Nov. 1, 1960 J. M. NOY ET AL 2,958,588

SUBLIMED OXIDE FURNACE

Filed June 20, 1958 5 Sheets-Sheet 1

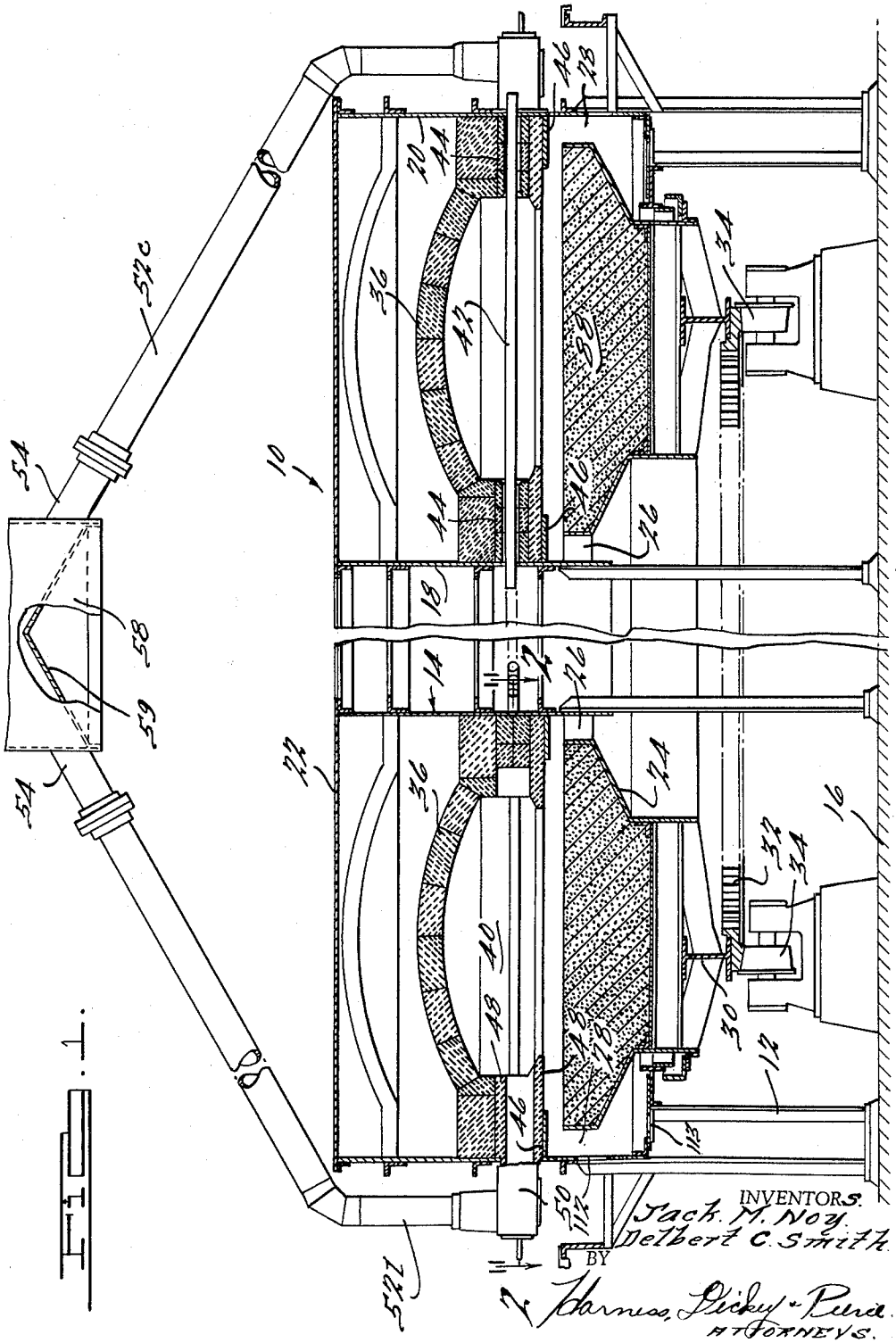


FIG. 1.

