

## RECOVERY OF CHLORINE FROM FERRIC CHLORIDE

By Richard S. Olsen<sup>1</sup>

U.S. Department of the Interior  
Bureau of Mines  
Albany Research Center  
Albany, Oregon 97321  
USA

### Summary

Chlorination of ilmenite to produce titanium products results in large quantities of byproduct ferric chloride. This finely divided, deliquescent material is extremely corrosive and presents a difficult waste disposal problem. In particular, it is difficult to transfer accurately and reliably for processing in fluidized bed operations for chlorine recovery. Compacting ferric chloride into hard, dense pellets reduces its tendency to pick up moisture and allows it to be easily fed into oxidation units where it can be converted to iron oxide and recyclable chlorine. Pelletizing also allows the incorporation of promoters such as sodium chloride and fuels such as carbon or metallic iron within the feed pellets. Bench-scale studies in fluidized-bed oxidation reactors are described and discussed. A product gas containing 80 pct chlorine was produced at 800° C in a reactor where pelletized ferric chloride was fed to the bottom of a fluidized bed of iron oxide.

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<sup>1</sup>Chemical engineer.

## Introduction

Titanium tetrachloride can be made either from rutile ( $TiO_2$ ) or from more plentiful, less expensive ilmenite ( $TiFeO_3$ ). When ilmenite is used, large quantities of ferric chloride are produced. This deliquescent, corrosive byproduct is very difficult to dispose of and is a major deterrent to the use of ilmenite for the production of titanium tetrachloride.

The chlorine in ferric chloride can be recovered and the iron converted to useful, or at least disposable, iron oxide by reaction with oxygen. However, ferric chloride's deliquescence, low melting point, low boiling point, and negligible heat of reaction with oxygen make its conversion to ferric oxide and chlorine a challenging technological problem.

Considerable effort has been expended to carry out the ferric chloride oxidation reaction in fluidized-bed reactors. DuPont patents by Reeves (1) and Haack (2) recommend a circulating, or fast, fluidized-bed reactor in which the bed material is continually blown from the reactor, separated in cyclones, and most of the solids recycled to the bottom of the fluidized bed. Ferric chloride, with about 2 pct NaCl, was fed to the bottom of the reactor with the recirculating iron oxide. This approach has been made to work in a large unit (1.5 to 3.5 ton/h) but has not, as of this date, been used in production to recover chlorine from ferric chloride.

Temperatures of 550° to 750° C are required to maintain the dechlorination reaction at reasonable rates, and some type of auxiliary heat must be supplied to supplement the small heat of reaction of  $FeCl_3$  with oxygen. The DuPont patent (2) calls for burning a specially prepared coke in the dechlorination unit to maintain the reaction temperature. This coke was made from subbituminous coals and must contain at least 0.5 pct hydrogen to provide a stable ignition temperature no higher than 500° C. The coke must contain less than 2.5 pct hydrogen, so that excessive amounts of water are not formed. Any water is absorbed by ferric chloride to make a sticky, low-melting hydrate that will collect and foul the reactor.

Similar work on fluidized-bed dechlorination of ferric chloride was done by the Bureau of Mines. Henderson (3) reacted ferric chloride and oxygen in a fluidized bed of freshly prepared iron oxide and obtained a product gas containing 90 pct  $Cl_2$  at 650° C. Paige (4) and Harris (5) used a larger scale bubbling, or dense, fluidized-bed device and reacted ferric chloride with oxygen in a bed of well-used iron oxide. They introduced fresh iron oxide that had been treated with 0.5 pct NaCl along with the  $FeCl_3$  feed in a 1:3 ratio. Chlorine levels in the exhaust of around 65 vol pct were obtained. External heating was used to maintain temperatures around 500° C.

In the present effort an attempt was made to carry out the dechlorination reaction in such a way that the difficulties experienced with the previous approaches were avoided. A dense phase, bubbling fluidized bed was used to avoid the very large and complex equipment required in the DuPont process. Feeding of the dusty, finely divided ferric chloride was made much easier by pelletizing it into hard, uniform, cylindrical pellets. This allowed accurate, reliable feed rates and, more importantly, allowed the ferric chloride to be injected into the fluidized bed before any melting or vaporization could take place. It was originally thought that pelletizing might allow top feeding of the fluidized-bed reactor, and some success was obtained by dropping the pellets into a fluidized bed. However, much better conversions were obtained when the pellets were

injected into the bottom of the fluidized-bed reactor in a high-velocity stream of oxygen.

