

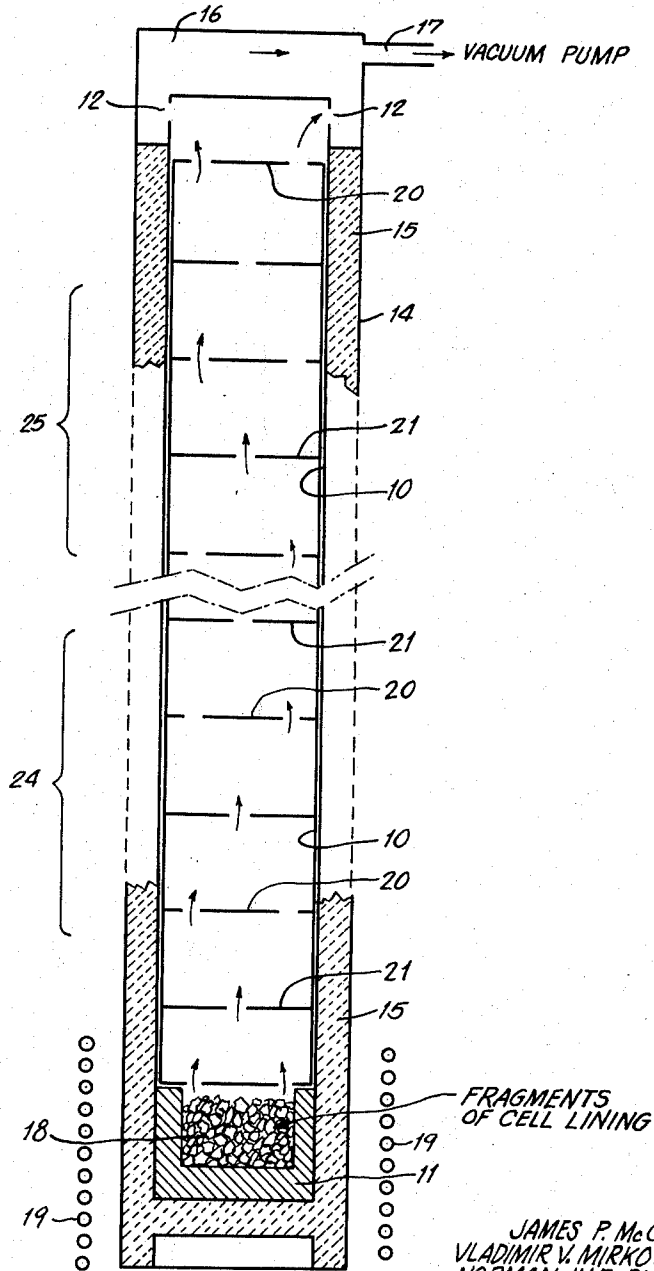
Oct. 28, 1958

J. P. McGEER ET AL

2,858,198

RECOVERY OF MATERIAL FROM ALUMINUM REDUCTION CELL LINING

Filed Oct. 19, 1954



BY Robert S. Drubshaw

ATTORNEY

1

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RECOVERY OF MATERIAL FROM ALUMINUM REDUCTION CELL LINING

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Application October 19, 1954, Serial No. 463,214

8 Claims. (Cl. 23—294)

This invention relates to the treatment of aluminum reduction cell linings for the recovery of valuable materials that have become contained in such lining during the operation of the cell.

Aluminum reduction cells are those which are employed for production of metallic aluminum by electrolysis of a fused bath containing fluoride or fluorides, i. e. conventionally cryolite, and alumina. In operation, electric current is passed through such cells from carbon anodes which project downwardly into the bath, to the carbon lining, which constitutes the cathode of the cell and in which steel or other collector bars are embedded to provide means for conducting electric current to the lining. The aluminum metal collects in molten form in the bottom of the cell, being drawn off from time to time. Fresh quantities of alumina for reduction are progressively added, and likewise as necessary additional cryolite or other fluoride to maintain the desired molten and other characteristics of the electrolyte.

