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DECHLORINATION

OF POLYVINYLCHLORIDE

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DECHLORINATION
OF POLYVINYLCHLORIDE

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CANMET
Energy Research Laboratories
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Nepean, Ontario

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August 24, 1992



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INTRODUCTION:

The purpose of these experiments is to determine the optimum conditions for chlorine removal in polyvinylchloride (PVC). The conditions examined were experimental temperature, heating time and particle size of the PVC. A few modifications to the experimental procedure were also examined.

Dechlorination of PVC is an important process since the dechlorinated product can be a useful source of hydrocarbons for further processing in a refinery. A dechlorination process is necessary since during the decomposition of the PVC any chlorine left in the plastic is released as hydrochloric acid which is very corrosive and damages the refinery equipment. It is also necessary to keep the dechlorination process simple so that it can be used with all the different types of plastics without causing them to decompose. This will help to keep the handling cost low as sorting of the different plastics will not be necessary.

PROCEDURE:

The glassware set up is as seen in Figure 1. It is important to note that the position of the thermocouple has a very large effect on the temperature reading. Therefore it should be placed in the same position each time, and as close to the bottom of the flask and the sample as possible. Before this system is set up the weight of all the glassware including the glass junctions is recorded. Once the system is set up, the 10 g of polyvinylchloride is added to the three armed flask (the Reaction Flask). The Reaction Flask is secured to the retort stand and then wrapped in glass wool up to the branch going to the Trap Flask. Elastics are used to secure all the glass junctions because the build up in pressure can break these seals. The second flask is the Trap Flask; some of the heavier vapours settle here. 400ml of a 1.0 N sodium hydroxide solution is added to the third 500ml flask. This scrubbing solution collects most of the inorganic chlorine as it passes through in a gaseous form. The last two condensers are used to condense any remaining vapours. The first condenser is placed in a beaker of ice water and the second in a mixture of dry ice and acetone. It is important to dry these condensers thoroughly, since any remaining water will freeze and plug the vapours exit route, causing a back flow of the scrubbing solution. The glassware can be dried by rinsing it with acetone and using compressed air to evaporate off the acetone.

Once the system is set up and secure, the purge gas, nitrogen,

is ran through at a very slow rate (< 0.1 L/min). Then by plugging the exit tube any leaks can be detected. If the rotameter reading drops to zero then there are no leaks in the system. If there is a leak check all the connections and try again. This also ensures that nitrogen, which is inert, is the only gas present in the reaction flask.

The reaction flask is heated until its close to the desired temperature using the UDS 2000 set with Gain-1000, Rate T-0, I Min-2 and Cycle-10. When the temperature is about 30°C lower than the set point, the tuning parameters are changed to Gain-20, Rate T-2, I Min-4.5 and Cycle-5. The temperature at which these settings are changed also depends on the desired final temperature, so this generalization might not work for all temperatures. This still appears to be the best heating method, however, as other methods have been tried. The desired outcome is a rapid heating rate (under 10 min ideally) and a steady end point with little fluctuation ($< \pm 2^{\circ}\text{C}$).

Once the reaction flask has been held at this temperature for the desired length of time the heating mantle is turned off and the nitrogen gas turned on at a rate of 0.2 L/min. After 1 minute the heating mantle is lowered and the glass wool removed from the reaction flask. Then the reaction flask is cooled with compressed air until it reaches 100°C . Once the flask has cooled to room temperature the nitrogen is turned off and all the glass pieces are weighed again before analysis of the fractions. During the systems cool down a gas volume reduction, caused by the decrease in

temperature occurs which leads to a pressure drop. Therefore, it is important to disconnect the glass junctions when the nitrogen is turned off, so that the drop in pressure, does not cause a back flow and contaminate the trap material.

At the conclusion of this reaction there is a black, porous solid present in the reaction flask. There are often a few crystals at the top of the reaction flask. These crystals are long and needle like with a faint yellow colour. There are also lots of crystals present in the glass junctions. These crystals are also located in the trap flask but are not found any farther down in the set up. The trap flask also collects a dark brown oily, organic material. During an experiment using PVC chips this brown material turns white due to a suspended powder, when water is added. The scrubbing solution usually remains colourless. A slight yellow tint is apparent in the scrubbing solution when the reaction is carried out at 400°C. This is probably due to the decomposition of the plastic that occurs at this temperature. Usually the condensate found in the condensers, if it is visible at all, is only a few drops of clear liquid at the bottom of the second condensor. Each of these products is analyzed for chlorine content.

To collect the chlorine in the condensers they are rinsed, twice, with distilled water and the rinsings are transferred to a 50 ml flask. 5 ml of a 1.0 N NaOH solution is added to the flask and then this solution is back titrated with 0.5 N HCl to determine the Cl content.

The glass junctions can be rinsed with a gentle stream of

distilled water into the trap flask. This collects the water soluble Cl but does not dislodge the crystals present in the junctions. These crystals can then be collected using acetone. The acetone evaporates off leaving the washed crystals.

The trap flask is rinsed with distilled water and the rinsings are transferred to a separatory funnel. Then the flask is rinsed with hexane and this rinsing added to the separatory funnel. The lower aqueous phase is drained into a flask. The organic phase washed once more and again the aqueous phase is added to the flask. Then this aqueous solution is back titrated utilizing the same procedure used for the condensers. In the case of the PVC chips the aqueous phase is centrifuged before the titration is carried out to remove the white powder that forms in this phase. The organic phase is collected in a round bottom flask and then evaporated with the rotoevaporater to remove the hexane. Then the remaining organic matter is transferred to a sample vial.