#### Equipment and Procedures

Ferric chloride pellets were made by compacting technical-grade anhydrous chloride in a California Pellet Mill<sup>1</sup>. This rotary mill was sealed at the feed inlet and the pellet collection flask, and purged with dry carbon dioxide to prevent hydration of the ferric chloride. Interchangeable dies allowed pellets to be made in 3.2, 1.6, and 1.0 mm diameters. The pellets were screened in a dry box to remove any fines or lumps and stored in a desiccator.

Iron oxide bed material was prepared from finely divided commercial ferric oxide by sintering it in air at 850° C. The charge was brought up to temperature, held over a weekend at 850° C, and cooled in the furnace. The sintered oxide was then crushed and screened to the minus 80-plus 270-mesh size fraction used as bed material for the fluidized bed dechlorination units.

The top-fed, fluidized-bed dechlorination unit is shown in figure 1. The reactor consisted of a 5-cm-diameter Vycor<sup>1</sup> tube mounted in an electric furnace. Oxygen, preheated to help maintain the desired reaction temperature, was admitted through a fritted glass bed support at 2 L/min to fluidize a bed of minus 80-mesh iron oxide. Pelletized ferric chloride was added to the top of the fluidized bed with a screw feeder. The feed tube was purged with 150 mL/min nitrogen to prevent reaction in the tube. Chlorine-containing off-gases, along with some vaporized ferric chloride and iron oxide dust, left the top of the reactor. Unreacted ferric chloride and dust were removed from the gas stream prior to analysis of the gases in an ultraviolet-light-absorption gas analyzer. All tests done in the top-fed fluidized-bed reactor were relatively short term, and no provision was needed to remove oxide from the bed during the run. Ferric chloride conversion was determined by weighing and analyzing the contents of the reactor and the solids-collecting vessels downstream from the reactor.

The bottom-fed reactor is shown in figure 2. As with the top-fed, fluidized-bed reactor, a bubbling fluidized bed was maintained with as little entrainment of solids as possible. Both oxygen at 15 L/min and 1-mm-diam FeCl<sub>3</sub> pellets were introduced to the 9.5 cm diameter bed through a 3.2-mm-ID stainless steel tube. A pressure drop around 5 psi over the length of the tube caused the pellets to be swept up the tube. The desired feed rate, around 140 g/min, was maintained by the pressurized screw feeder that moved the ferric chloride pellets into the chamber containing the lower end of the feed tube. Iron oxide dust and unreacted ferric chloride were collected in the solids separation equipment downstream from the fluidized-bed reactor. Preliminary tests were again short enough that no bed overflow was required, and iron oxide product built up in the bed. Conversions were calculated based on the amount of chloride fed and the amount of unconverted ferric chloride collected in the dust traps downstream from the reactor.

In both cases, a run was made by preheating the reactor to the desired temperature while the bed material was fluidized with oxygen or air and then introducing the feed material at the desired rate. Chlorine in the

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<sup>1</sup>Reference to specific products does not imply endorsement by the Bureau of Mines.

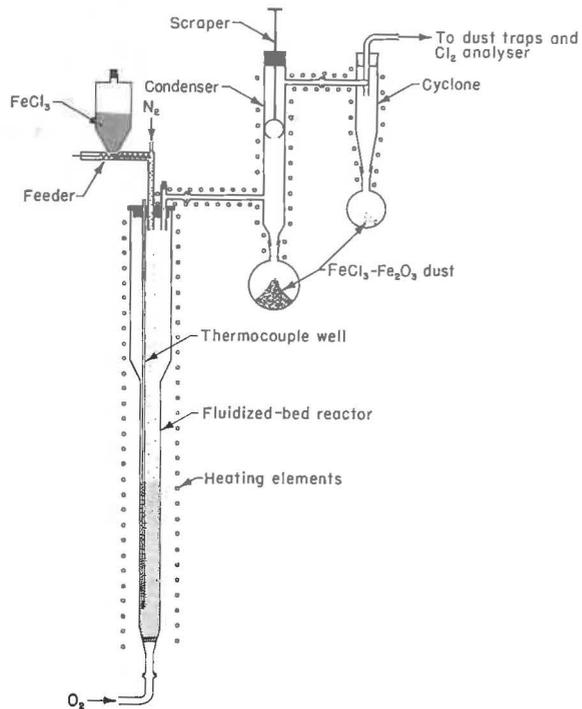


Figure 1 - Top-fed, fluidized-bed reactor.