The innermost lining of such cells, directly receiving the molten bath, is conventionally composed essentially of carbon, e. g. as may be derived from appropriately pure or purified coke and carbonaceous binders such as tar or pitch, in a layer of substantial thickness, such as 10 to 20 inches. The carbon lining may be and in modern practice invariably is surrounded by an outer layer of material having thermal insulating properties, e. g., insulating brick or granular alumina, bauxite or magnesite, the inner carbon lining being originally deposited or constructed as a coherent, solid mass, and the insulating layer in a manner appropriate to the particular material used. The carbon lining and insulation are contained in an appropriate steel or other shell.

It has long been known that in the course of operation of such cells, which may be in service continuously for periods of one to several years, considerable amounts of combined fluorine, chiefly as fluorides of aluminum and sodium, become contained in the lining, by absorption or similar accretion from the molten bath. Sooner or later, each reduction cell becomes unserviceable, usually by reason of cracking or deterioration or other failure of the lining at one or more localities, so that use of the cell must be interrupted and its contents and lining removed for ultimate replacement of a new lining and restoration of the cell to service.

Heretofore when it has been found desirable or economical to recover the contained fluoride material from the used lining of a cell that has been taken out of service for reconstruction, conventional practice has been to grind the used lining to a fine condition and then subject it to a wet extraction process, i. e. leaching with aqueous solution of sodium hydroxide or sodium aluminate, the aluminate solution being, for instance, some of the caustic liquor taken from an alumina-producing system utilizing the Bayer process.

With such leaching, it is possible to separate a considerable amount of fluoride from the used lining, but the process is long and rather costly in a number of respects. In the first place, the lining material, which is commonly

2

called pot lining and may sometimes be so identified herein, must first be comminuted to a very fine state, e. g. usually smaller than 100 mesh, in order to obtain reasonably satisfactory dissolution of the fluoride material in the leach liquor. In the second place, actual recovery of such fluoride from the liquor is difficult or inconvenient, for example in requiring evaporation or other treatment to precipitate the fluoride as cryolite. When so separated, the cryolite precipitate is usually in an inconveniently fine state of subdivision and may carry with it considerable impurity which was also leached from the pot lining. Some purification steps are then often necessary. Eventual drying and calcining of the separated fluoride material is required if it is to be reintroduced to the aluminum producing process.

To the end of providing an improved and more economical process of recovering valuable materials from aluminum reduction cell lining, the present invention embraces the discovery that such material, especially the desired fluorides, may be recovered by distillation, specifically by breaking up the old lining into coarse pieces and then heating it in an appropriate crucible or furnace to an elevated but conveniently attained temperature, viz. in the neighborhood of 1000° and preferably upwards thereof, under subatmospheric pressure. By such step, it is found that valuable fluorides are volatilized from the lining, yielding a vapor which contains them, usually, as a mixture of cryolite and sodium fluoride. Conducted away from the heating region under the stated pressure, the vapor is passed to a condensing region, where the fluorides condense and can be removed in a dry, highly pure and physically convenient state.

A particularly important feature of the invention is the discovery that by such process the produced vapor contains not only the volatilized fluorides, but generally also metallic sodium in vapor form. Thus the procedure more specifically comprises the conduction of the vapors, i. e. of fluorides and sodium, through the condensing region where successively lower temperatures are maintained, along the path of vapor travel. By maintaining a zone of relatively high condensing temperature in the first part of such path, all of the fluoride is condensed while the sodium remains in the vapor state, and by maintaining a future zone of the condenser at a relatively low temperature, at least sufficiently low for condensation of sodium, this metal is thus condensed, separately from the fluorides, in a relatively very pure condition.

