F. D. S. ROBERTSON.

PROCESS FOR OBTAINING VOLATILIZABLE METAL OXIDES.

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Fig. 2.

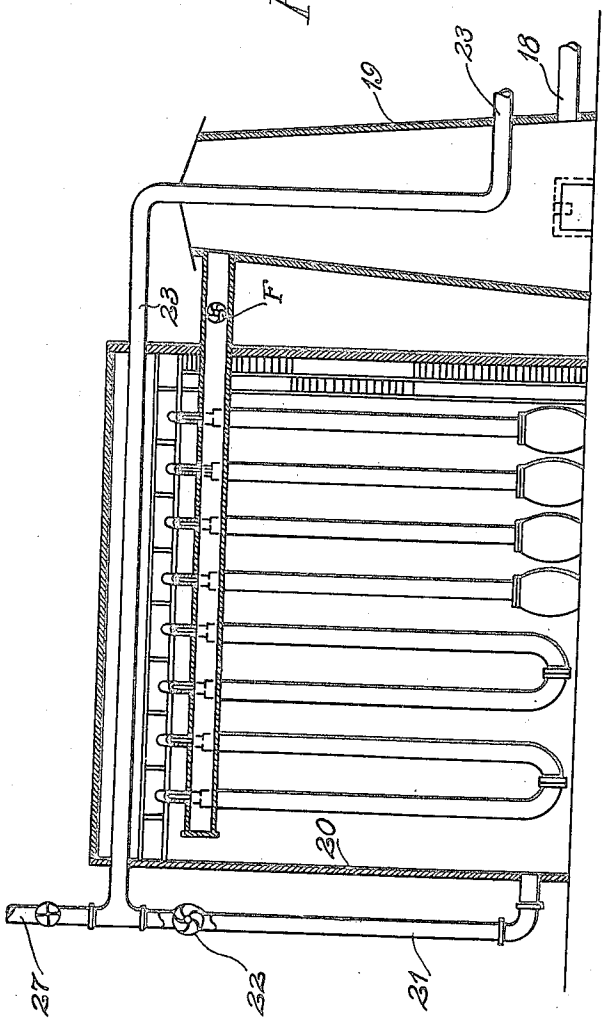
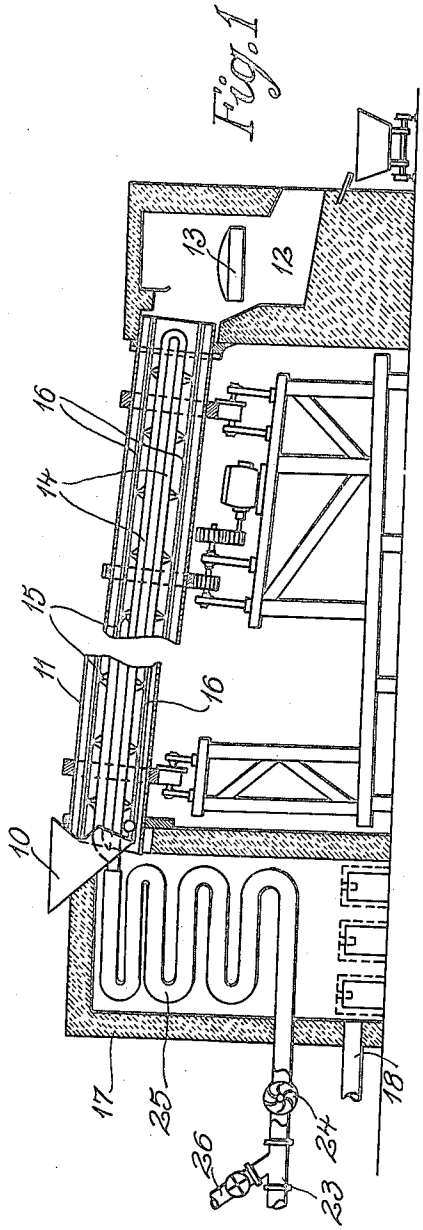


Fig. 1



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PROCESS FOR OBTAINING VOLATILIZABLE METAL OXIDES.

1,426,602. Specification of Letters Patent. Patented Aug. 22, 1922.

Application filed December 16, 1918. Serial No. 266,984.

To all whom it may concern:

Be it known that I, FREDERICK DUNCAN STRUAN ROBERTSON, a subject of the King of Great Britain, residing at Toronto, Province of Ontario, Dominion of Canada, have invented certain new and useful Improvements in Processes for Obtaining Volatilizable Metal Oxides, of which the following is a specification.

