

## 6. ENVIRONMENT

The aluminum industry has, of necessity, made huge strides in environmental performance over the past 50 years. The papers selected in this section document the progress in our understanding of how gaseous emissions are generated and measured, and the technologies and practices to minimize their release to the environment. They are, as the industry has been, primarily focused on gaseous and particulate fluorides, SO<sub>2</sub>, and the greenhouse gases, especially perfluorinated carbons (PFCs).

On the topic of fluoride emissions, the papers show how our understanding of the fundamentals of fluoride generation has advanced, and how this knowledge has translated to the potroom in the development of practices and technologies (for example, around cell covering and anode butt cooling). The challenges of executing highly efficient exhaust gas cleaning are highlighted. The gas treatment plant is more than just a bolt-on extra; it is an integrated component of the whole smelting process, supplying the feedstock for the reduction cells.

The issue of PFC emissions emerged in the 1980s. There is no capture technology available for PFCs, so the only solution is to prevent their generation. The remarkable progress in reducing anode effects and consequent PFC emissions can be followed in the papers presented here. This progress was enabled by the development of techniques and protocols to monitor PFCs and the industry's commitment to this initiative. New challenges are appearing: Can PFCs be generated outside of anode effect? How do we deal with the other major greenhouse gas, carbon dioxide?

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## A Study of Factors Affecting Fluoride Emission from 10,000 Ampere Experimental Aluminum Reduction Cells

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### Abstract

Results are described of experiments aimed at obtaining a better understanding of factors affecting fluoride emission from aluminum reduction cells. The general nature of fume generation is considered with the emphasis placed on the effect of cell operating variables on fluoride emission rate. The study, carried out with 10,000 amp experimental reduction cells with prebaked anodes, has shown that cell temperature, bath ratio, and alumina concentration are the most important factors affecting fluoride emission rate. Gaseous hydrogen fluoride generation results principally from the reaction of molten or vaporized bath materials with moisture in the air and hydrocarbons in the carbon anodes. Relatively little hydrogen fluoride is formed from the reaction with moisture in the alumina fed to the cells. The large number of variables involved in aluminum reduction cell operation make it difficult to establish with a high degree of certainty the correlation of operating factors with fluoride emission. This difficulty exists even with small-scale experimental reduction cells operated by research personnel.

### Introduction

The emission of fumes from the Hall-Héroult reduction cell has always been a concern of aluminum producers from the standpoint of health and comfort of the workers, air pollution, equipment corrosion, and loss of materials. A great deal of time and money has been expended both here and abroad in the area of fume recovery and air pollution abatement.

Relatively little information is found in the published literature on the subject of chemical constitution of reduction cell fumes or concerning the effect of operation variables on the emission of fumes. The relative complexity of the subject probably accounts for the fragmentary nature of many of the investigations and the amount of disagreement found.

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In order that we may best control the generation of fumes and most efficiently handle them by means of filters, cyclones, and scrubbers, it is important to understand the nature of the fumes and their generation.

The purpose of this paper is to discuss generally the nature of the fumes emitted during aluminum reduction and to describe the results of experiments aimed at obtaining a better understanding of the factors affecting fluoride emission.

### The Nature of Reduction Cell Fumes

Reduction cell fumes may be described as a mixture of gases and particulate matter which is being constantly emitted during the electrolysis of alumina in solution in fused cryolite. The particulate matter, in turn, is composed of airborne solids of very small particle size.

The gaseous phase, before coming into contact with air, consists principally of carbon dioxide and carbon monoxide which are formed by the consumption of carbon anodes during the electrolysis. Several publications have treated the subject of formation of these gases including the work of Mashovets et al.,<sup>1</sup> Pruvot,<sup>2</sup> and Pearson and Waddington.<sup>3</sup> Although the mechanism of formation is still subject to argument, it is generally known how the volume ratio of the two gases varies during the normal cycle of cell operation. The ratio of CO<sub>2</sub> to CO is decreased when the cell temperature is abnormally high and also during the brief periods when the anodes are polarized (anode effect). The carbon dioxide content of the unburned gas varies between about 60 and 85%. The balance is largely carbon monoxide. Contact of these hot gases with air results in a substantial decrease in the carbon monoxide through combustion.