It is found that for effective volatilization and separation of the valuable absorbed material, the pot lining need not be crushed or ground to any fine state of subdivision; relatively coarse or even very large pieces can be efficiently treated, so that the expensive grinding step of the prior wet method is avoided. At the same time, the desired fluorides are recovered in a highly pure form and in an entirely dry, water-free state. A higher actual recovery of fluorine from the pot lining has been found achievable than with the usual wet processes. Finally, the separate recovery of substantially pure sodium metal represents a particularly new aspect of the process, the quantities of sodium thus produced being relatively substantial and suitable for use or sale.

Practice of the process is essentially simple and can be performed in various types of equipment. The pot lining from one or more used cells is first subjected to rough crushing or other operation to break it up into coarse pieces, the maximum size of which will depend upon the nature of the pot lining and on the rapidity with which it is desired to recover the absorbed materials. Thus in some cases the pot lining may exhibit numerous small cracks and fissures which permit rapid escape of volatile materials from relatively large lumps while in other cases the pot lining may be relatively

dense, thus requiring crushing to a smaller size to permit the same rate of removal of volatiles. However, in any case the particle size may be many times that required by the wet extraction method and in general need never be smaller than an average diameter of about 2 inches. In especially favorable cases the lining may be treated in blocks of a foot or more in each dimension. Fine crushing can be employed if desired, but evidence indicates that there is ordinarily no advantage in so doing and, owing to the expense of crushing, the size used will ordinarily be the largest which will give an adequate rate of vaporization.

In its broken-up state, the pot lining is introduced into a suitable furnace, which is closed except for connection to a condenser. The system has appropriate means for maintaining a sub-atmospheric pressure, as with a suitable vacuum pump connected to the remote or furthest end of the condenser whereby the pressure in the entire system, including the heating zone of the furnace, is kept at the desired value and the desired conduction of vapors from the furnace into and along the condenser is achieved. The pot lining material is then heated in the furnace in any of various ways, e. g. by electrical induction, or by passing electric current through the charge between electrodes extending into it, or by combustion of suitable fuel in regions surrounding the crucible or the like in which the charge may be placed.

As will be appreciated, the pressure to be maintained for effective volatilization depends on the vapor pressure of the materials being distilled and thus varies with temperature, being about 0.001 atmosphere when the charge is only heated to about 1000° C., and ranging up to 0.1 atmosphere for distillations at about 1400° C. (being of the order of 0.01 atmosphere for operations at 1200°), somewhat higher pressures, but still preferably sub-atmospheric, being usable at higher temperatures, such as 1500° C., or above. Thus it will be understood that at increasing temperatures, higher vapor pressures are achieved and more rapid vaporization results. However, energy losses are excessive at high temperatures and fluoride materials are corrosive to practically all construction materials. Since the difficulties in these respects increase rapidly as temperature is increased, it will generally be found advisable to maintain temperatures within the range from about 1000° C. (i. e. not substantially below such value) to about 1500° C., and preferably at 1050° to 1300° C. Within this range evaporation rates increase with increasing temperature but so do energy losses and costs for maintenance of equipment. At temperatures substantially below 1000° evaporation rates are, in general, too slow to be practical while above 1500° C. energy losses and deterioration of equipment are apt to be excessive. In any given instance a satisfactory balance between the two factors, depending on the specific conditions in existence, can readily be struck at some temperature which falls in the above stated range; indeed special advantage will usually accrue by operation in the range of 1050° to 1300° C.

The total time of treatment for production of the desired vapor, i. e. in order to secure optimum recovery of fluoride, varies somewhat with the temperature and pressure conditions, longer times being usually required at lower temperatures. In general, distillation times of the order of one-half hour to several hours appear entirely adequate. From the furnace, where the described vapors are evolved throughout the heating period, such vapor is passed, e. g. by simple conduction under the influence of the exhaust pump, into and through a condensing region, which may consist of an elongated chamber having appropriately cooled walls and having its effective length, if desired, increased by appropriate baffles or the like.