To analyze the scrubbing solution, first the glass junction entering the scrubber is rinsed using some of the solution from the flask. For this a pasteur pipette is used and the rinsings added back into the scrubbing flask. This junction should not be rinsed into the trap flask as some of the NaOH present would wash off also. Then 5 ml samples of the scrubbing solution can be titrated directly with the 0.5 N HCl.

The residue in the reaction flask is collected by breaking it into smaller pieces if it is brittle, or by pulling it through the opening with pliers if it is rubbery.

After these analyses are completed the rest of the solution is discarded and the glass set up is washed with water and finally acetone. The reaction flask may need scraping or even firing in a blast furnace to remove the rest of the residue.

RESULTS AND DISCUSSIONS:

The data gathered from these experiments shows distinct trends in the chlorine loss that occurs. There is still a problem of confirming if the value of 25.87% Cl by weight, is the true value of the amount of chlorine in the PVC powder. This value was determined by the Oxygen Bomb Combustion Method. The PVC chips have been determined, by the same method, to contain 27.2% chlorine.

The mass loss from the PVC chips and powder can be accounted for by the trap material, the crystals and the chlorine loss. The distribution of this mass loss can be seen in Table 2. The mass loss accounted for by the trap and crystals is usually around 30-40%. These products are formed due to the additives and stabilizers present in the PVC. The remaining mass is lost as hydrochloric acid.

From Figure 2 it is apparent that at 400°C, 5 minutes is sufficient time to release all the chlorine present in the PVC powder (assuming this value of 25.87% to be correct). The PVC chips still released slightly more chlorine the longer the time interval that they were at 400°C. Both the PVC powder and the chips released more chlorine the longer the time interval that they were held at 270°C.

Comparing Figures 2 and 3 it is also apparent that the PVC chips, under the same conditions as the powder, release less chlorine than the powder. The difference between the chip's and the powder's chlorine loss is not quite as large as it seems since the

thermocouple during the first set of powder runs was too high, and therefore the actual temperature of the bottom of the flask was closer to 300-330°C. Runs 4B and 4C can more accurately be compared to run 18 to give an indication of the difference in chlorine loss. The chips released 13.27% of their mass as chlorine whereas the powder released about 20% of its mass as chlorine. This fact could be a result of the different shape and size of the chips compared to the powder but it could also be due to the slightly different composition of the chips. It was clear that the chips and the powder had different compositions, since the aqueous trap phase, in the case of the chips formed a white powder upon the addition of distilled water. The powder's aqueous trap phase remained clear when distilled water was added. Also during the runs using PVC chips, the chlorine settled sooner in the apparatus, therefore more chlorine was found in the glass junctions and the trap flask.

From these observations it was determined that a flask temperature of 270°C is sufficient to release. At a flask temperature of 400°C the chlorine is released more quickly. The problem with running the reaction at 400°C is that more decomposition of both the powder and the chips occur. This can be seen as the dramatic increase in weight loss in both Figures 4 and 5 when the temperature is raised to 400°C.

The heating time of the PVC can also explain the phenomenon of less chlorine being released from the chips than the powder under similar conditions. Since the chip pieces are larger, it is understandable that they would take longer to heat up due to the

less surface area in contact with the heat source. There is another explanation for the lower release of chlorine from the chips. After HCl cleavage the PVC is left with alternating double bonds. These double bonds can cross link and cause polymerization with adjacent PVC polymers. Therefore, the chlorine's path can be further hindered by this polymerization which forms a barrier that traps these vapours. This effect is more of a problem with the PVC chips than the powder since the chlorine gas has farther to travel in the PVC chips.

Runs 14, 14B and 14C were performed to observe the effect of adding Cold Lake vacuum bottoms to the PVC chips. It was observed that this addition might increase the release of the chlorine from the PVC as values of 19.14%, 20.69% and 13.37% were obtained for runs at 300°C for 15 min. These first two values are similar to the values obtained at 400°C for 15 min without any Cold Lake vacuum bottom added. These experiments were also performed to determine if the presence of Cold Lake vacuum bottoms would cause any secondary reactions resulting in the production of noxious chlorinated compounds. This would be observed as any decrease in the amount of chlorine released as HCl. The opposite effect was observed indicating that the Cold Lake vacuum bottoms do not interfere with the usual reaction.

The pH experiment was performed to see if monitoring the change in pH of the scrubbing solution could be used to follow the release of chlorine from the PVC. The scrubbing solution used was distilled water and the drop in pH is directly related to the

increase in chlorine in the scrubbing solution. Figures 6 and 7 show these trends. This method shows some promise as all the rinsings could be added to the scrubbing solution once their volume was taken and then a simple calculation would indicate the amount of chlorine present. This simplifies the dechlorination reaction as the many titrations are no longer necessary. A strip chart can also be used to plot the change in pH (representing chlorine loss) against time to give a further understanding of when the chlorine is lost. Figures 6 and 7 show that most of the chlorine loss occurs within the first 3 to 10 minutes. Then the rate of release slows down and tapers off. These figures also show that the nitrogen does in fact clear out, more rapidly, some off the chlorine gas in the reactor.

There seems to be at the best a '2% margin of error in these experiments. There are many factors that need to be carefully controlled so that the much larger deviations in results are kept to a minimum. Some of the factors that cause these deviations are: pressure build up, non uniform heating rates and non uniform cooling rates. Throughout these experiments most of the deviations can be explained. Any differences in the experimental runs are summarized in Table 5. Although some modifications were made throughout these experiments they were almost always made at the end of a block with similar conditions and thus the results can still be used for comparisons.

CONCLUSIONS:

270°C is sufficient heat to start the dechlorination of both the PVC chips and the powder. The dechlorination reaction is sped up at higher temperatures. Around 400°C decomposition of the PVC also begins to take place. The addition of Cold Lake Vacuum Bottoms to the PVC chips does not hinder the release of chlorine, in fact it seems to increase the chlorine loss.