This invention relates to processes for obtaining volatilizable metal oxides. More particularly the invention has to do with the formation or separation and obtaining of volatilizable metal oxides in relatively pure condition from substances and minerals capable of yielding the same, whereby larger yields of pure products may be obtained more expeditiously and with less labor, fuel and handling than has been possible in processes heretofore proposed. In general, the process of the invention involves a cyclical procedure, and in a desirable embodiment of the invention, it comprises establishing a gas current traveling in a circuit, charging the gas current at one locality in the circuit with a metal oxide volatilized from a substance or mineral capable of yielding a volatilized metal oxide under the conditions of operation, separating the major portion of the volatilized oxide from the gas current at a succeeding locality in the circuit, and returning at least a portion of the residual gas or gases to the first mentioned locality in the circuit to pick up more of the volatilized metal oxide, and so on, the steps of charging the gas current with oxide and removing oxide therefrom occurring cyclically. Although the gaseous fluid employed as the carrier or vehicle in the described circuit may be inert or neutral in character with respect to the substance undergoing treatment, it is more desirably oxidizing or oxygen-yielding in character, whereby the oxide desired to be volatilized and recovered may be of comparatively uniform composition, even though the particular metal of the oxide, such as molybdenum, for example, may be capable of forming a number of oxides. The pressure under which the gaseous carrier is maintained at any point in the circuit may be normal, subatmospheric or superatmospheric, as may be desired to produce certain results. In some cases it is especially desirable that the volatilization of the metal oxide into the gaseous carrier be effected under subatmospheric pressure.

The process of the invention is of special value in the obtaining of molybdenum oxide from ores or other materials containing the same, such as concentrates, middlings or tailings, for example. In particular, the process can be very advantageously employed in the treatment of molybdenite (MoS_2) for the production of a volatilizable molybdenum oxide such as molybdenum trioxide (MoO_3), and a specific example illustrating the process as applied to the treatment of molybdenite will therefore be more particularly hereinafter described for the purpose of a concrete example embodying the principles of the invention.

In the treatment of molybdenite according to the invention, the molybdenite is placed in a suitable receptacle and heated in an oxidizing gas current at a sufficiently high temperature to bring about oxidation of the sulfur to sulfur dioxid and to produce a volatile molybdenum oxide, such as molybdenum trioxide. When the reaction takes place under ideal conditions, it may be represented as follows:

$$MoS_2+7O=MoO_3+2SO_2.$$

The gas current laden with fumes of the molybdenum oxide and also containing sulfur dioxid is conducted from the heating receptacle and is cooled or is allowed to cool sufficiently to deposit the greater part of the molybdenum oxide as a sublimate, any suitable means of separating the suspended sublimate from the gas current, such as filter bags, water sprays, or the like, being employed. The residual carrier gas current, which may still contain a substantial amount of sensible heat and which also necessarily carries uncondensed molybdenum oxide to an extent depending upon the temperature of the gases and the effectiveness of the fume separating means, is then conducted back to the heating receptacle and is employed again as an oxidizing agent and also as a carrier for volatilized molybdenum oxide. The described cycle of steps may be carried out repeatedly, assuming that suitable provision is made for maintaining the desired oxidizing character of the gas current in the circuit. As the process continues, the gas current tends to become charged with sulfur dioxid and to be de-

prived of its available oxygen; and hence it is necessary to withdraw or discharge from the circuit proper proportions of the more or less spent or depleted gases, and to introduce air, for example, in such manner as to maintain the gas current suitably oxidizing in character for the purposes in view. The sulfur dioxid resulting from the process may be employed in the manufacture of sulfuric acid or utilized in any other desired manner.

The heating and cooling of the circulating gas current may be accomplished in any suitable manner. As regards heating, the necessary heat units which may be necessary in practice to supplement those available from the oxidizing reaction in the case of molybdenite, may be supplied to the volatilizing zone either by external or internal heating means, such additional heat being provided either by combustion or electrically as may be desired. If the employment of special means for cooling the gases to deposit the sublimed oxid be desirable, various expedients may be adopted. For example, the cooling may be effectively accomplished by introducing liquid oxygen or liquid air at the precipitating zone or sphere, thus attaining both the refrigerating effect consequent upon expansion of the liquid and the enriching of the gas current with oxygen prior to its return to the volatilizing zone.

One form of apparatus in which the process of the invention may be carried out is illustrated in the accompanying drawings in which—

Figures 1 and 2 represent more or less diagrammatically and in longitudinal section a rotary roasting furnace with its cooperating fume collecting system. It will be understood that this apparatus is only one of various forms adapted for successful practice of the novel cyclical process here involved.