Other gases are found in relatively small amounts during normal cell operation. These include sulfur dioxide (SO<sub>2</sub>), hydrogen sulfide (H<sub>2</sub>S), carbonyl sulfide (COS), carbon disulfide (CS<sub>2</sub>), silicon tetrafluoride (SiF<sub>4</sub>), hydrogen fluoride (HF), and water vapor. These species have been identified and discussed previously by the author.<sup>4</sup>

During the period of anode effect, fluorocarbons are known to be produced. These relatively inert gases consist almost entirely of carbon tetrafluoride (CF<sub>4</sub>) together with very small amounts of hexafluoroethane (C<sub>2</sub>F<sub>6</sub>). Although these compounds contain a high percentage of fluorine, they do not constitute a serious material loss because the period of anode effect is relatively short and they are not formed during normal cell operation.<sup>5</sup>

The particulate phase of reduction cell fumes may be considered as the material derived from dusting of the alumina and other raw ma-

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terials during the feeding operation of the cells and solid matter which originates from the volatilization of the fused salt bath. The greatest portion of the airborne particulate consists of alumina from dusting, but some of the alumina is formed by thermal hydrolysis of the volatilized bath materials. Carbon particles result from the mechanical and electrochemical dusting of the anodes.

Other components have been identified in the particulate matter including cryolite ( $\text{Na}_3\text{AlF}_6$ ), aluminum fluoride ( $\text{AlF}_3$ ), calcium fluoride ( $\text{CaF}_2$ ), chiolite ( $\text{Na}_5\text{Al}_3\text{F}_{14}$ ), and iron oxide ( $\text{Fe}_2\text{O}_3$ ). Elemental sulfur has been found in particulate collected in the absence of air.

No attempt was made in this work to identify the compounds in the particulate phase. Only the total fluoride content of this solid material was measured.

The ratio of gaseous to particulate fluoride in reduction cell fumes varies over a range of about 0.5 to 1.3. These values are derived from the analysis of fumes which have burned in contact with air at openings in the crust. Pure unburned fumes usually show a lower ratio of about 0.3. Burning of the hot gas-particulate mixture when it contacts air results in thermal hydrolysis of some of the particulate fluoride with the formation of additional hydrogen fluoride.

Laboratory experiments have shown that gaseous hydrogen fluoride is "picked up" by particulate matter when they are in intimate contact. Chemical reaction is responsible for some of this pickup, while some is due to chemisorption, adsorption, and absorption.

Although the determination of total fluoride content of fumes may be quite reliable, estimation of the distribution of fluoride between gas and particulate is subject to variation. This variation is due to such factors as the degree of thermal hydrolysis during burning of the gas and method of separation of gas and particulate during sampling.

### Origin of Gaseous Hydrogen Fluoride

Thermal hydrolysis of volatilized bath materials appears to be responsible for a substantial part of the hydrogen fluoride found in reduction cell fumes. This thermal hydrolysis or reaction of solid or vaporized fluorides with water vapor at elevated temperature takes place primarily at the point where the hot gases escape through vents in the crust.

Relatively pure unburned reduction cell gas which has been sampled before it has had a chance to vent through crust openings is found also to contain gaseous hydrogen fluoride. The nitrogen and argon content of this gas (mass spectrometric analysis) indicates that some air enters the reduction cell in spite of the positive gas pressure.

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Moisture contained in this air is quite insufficient, however, to account for the amount of hydrogen fluoride found.

Obviously a source of hydrogen is necessary for the generation of hydrogen fluoride. Water vapor in the air is a contributor of part of this hydrogen. Other sources include residual moisture in alumina and bath raw materials and hydrocarbons in the carbon anodes.

A simple calculation can show that sufficient moisture is introduced in the alumina to more than account for the hydrogen fluoride generated.