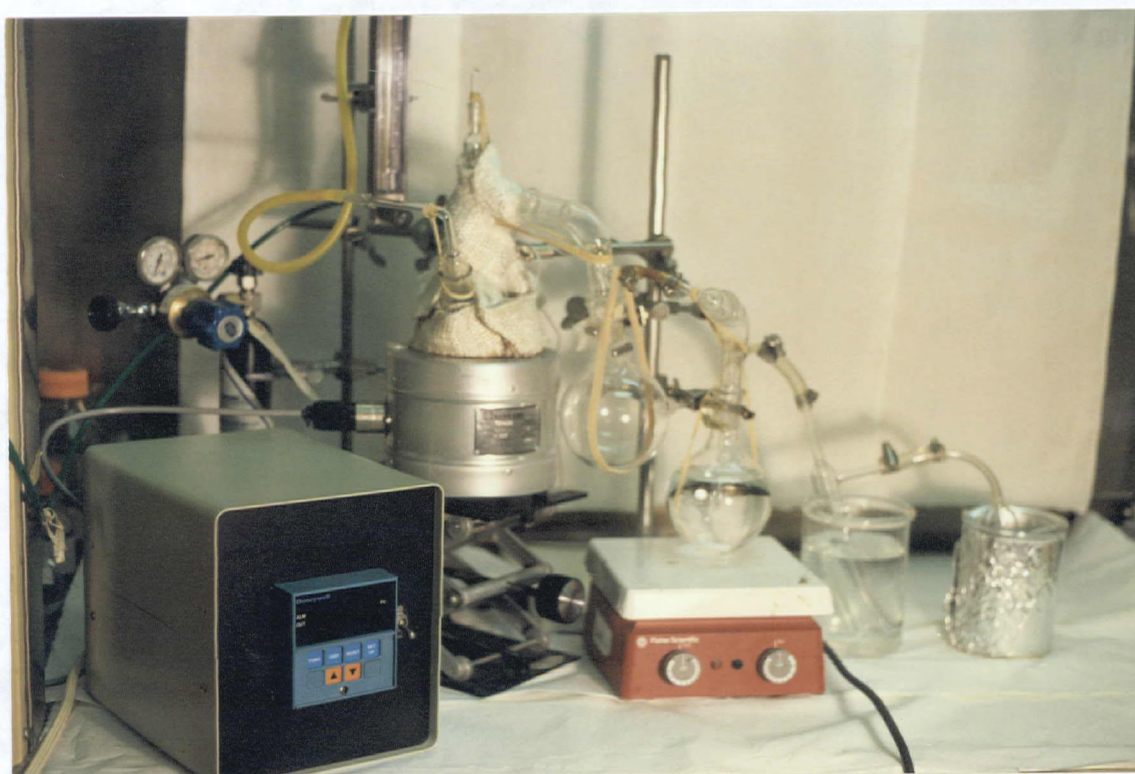


Figure 1:
DECHLORINATION APPARATUS

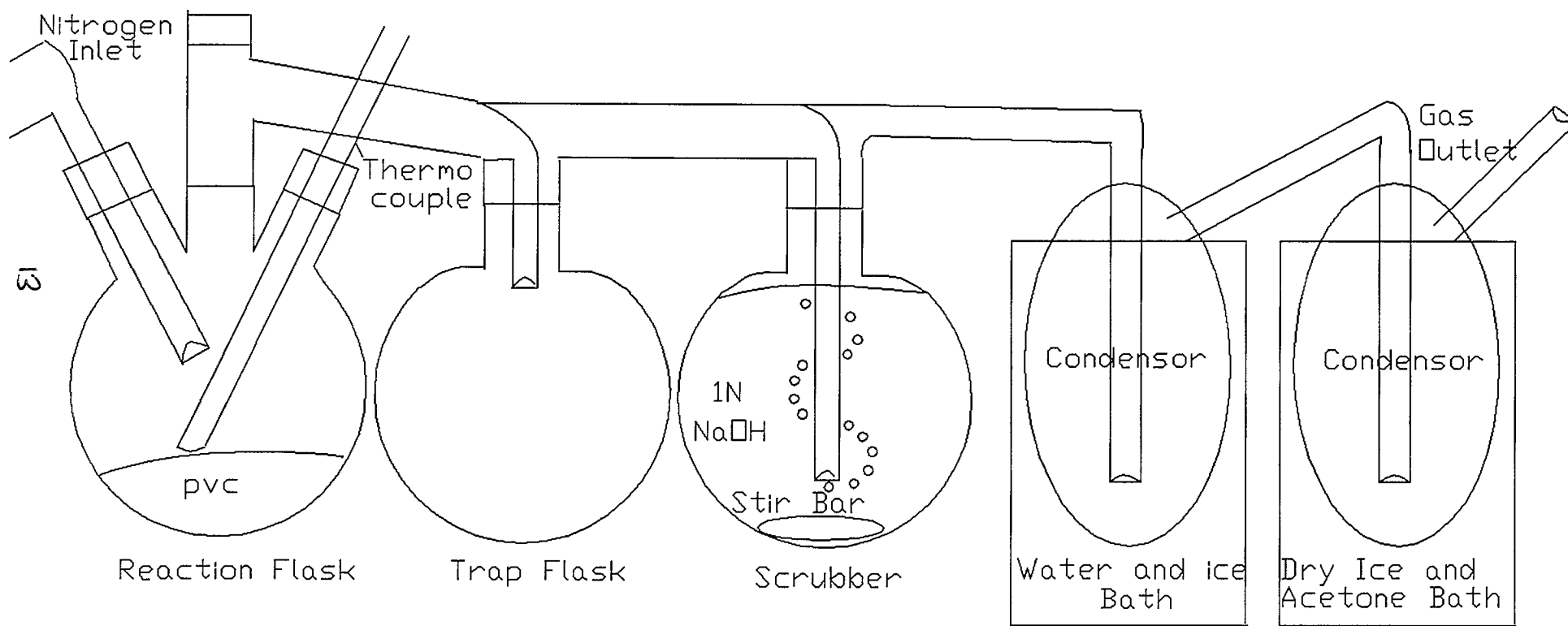
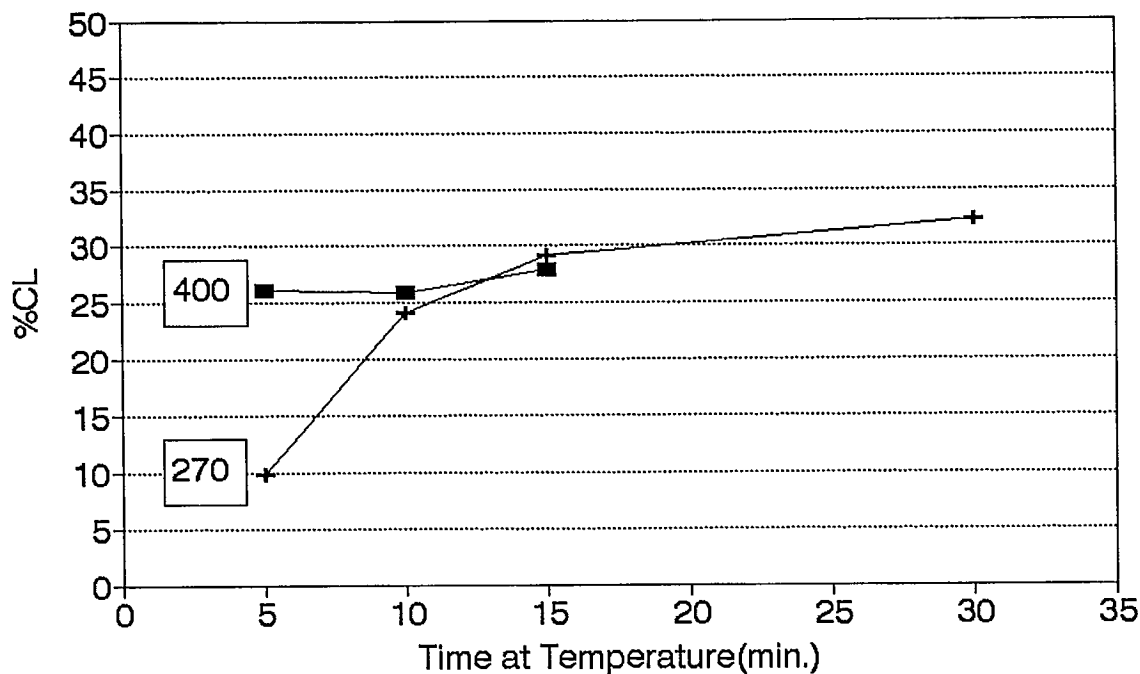


Figure 2: PVC Powder