When the form of apparatus shown in the drawings is employed in carrying out the process as applied to molybdenite, the ore, crushed to say 10 mesh size, is fed into the hopper 10 and passes into the inclined rotary cylindrical roaster 11, being heated to progressively higher temperatures as it approaches the lower end of the roaster, from which it drops in spent condition into chamber 12, going thence to any convenient place of discharge. Hot gases from a fire place or other suitable firing means located in chamber 13, which may be either within or at one side of the chamber 12, pass into the lower end of the rotary roaster, traveling in a direction opposite to the descending stream of ore. From pipes 14 which extend longitudinally within the roasting cylinder and are provided with jet openings or twyers 15, preheated gas containing available oxygen in proper proportion is blown into the roaster and into intimate contact with the ore passing downwardly therethrough. The roasting cylinder is provided internally with longitudinal ribs or riffles 16 which serve to carry the ore upwardly as the cylinder rotates, and to shower it down through the current of hot oxidizing gases traveling toward the upper end of the roaster. In the upper portion of the roasting cylinder, most of the sulfur is oxidized with production of a large amount of heat which is available to aid in maintaining the roaster at the proper temperature. As the solid material continues to move toward the lower end of the roaster, the resultant molybdic oxide is volatilized and is swept out of the upper end of the roaster by the current of hot gases. By means of fan F, the gases and fume leaving the roaster are drawn down through a preliminary cooling chamber 17, in which a part of the molybdenum oxide may be condensed, and thence through outlet pipe 18 into the fume collecting system proper, consisting in this instance of a cooling tower 19 and bag house 20, where the temperature of the gases is lowered sufficiently to cause deposition of most of the molybdenum oxide or oxides still remaining in suspension. The residual gases, carrying more or less uncondensed molybdenum oxide, are drawn from the bag house through pipe 21 by fan 22, and are forced thence by way of return pipe 23, which may pass through the bag house and the cooling tower for purposes of heat conservation, to the twyer pipes 14 in the cylindrical roaster. To further ensure efficient circulation, a fan 24 may be interposed in pipe 23 close to where said pipe enters the chamber 17. Said pipe may be disposed in the form of coils 25 within said chamber, as shown, this arrangement being effective to recover heat from the furnace gases and fumes and to preheat the oxidizing gases blown into the furnace through pipes 14. A valved intake 26 may be provided leading into the pipe 23 on the suction side of fan 25, whereby oxygen or air may be introduced as may be necessary to maintain the gas current in the circulating system oxidizing to a sufficient degree. At 27 is provided a valved discharge through which any desired proportion of the more or less spent gases leaving the bag house may be discharged to waste when this is necessary to aid in maintaining the proper oxidizing character of the gases in the circuit.

It will be observed that in carrying out the process as above described, not only is it possible to conserve a substantial amount of heat that would otherwise be lost by discharging to waste all the residual gases leaving the bag house, but there is also the further advantage that the gas current returning in the circuit to the roaster already

carries with it the oxidized molybdenum which escaped deposition in the fume collecting system. It will thus be seen that the gaseous carrier picks up in the roaster an amount of volatilized molybdenum oxide corresponding rather closely to the amount which it later deposits in the fume collecting system; whereas if the gaseous carrier were composed wholly of gas introduced for the first time into the roaster, it would pick up in addition the further amount of molybdenum oxide corresponding to that necessarily escaping deposition, this further amount therefore being lost by discharge of the gases to waste after passing through the bag house. The economy thus effected is substantial. Furthermore, it has been found that in the practice of my process, employing a gaseous carrier in the circuit in the manner described, the fume or sublimate obtained is more easily and efficiently separated frm the gaseous carrier.

While the invention has been more particularly described in connection with the obtaining of molybdenum oxide, the process is applicable also to other volatile metal oxides, such as vanadium oxide, arsenious oxide, or the like.

What I claim is:

1. The process of obtaining volatilizable metal oxide from a substance capable of delivering the same, which comprises circulating a gaseous fluid in a circuit, heating to a sufficiently high temperature at one point in said circuit and in operative contact with said fluid a substance capable of delivering volatilizable metal oxide, and allowing such metal oxide to be deposited from said fluid at another point in said circuit.

2. In the process of recovering volatilizable metal oxide, the step which comprises establishing a current of gas traveling in circuit and capable of delivering oxygen, progressively heating and cooling the fluid at succeeding points in its travel, charging the gas with the vapor of a metal oxide at the heat delivering sphere and depositing the oxide as a sublimate at the cooling sphere.

3. In the process of recovering volatilizable molybdenum oxide, the step which comprises establishing a current of gas traveling in a circuit and capable of delivering oxygen, progressively heating and cooling the gas at succeeding points in its travel, charging the gas with the vapor of a molybdenum oxide at the heat delivering sphere and depositing the molybdenum oxide as a sublimate at the cooling sphere.

4. The process of recovering volatilizable metal oxide from substances containing the metal the products of which is desired, which comprises subjecting the substance to the action of heat while in the presence of a moving body of gaseous fluid, capable of yielding oxygen, under subatmospheric pressure, conveying the fluid laden with the vapor of the product desired away from the heat delivering sphere, and abstracting heat from the fluid conveyor at a different point to such a degree that the major part of the vaporized product is deposited as a sublimate.