### Experimental Procedure

The study reported herein was carried out by the employment of experimental prebaked anode reduction cells of 10,000 amp capacity. These cells were well suited for the study of fluoride emission since it was possible to exercise much better control over their operation than would be possible with larger production units. The cells were powered by individual rectifiers with powerstat controls. These cells were essentially the same in design as large production cells except for physical size and amount of thermal insulation.

### Continuous Fume Collection

Cell gases were allowed to escape from a small hole located in the crust in the geometrical center of the cell. All alumina and bath materials were fed through this hole. A hood and blower assembly removed the fumes to a cyclone and baghouse filter and finally to a water scrubbing tower. By means of this arrangement and the chemical analysis of solids and scrubber liquid, it was possible to determine the fluoride emission with reasonable accuracy over periods of time from a few days to two weeks.

### Differential Fume Sampling

In order to study the effect of certain reduction cell variables on the rate of emission of fluorides in pure unburned fumes, a special technique was developed. By this method it was possible to sample sufficient gas and particulate in one minute for a satisfactory determination of fluoride content.

Pure cell gas samples were taken by use of an Inconel funnel attached by an interrupted-screw coupling to a water-cooled Inconel pipe 2 in. in diameter and approximately 3 ft long. At the opposite end of this condenser a second interrupted-screw coupling allowed rapid connection and disconnection to an electrostatic precipitator and a large polyethyl-

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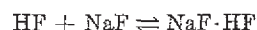
ene gas-collection bag. The gas-collection bags were fabricated from 0.006 in. polyethylene tubing, 36 in. in width. The tubing was sealed into a flat gas bag by use of an ordinary household electric iron. The bags' capacities were about 1 to 2 cu ft.

Gas samples were taken by placing the 5-in. diam Inconel funnel over the hole in the cell crust and carefully covering it with alumina or powdered cryolite. Gas was allowed to pass through the assembly for about 30 sec before the empty bag was attached. Within 1 min sufficient gas was collected in the bag and could be removed to the laboratory for analysis.

At the same time the funnel, condenser, and precipitator assemblies were removed from the cell and the particulate matter removed with a small brush for chemical analysis. Tests showed that the separation and recovery of particulate was nearly quantitative.

## Fluoride Analysis

Removal of hydrogen fluoride from the gas contained in the polyethylene bag was accomplished by chemisorption on sodium fluoride pellets. Hydrogen fluoride is quantitatively chemisorbed by sodium fluoride at room temperature and desorbed at elevated temperature,



The gas was pumped through two Inconel U-tubes in which was located a bed of sodium fluoride pellets. A sigma motor was employed to pull the gas from the bag through the tubes and finally through an oil-filled wet test meter for volume measurement. Half a cubic foot of gas was ordinarily sufficient for accurate analysis.

The hydrogen fluoride was then desorbed from the sodium fluoride into a stream of pure dry argon at 270–350°C which was passed through absorption tubes containing standard alkali solution. The fluoride was then determined directly by titration or by the Willard and Winter method. Any silicon tetrafluoride was removed from the gas by the sodium fluoride but was not desorbed below about 500°C.

The particulate matter was analyzed for fluorine content by the Willard and Winter procedure.

Extensive laboratory tests showed that the system was highly satisfactory for the purpose of fluoride sampling and analysis. The polyethylene film bag was not attacked by the gases. Sodium fluoride did not react with any of the other gaseous components.

Carbon dioxide, carbon monoxide, oxygen, and nitrogen were determined on gas samples collected in a glass bulb.

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This system made it possible to collect nearly the entire output of pure fume from the 10,000-amp cell (about 1.4 ft<sup>3</sup>/min at standard temperature and pressure) during the 1-min sampling period. Replication on successive samples was very good.

## Effect of Cell Temperature, Bath Ratio, and Alumina Concentration on Total Fluoride Emission

A total of 100 samples was taken from an experimental cell and analyzed for gaseous fluoride only. Sixty-six samples taken were analyzed for both gaseous fluoride and particulate fluoride. Cell temperature was measured during each run, and bath samples were taken for chemical analysis.