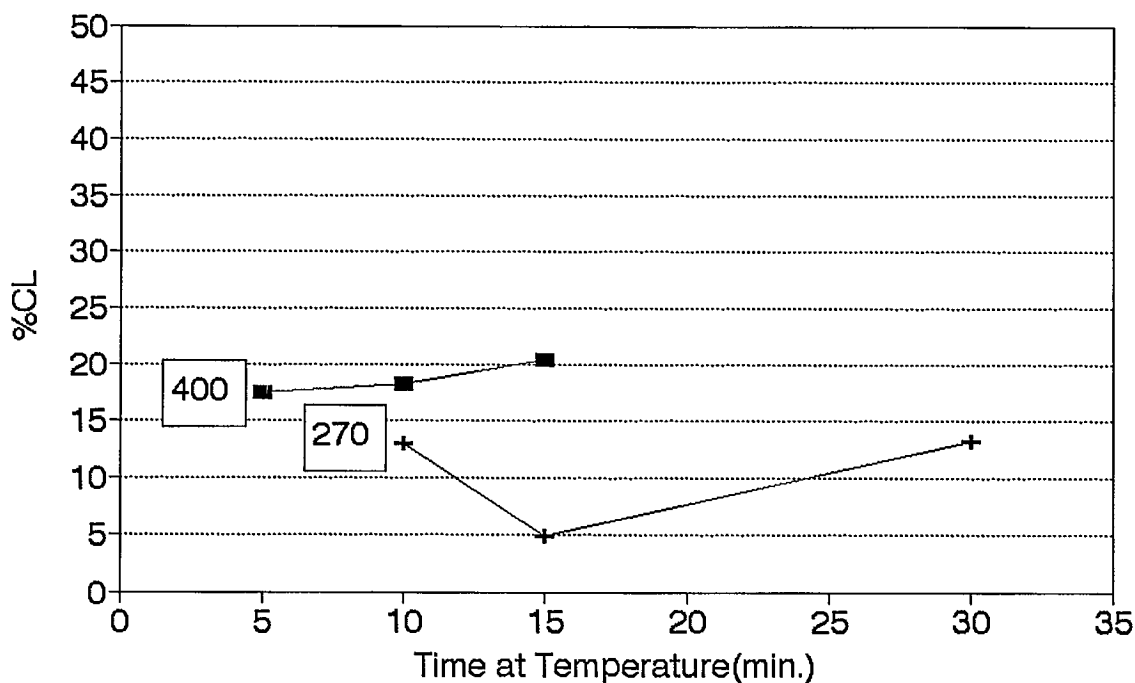
Cl recovered vs. Time at 400C and 270C



NOTE: The %Cl here is calculated as the sum of the water soluble Cl, in the trap, the scrubber and the condensers over the original PVC mass.