As will now be understood, temperature conditions in the condenser are suitably correlated with the temperature of volatilization and the pressure, so as to achieve

the desired condensation of the vapor, essentially in its entirety. Thus the condenser is conveniently arranged so that throughout the first part of its wall and other structure traversed by the gas, say about one-half of the total extent, the fluorides are condensed but essentially none of the sodium. In the further part of the condenser, where the vapor contains essentially only sodium, the temperature is kept considerably lower (though in most cases preferably above the melting point of sodium, 98° C., with resulting condensation of the metallic sodium.

At the end of the run, the condenser is opened and the distillates removed, that in the first zone is generally found to consist essentially of cryolite (sodium aluminum fluoride) and sodium fluoride, the complex fluoride usually predominating in amount. The metallic sodium is removed from the further region of the condenser, with appropriate precautions to keep it dry and avoid excessive contact with air. Actual removal of the distillates can be accomplished in any suitable way as by mechanical scraping or the like. Alternately, if desired, the recovery process may be made continuous by provision of means well known in themselves, such as pressure locks, for the addition of pot lining to the furnace and for the removal of spent pot lining and recovered fluorides and sodium, without interruption of distillation.

The efficiency of the process and production of the desired fluorides is unusually satisfactory, an important feature being that they are ordinarily recovered in a relatively pure state in the condensate, and with little or no oxide or moisture, as is encountered in the wet recovery process. In general, with the process, there is no difficulty in recovering about 90% or more of the total fluoride contained in the lining.

The production and separate recovery of metallic sodium is an unexpected and particularly valuable attribute of the process. Experimentation following this discovery tends to indicate that such sodium is present in the used pot lining in a combined form, most probably as sodium oxide or sodium carbonate. Although the invention is not limited to any particular theory, a likely hypothesis is that under the conditions in the furnace, the sodium of the compound or compounds named is reduced to metallic form by the instrumentality of the carbon or of a carbide, such as aluminum carbide, which may be present. Since it is noted that substantial amounts of metallic sodium are produced from those portions of pot lining which are essentially composed of alumina (i. e. the thermal insulating portions outside the heavy carbon layer), it would appear that there is sufficient carbon present from the original association with the carbon layer, or alternatively or perhaps additionally sufficient carbide present (in the alumina layer), in order to achieve the stated reduction. Indeed during the life of a reduction cell, if reasonably long, the insulating layer tends to lose its identity and to form a more or less monolithic block with the carbon layer, characterized by some mutual penetration of the respective materials. Thus while a large concentration of carbon usually remains adjacent the inner surface, certain amounts of carbon are almost certain to be present even in lining portions near the outer surface.

The process in its most complete form, i. e. including the recovery of sodium as well as fluorides, is therefore applicable to each of the more or less distinguishable parts of the reduction cell lining, namely the insulating portion and the carbon portion. Hence for any given run of the process, the furnace charge may consist of either one of those portions alone, or alternatively may consist of an unsorted mixture of the parts of the lining, e. g. such as might be obtained by appropriately breaking up the entire lining of a single pot into a unitary mass of unclassified pieces.

In some cases refractory brick of a silica-containing type, or other silica-containing material is employed for cell lining purposes, e. g. in regions of thermal insu-

lation outside the necessary interior carbon lining. For example, in some cases bottom insulation for aluminum reduction cells has been made of such refractory brick instead of alumina-type material, e. g. bauxite or pure alumina. While distillation of this material of high silica content from a used pot lining has been found to yield a fluoride condensate, the fluoride content of such material is usually low and experiments tend to indicate that a considerable contamination of silica (or equivalent combined silicon) will then appear in the condensate, while recovery of fluoride is considerably poorer than in treatment of the more usual pot lining materials mentioned above. These effects, particularly the lowered recovery, may in part be due to the fact that this silica-containing refractory fuses in the furnace or crucible, and it appears that the present process is less than completely suitable when the sole material under treatment is a silica-containing portion of refractory material. While somewhat better results are achieved where the last-mentioned material is mixed with carbon lining (the furnace charge being a mixture of pieces of both portions of the complete lining) in that the silica contamination of the fluoride distillate is somewhat reduced from that found in using the refractory brick material alone, the presence of silica in material to be treated is generally to be avoided. Such silica-containing material can ordinarily be segregated by simple mechanical means.