During this test the cell temperature varied from 956 to 991°C, the alumina concentration from 1.2 to 6.1%, and the bath ratio (weight ratio NaF:AlF<sub>3</sub>) from 1.19 to 1.88. The variation in temperature and alumina concentration was normal for cell operation, but the extreme ratio variation was brought about deliberately.

The data were analyzed statistically by both multiple linear correlation (mathematically) and multiple curvilinear correlation (graphically).

Approximately 75% of the variation in fluoride emission was found to be correlated with the three variables considered. Although bath ratio appears to have the greatest effect on gaseous fluoride emission, all three variables are equally important with regard to particulate fluoride. Generally, the fluoride emission rate is increased by increased temperature, decreased bath ratio, and decreased alumina concentration.

Table I illustrates the calculated change in total fluoride emission rate brought about by variation in cell temperature, bath ratio, and alumina concentration. The ratio of gaseous fluoride to particulate fluoride for the unburned fume samples averaged about 0.3.

TABLE I  
Effect of Cell Temperature, Bath Ratio, and Alumina Concentration on Total Fluoride Emission

Change in cell variable	Total fluoride emission
Bath ratio 1.44–1.54 (4% Al <sub>2</sub> O <sub>3</sub> , 975°C)	31% decrease
Alumina 3–5% (1.50 ratio, 975°C)	20% decrease
Temperature 982–972°C (1.50 ratio, 4% Al <sub>2</sub> O <sub>3</sub> )	24% decrease

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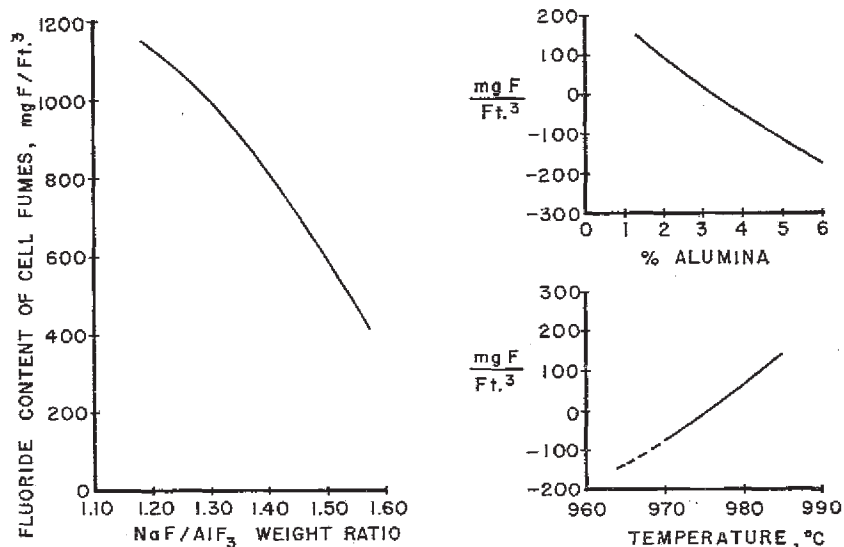


Fig. 1. Graphical relationship of total fluoride emission as a function of bath ratio with corrections for temperature and alumina concentrations.

Figure 1 shows the graphical relationship between total fluoride emission and bath ratio, together with the graphical correction for alumina concentration and cell temperature.

TABLE II  
Comparison of Calculated and Measured Fluoride Emission from Experimental Cells

No. of 4 week test periods	Bath ratio, avg	Alumina avg, wt-%	Temp. avg, °C	Total fluoride emission, (lb F/lb Al)	
				Calc.	Measured
9	1.50	2.4	975	0.020	0.019
4	1.34	2	976	0.030	0.024
4	1.20	2	973	0.034	0.029
3	1.49	4.9	971	0.015	0.015

Fluoride emission calculated from these data for the 10,000-amp cells compared well with values measured by means of the continuous fume collection system. Table II shows the comparison of calculated and measured fluoride emission for several four week test periods during which bath ratio and alumina concentration were held at different values.