Figure 3: PVC Chips

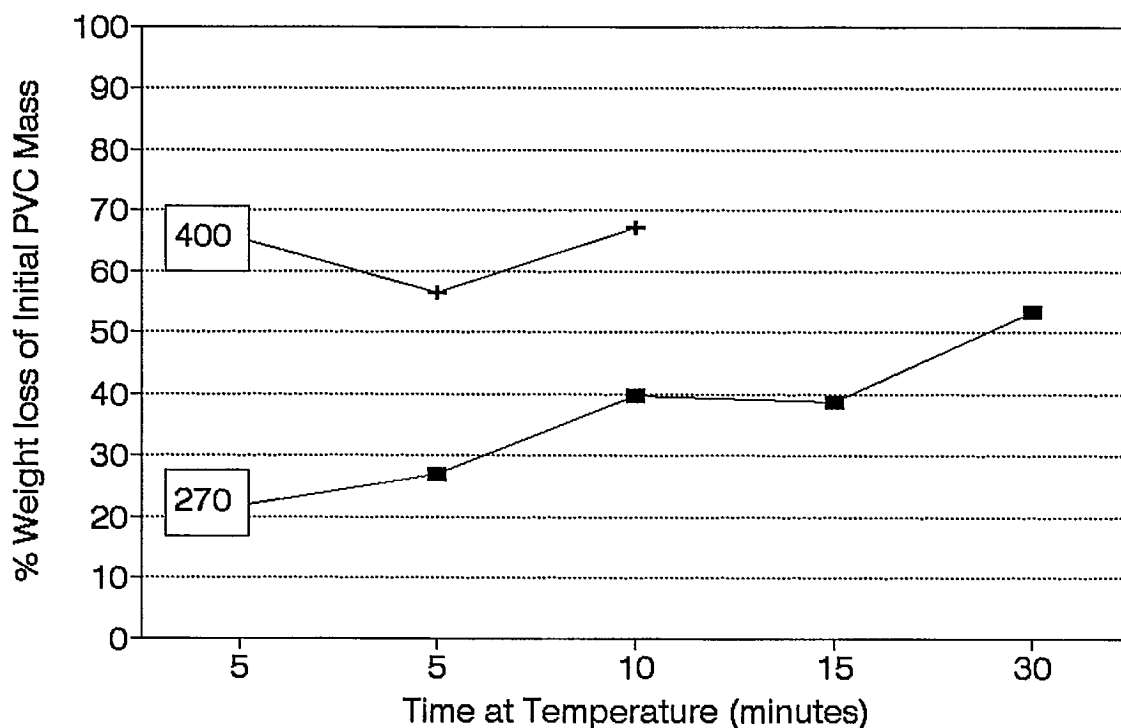
Cl recovered vs. Time at 400C and 270C



NOTE: The %Cl here is calculated as the sum of the water soluble Cl, in the trap, the scrubber and the condensers over the original PVC mass.

Figure 4: PVC Powder

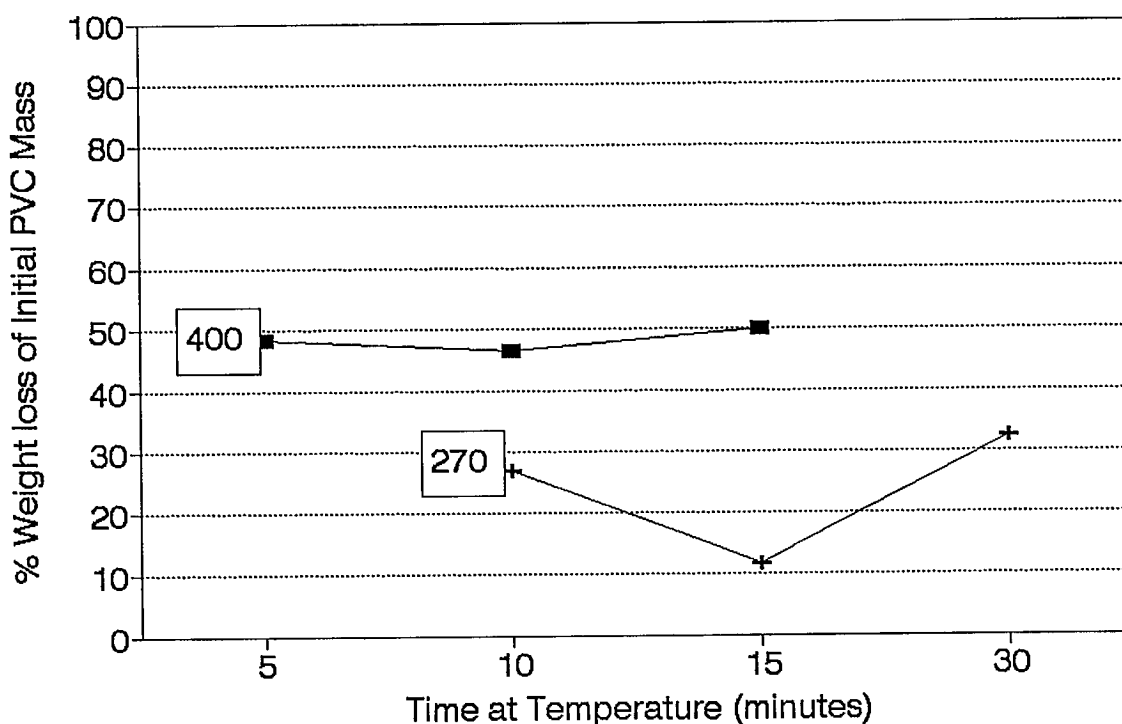
%Weight Loss vs. Time at 400C and 270C



NOTE: The weight loss of the PVC is based on the difference between the weight of the reaction flask before and after the reaction

Figure 5: PVC Chips

%weight Loss vs. Time at 400C and 270C



NOTE: The PVC weight loss is based on the difference in the weight of the reaction flask before and after

Figure 6: Change in Millivolts

Representing CL Loss Min. vs. mV.