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SUBLIMED OXIDE FURNACE

Filed June 20, 1958 5 Sheets-Sheet 2

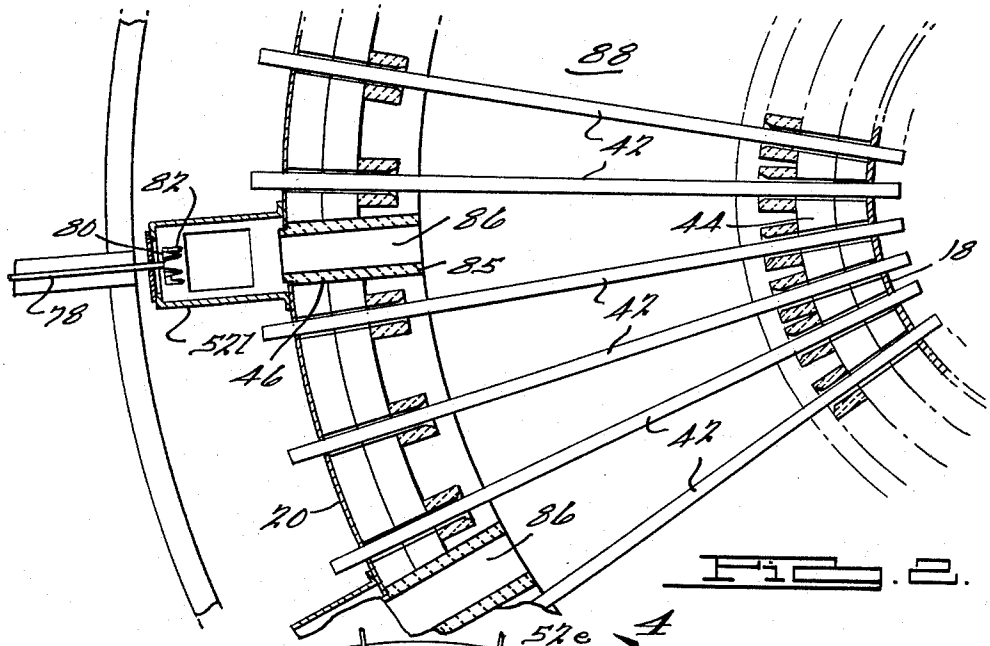


FIG. 2.

