment of Ra by C.E., possibly due to a complexing interaction between the C.H. molecule and the benzene ring attached to the C.E.

### §8 Reverse-Osmosis (R.O.) membrane separation study:

A series of studies was conducted in NRC, where 4 different R.O. membranes and 2 nanofiltration membranes were tested for the separation of C.H. The effect of C.H. on the membrane material was first assessed by immersing the membrane coupons (2 of each membrane) in a 10 % C.H. solution for 7 days, and performing a 3500 ppm NaCl separation test (at room temp., about 250 PSI) before and after this immersion period. The results (table 6, figure 2) do not indicate any effect of the C.H. on the membranes performance.

The study of membrane separation of 0.2 % and 2 % C.H. solutions was conducted by simultaneously passing approximately 8 litres of the studied solution across the membranes, using two coupons of each of the studied membranes. The permeate was collected while the reject was recycled back into the feed solution. The feed and the permeate were analyzed for C.H. using the spectrophotometric method described in section 3. Another NaCl (3500 ppm) run was then performed. The results for the NaCl separation following the C.H. separation runs are in table 7. The flow characteristics across the membranes are shown in figures 3 and 4. The C.H. separation results are in table 8. It is evident that C.H. separation is less efficient than that of NaCl, ( 5% vs. 1% respectively). No clear effect of the increased concentration (from 0.2 to 2 %) on the separation is indicated.

### §9 Chloral hydrate osmotic pressure:

The studies performed in NRC allowed the estimation of the osmotic pressure exerted by C.H. solutions of 0.2 and 2 %, from knowledge of the productivity flux rate of C.H. (PR, table 9), the applied pressure ( $\Delta P$ ) and the pure water flux rate (PWP, table 9). The approximate relation between the above, expressed in  $m^3/m^2/day$ , and the osmotic pressure ( $\Delta\pi$ ) in psi, is:

$$PR = PWP/\Delta P(\Delta P - \Delta\pi)$$

The osmotic pressure was estimated based on the data from the runs on the DS-3B and FT-30 membranes, and the results for 0.5% and 2.3% solutions are 30.3 psi and 136.5 psi respectively (see figure 5 and table 9). C.H. osmotic pressures are found to be similar to those exerted by methanol, are slightly higher than those of ethylene glycol and acetic acid and significantly higher than those of TCA of the same concentrations. Thus it seems that the alcohol group, rather than the molecular size and the chloride substituents, is the chief determining factor in the osmotic pressure of C.H. at these (low) concentrations.

NaCl of similar concentrations yields osmotic pressures of 45 and 264 psi respectively (Sourirajan, 1969). As suggested earlier, since the molecular weight of NaCl is a third that of chloral hydrate, and since the later does not ionize in solution while NaCl is fully dissociated into  $Na^+$  and  $Cl^-$  ions, the ratio of the respective osmotic pressure/g material for NaCl and C.H. would be expected to be 6:1 in a case where the osmotic pressure is solely a function of no. of dissolved molecules/ions. We find though that the ratio of osmotic pressure/g material between the two is only 2:1, so that a chemical effect on the osmotic pressure is apparent.

### §10 Mechanical testing of acrylic and polypropylene:

Samples of OP4 acrylic (UV transparent) and polypropylene were aged in water, 1% Chloral Hydrate 10% C.H., 1% NaCl and 10% NaCl (% weight) at 55°C for 21 days. Three types of samples were used in order to assess a) weight gain, b) tension, and c) crazing.

#### a) Weight Gain Results

Solutions of 1% and 10% solutions of both NaCl and C.H. were prepared using milli Q water and were placed in glass jars (four jars of each of the above solutions). A control consisting of milli Q water was also prepared. Bars of acrylic and polypropylene of dimensions  $11 \times 3.5 \times 5 \text{ cm}^3$  and  $11 \times 3.5 \times 0.6 \text{ cm}^3$  respectively were cut, washed with soap, wiped dry, air dried overnight and weighed. two-three bars of acrylic were placed in half of the jars and two-three bars of polypropylene were placed in the other half. the jars were then stoppered with a teflon lined cover.

and placed in an oven at 60°C for a period of one month. Following a month at elevated temperatures the bars were rinsed with ultrapure water, wiped dry and reweighed. The results are in table 10.

Acrylic

For all solutions but the 10% C.H., the increase in weight was below 1.7% of the original weight. Lower value for the NaCl 10% solution suggests an exclusion of salt from the acrylic, probably resulting in an osmotic pressure gradient buildup, thus hindering further absorption of water. For the C.H. 10% solutions, the average increase in weight is  $2.48 \pm 0.05$  %, above the critical 2% weight increase recommended by Bosich (1970) as the indicator for solvent corroding potential.

Polypropylene

No significant weight increase was observed for these samples.

b) Tension Test Results

The testing of the effect of tension was conducted at room temperature using a strain rate of 0.2 inches/minute. A 2" clip gauge was mounted to measure the strain.

Acrylic

Ultra pure water - These samples broke at 8400 - 9400 psi at a strain of 3.3 to 4.3%. No necking occurred and plastic deformation was limited to 5%.

10% C.H. - these samples had the same ultimate load as the water but there was a marked difference in the yield and plastic behaviour. All of the samples necked and experienced strains in excess of 20% before failure (in some cases the test were stopped to preserve a necked acrylic specimen). This material would be considered elastic-plastic.