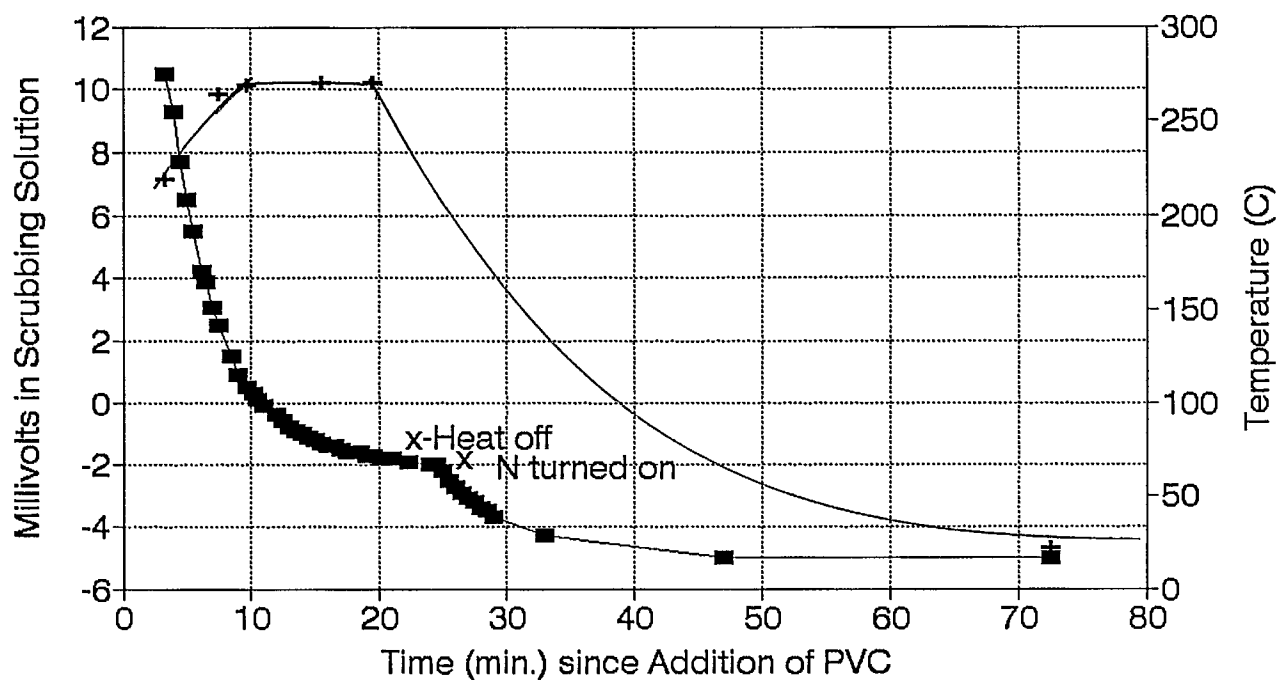


Figure 7: Change in Ph Representing

Chlorine Loss Min. vs. Ph

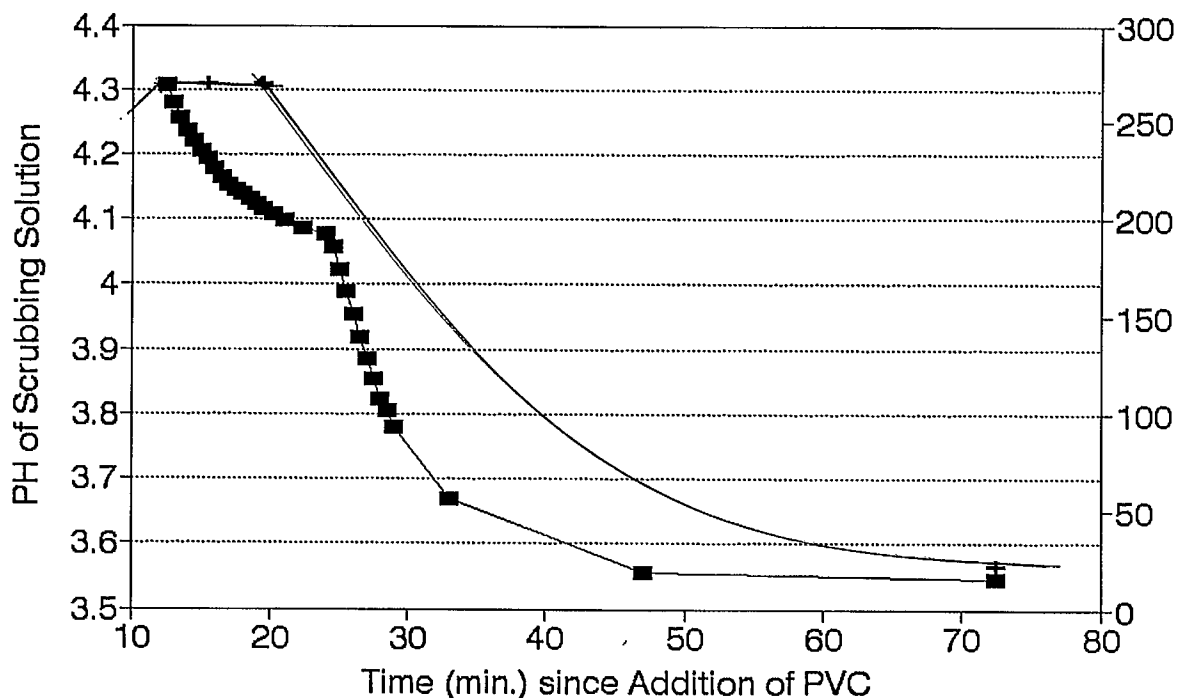


TABLE 1

Experimental Conditions and Result For
Dechlorination Reactions

Trial	Temp. 'C	Time to Heat min.	Time at Temp. min	%Cl of PVC Mass	%Cl Recovered	Mass Balance	PVC Form	
1	400	25	15	27.92	107.91	101.48	powder	
2B	400	21	10	25.86	99.93	95.85	powder	
3	400	20	5	26.10	100.87	99.77	powder	
4	270	7	30	32.27	124.72	101.43	powder	
4B	270	8	30	21.38	82.63	94.08	powder	+
4C	270	9.5	30	18.94	73.20	96.92	powder	+
5	270	8	15	29.15	112.66	97.77	powder	
6B	270	7	10	24.09	93.10	100.03	powder	
7B	270	10	5	14.13	54.61	98.37	powder	
18	270	14	30	13.27	51.29	95.83	chip	
8	270	15	15	4.81	18.59	96.31	chip	
8R	270	16	15	7.98	30.84	97.47	T8 residue	
9	270	9	10	13.01	50.28	97.47	chip	
16	400	23	15	20.46	79.07	96.47	chip	
19	400	14	10	18.28	70.65	94.69	chip	
20	400	15	5	17.45	67.45	94.31	chip	
14	300	10	15	19.14	73.97	98.2	chip+CLV	
14B	300	8	15	20.69	79.96	98.05	chip+CLV	
15	300	8	15	13.37	51.67	99.4	chip+CLV	

NOTE: The %Cl recovered is based on a lab result of 25.87%

+ These runs were performed after the thermocouple was lowered

TABLE 1 Continued

Trial	Temp. 'C	Time to Heat min.	Time at Temp. min	%Cl of PVC Mass	%Cl Recovered	Mass Balance	PVC Form	
2	400	25	10	21.04	81.32	93.10	powder	*
6	270		10	16.44	63.54	93.31	powder	^
7	270		5	9.95	38.46	92.34	powder	^
PH1	270		10	16.65	64.35	91.89	powder	^
Tr2	300	5	15	25.94	100.25	97.44	powder	
17	300	17	30	12.10	46.76	89.79	powder	

^The PVC was added after heating and loss occurred at this time

