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TECHNICAL MEMORANDUM REPORT

Removal of Hydrogen Fluoride at Low Concentrations in Air Streams by Activated Charcoal and Sodium Fluoride

Object

To determine the efficiency of the removal of low concentrations (0.1 to 0.4 per cent by volume) hydrogen fluoride gas from air streams by adsorption on charcoal and chemisorption on sodium fluoride and to correlate the capacity and efficiency characteristics of these substances.

Experimental

A schematic diagram of the apparatus is shown in Figure 1. Connections, fittings, and tubing in the apparatus were saran plastic. The valves used were Hoke No. 341. The absorption tower was fabricated from a screw-cap cylindrical lucite container, 11 inches long and 1.75 inches in

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diameter. The gas flow path was lengthwise in an upward direction and the tower beds were held in place with perforated teflon disks.

A polythene bubbler filled with mineral oil gave an indicative means of HF flow control. Mineral oil was selected as the bubbler liquid, because of its low reactivity and low hydrogen fluoride solubility.

Runs were made on sodium fluoride, activated charcoal and a mixture of the two. The sodium fluoride was Baker's C. P. powder and the charcoal was Fisher's Code 5-685 activated charcoal of 6-14 mesh. Both the sodium fluoride and the fluoride-charcoal composite were mixed with lucite turnings to prevent packing and high pressure drop. The hydrogen fluoride used was obtained from the Harshaw Chemical Company.

The apparatus system allowed routing the gas stream either through the tower or through an open line to the dilute sodium hydroxide wash. Variance of pH in the dilute sodium hydroxide wash, a measure of HF in the air stream, was measured with a Model G Beckman pH meter employing an 1170 calomel electrode and an 1190-E glass electrode.

The gas mixture was passed directly into the dilute sodium hydroxide wash and the time recorded for a pH drop from 11 to 10 in two liters of solution. After replacing the wash with fresh solution, the gas stream was rerouted through the tower and the time recorded again for pH drop from 11 to 10. Average times were taken with blanks being made both before and after each absorption run.

The concentrated sodium hydroxide wash was attached directly to the tower line during hydrogen fluoride passage between measurements.

An estimation of the weight of hydrogen fluoride in the tower was made at each measurement of per cent removal, from the product of the HF flow (calculated from the pH change in the open line determinations), the time elapsed since the previous absorption run, and the fraction of HF being removed by the tower sample.

After the final measurements were taken, the contents of the tower were checked for HF content by acidity measurements and an average was made between the pH and acidity values to determine the grams of HF present in the tower.

Data for the runs are contained in Tables I, II, and III. The densities of the tower beds are listed in Table IV.

Results and Discussion

Gas mask cannister specifications state that charcoal should adsorb 1.75 moles of HF/ml of solid (i.e. 0.09 grams HF/gram of charcoal). The efficiency of the charcoal adsorber decreased rapidly at a tower load of 0.08 to 0.12 gram HF/gram adsorbent (Figure 2).

Results were not reproducible between different beds for the powdered sodium fluoride due to the channeling effects in the supported solid, and

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the removal of HF from the air stream varied from 96 to 78 per cent at 0.0 grams HF/gram NaF. One bed was carried through the entire range of the sodium fluoride absorber to determine the shape of the curve and total capacity as shown in Figure 2. The capacity of the sodium fluoride for removing HF at low concentrations from air streams is 0.4 - 0.5 grams HF/gram NaF.

Sodium fluoride has been used in pellet form in pilot plant operation for the removal of HF from fluorine gas streams.(1) The pilot plant efficiency was near 100% until the HF/NaF weight ratio equaled 0.12. This higher efficiency resulted from pilot plant use of a weight ratio (NaF in Tower/HF in air stream), six times larger than used in our laboratory studies.

At the higher HF percentages used in the pilot plant (4 per cent by volume), the sodium fluoride formed semi-fluid polyacid polymers below 100°C; a phenomena not obtained at low HF percentages in the laboratory.