1% C.H. - Results were similar to the results for 10% C.H., however not all samples necked before failure, though some specimens did not have the range of plastic deformation as the 10% C.H.

10% NaCl - These samples behaved the same as the water samples.

1% NaCl - These samples behaved the same as the water samples.

Polypropylene

All samples behaved in an identical manner and no samples broke. Necking did occur and the tests were stopped after a strain of 15% to 35%.

c) Crazeing

The crazeing test setup consisted of applying a tensile strain to the outside of two samples by bolting them together with a spacer in between them. The tensile stress was to be  $13.8 \text{ N/mm}^2$  (2000 psi) at the end of the test. The specimens were 20 mm X 120 mm with holes for bolting together, 100 mm centre to centre. The spacer was selected to take into account the creep that would occur due to the elevated temperature over a period of 40 days. This was based on information supplied by Rohm for plexiglass 233. The spacer thickness was 3.94 mm (0.155"). After 21 days a pair was removed and unbolted to measure the creep and estimate the stress in the sample. The residual stress was estimated to be  $3.1 \text{ N/mm}^2$  (450 psi). Only one sample in 10% C.H. had any evidence of crazeing, which was a single



craze of a few mm in length and less than 1 mm deep.

A new test was devised to establish a crazing limit curve as a function of applied stress and time at an elevated temperature. It is assumed that the elevated temperature will accelerate crazing especially for the lower stresses. This method was chosen because enough data can be collected from tests conducted over a few days to prepare a curve. The test consisted of subjecting a beam to three point bending while immersed in the solution of interest. The beam is to be conditioned in the solution prior to the test. A drawback of this test is that the tensile stress distribution through the sample is linear going from compression on one side to tension on the other. However this is the same as for the crazing tests conducted by J. Stachiw when investigating cleaning agents for acrylic. A better test may be to subject the entire sample to uniform tensile stress. The above described tension test specimens were examined for evidence of crazing and none was observed. yet it is possible that failure initiated at the first crazing site.

The first series of tests for crazing did not condition the samples properly. The samples were held in a bath of water at 70°C and then tested in the solution of interest at 45°C. Table 11 gives the time for crazing to initiate for water and a 10% C.H. solution. One should be cautious in the interpretation of these results because of the possibility of higher surface stresses in the 10% C.H. due to liquid absorption and temperature difference. The above tests were repeated with samples that have been conditioned at a temperature of 60°C for 3 days. These tests show similar crazing results for water and brine. It is interesting that the samples conditioned in C.H. did not craze. A possible explanation for this is that the C.H. acts to blunt or heal any cracks that are present on the surface of the acrylic so that they do not grow.

#### Effect of C.H. on optical properties of acrylic

Samples of acrylic which were subjected to accelerated aging in C.H. were to be sent to AECL in order to examine any change in their optical properties. However, these samples were kept out of the C.H. solutions for about a month before shipping, and at that stage extensive crazing of the acrylic bars was noticed. It is not clear whether this crazing would have occurred at 100% humidity (the conditions desired in the SNO cavity during the experiment).

#### Future tests

A) The situation where C.H. is first introduced to the acrylic vessel should be investigated. This would be done by conditioning acrylic samples in water and then conducting a crazing test in a C.H. solution. The same condition will be examined for brine.

B) The situation where stratified filling up of the acrylic vessel is taking place and there is a region of no C.H. and a region of high C.H. concentration should be considered. There is a concern that due to different absorption rates of the water and C.H. solution by the acrylic, stresses due to differential expansion could take place. An appropriated test would use a thin sheet of material with water on one

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side and two solutions on the other side separated by a barrier of some sort. It may be possible to simplify the test to one of two different solutions on either side and measure the deflection of the acrylic due to the different absorption rates.

C) A third test would simulate the condition of  $H_2O$  on one side and  $D_2O$  on the other side.

D) crazing tests should be conducted on slush cast acrylic material from RPT using samples conditioned in water and then submitted to a stress in the presence of a C.H. or brine solution.

E) Test D should be repeated using samples conditioned in test solution.

F) Mechanical strength test of slush cast material from RPT after being conditioned in water, C.H. and brine solutions should be carried out.

§11 Concluding remarks:

The studies performed so far indicate that C.H. is acceptable as a neutral current additive in terms of the transparency of its solutions (at 2 weight % or less) and the availability of a convenient method of assaying it.

Aging tests indicated that C.H. solutions are somewhat unstable as witnessed by the decrease in pH values and the increase in chloride concentrations in the aged solutions. Studies of MnO<sub>2</sub> beads exposed to C.H. indicated some deterioration of the beads as compared with similar studies with water and with NaCl solutions. An adverse effect on radium adsorption by MnO<sub>2</sub> was found, but the K<sub>D</sub> value for the C.H.-MnO<sub>2</sub>-Ra system was still in the acceptable order of 10<sup>5</sup>. Conversely, the effect of C.H. on radium adsorption by 18-6 crown ether fused with silica is more pronounced, lowering the K<sub>D</sub> to 10<sup>3</sup>.

C.H. separation by RO was found to be less efficient than that of NaCl of a similar weight concentration (the permeate weight concentrations for C.H. solutions of 2% and NaCl solutions of about 3% were found to be 5% and 1% of the feed solution concentration, respectively) even though the osmotic pressure generated by a given weight concentration of C.H. was extrapolated to be lower than that generated by the same weight concentration of NaCl.