*This run leaked at the condensor

TABLE 2

Experimental Summary of PVC Powder Runs

Run Number and Conditions	Mass Loss (g)	Trap Mass (g)	Mass of Crystals (g)	Cl recovered (g)	% of Mass Loss Accounted for by the Trap and Crystals	% of Mass Loss Accounted for by the Cl Loss	% of Mass Loss Accounted for
T2-300-15	4.46	0.12	1.26	2.81	31.03	62.93	93.96
1-400-15	6.98	3.20	0.86	2.98	58.22	42.74	100.96
2-400-10	7.08	1.36	0.91	2.18	32.07	30.84	62.90
2B-400-10	5.80	1.26	1.38	2.73	45.56	47.08	92.65
3-400-5	6.59	3.16	0.70	2.71	58.55	41.09	99.65
4-270-30	5.59	1.19	1.09	3.47	40.71	62.15	102.86
4B-270-30	4.08	0.79	0.50	2.21	31.62	54.15	85.77
4C-270-30	3.37	0.58	0.57	1.96	34.12	58.25	92.38
5-290-15	3.99	0.21	0.51	3.09	18.01	77.55	95.55
6-270-10	3.44	0.25	0.76	1.74	29.24	50.67	79.92
6B-270-10	4.08	0.75	0.79	2.54	37.76	62.30	100.06
7-270-5	2.69	0.21	0.69	1.02	33.34	38.01	71.35
7B-270-5	2.21	0.22	0.34	1.48	25.34	67.11	92.45

Trap floo

Leaked

Leaked

19

TABLE 3

Experimental Summary of PVC Chip Runs

Run Number and Conditions	Mass Loss (g)	Trap Mass (g)	Mass of Crystals (g)	Cl recovered (g)	% of Mass Loss Accounted for by the Trap and Crystals	% of Mass Loss Accounted for by the Cl Loss	% of Mass Loss Accounted for
18-270-30	3.27	0.95	0.52	1.38	45.08	42.06	87.14
8-270-15	1.17	0.08	0.22	0.50	25.64	42.72	68.36
8R-270-15	2.24	0.76	0.58	0.68	60.00	30.58	90.58
9-270-10	2.70	0.76	0.51	1.34	47.04	49.70	96.74
16-400-15	5.03	2.19	0.37	2.11	50.91	42.04	92.95
19-400-10	4.66	1.95	0.61	1.88	54.94	40.34	95.28
20-400-5	4.83	2.28	0.52	1.80	57.97	37.27	95.24