5. The process of recovering volatilizable molybdenum oxide from substances containing molybdenum oxide, which comprises subjecting the substance to the action of heat while in the presence of a moving body of gas, capable of yielding oxygen, under subatmospheric pressure, conveying the gas laden with the vapor of the molybdenum oxide desired away from the heat delivering sphere, abstracting heat from the gas conveyor at a different point to such a degree that vaporized molybdenum oxide is deposited as a sublimate.

6. The process of recovering volatilizable metal oxide from substances capable of delivering the same, which comprises establishing a traveling circuit of oxygen delivering gaseous fluid, heating the traveling fluid at one or more points to the temperature at which the metal oxide desired volatilizes under the existing conditions and subjecting the metal oxide delivering substance to the action thereof, exposing the moving fluid carrying the volatilized metal oxide to refrigeration or cooling at a succeeding point to deposit volatilized metal oxide as a sublimate, enriching the fluid with oxygen, again heating the fluid and charging it with volatilized metal oxide at a succeeding point in the circuit and again cooling and precipitating metal oxide in the continuous progress of the process.

7. The process of recovering volatilizable molybdenum oxide from substances capable of delivering the same, which comprises establishing a traveling circuit of oxygen delivering gaseous fluid, heating the traveling fluid at one or more points to the temperature at which the molybdenum oxide desired volatilizes under the existing conditions and subjecting the molybdenum oxide delivering substance to the action thereof, exposing the moving fluid carrying the volatilized molybdenum oxide to refrigeration or cooling at a succeeding point to deposit volatilized molybdenum oxide as a sublimate, again heating the fluid and charging it with volatilized molybdenum oxide at a succeeding point in the circuit and again cooling and precipitating molybdenum oxide in the continuous progress of the process.

8. The process of recovering volatilizable molybdenum oxide from substances capable of delivering the same, which comprises establishing a traveling circuit of oxygen delivering fluid, heating the traveling fluid at one or more points to the temperature at which the molybdenum oxide desired vola-

tilizes under the existing conditions and subjecting the molybdenum oxide delivering substance to the action thereof, exposing the moving fluid carrying the volatilized molybdenum oxide to refrigeration or cooling at a succeeding point to deposit volatilized molybdenum oxide as a sublimate, enriching the fluid with oxygen, again heating the fluid and charging it with more volatilized molybdenum oxide at a succeeding point in the circuit and again cooling and precipitating molybdenum oxide in the continuous progress of the process.

9. The process of obtaining a molybdenum oxide which comprises establishing an oxidizing gas current traveling in a circuit, charging said gas current at one locality in said circuit with a molybdenum oxide by heating in operative contact with said gas current a substance capable of delivering a molybdenum oxide thereto, lowering the temperature of the charged gas current at another locality in said circuit and thereby separating a molybdenum oxide therefrom, and repeating the charging and separating steps cyclically.

10. The process of obtaining a molybdenum oxide which comprises establishing an oxidizing gas current traveling in a circuit, charging said gas current at one locality in said circuit with a molybdenum oxide by heating in operative contact with said gas current a substance capable of delivering a molybdenum oxide thereto, lowering the temperature of the charged gas current at another locality in said circuit and thereby separating a molybdenum oxide therefrom, repeating the charging and separating steps cyclically, and maintaining the desired oxidizing character of the gas current by removing spent gas from the circuit and introducing fresh oxidizing gas thereinto as may be required.

11. The process of treating molybdenite which comprises heating the same in an oxidizing gas current to a temperature sufficiently high to volatilize a molybdenum oxide, separating a molybdenum oxide from the gas current at a later stage of its travel, and returning the gas current into operative contact with more heated molybdenite.

12. The process of treating molybdenite which comprises heating the same in an oxidizing gas current to a temperature sufficiently high to volatilize a molybdenum oxide, separating a molybdenum oxide from the gas current at a later stage of its travel, returning the gas current into operative contact with more heated molybdenite, discharging spent gas from the circuit and introducing fresh oxidizing gas thereinto as may be required to maintain the desired oxidizing character of the gas current.

13. The process of treating molybdenite which comprises heating molybdenite in an oxidizing gas current to temperatures high enough to oxidize the molybdenite and volatilize a molybdenum oxide, passing the resultant fume-laden gases through a fume collector to deposit a molybdenum oxide, returning at least part of the gases leaving the fume collector into contact with more heated molybdenite to pick up more oxide, and introducing oxygen into the gas current in sufficient proportions to maintain the desired oxidizing character of the gas current.

In witness whereof, I affix my signature in the presence of two witnesses.

FREDERICK DUNCAN STRUAN ROBERTSON. [L. S.]

Witnesses:

F. L. WHOITNER,

R. H. DOVE.