Mechanical studies involving polypropylene did not reveal any effect of C.H. on the former. Similar studies with acrylic indicated interaction between C.H. and acrylic which might be of a beneficial nature (i.e. increase in the ductility of the acrylic) but is not yet completely understood.

The above suggest that while C.H. can not be ruled out as an option for a neutral current additive, further studies on the nature of its interaction with acrylic and radium adsorbers and on its concentration and separation by RO membranes should be conducted.

§12 References

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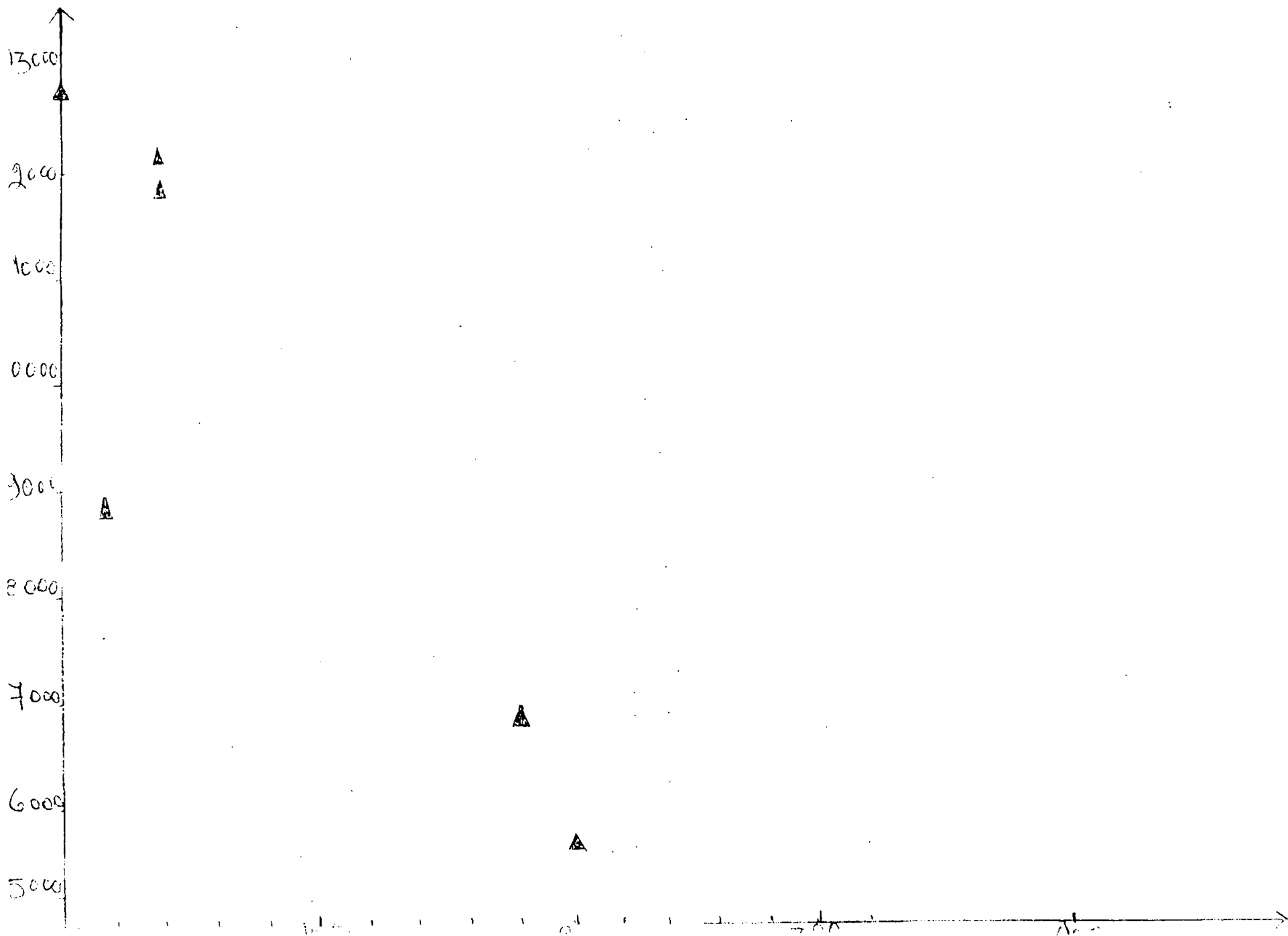
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<sup>226</sup>Ra counts/3hrs