Conclusions

Activated charcoal (Fisher's Code 5-685, 6-14 mesh) deposited in a 6 inch thick bed removed 96.5 per cent of the HF from an air stream containing 0.2 to 0.4 mole per cent HF and flowing at 25.7 ft/min. and a volume velocity of 51.5 volumes/min. The efficiency diminished as the amount of HF adsorbed increased with rapid decrease in efficiency occuring between the tower loadings of 0.08 and 0.12 grams HF/gram adsorbent. At 0.15 gram HF/gram of adsorbent the removal efficiency was 50 per cent.

Sodium fluoride powder supported on lucite turnings exhibited a greater capacity than charcoal; at 50 per cent efficiency, the sodium fluoride had adsorbed approximately 0.3 grams HF/gram of sodium fluoride. The maximum removal efficiency of this run was only 85 per cent. Air flow rates of 25.7 ft/min., volume velocity of 61.2 volumes/min. and HF mole percentage in air of 0.2 to 0.4 were employed.

A mixture of sodium fluoride and activated charcoal on lucite turnings had a slightly higher efficiency (97.7 per cent) than the charcoal alone, but the total capacity of the mixture was not significantly greater than that of the contained quantity of sodium fluoride alone. At 47.7 per cent efficiency sodium fluoride contained 0.342 grams HF/gram NaF, while at 47.2 per cent efficiency the mixture of charcoal and sodium fluoride contained 0.354 grams HF/gram of contained NaF.

(1) Froning, Richards, Stricklin, and Turnbull, Industrial and Engineering Chemistry, 39, 275-6 (1947).

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Table I -- Adsorption of HF by Activated Charcoal(1)

Time (Hours)	grams HF/gram Charcoal(2)	grams HF/gram Charcoal(3)	grams HF/gram Charcoal (average)	Efficiency Per Cent(4)
0	0	0	0	
2 3/4	0.059	0.037	0.048	96.5
5 1/4	0.113	0.071	0.092	91
7 3/4	0.167	0.105	0.136	90
8 3/4	0.189	0.118	0.153	60
				50

(1) 100 grams; bed height - 6 inches; air flow - 0.43 CFM; HF concentration - 0.2 to 0.4 per cent by volume.
(2) Determined by pH change in open line gas wash and operating time.
(3) Determined by filtration of final tower contents with standard NaOH.
(4) Determined by time of pH change ratio in open line and tower effluent gas wash.

Table II -- Absorption of HF by Sodium Fluoride(1)

Time (Hours)	grams HF/gram NaF(2)	grams HF/gram NaF(3)	grams HF/gram NaF (average)	Efficiency Per Cent(4)
0	0	0	0	
3	0.108	0.113	0.111	86
5	0.173	0.182	0.178	78.8
7	0.233	0.245	0.239	70.4
9	0.286	0.300	0.293	59.7
11 1/2	0.333	0.349	0.341	55
16 1/2	0.420	0.44	0.43	47.7
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(1) 50 gram of powdered sodium fluoride in lucite turnings; bed height 5-6 inches; air flow - 0.43 CFM; HF concentration - 0.2 to 0.4 per cent by volume.
(2) Determined by pH change in open line gas wash and operating time.
(3) Determined by filtration of final tower contents with standard NaOH.
(4) Determined by time of pH change ratio in open line and tower effluent gas wash.

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Table III -- Absorption Using 50/50 Mixture of Sodium Fluoride and Activated Charcoal[1]

Time (Hours)	grams HF/gram mix.[2]	grams HF/gram mix.[3]	grams HF/grams mix. (average)	grams HF/gram NaF (average)	Efficiency Per Cent[4]
0	0	0	0	0	97.7
3	0.065	0.051	0.058	0.116	95.0
6	0.125	0.098	0.112	0.224	88.0
8 1/2	0.174	0.137	0.156	0.312	68.6
11	0.20	0.157	0.177	0.354[5]	47.2

(1) 50 grams of sodium fluoride and 50 grams of 6-14 mesh activated charcoal mixed in lucite turnings; bed height 6-7 inches, air flow - 0.43 CFM; HF concentration - 0.2 to 0.4 per cent by volume.
(2) Determined by pH change in open line gas wash and operating time.
(3) Determined by filtration of final tower contents with standard NaOH.
(4) Determined by time of pH change ratio in open line and tower effluent gas wash.
(5) Compares with 11 1/2 hour point on sodium fluoride alone. See Table II.