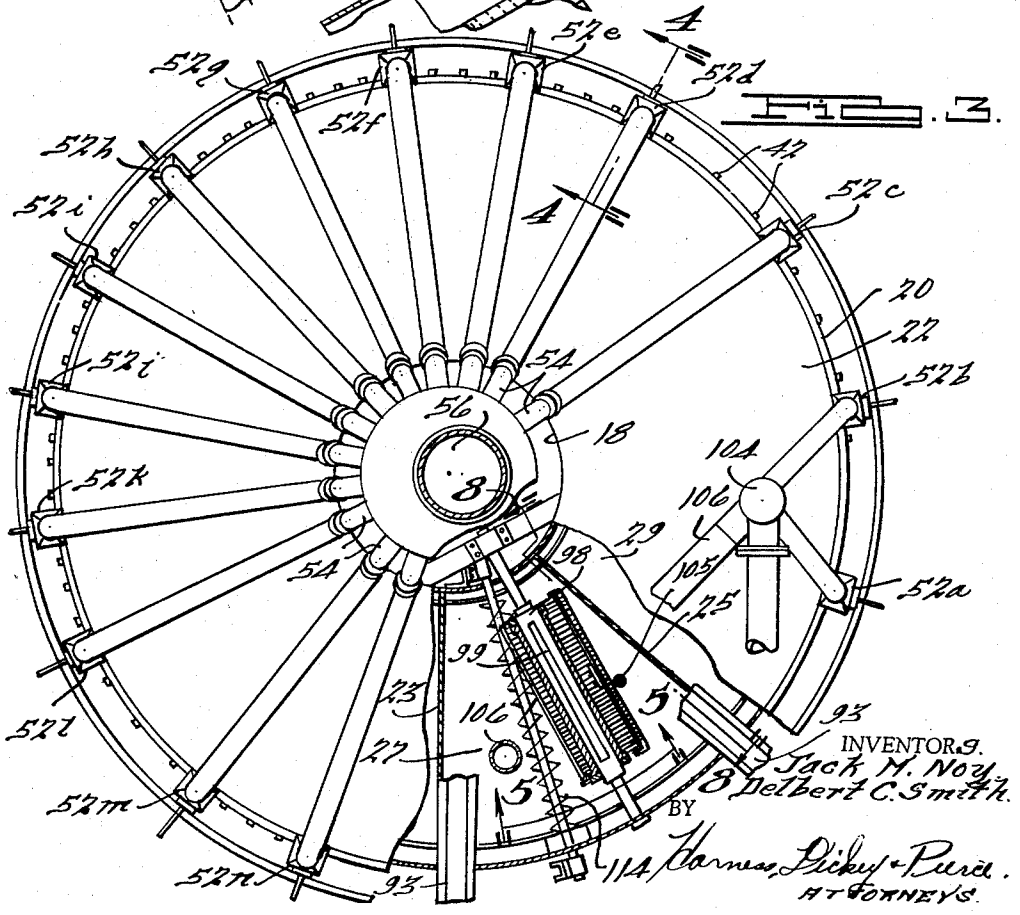


FIG. 3.

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SUBLIMED OXIDE FURNACE

Filed June 20, 1958 5 Sheets-Sheet 3

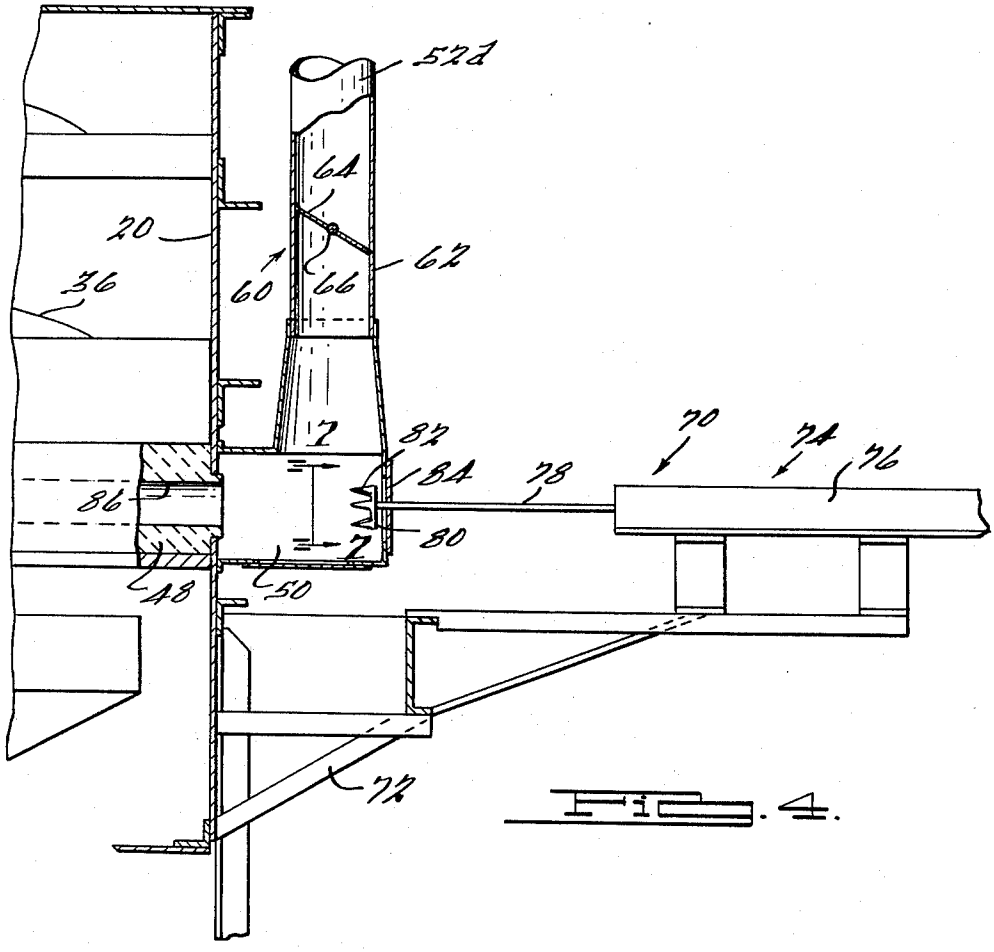


FIG. 4.

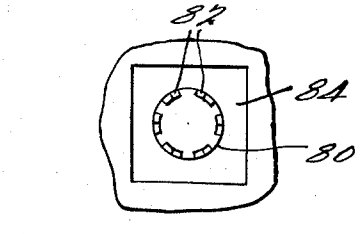


FIG. 7.