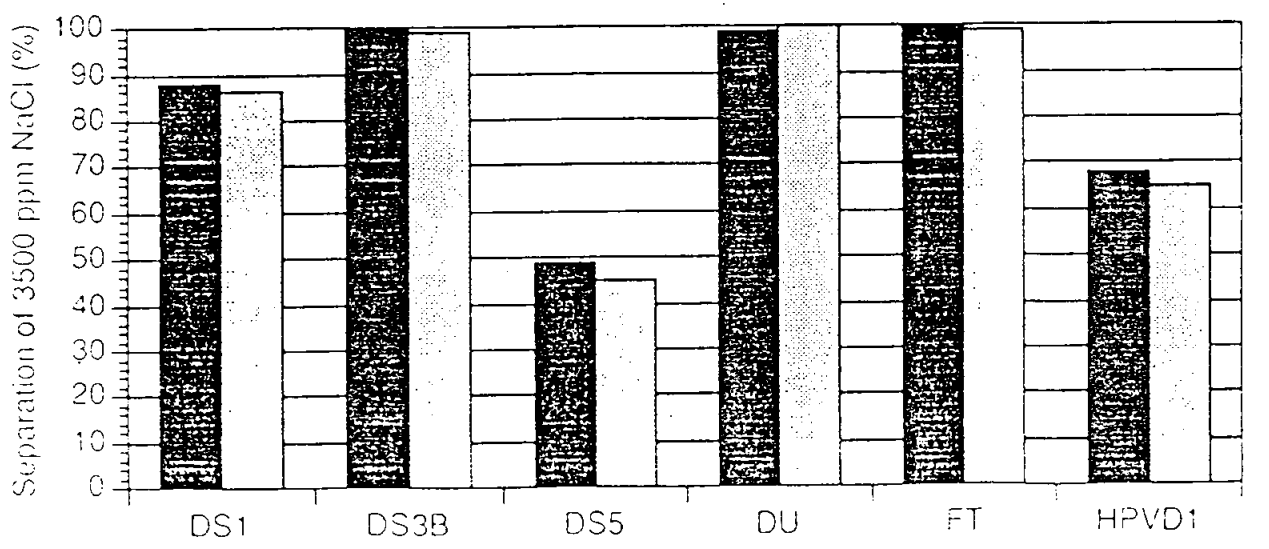
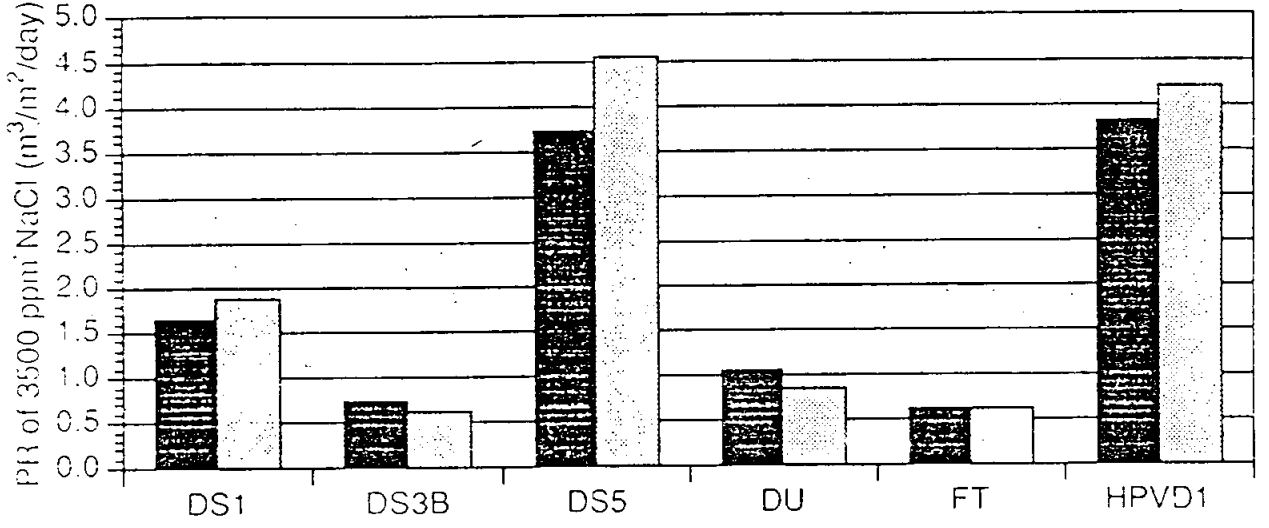
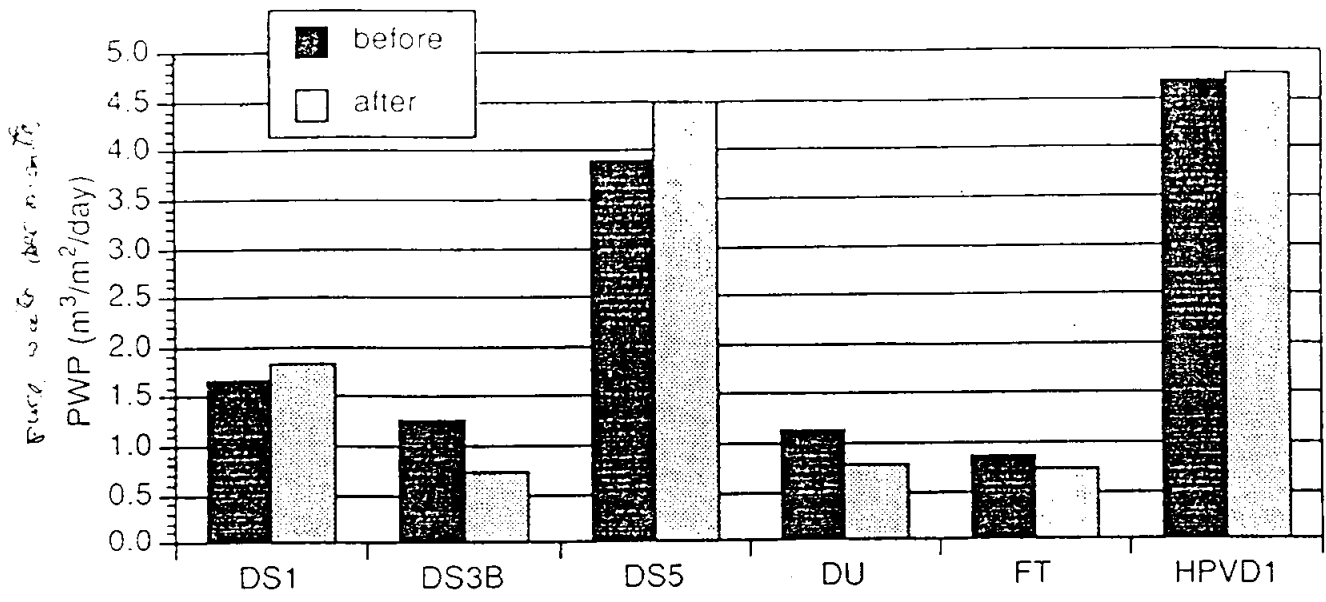
Figure 1