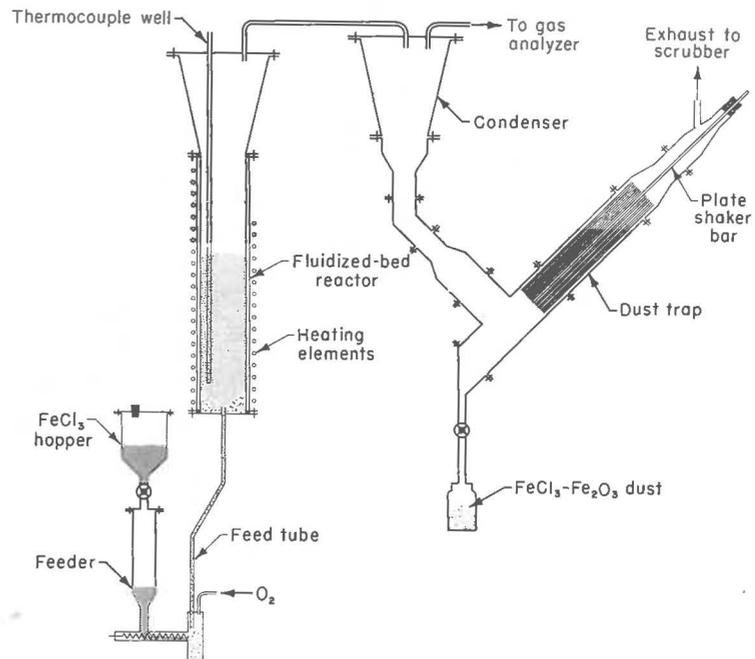


Figure 2 - Bottom-fed, fluidized-bed reactor.

55 pct with 0.5 pct sodium chloride was accompanied by large amounts of a tough, dense wall deposit above the fluidized bed. This wall deposit could not be tolerated during continuous operation and would prevent the use of sodium chloride, at least in small, top-fed, fluidized-bed reactors.

The effect of more, or less, ferrous chloride in a potential ferric chloride waste product was considered. A set of tests was done that was identical to the first, only using anhydrous ferrous chloride rather than ferric chloride. The results shown in table 2 and illustrated in figure 4 are very similar to those obtained with ferric chloride. However, conversions were considerably higher when ferrous chloride was used. This is most likely due to the much higher boiling point of ferrous chloride and its longer retention in the fluidized-bed reactor.

TABLE II. - Preliminary tests for  $\text{Cl}_2$  recovery from  $\text{FeCl}_2$  in top-fed, fluidized-bed reactor<sup>1</sup>

Pellet diam, cm	Bed temp, °C	Bed height, cm	NaCl in pellets, pct	Wall deposits, pct of $\text{Fe}_2\text{O}_3$ produced	Conversion of $\text{FeCl}_3$ , pct	Chlorine in exhaust, pct
3.2	650	18	0	2.3	62	57
1.6	750	18	0	1.3	59	45
1.6	650	30	0	1.4	43	30
1.6	750	30	.5	67.5	61	61
3.2	750	30	0	.9	51	39
1.6	650	18	.5	35.3	56	55
3.2	650	30	.5	50.4	56	50
3.2	750	18	.5	24.7	68	65

<sup>1</sup> $\text{FeCl}_3$  feed rate = 11.3 g/min;  $\text{O}_2$  feed rate = 2.0 L/min.

The important factors identified in the screening test series were examined further. These were the effects of bed temperature and sodium chloride additions to the ferric chloride feed pellets. Operating conditions and results for these tests, along with several tests in which metallic iron was added to the ferric chloride feed pellets, are shown in table 3. The tests with metallic iron in the feed pellets were done to explore its use as a fuel to heat the reactor. Although more expensive than carbon, metallic iron has the advantages of being more reactive and igniting at a much lower temperature in the presence of chlorine, and that its oxide is a solid and not a gas that would dilute the chlorine product. The temperatures shown in the table are those maintained before the pellets containing metallic iron or carbon were fed. The temperature rise obtained is shown in the seventh column.