Leaked

TABLE 4

Location and Amount of Chlorine for Each Run

Run Number and Conditions	Chlorine Content Determined by Titration							Form of PVC
	Total	Scrubber		Trap		Condensors		
	g	g	%	g	%	g	%	
T2-300-15	2.81	2.65	94.31	0.16	5.69	0.00	0.00	powder
1-400-15	2.98	2.90	97.11	0.09	2.89	0.00	0.00	powder
2-400-10	2.18	0.25	11.62	1.93	88.38	0.00	0.00	powder
2B-400-10	2.73	2.70	98.93	0.03	1.00	0.00	0.07	powder
3-400-5	2.71	2.64	97.64	0.06	2.26	0.00	0.09	powder
4-270-30	3.47	3.43	98.69	0.04	1.28	0.00	0.03	powder
4B-270-30	2.21	2.19	99.27	0.01	0.65	0.00	0.08	powder
4C-270-30	1.96	1.91	97.21	0.05	2.74	0.00	0.06	powder
5-290-15	3.09	2.91	94.03	0.18	5.87	0.00	0.10	powder
6-270-10	1.74	1.74	99.68	0.00	0.23	0.00	0.09	powder
6B-270-10	2.54	2.52	99.12	0.02	0.76	0.00	0.12	powder
7-270-5	1.02	1.01	98.56	0.01	1.39	0.00	0.05	powder
7B-270-5	1.48	1.48	100.00	0.00	0.00	0.00	0.00	powder
18-270-30	1.35	1.25	92.73	0.10	7.13	0.00	0.14	Chips
8-270-15	0.50	0.49	9.00	0.01	2.58	0.00	0.03	Chips
8R-270-15	0.68	0.54	78.47	0.15	21.30	0.00	0.23	Chips
9-270-10	1.32	1.15	87.06	0.17	12.92	0.00	0.02	Chips
17-300-30	1.23	1.11	90.18	0.12	9.67	0.00	0.15	Chips
16-400-15	2.11	1.78	84.37	0.33	15.57	0.00	0.06	Chips
19-400-10	1.88	1.55	83.27	0.31	16.65	0.00	0.08	Chips
20-400-5	1.80	1.46	81.27	0.34	18.73	0.00	0.00	Chips
14-300-15	0.29	0.29	97.54	0.00	1.46	0.00	0.99	Chips+CL
14B-300-15	0.32	0.31	98.97	0.00	0.85	0.00	0.18	Chips+CL
15-300-15	0.21	0.20	95.20	0.01	3.24	0.00	1.56	Chips+CL

TABLE 5

Variations or Problems in Each Run

Run #	Variation or Problem that Arose	Date Performed
T2	Overshot to 330C	21/05/92
5	Overshot to 290C, residues weight not found Therefore it was estimated	22/05/92
4	The Nitrogen was turned on as a back flow started	28/05/92
PH1	The PVC was added after the flask reached 200. A PH probe was used to measure the chlorine loss.	05/06/92
6	The PVC was added after the flask reached 270C. Mass loss occurred.	16/06/92
7	The PVC was added once the flask reached 250C. There was a leak at the reaction flask	09/06/92
1	A back flow started so the nitrogen was turned on barely	10/06/92
2	A backflow started so the nitrogen was turned on barely. There was a leak.	11/06/92
3	This run was without problems.	16/06/92
6B	This run was without problems.	17/06/92
7B	The trap material was lost so it was estimated to be the same as in run 7. This value is probably a little low however.	18/06/92
2B	The thermocouple was lowered.	19/06/92
8	This run was without problems. Centrifuging was necessary for the trap material during PVC chip runs	22/06/92
8R	This run was without problems.	24/06/92
16	This run was without problems.	25/06/92
17	This run leaked.	29/06/92
18	This run was without problems.	30/06/92
14	This run used PVC chips and CLVB. Centrifuging was not required.	02/07/92
15	This run was without problems.	06/07/92
14B	This run was without problems.	08/07/92
4B	There was a low mass balance determined but it seems to be due to error in crystal mass.	09/07/92
4C	This run overshot to 290C. A backflow started so the nitrogen was turned on barely.	14/07/92
9	This run overshot to 280C. The junctions starting with this run were also rinsed with distilled water and added to the trap.	15/07/92
19	This run was without problems.	17/07/92
20	This run was without problems.	27/07/92

APPENDIX 1:

Brief Summary of UDS 2000 Operation

This machine is used to control the heating mantle. The only flaw with this control is it is nearly impossible to have a rapid heating rate and a steady end point, with little overshoot, at the same time.

To set the end point temperature press the "DISP" key. This shows the current set point, which can be changed by pressing the up or down arrow key. To raise this value more quickly hold the up arrow key in and press the down arrow key once. This changes the increase to the next digit in the temperature. Pressing the down arrow key again moves the increase to the next left digit. Releasing the up arrow key returns you to the rightmost digit again. The same is true for lowering the temperature but using the opposite arrow keys. To return to a display of the current temperature press the "FUNC" or "RESET" key.

Once displaying the current temperature the parameters can be displayed by pressing the "SET UP" key. The words "SET UP TUNING" will appear on the display first. This will probably be the only set of parameters that need to be adjusted but further pressing of the "SET UP" key shows the other parameter groups. Once "SET UP TUNING" appears on the display pressing the "FUNC" key will scroll through the tuning parameters. These parameters can be adjusted by pressing the up and down arrow keys.

The first tuning parameter is "GAIN". It has a range from 0.1 to 1000. A value of 0.1 gives the least temperature gain rate and a value of 1000 results in a very high rate of gain. A high value for the "GAIN" leads to more overshoot, however, as the temperature is increasing in large increments.

The next parameter is "RATE T". It has a range from 0 to 10. A value of 0.08 to 0.00 gives the fastest rate of increase. Again, a very fast rate also gives the most overshoot.

The remaining tuning parameters are "I MIN" and "CYCLE". The "I MIN" controls how much output the controller puts out based on the deviation of the actual temperature from the set point temperature. The "CYCLE" key determines the length of the output relay cycle. Therefore, the lower the values for these parameters, the more carefully controlled the output of the controller is.

All these tuning parameters affect each other, so it is necessary to find values that, all together, give you the result you want. A setting, for example, that gives you a very stable set point with no overshoot, but takes 25 min to reach this point is GAIN-20, RATE T-2, I MIN-4.5 and CYCLE T-5. One way to speed this heating time is to use a setting for rapid heat and change to these parameters when the actual temperature is closer to the set point.

*For a more detailed description see UDS 2000 Operation Manual