# Effect of Seven Days Exposure to a 10% Chloral Hydrate Solution

m.g. 2

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### Effect of 0.2% and 2% Chloral Hydrate Solutions on Product Rates of Membranes

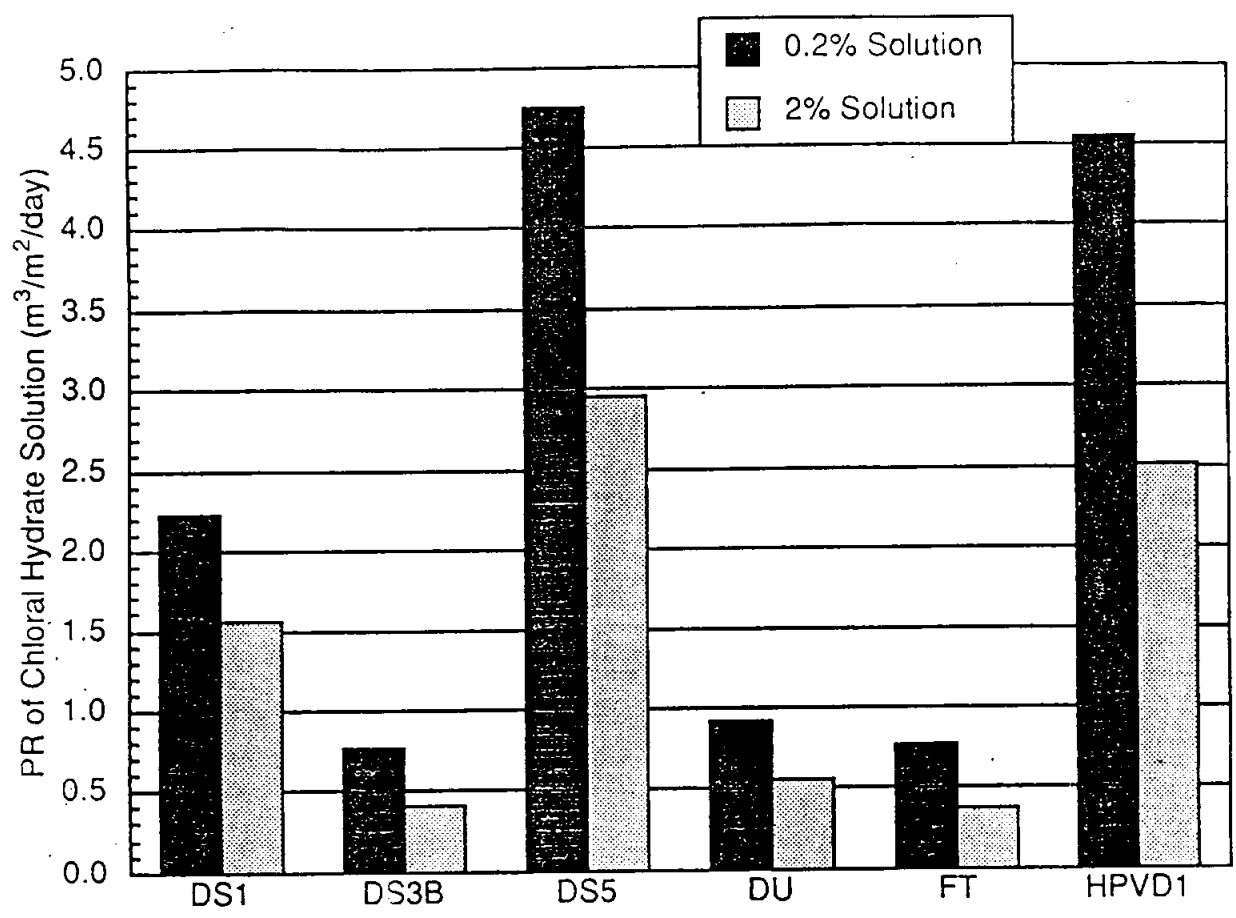
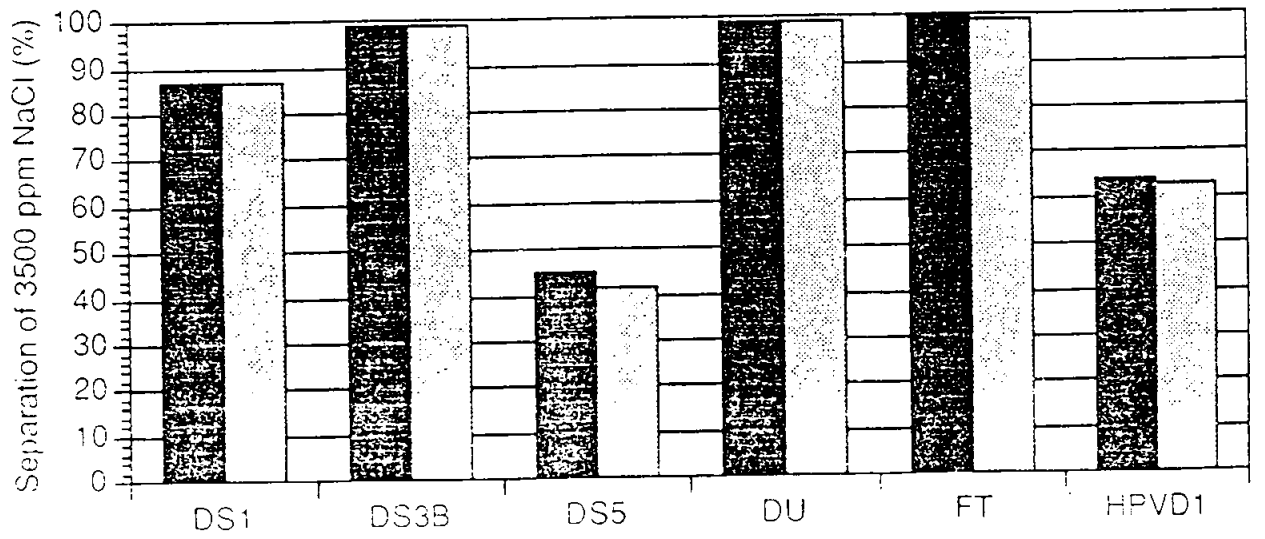
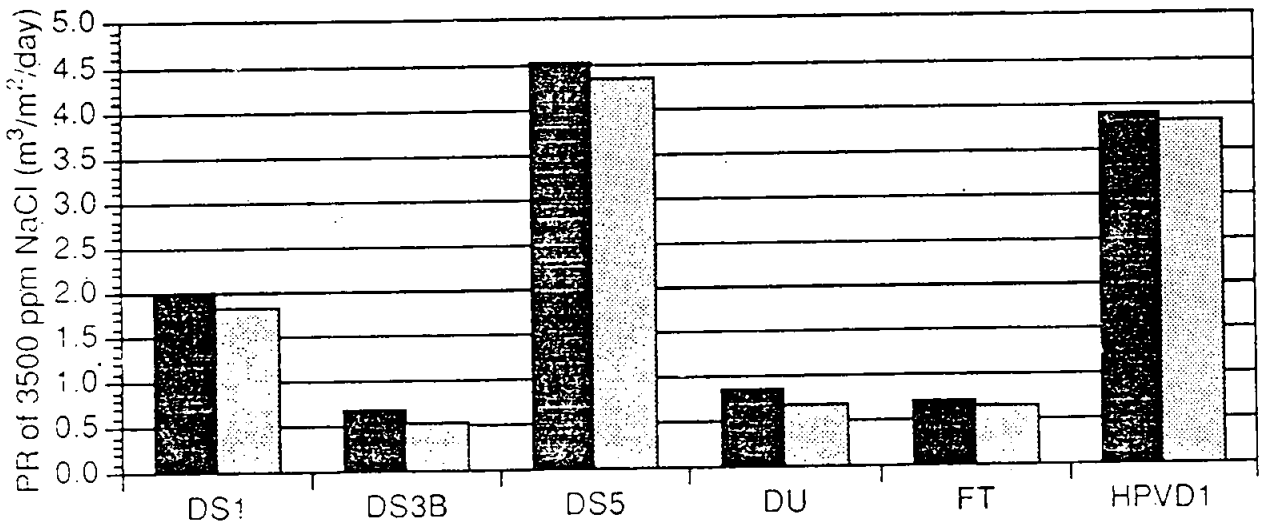
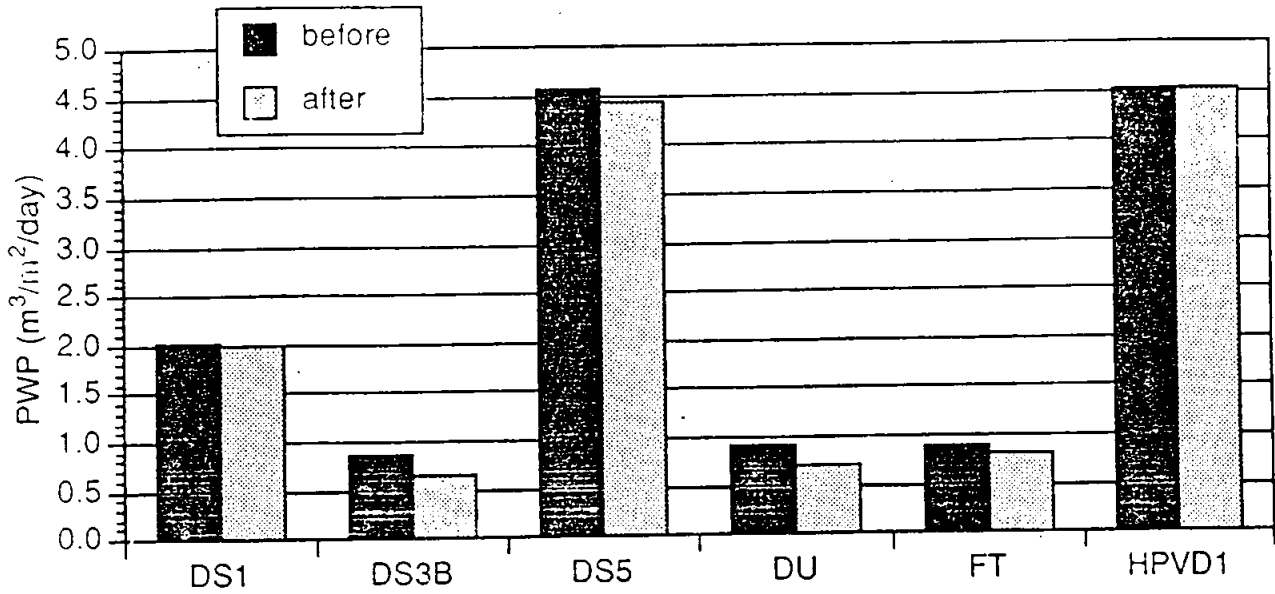