By way of example, some specific operations in accordance with the invention are described below, representing actual distillations with various examples of pot lining, for recovery of fluorides and metallic sodium.

Referring to the drawing, the figure is a diagrammatic illustration, as in vertical section, of one arrangement of a crucible, including evaporator and condensing section, that has been successfully employed.

Structural details are omitted from the drawing, for the sake of simplicity and because they may obviously assume a variety of forms. The apparatus essentially comprises an upright, cylindrical, inner shell or vessel 10 having a cup-shaped evaporator section 11 at the bottom and openings 12 at the top for withdrawing permanent gas, i. e. gases that may be present or may be released by the treated material and that are not condensed at normal temperature and pressure, such as hydrogen, oxygen, carbon dioxide or the like. The vessel 10 is enclosed within an appropriate outer housing 14, the space between the latter and the inner shell 10 being filled with insulating material 15, say of a refractory nature such as granular zirconia, or other divided or cohered refractory material. At the top, the openings 12 communicate with a head chamber 16, having a connection 17 to a vacuum pump (not shown) by which air may be exhausted from the system and the desired, reduced pressure maintained during operation. The reduction cell lining material 18 to be treated, is disposed in the evaporator 11. Heat is applied by suitable means such as electrical induction; for instance, the lower part of the apparatus may be surrounded by a water-cooled coil 19 through which high frequency electric current is conducted from an appropriate source (not shown).

To facilitate condensation the interior of the vessel 10, above the evaporator 11, may have a number of horizontal baffles 20, 21, apertured to afford a tortuous path for the vapor drawn from the material 18. Although in some cases mechanical scraping devices or the like may be permanently installed for periodic collection of distillate from the interior surfaces of the condenser, simple forms of the apparatus may be appropriately designed, as in a number of sections, to permit disassembly for recovery of the deposited material. For instance, the baffle plates 20, 21 and appropriate spacing sleeves between them, can be made to be removable, when the vessel is opened.

In the various procedural examples below, apparatus 75

of the sort shown was utilized, having an inside diameter of about 3.5 inches. In one instance, the height of the condensing section, above the evaporator, was about 18 inches, and in another instance, affording somewhat better results, was about 28 inches. The evaporator section 11 was advantageously made of steel, particularly stainless steel. Although useful results were obtained with a condensing section of graphite, particularly satisfactory operation, including ready removal of distillate, was achieved when the various parts of condensing section 10 were made of steel. The horizontal baffles (20, 21) were spaced about two inches apart in all cases below. In one particularly effective arrangement, the baffle plates 21 had a central opening about 0.5 inch in diameter and the other plates 20 had an annular row of six holes each about 0.25 inch in diameter.

In operation, the evaporator 11 is charged with appropriate pieces of pot lining, and heat is applied by induction from the coil 19. Under the influence of the vacuum pump, air is essentially removed from the system, and as and after the temperature for evaporation is reached, continued pumping removes permanent gases which are evolved, and maintains the selected low pressure in the system.

Many runs have been made with apparatus such as described, utilizing pot lining material either in large pieces or crushed to a coarse state of subdivision, e. g. of the order of $\frac{1}{4}$ inch down to 28 mesh (openings per linear inch).

Temperatures stated hereinbelow generally represent the approximate temperature of the charge itself during evaporation, i. e. as measured by thermocouple extending into the charge. Thermocouple measurements at the bottom of the steel evaporator section 11 showed somewhat higher temperatures in all cases, i. e. usually about 60° to 80° C. more.