Figure 5 shows the effect of temperature on chlorine content in the fluidized-bed reactor exhaust. This shows the same trend as earlier work (3), but conversions are not as high owing to the poor efficiency of the top-fed, fluidized-bed reactor. Chlorine in the exhaust was 45 pct at a bed temperature of 800° C.

Figures 6 and 7 show the effect of sodium chloride in the feed on chlorine in the exhaust, and the buildup of deposits on the reactor wall above the fluidized bed. The catalytic effect of sodium chloride appears to be linear with concentration, with 0.5 pct NaCl increasing the chlorine in the exhaust gas from 38 to 65 pct. However, the accumulation of wall deposits also shows a linear trend and may preclude the use of sodium chloride as a catalyst in the ferric chloride oxidation reaction.

The effect of metallic iron addition to the ferric chloride feed is shown in table 3 and illustrated in figures 8 and 9. The iron was added to

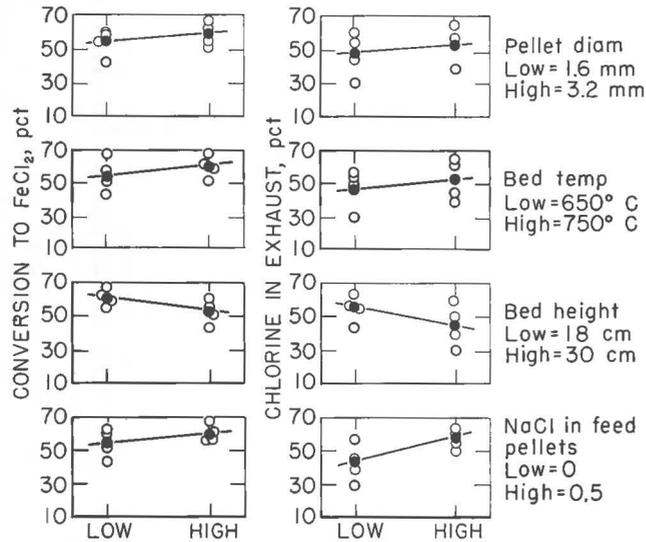


Figure 3 - Dechlorination of ferric chloride in top-fed, fluidized-bed reactor.

Open dots represent actual responses  
 Closed dots represent averaged responses

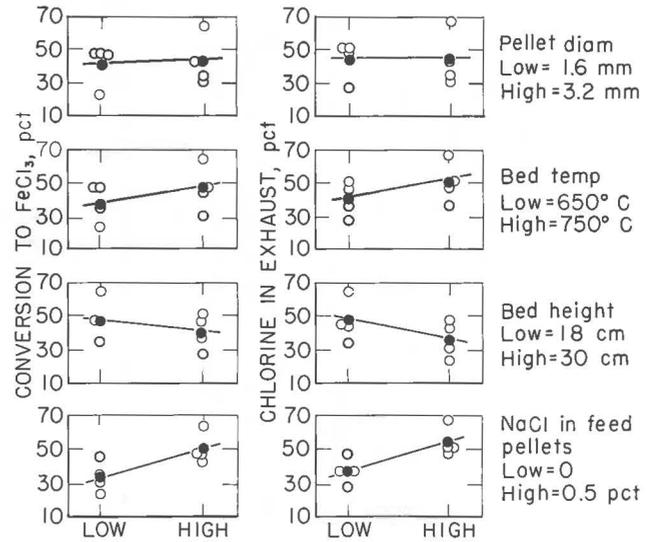


Figure 4 - Dechlorination of ferrous chloride in top-fed, fluidized-bed reactor.

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3.2	650	18	0	2.3	62	57
1.6	750	18	0	1.3	59	45
1.6	650	30	0	1.4	43	30
1.6	750	30	.5	67.5	61	61
3.2	750	30	0	.9	51	39
1.6	650	18	.5	35.3	56	55
3.2	650	30	.5	50.4	56	50
3.2	750	18	.5	24.7	68	65

<sup>1</sup> $FeCl_3$  feed rate = 11.3 g/min;  $O_2$  feed rate = 2.0 L/min.