FIG. 3

### Effect of Exposure to a 0.2% and 2% Chloral Hydrate Solution Test Run





## Amount of leaching (litres)

	0	22 (1%)	20 (1%)	20 (7%)	20 (1%)	19 (1%)
a 226 Counts/ 3 hrs)	12675 pm310	8490 pm275	11648 pm320	6684 pm584	5105 pm549	4322 pm593

%) : weight % of chloral hydrate solution

m : plus/minus

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	C.H. concentration (%)	pH	density g/cc)
before MnO2 column	7 pm 0.5	3.86 pm 0.1	1.028
after MnO2 column	7 pm 0.5	3.95 pm 0.1	1.029

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pm): weight % of chloral hydrate solution

pm : plus/minus

	C.H. (mg/cc)	pH
MnO <sub>2</sub> +1% C.H. (*)	7.4 pm 0.4	7.2 pm 0.1
1% C.H.	12.8 pm 0.6	3.7 pm 0.1
MnO <sub>2</sub> +H <sub>2</sub> O	/	9.0 pm 0.1

(\*) : All samples were shaken for 24 hours on a reciprocal shaker.  
1% samples are prepared with an error in concentration of  
0.1 %.

pm : plus/minus

C.H. sample	pH (25 C)	conductivity (microsimens) (25 C)
50% Sigma, 2 months after preparation	1.8 pm 0.1	849
10% Sigma, 5hrs sonication, 2 months after preparation, (26.11.91)	2.6 pm 0.1	615
10% Sigma, (23.12.91)	2.8 pm 0.1	156
10% Sigma, 28 days at 60 C with acrylic, (23.12.91)	2.4 pm 0.1	
10% Sigma, (24.2.92)	3.0 pm 0.1	
10% Sigma, 30 days at 60 C, (25.3.92)	2.7 pm 0.1	
10% Fisher, 3 yrs old	4.1 pm 0.1	
1 % Sigma, (23.12.91)	3.8 pm 0.1	
1 % Sigma, 28 days at 60 C with acrylic, (23.12.91)	3.3 pm 0.1	
1 % Sigma, (24.2.92)	4.1 pm 0.1	
1 % Sigma, 30 days at 60 C, (25.3.92)	3.2 pm 0.1	
0.1% Sigma, (5.12.91)	5.3 pm 0.1	4.8

pm : plus/minus

pH measured by a Brenant pH electrode.

	C.H. (mg/cc)	pH	Chloride (mg/l)
8% C.H.	10.6 pm 0.5	4.12 pm 0.1	0 pm 8.7
10% C.H.	90. pm 4.5	3.04 pm 0.1	0 pm 8.7
8% C.H., 60 C 30 days	10.8 pm 0.5	3.18 pm 0.1	39.2 pm 8.7
10% C.H., 60 C 30 days	88. pm 4.4	2.69 pm 0.1	93.9 pm 8.7

pm : plus/minus

22

January 1992

Coupons tested in system with a pressure of 250 psig, flow rate of 1.4 GPM, and a temperature of 20°C to 28°C.

All rates in m3/m2/day.