In one operation, a 100 gram charge of pot lining material containing 14.6% fluorine by weight was heated to 1125° C. for 15 minutes. In this test it was found that 89% of the fluorine in the pot lining was evaporated, such determination being conveniently made by analysis of the fluorine content of the crucible charge (i. e. the pot lining under treatment) before and after the described heating. Numerous other tests having shown that all of the evaporated fluorine is condensable, it is apparent that recovery of the order of 89% was achieved.

In another run, 100 grams of pot lining material of the same character were heated to 1130° C. for 60 minutes. Here 90% of the original fluorine, by weight, was evaporated and condensed for recovery. The fluoride part of the distillate contained 48.9% fluorine. At the same time 2.4 grams of metallic sodium were collected in a higher region of the condensing section. This and the preceding operation were performed with the graphite evaporator section in the first-described size of apparatus.

In another operation where 200 grams of the same pot lining were heated to 1040° C. for 60 minutes, 56% of the fluorine was evaporated. The resulting condensate showed that besides sodium and aluminum (as present to constitute cryolite and sodium fluoride) there were no metallic elements in the fluoride product except in traces or minute amounts: iron, manganese and vanadium, each less than 0.005%; copper and magnesium, each less than 0.0005%; silicon 0.03% and calcium 0.01%.

A further, extended series of runs was made to investigate the percentage recovery of fluorine, utilizing samples from several different localities of each of five (5) different pot linings, i. e. the carbon and insulating sections of aluminum reduction cell linings that had been in use for various periods ranging from 1.2 to 7.6 years. In these tests the crucible having the graphite-lined condensing section was employed, temperatures of evaporation being approximately 1135° C., and the pressure being kept below one millimeter of mercury. As in other cases, fluoride condensate was deposited in lower or

central portions of the condensing tower, while metallic sodium was separately condensed at localities above the fluoride condensate. As will now be appreciated, no particular temperature control, nor indeed any cooling means, was found necessary in order to effectuate condensation or to provide the desired separation of the distillates. The inherently progressive fall of temperature from the bottom to the top of the condenser column was sufficient to bring about the desired results in all cases.

Referring particularly to the series of tests last mentioned, the percentage recovery of fluorine, measured as percent of the total in the original pot lining, and actually obtained in the form of cryolite ($3\text{NaF} \cdot \text{AlF}_3$) and sodium fluoride (NaF), varied considerably, from values in the neighborhood of 75% up to 99%, the average being about 90%, and thus considerably better than the average recovery of about 78% that was obtainable from samples of the same pot linings by utilizing a standard wet method, i. e. the conventional caustic leach. By way of specific example, 100 gram portions of pot lining derived from the carbon bottom of a seven-year-old pot, and containing 17.9% fluorine, was heated to 1135°C . (for fluoride vaporization) for periods varying from 15 to 60 minutes, and showed fluorine recovery of 81 to 85.5%, these being apparently samples from which fluorine was rather difficultly releasable.

Another sample (from the same pot, deeper in the bottom) containing 19.2% fluorine was treated at the same temperature for 30 minutes, affording a fluorine recovery by distillation of 91%. As illustrative of further high recoveries, one sample (from a pot $2\frac{1}{2}$ years old) containing 9.4% fluorine yielded fluorine recoveries of 96% and 98.5% on distillation for 60 and 120 minutes respectively, at 1135°C . Essentially similar results were obtained with another lining sample containing 33.5% fluorine, from a pot somewhat over a year old.