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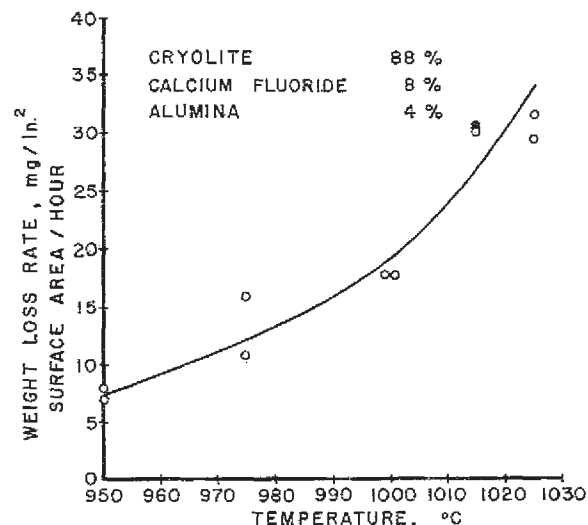


Fig. 2. Relative volatility of molten reduction cell bath as a function of temperature.

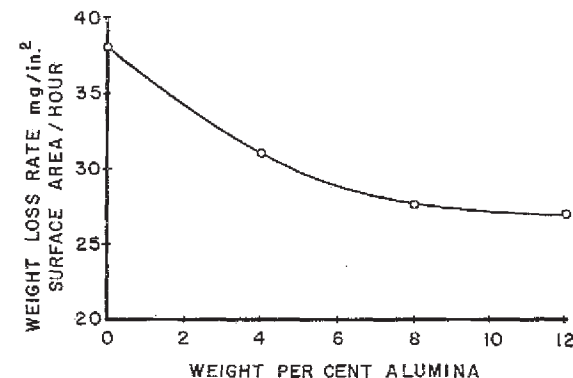


Fig. 3. Relative volatility of molten cryolite as a function of alumina concentration (1015°C).

The effect of temperature, ratio, and alumina concentration on actual fluoride emission is in good agreement with laboratory measurements of relative volatility of fused reduction bath. Dry argon was passed through a sealed Inconel canister containing an open graphite crucible of molten bath. Weight losses were carefully determined in various experiments in which the temperature, alumina concentration, calcium fluoride con-

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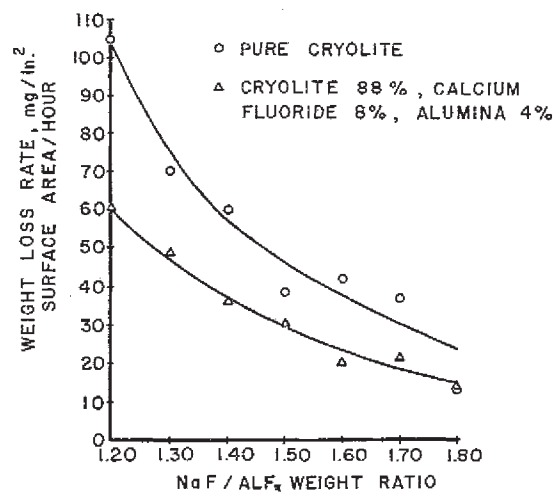


Fig. 4. Relative volatility of molten cryolite and reduction cell bath as a function of NaF/AlF<sub>3</sub> weight ratio (1015°C).

centration, and ratio were varied. Figures 2, 3, and 4 illustrate the effect of these variables on the volatility. The volatility is a relative measure expressed in milligrams weight loss per square inch of melt surface per hour.

#### Effect of Moisture in Alumina on Gaseous Fluoride Emission

The Bayer alumina fed to aluminum reduction cells varies greatly in moisture content. Laboratory studies revealed that most of this moisture is adsorbed and may be removed by heating to 200°C. Some of the water is held chemically by the residual sodium carbonate (expressed usually as Na<sub>2</sub>O) and requires a slightly higher temperature for removal. Water continues to be evolved from the alumina at temperatures up to 1500°C, but relatively little above 1000°C.