Table IV -- Densities of Charcoal and Sodium Fluoride Tower Beds

Charcoal	=	0.41 grams/ml
Sodium Fluoride (powder)	=	0.97 grams/ml
(in lucite turnings)	=	0.26 grams/ml
50/50 mix. charcoal and sodium fluoride	=	0.516 grams/ml
(in lucite turnings)	=	0.36 grams/ml

APPARATUS FOR HF REMOVAL FROM AIR STREAM STUDIES

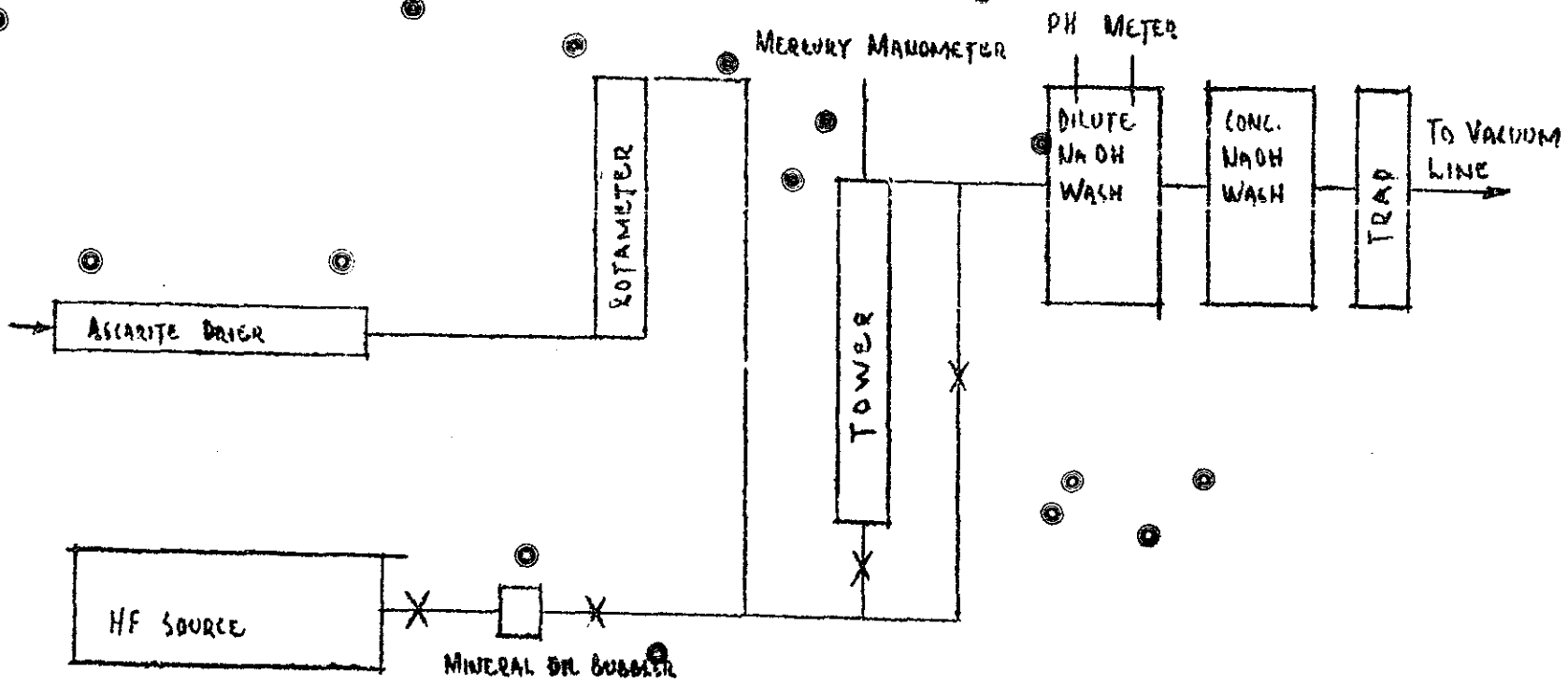

FIG. #1

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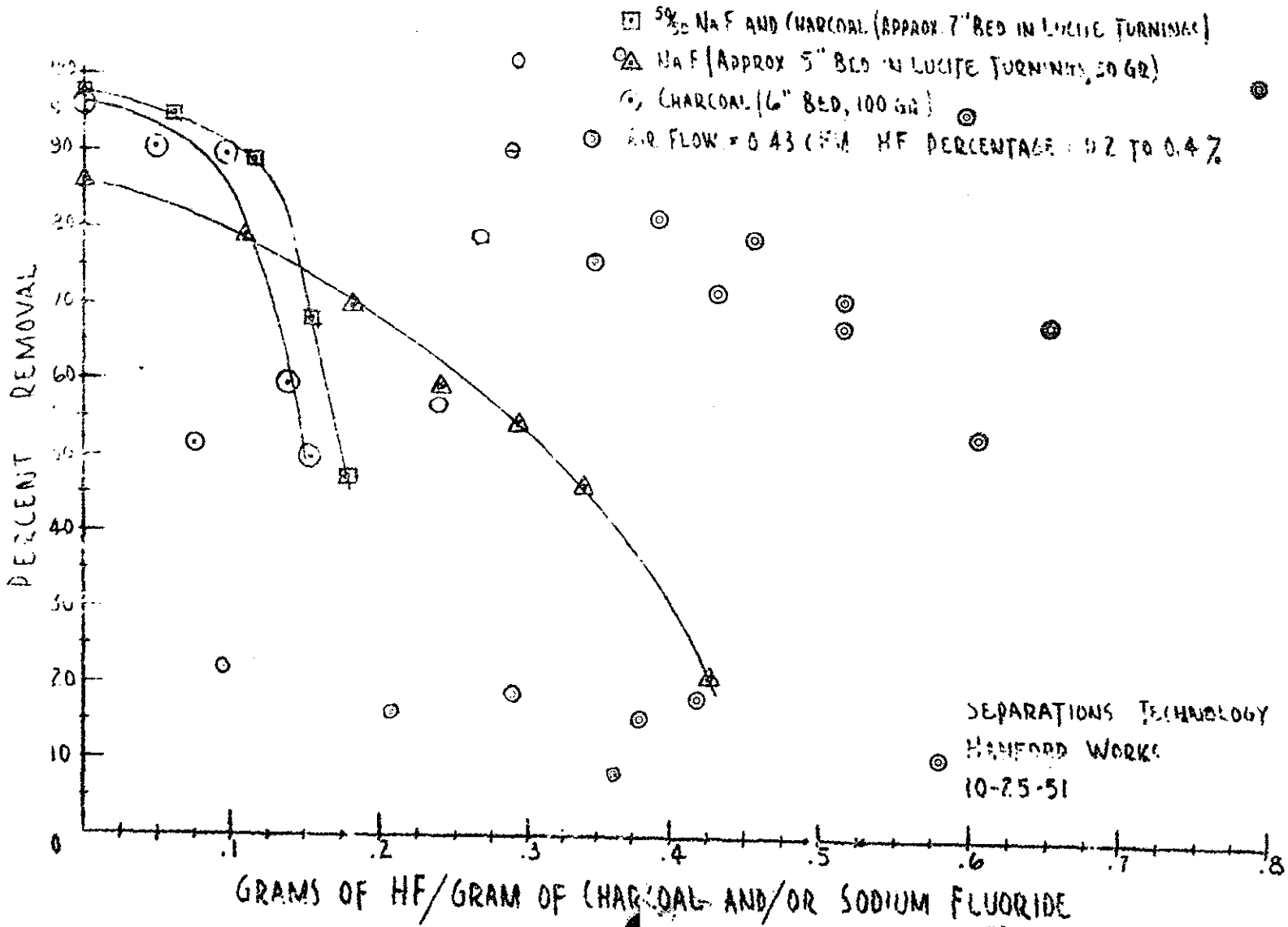
REMOVAL OF HF FROM AIR STREAM



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