In another series of operations, the compositions of the distillates were further investigated, utilizing material from the same pot linings, and performing distillations at temperatures from 1050° to 1200°C . The apparatus was of the larger size mentioned above, utilizing the steel condensing section. The distillation times were recorded as varying from 2 to 5 or 6 hours, but included all of the time beginning with the attainment of a temperature of 950°C . in the course of heating up the apparatus. In every case there was at least one-half hour of operation at the maximum temperature selected, viz. 1050° , 1100° , 1150° or 1200° , in different cases. In these tests, the

tion of sodium fluoride appears to increase relative to the cryolite, and in general the greatest concentrations of sodium fluoride appear to occur in the side insulation and at the carbon corner at the collector bar level, the least concentrations being in the carbon directly under the bath. Thus in one instance values of 45.5% cryolite and 54.5% sodium fluoride were obtained and in another instance proportions as low as 39.9% cryolite and 61.1% sodium fluoride, but in many cases the cryolite considerably exceeded the sodium fluoride, up to situations where the proportion of cryolite to sodium fluoride was approximately 90% to 10% and even higher, e. g. of the order of 97% to 3%. The total weight of the fluoride condensate (excluding the separate sodium) appeared to average about 35% of the weight of the lining treated, ranging as low as 12% and as high as 58% among the various samples used.

In these same tests measurement was also made of the amount of metallic sodium distilled and recovered from the pot lining, it being found (as explained above) that the distillate was usually in two distinct zones, viz. the fluorides and above them (in the apparatus shown) the sodium. In some cases, in addition to the separate sodium distillate, it was found that a small amount of sodium appeared in the fluoride distillate portion, e. g. equalling not more than a few percent of the weight of the fluoride distillate, and also being quite small in quantity compared with the valuable, separately collected sodium condensate mentioned above. Referring more particularly, however, to the separate sodium condensate it was noted that as a general average, about 10% of the weight of the cell lining charged to the distillation apparatus was recovered as sodium metal, the sodium condensate ranging from 2% to 18.5% of the original lining weight in these tests. The amount of recoverable sodium varies in different parts of the lining, and appears to increase with the age of the pot. It is nevertheless usually recoverable from all portions, except that the outer insulation layer of a short-lived pot tended to yield little or no sodium. No difficulty was encountered in removing the condensed sodium from the condenser, as metal and in a high state of purity, adaptable for many uses.

As further illustration, data from some of the above tests are given in the following table, the figures in each case being related to 100 grams of pot lining under treatment, and the distillation times being those required to carry the temperature from 950° to the named maximum but including one-half hour at such maximum:

Exp. No.	J271	J217	J220	J304	J282	J306	J296	J308
Max. temp., $^\circ \text{C}$	1,200	1,200	1,200	1,150	1,150	1,100	1,150	1,050
Time (hours)	3.75	2	2.25	4	5.25	1.75	4.75	4.5
A. Wt. loss (g.)	41	51	68	61	59	48	42	77
B. Fluoride condensate (g.)	30	35	40	52.5	50	23	28	58
C. Sodium condensate (g.)	9	15	17	6	9	16	11.5	11
D. Percent of F in lining distilled	85.5	93.7	82.8	97.5	95.7	98	94	97.6
E. Percent of distilled F condensed	100	100	95.5	100	95	100	100	90
F. In Fluorides of condensate:								
Percent cryolite	97.1	99+	85.7	43.5	92.7	25.2	93.2	96.6
Percent NaF	2.9	0-1	14.3	56.5	7.3	74.8	6.8	3.4

fluorine recovery ranged as high as 98 to 99% in many cases and averaged about 90% or better, thus agreeing with the operations described above.

Analysis of the fluoride distillate (in the above tests) both by X-ray and by chemical leaching procedures, showed it was essentially composed of cryolite and sodium fluoride in practically all cases. In one or two instances there appeared to be practically no sodium fluoride present (the entirety of the fluoride being essentially cryolite), but in general the mutual proportions of these compounds varied considerably, somewhat in dependence on the age of the pot lining sample and the locality from which it had been taken in the entire body of lining. As the age of the pot increases, the propor-

As will be apparent, the weight loss A is that of the lining under treatment, while items B and C are the weights of the two condensates. Item D is the percentage of the total fluorine, in the lining, which was removed in vapor. Measured on the fluorine thus distilled, item E represents the percentage condensed. The table also gives (item F) the composition of the condensed fluoride material, in specific compounds. While it will be appreciated that in some of these and other runs the fluorine recovery was appreciably below 90%, a number of the above and still other tests showed recoveries well beyond such figure, which represents the average over a large variety of pot lining samples, of different ages and characteristics.