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Nov. 1, 1960 J. M. NOY ET AL 2,958,588

SUBLIMED OXIDE FURNACE

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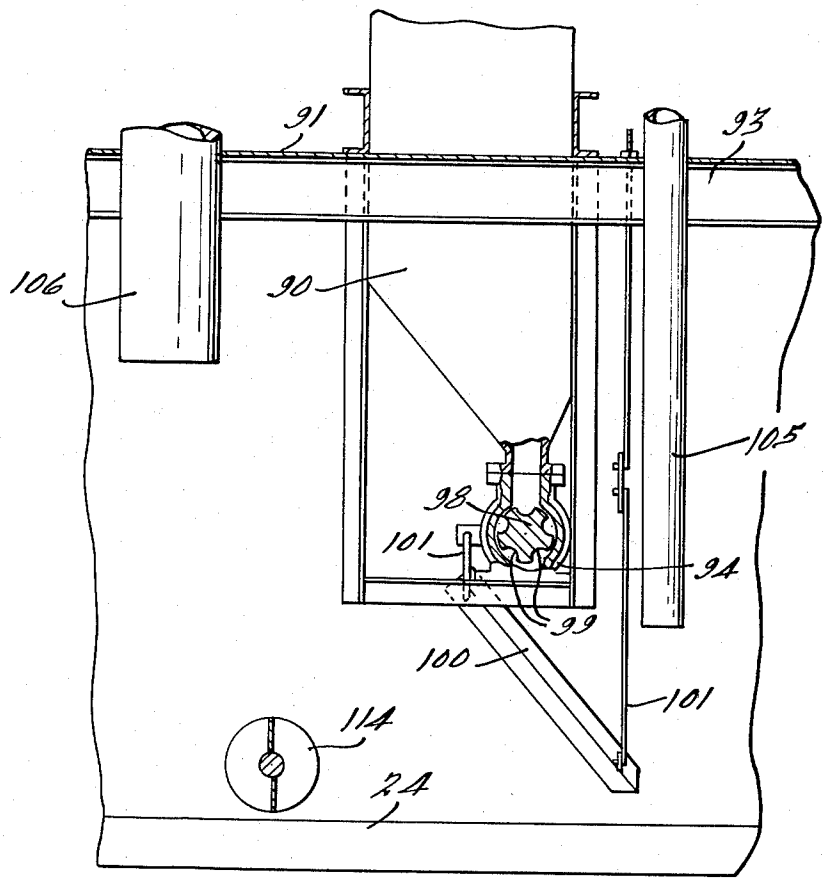


FIG. 5.

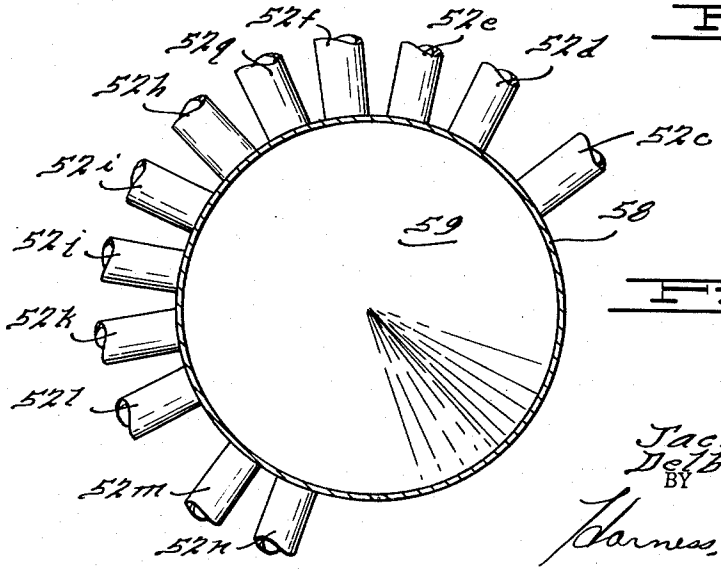


FIG. 6.