Membrane	Supplier	Type	Nominal NaCl Separation (5000 ppm)	Before			After		
				PWP	3500 ppm NaCl		PWP	3500 ppm NaCl	
					pr	sep		pr	sep
DS1-12	Desalination Systems Inc.	HF, thin-film composite	97-99%	1.92	1.85	76%	2.23	2.20	72%
DS1-20				1.42	1.41	100%	1.39	1.52	99%
DS3B-4	Desalination Systems Inc.	HF, thin-film composite	98.5%	1.32	0.78	99%	0.79	0.68	99%
DS3B-13				1.22	0.70	100%	0.66	0.58	99%
DS5-19	Desalination Systems Inc.	NF, thin-film composite	50-70%	3.84	3.69	51%	4.40	4.47	46%
DS5-27				3.97	3.77	47%	4.57	4.60	43%
DU-30	Dupont	HF, Amamide	95%	1.11	1.03	99%	0.78	0.83	99%
DU-33				1.18	1.13	99%	0.76	0.86	100%
FT-39	Filmtech Corp.	HF, thin-film composite	99%	0.84	0.60	100%	0.73	0.61	99%
FT-45				0.89	0.61	99%	0.71	0.60	99%
HPVD1-1	Hydranautics	NF, Polyvinyl alcohol derivative	92%	4.32	3.67	80%	4.52	4.09	77%
HPVD1-8				5.05	3.96	56%	4.97	4.30	53%

same

no

Over-saturation requirements  
(m<sup>3</sup>/m<sup>2</sup> day)

Over-saturation requirements

February 1992

Coupons tested in system with a pressure of 250 psig, flow rate of 1.4 GPM, and a temperature of 22°C to 24°C.

All rates in m<sup>3</sup>/m<sup>2</sup>/day.

Membrane	Supplier	Type	Nominal NaCl Separation (5000 ppm)	Before			After		
				PWP	3500 ppm NaCl		PWP	3500 ppm NaCl	
					pr	sep		pr	sep
DS1-12	Desalination Systems Inc.	HF, thin-film composite	97-99%	2.47	2.32	75%	2.45	2.15	75%
DS1-20				1.56	1.61	99%	1.54	1.47	99%
DS3B-4	Desalination Systems Inc.	HF, thin-film composite	98.5%	0.93	0.76	99%	0.73	0.60	99%
DS3B-13				0.78	0.63	99%	0.56	0.46	99%
DS5-19	Desalination Systems Inc.	NF, thin-film composite	50-70%	4.57	4.53	44%	4.44	4.32	41%
DS5-27				4.57	4.53	46%	4.44	4.34	42%
DU-30	Dupont	HF, Amamide	95%	0.93	0.86	99%	0.66	0.70	99%
DU-33				0.93	0.88	99%	0.76	0.66	99%
FT-39	Filmtech Corp.	HF, thin-film composite	99%	0.91	0.71	100%	0.81	0.65	99%
FT-45				0.86	0.71	99%	0.79	0.65	99%
HPVD1-1	Hydranautics	NF, Polyvinyl alcohol derivative	92%	4.52	3.86	76%	4.72	3.91	73%
HPVD1-8				4.50	3.92	52%	4.32	3.69	53%

Table 8

Membrane Performance for chloral hydrate

Membrane	0.2% feed			2.0% feed		
	Perm conc.	% of feed	% reject	Perm conc.	% of feed	% reject
DS5-27	1350	67.5	32.5	12000	60	40
DS5-19	1375	68.8	31.2	13000	63	35
HPVD1-8	850	42.5	57.5	9700	48.5	51.5
HPVD1-1	490	24.5	75.5	25000	100	0
DS1-10	160	8	92	1310	6.6	93.4
DS1-12	590	29.5	70.5	5700	28.5	71.5
DS3-1	91	4.6	95.4	1310	6.6	93.4
DS3-4	122	6.1	93.9	1070	5.4	94.6
DU30	177	8.9	91.1	1210	6	94
DU33	198	9.9	90.1	1740	8.7	91.3
FT45	111	5.6	94.4	1310	6.5	93.5
FT39	96.5	4.8	95.2	1170	5.8	94.2



25

Table 9

Membrane	PWP (m <sup>3</sup> /m <sup>2</sup> /day)	PR (m <sup>3</sup> /m <sup>2</sup> /day) for 0.2 % Chloral Hydrate	PR/PWP	$\Delta\pi$ (psi)	PR (m <sup>3</sup> /m <sup>2</sup> /day) for 2.0 % Chloral Hydrate	PR/PWP	$\Delta\pi$ (psi)
Desalination DS-1	2.47	2.78	1.13	-31.38	1.97	0.80	50.61
	1.56	1.69	1.08	-20.83	1.14	0.73	67.31
Desalination DS-3B	0.93	0.84	0.90	24.19	0.46	0.49	126.34
	0.78	0.68	0.87	32.05	0.36	0.46	134.62
Dupont B-10	0.93	0.91	0.98	5.38	0.56	0.60	99.46
	0.93	0.91	0.98	5.38	0.55	0.59	102.15
Filmtec FT-30	0.91	0.78	0.86	35.71	0.38	0.42	145.60
	0.86	0.76	0.88	29.07	0.38	0.44	139.53
Average for DS-3B, FT-30				30.26		0.31	136.52
				0.51	30.26		
				2.31	136.52		

Solution	Weight gain in %			
	Acrylic		Polypropylene	
milli Q water	1.71 pm 0.05	n=7	le 0.01	n=2
NaCl 1%	1.64 pm 0.06	n=6	"	n=5
NaCl 10%	1.35 pm 0.04	n=5		
C.H. 1%	1.61 pm 0.06	n=6	"	n=5
C.H. 10%	2.48 pm 0.05	n=6	"	n=7

pm: plus/minus

le: less or equal

n : number of replicates

Table 11

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stress psi)	Time to observe crazing (sec.)	
	water	10% C.H.
500	1-6	1
600	130-863	5-37
600	1815-3060	10-19

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