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TABLE III. - Chlorine recovery from  $\text{FeCl}_3$  in top-fed, fluidized-bed reactor<sup>1</sup>

Bed temp, °C	Additives to pellets, wt pct				$\text{FeCl}_3$ feed rate, g/min	Temp rise when fuel added, °C	Wall deposits, pct of $\text{Fe}_2\text{O}_3$ produced	Conversion of $\text{FeCl}_3$ , pct	Chlorine in exhaust, pct
	NaCl	$\text{Fe}_2\text{O}_3$	C	Fe					
650	0	0	0	0	22.4	NA	ND	24	23
700	0	0	0	0	20.5	NA	ND	27	27
750	0	0	0	0	20.0	NA	ND	37	38
800	0.1	0	0	0	20.0	NA	ND	45	46
750	0.1	0	0	0	20.5	NA	17	42	44
700	0	0	0	0	19.5	NA	18	34	33
650	0	52	0	0	18.2	NA	ND	ND	20
750	0	52	0	0	19.2	NA	ND	54	31
650	0	0	5	0	19.0	0	ND	ND	15
750	0	0	5	0	19.0	10	ND	ND	20
720	0	10	0	0	15.4	NA	ND	32	28
650	0	10	0	0	18.0	NA	ND	24	21
650	0	0	0	5	16.7	15	ND	29	25
750	0	0	0	5	17.5	20	ND	46	39
650	0	10	0	5	16.5	10	ND	28	30
750	0	10	0	5	17.0	15	ND	30	28
650	0	0	0	10	18.9	40	ND	25	31
750	0	0	0	10	18.0	35	ND	37	46
650	0	0	0	15	17.8	60	ND	27	41
750	0	0	0	15	18.6	55	ND	41	58

NA = Not applicable; ND = Not determined; <sup>1</sup>Bed height = 18 cm; Pellet diameter = 3.2 mm;  $\text{O}_2$  Feed rate = 3 L/min.

TOP-FED, FLUIDIZED-BED RESULTS

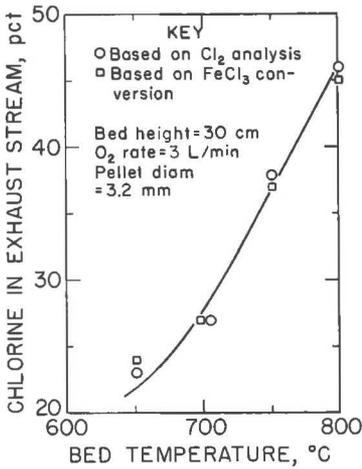


Figure 5 - Chlorine in exhaust as a function of bed temperature.

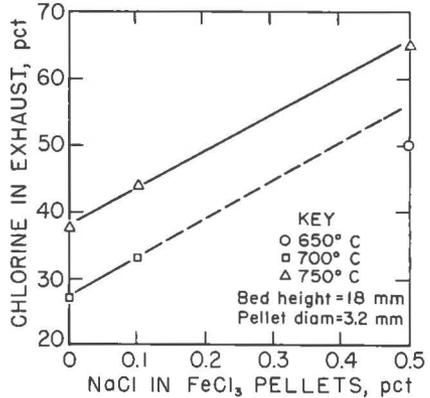


Figure 6 - Chlorine in exhaust as a function of NaCl in  $\text{FeCl}_3$  pellets.

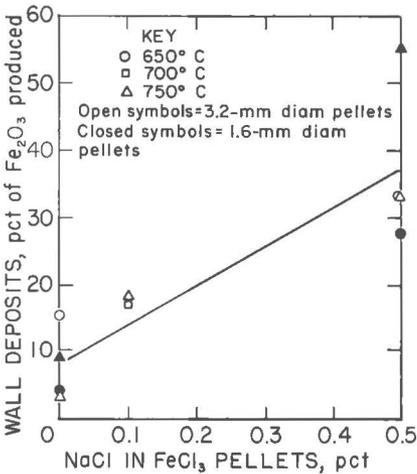


Figure 7 - Wall deposits as a function of NaCl content in  $\text{FeCl}_3$  pellets.