It is customary to preheat the alumina on the bath crust prior to breaking it into the bath. Temperature measurement of the alumina on the crust shows it to vary from 430 to 550°C in prebaked anode cells and 410 to 615°C in Soderberg anode cells. On the basis of moisture loss curves for various lots of Bayer alumina, the water content of the alumina just prior to break-in ranges from 0.2–0.5 wt-%. Should all of this water react with the molten bath to produce hydrogen fluoride, a total of 15 lb of HF per cell-day would be generated by a 60,000-amp

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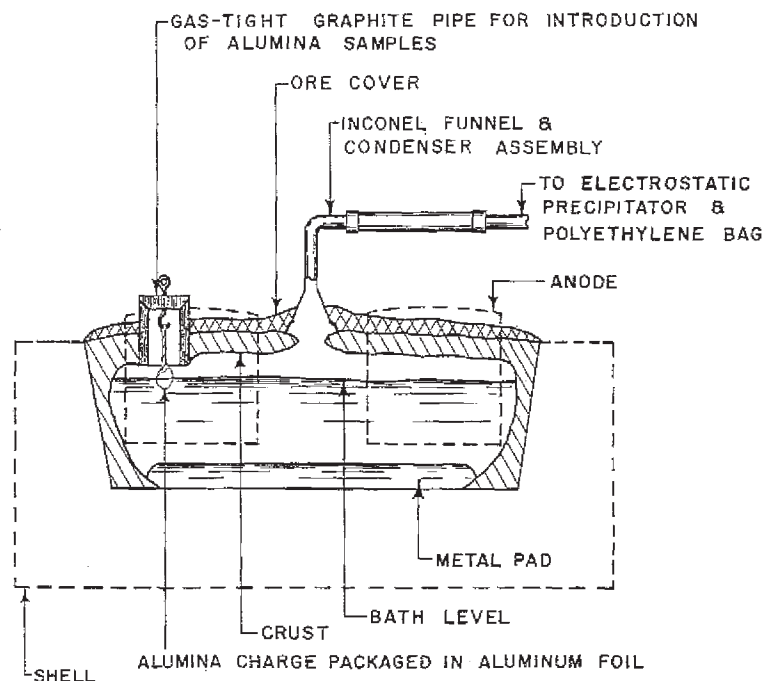


Fig. 5. Experimental arrangement for measurement of gaseous fluoride emission rate during the feeding of Bayer alumina.

cell (0.018 lb F/lb Al). Fluoride emission measurements of gaseous HF (0.006 to 0.009 lb F/lb Al) show that this reaction is far from complete.

Experiments were conducted with the 10,000-amp cell to determine the relative importance of moisture content of alumina on gaseous fluoride emission. The experimental arrangement is shown in Figure 5.

A graphite tube with tight-fitting cap was sealed into the crust for the introduction of weighed amounts of Bayer alumina of known moisture content. Gas samples were taken with the Inconel funnel-polyethylene bag assembly just before, during, and just after the addition of alumina packaged in alumina foil.

Water was expelled rapidly when the cold alumina was submerged beneath the bath surface. When a 1 lb charge of alumina was employed, several grams of water condensed in the sampling assembly. Fifty gram charges of alumina resulted in a less violent evolution of steam, but the water vapor evolved made it necessary to employ a wet method of gas collection. In this connection, 200 cu cm of distilled water was placed

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in the polyethylene bag. After the gas was collected, the water was shaken to dissolve the hydrogen fluoride. The gas was then removed for volume measurement and an aliquot of the solution analyzed for fluoride by the Willard and Winter method.

In each test a blank was run in which aluminum foil alone was introduced into the bath. Approximately 5 min later when the collection system was cleaned and reassembled, the sample of alumina wrapped in foil was introduced and the gas was collected simultaneously. After about 40 sec the first polyethylene bag was replaced by a second and later by a third. In this manner it was possible to determine the normal evolution rate and the increased rate during the first, second, and third 40 sec interval. The rate was higher during the first 40 sec but closely approached the normal rate within the second or third period.

The results of these tests are shown in Table III. It is seen that the water in the 50 g charges of alumina reacts to the extent of about 5% on the average.