Although the localities of the condensates varied considerably among the tests, typical positions for the fluoride and sodium fractions are indicated respectively at 24 and 25 in the drawing.

It is to be understood that the invention is not limited to the specific procedures herein illustrated and described but may be carried out in other ways without departure from its spirit.

We claim:

1. A method for the recovery of valuable fluoride material from used aluminum reduction cell lining, comprising heating the lining at a temperature in the range of about 1000° C. to about 1500° C. and under pressure reduced below atmospheric, to volatilize fluoride material from the lining into vapor condensable as fluoride material of the class consisting of cryolite and sodium fluoride, conducting the resulting vapor to a condensing region and there condensing fluoride material of said class from the vapor.

2. A method for producing fluoride material of the class consisting of cryolite and sodium fluoride, comprising heating used aluminum reduction cell lining at a temperature in the range of about 1000° C. to about 1500° C., under suitable subatmospheric pressure in the range of 0.001 atmosphere and above and for a sufficient time to vaporize fluoride material therefrom, and condensing fluoride from said vapor, consisting essentially of fluoride material of the aforesaid class.

3. A method as defined in claim 2 in which the lining is heated at a temperature in the range of 1050° C. to 1300° C.

4. A method for the recovery of valuable material from used aluminum reduction cell lining, comprising volatilizing metallic sodium and fluoride condensable as fluoride material of the class consisting of cryolite and sodium fluoride, from said lining, by heating the lining at a temperature in the range of about 1000° C. to about 1500° C. and under pressure reduced below atmospheric, conducting the produced vapor into and along a condensing path which is characterized by decrease of temperature along it, and condensing said fluoride material and metallic sodium from the vapor respectively at successive regions of decreasing temperature along said path.

5. A method for the recovery of valuable material from used aluminum reduction cell lining, comprising volatilizing fluoride material and metallic sodium from the lining by heating the lining at a temperature in the range of about 1000° C. to about 1500° C. and under suitable subatmospheric pressure in the range of 0.001 atmosphere and above, to produce vapor containing fluoride material condensable as material of the class consisting of cryolite and sodium fluoride, and metallic sodium, conducting said vapor into and along a condensing path which is characterized by decrease of temperature along it, and condensing fluoride material of said class and metallic sodium from the vapor respectively at successive regions of decreasing temperature along said path.

6. A method for producing fluoride material and metallic sodium, said fluoride material being of the class consisting of cryolite and sodium fluoride, comprising heating used aluminum reduction cell lining at a temperature in the range of about 1000° C. to about 1500° C., under suitable subatmospheric pressure and for a sufficient length of time to vaporize fluoride material and metallic sodium therefrom, conducting the produced vapor into and along a condensing path which is characterized by decrease of temperature along it, and condensing fluoride and metallic sodium from the vapor respectively at successive regions of decreasing temperature along said path, said fluoride condensate consisting essentially of fluoride material of the aforesaid class.

7. A method as defined in claim 6, in which the lining is heated at a temperature in the range of 1050° C. to 1300° C.

8. A method as defined in claim 7, in which the lining is heated and the vapor withdrawn therefrom, under a pressure between 0.001 and 0.1 atmosphere.

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UNITED STATES PATENT OFFICE
Certificate of Correction

Patent No. 2,858,198

October 28, 1958

James P. McGeer et al.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 44, for "future" read —further—; column 4, line 10, for "C.," read —C.)—.

Signed and sealed this 28th day of April 1959.

[SEAL]

Attest:

T. B. MORROW,
Attesting Officer.

ROBERT C. WATSON,
Commissioner of Patents.