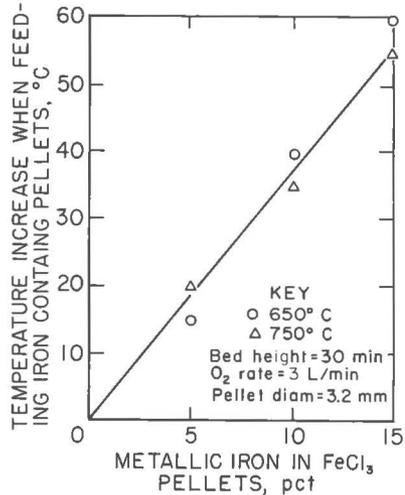


Figure 8 - Temperature rise in fluidized-bed dechlorination as a function of metallic iron content in  $\text{FeCl}_3$  pellets.

the pellets by blending finely divided iron powder with the ferric chloride prior to pelletizing. The iron powder did not interfere with the pelletizing operation when present in quantities up to 15 wt pct. Figure 8 shows the temperature rise in the fluidized-bed reactor when the feed was changed from pure ferric chloride pellets to pellets containing metallic iron. As might be expected, the temperature rise was a linear function of iron content and amounted to 3.8° C for each percent of iron added. Figure 9 shows that chlorine in the exhaust rises from 38 pct to 57 pct at 750° C when 15 pct iron is added. This is due in part to better conversion, but mostly to consumption of oxygen to form iron oxide. This indicates that if metallic iron can be purchased, or perhaps reduced on site inexpensively, it may provide a good means of supplying the necessary heat for the dechlorination reaction and also increase the percentage of chlorine in the exhaust gases.

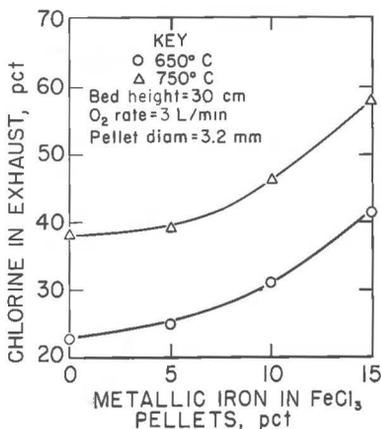


Figure 9 - Chlorine in exhaust as a function of metallic iron content in  $\text{FeCl}_3$  pellets.

#### Bottom-Fed, Fluidized-Bed

Shakedown tests have been done on a bottom-fed, fluidized-bed dechlorination unit. Results of these preliminary tests are shown in tables 4 and 5, and in figure 10, and should be considered as semiquantitative due to erratic feeding or poor temperature control in the early tests. In the cases where sodium chloride or metallic iron were used, the results of which are shown in table 5, the reactor was brought to steady state with ferric chloride feed pellets, and then pellets containing the additive were used as feed. The results may well be different when feeds containing metallic iron or sodium chloride are used throughout the run over an extended period.

Table 4 and figure 10 show the results of tests using ferric chloride pellets with no additives. As shown in figure 10, the chlorine concentration in the exhaust trends upward with increasing temperature, reaching about 80 pct at 800° C. This compares with chlorine levels of 46 pct in the top-fed, fluidized-bed unit. The data in figure 10 also indicate that the chlorine level in the exhaust is strongly dependent on bed height

TABLE IV. - Chlorine recovery from  $\text{FeCl}_3$  in bottom-fed, fluidized-bed reactor<sup>1</sup>

Bed height cm	Reaction temp, °C	$\text{FeCl}_3$ feed rate, g/min	$\text{O}_2$ to $\text{FeCl}_3$ , stoichio-metric ratio	Cumulative bed use time, min	Conversion of $\text{FeCl}_3$ , pct	$\text{Cl}_2$ in exhaust, pct
29	705	100	1.42	56	67	64
21	745	142	1.00	108	42	51
29	800	146	.97	158	73	86
36	725	165	.86	33	78	95
29	800	127	1.12	256	66	75
29	738	139	1.02	351	53	68

<sup>1</sup>Bed height measured in unfluidized state;  $\text{O}_2$  feed rate = 14.7 L/min in all runs.

or condition of the bed material, or both. Ninety-five pct chlorine was obtained from a 36-cm-deep bed at 725° C. However, the iron oxide bed material used in this test was freshly prepared and may have been more active and caused these excellent results. In contrast, a 21-cm-deep bed produced an exhaust containing only 51 pct chlorine. This may have been due to the short bed or perhaps to plugging problems in the crossover from the reactor to the condenser that occurred during this run.