In a second test both gaseous and particulate fluoride emission was measured by the continuous fume collection system during four week test periods in which alumina of different moisture contents was fed to the cell. During one test period hot calcined alumina was fed directly

TABLE III

Increase in Gaseous Fluoride Emission Caused by Moisture in Alumina

Alumina employed	Moisture content of alumina, mg H <sub>2</sub> O/50 g	Excess HF <sup>a</sup> produced, mg F		
		Theoret.	Measured	Theoret., %
Sample C (as received)	1405	2970	109	3.7
Sample A (as received)	1010	2130	136	6.4
			116	5.4
			96	4.5
			97	4.6
Sample A (preheated to 200°C)	290	612	26	4.2
			25	4.1
			58	9.5
			37	6
Sample A (preheated to 1000°C)	90	190	1	0.5
			6	3.2

<sup>a</sup> Excess HF above normal emission rate expressed as milligrams of fluorine.

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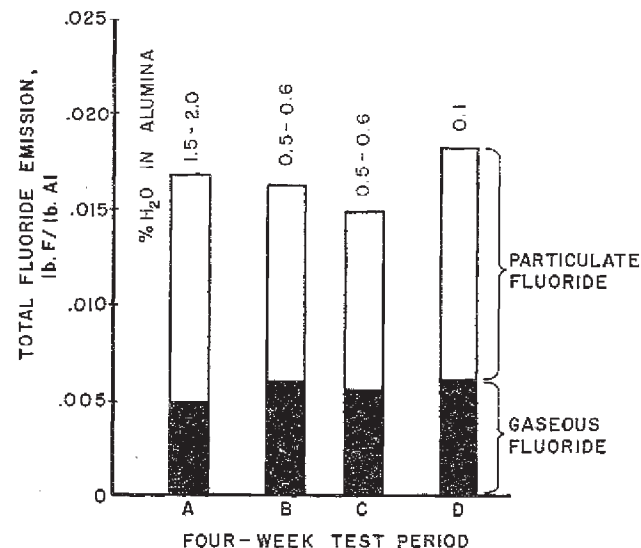


Fig. 6. Gaseous and particulate fluoride emission as influenced by moisture content of alumina.

to the cell. The alumina was calcined at 700°C by passage through a vertical-tube furnace into Inconel buckets where it was held at this temperature by a Globar furnace until it was fed. The results of these tests are given in Figure 6.

Test periods B and C were carried out with the same alumina in exactly the same manner. Data from these test periods illustrate the degree of reproducibility of the system under as nearly identical conditions as were able to be attained. The two runs differ by about 10%. This difference reflects all variations in cell operation, fume recovery, sampling, and analysis. A variation this large would mask the difference in fluoride emission caused by alumina of different moisture contents if the water reacted only to the extent of about 5%.

No difference is seen in fluoride emission although a twentyfold difference in moisture content of alumina was employed. These data appear to confirm the results of the previously described test.

A third test was conducted during the same time that alumina of various moisture levels was being routinely fed to the cell. In this test, 16 differential gaseous fluoride samples were taken during the test period in which alumina of 1.5 to 2% moisture content was being fed, and 12 samples taken during the period of feeding the hot calcined alumina.

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TABLE IV  
Effect of Moisture in Alumina on Gaseous Fluoride Emission Several Minutes After Feeding

Alumina employed	Moisture content, wt-%	No. of samples taken	Gaseous HF, mg F/ft <sup>3</sup>
Sample C (crust heated)	1.5-2	16	107
Sample D (precalcined and fed hot)	0.1	12	101

In each period samples were taken 15 min. before and 15 min. after the hourly alumina feedings.

The purpose of this test was to determine if any significant difference in fluoride emission could be detected several minutes after the moisture was expelled from the alumina.

Data in Table IV show that no significant difference was found.

#### Effect of Atmospheric Humidity on Gaseous Fluoride Emission

The effect of atmospheric humidity on hydrogen fluoride formation was studied on two 10,000 amp. experimental cells during a continuous five-

TABLE V  
Gaseous Fluoride Emission as a Function of Atmospheric Humidity

Test period	Avg gaseous fluoride emission, mg F/ft <sup>3</sup>	Bath ratio	Avg humidity, grains H <sub>2</sub> O/ft <sup>3</sup>
A, 1st week	135	1.49	5.03
A, 2nd week	137	1.49	2.56
B, 1st week	206	1.31	2.49
B, 2nd week	203	1.31	3.34
B, 3rd week	199	1.31	3.46
B, 4th week	209	1.31	2.07
C, 1st week	231	1.29	2.29
C, 2nd week	226	1.29	2.94
C, 3rd week	150	1.29	2.36
C, 4th week	224	1.29	2.38
D, 1st week	283	1.34	3.28
D, 2nd week	363	1.34	3.68
D, 3rd week	241	1.34	3.75
D, 4th week	279	1.34	3.86

month period. Humidity measurements were made by a psychrometer three to four times each 8 hr. shift and the hydrogen fluoride evolution measured by the analysis of scrubber brine from the continuous fume collection system.

The absolute humidity values were converted to grains of water per cubic foot of air and averaged for each week.

Some typical data are shown in Table V. Since other variables were introduced each test period, one may compare only the fluoride emission and humidity for the weeks within a given period. It is obvious that no correlation can be found. Unfortunately the range of humidity is small. Even if the range were larger, it is likely that any relationship would be obscured by other cell variables.

#### Effect of Hydrogen (Hydrocarbons) in Anode Carbon on Gaseous Fluoride Emission

Preliminary tests were conducted to determine what correlation might exist between gaseous fluoride emission and hydrogen content of pre-baked carbon anodes. Differential gas samples were taken during test periods in which anodes of slightly different hydrogen content were employed. One set of anodes was baked at 960°C and contained 0.08% hydrogen. The other set was baked at 1069°C and contained 0.06% hydrogen. The tests indicated no significant difference in the gaseous fluoride emission.

A second test was performed in which graphite anodes procured from the National Carbon Co. were used. These anodes contained only 0.01% hydrogen. Use of the graphite anodes made it possible to study a

TABLE VI  
Fluoride Emission Rate as Influenced by Hydrogen Content of Anodes

Cell	No. of samples	Anodes used, H, %	Ratio	Al <sub>2</sub> O <sub>3</sub> , %	Temp., °C	Fluoride emission, mg F/ft <sup>3</sup>		
						Gas-eous	Partic-ulate	Total
A	30	Carbon 0.06-0.08	1.48	4.23	976	130	504	634
C	12	Carbon 0.06-0.08	1.49	2.13	975	146	473	619
C	25	Graphite 0.01	1.46	3.09	971	65	345	410



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much wider difference in hydrogen content, the effect of which would be less likely to be masked by other variables.

Differential gas samples were taken at various time intervals while the cell was operating with the graphite anodes. The results are given in Table VI.

From these data it is seen that the evolution rate of gaseous hydrogen fluoride was substantially less when graphite anodes are employed. The rate was reduced to approximately half that of the regular carbon anode test periods. The decreased rate of particulate fluoride emission is unexplained.

### Summary

Determination of the effect of operating variables on the fluoride emission from aluminum reduction cells is difficult to accomplish with a high degree of certainty. This is true even with small-scale experimental cells operated by research personnel. It appears from the work reported here, however, that cell temperature, bath ratio, and alumina concentration are the most important variables affecting total fluoride emission.

Generation of gaseous hydrogen fluoride results primarily from the reaction of liquid and vaporized bath materials with hydrogen from the hydrocarbons in the carbon anodes and moisture in the air. Relatively little hydrogen fluoride can be attributed to the reaction with moisture contained in the alumina fed to the cells.

### References

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### Discussion

E. A. Hollingshead (*Aluminium Labs., Arvida, Quebec, Canada*): Our experience has shown that small quantities of gaseous HF are readily taken up by various solids. Is the author sure that appreciable percentages of HF were not lost in the Inconel piping and polyethylene bags used for collecting the unburned cell gases?

J. L. Henry: The gas was passed at high speed through an electrostatic precipitator rather than through a filter to minimize removal of HF by the particulate matter. Nevertheless, it is difficult to obtain reliable results for the ratio of gaseous to particulate fluoride. The results for total fluoride loss are more reliable.