Table 5 shows the effects of adding metallic iron or sodium chloride to the feed pellets. In run 2 the addition of 5 pct iron caused a 15° C temperature rise and caused chlorine in the exhaust to increase from 78 to 82 pct. During this run, conversion dropped from 62 to 59 pct because the solids feed rate was not reduced to compensate for the presence of the metallic iron in the feed. As a consequence, there was only enough oxygen available to react with 85 pct of the ferric chloride metallic iron pellets. Addition of sodium chloride showed little effect on conversion but tended to form a plug in the crossover between the reactor and the dust collector. The minimal effect shown by sodium chloride additions, particularly when compared with the strong effect shown in figure 6 for

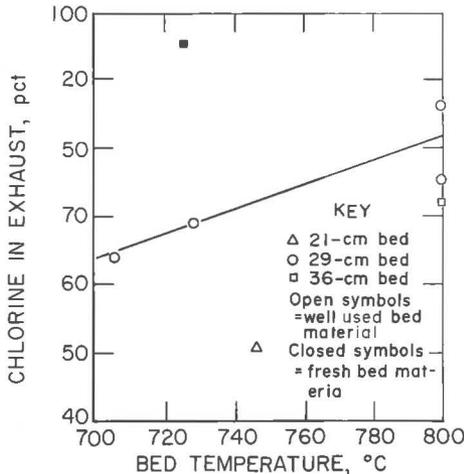


Figure 10 - Chlorine production in bottom-fed, fluidized-bed reactor as a function of temperature.

TABLE V. - Effects of sodium chloride and metallic iron additions on bottom-fed, fluidized-bed dechlorinator operation<sup>1</sup>

Additives to feed, wt pct		Reaction temp, °C	FeCl <sub>3</sub> feed rate, g/min	O <sub>2</sub> to FeCl <sub>3</sub> , stoichio-metric ratio	Temp rise from fuel use, °C	Cumulative time of bed use, min	Conversion of FeCl <sub>3</sub> , pct	Cl <sub>2</sub> in exhaust, pct
NaCl	Fe							
0.5	0	750	95	1.49	NA	70	73	66
0	0	760	147	.97			62	78
0	5	775	149	.85	15	196	59	82
0	0	710	143	.99	NA		45	62
0.5	0	705	143	.99	NA	94	51	68

NA = Not applicable,

<sup>1</sup>Bed height measured in unfluidized state = 29 cm; O<sub>2</sub> Feed rate = 14.7 L/min.

top-fed results, is probably the result of the short term runs in the bottom-fed reactor. Longer tests in which the bed material can become thoroughly conditioned with sodium chloride will provide a better measure of the effect.

#### Conclusions

The concept of pelletizing ferric chloride to provide easier operation of ferric chloride dechlorination units was demonstrated with encouraging results. A reactor in which pelletized ferric chloride was introduced to the bottom of a fluidized bed with oxygen yielded a 95-pct-chlorine product gas. This was consistent with the results of others (3) in that the high conversion was obtained with fresh bed material. Good results were also obtained with well-used bed material where 86-pct-chlorine product was obtained. This indicates that future research should be conducted on the effects of reaction time, temperature, and the presence of additives such as sodium chloride or metallic iron on the activity of the iron oxide in the fluidized bed.

Samples of fresh and well used fluidized-bed material were examined electron microscope at a magnification of 2,000. The fresh material appeared finely divided and amorphous, while the well-used material showed the presence of crystallites on the surface of the iron oxide particles. The presence of these hard, regular particles indicates that it may be necessary to introduce some fresh oxide as practiced by Paige (4) and Harris (5) to maintain high efficiency in the dechlorination reaction.

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# RECYCLE AND SECONDARY RECOVERY OF METALS

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Edited by

**Patrick R. Taylor**

Department of Metallurgical and Mining Engineering  
University of Idaho  
Moscow, Idaho

**Hong Y. Sohn**

Department of Metallurgy and Metallurgical Engineering  
University of Utah  
Salt Lake City, Utah

**Noel Jarrett**

Alcoa Technical Center  
Alcoa Center, Pennsylvania

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