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SUBLIMED OXIDE FURNACE

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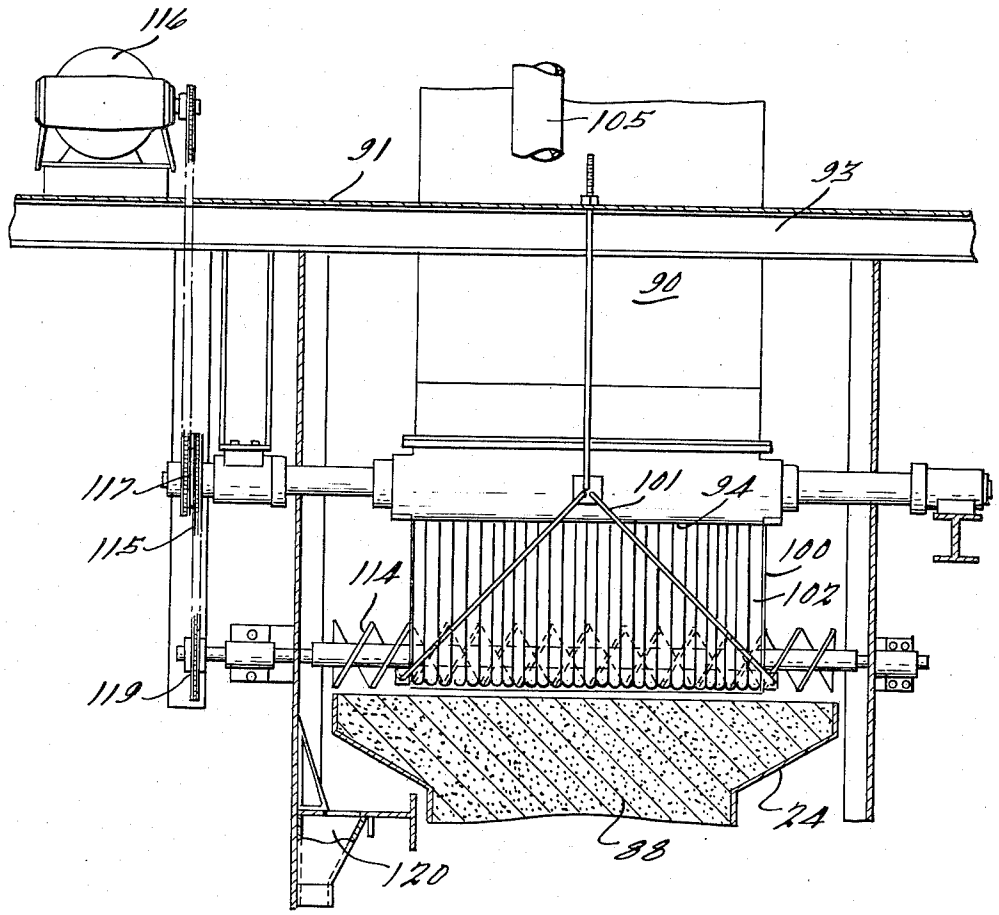


FIG. 8.

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SUBLIMED OXIDE FURNACE

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Filed June 20, 1958, Ser. No. 743,282

8 Claims. (Cl. 23—264)

This invention relates generally to furnaces and more particularly to a furnace for producing molybdenum trioxide (MoO_3), sometimes called molybdic anhydride or molybdic acid, and commonly referred to in the molybdenum processing field as "oxide," by subliming a high purity molybdenum trioxide from a lower purity feed material.

Prior furnaces for obtaining high purity oxide (better than 99% MoO_3) from technical grade oxide (approximately 90% MoO_3 and containing some MoO_2 and insoluble impurities such as aluminum oxide, silica and iron oxide) have utilized a rotating hearth located below the furnace heating units. Technical grade oxide, continuously fed to the hearth which is heated by radiant heaters to approximately 2100 degrees Fahrenheit, starts volatilizing at about 1100 degrees Fahrenheit which is approximately 300 degrees below its melting point. Air streams passed over different sections of the hearth sweep the sublimed oxide out of the furnace to be cooled, collected, densified and placed in containers at the bag-house. One of the principal problems in furnaces of this type has been that since uniform conditions do not exist at all sections of the hearth, it has been difficult to obtain oxide of uniform purity from all of the hearth sections. If the air stream at a hearth section is too large, it picks up impurities from the oxide on the hearth so that the resulting oxide is below the required purity, and cools the oxide on the hearth to an extent such that the production rate is lowered. If the air stream is too small, the oxide bed on the hearth becomes molten, rather than remaining in the desired sticky non-flowable mass, and the rate at which oxide is removed from the hearth section is reduced so that the output of the furnace is lowered. In a furnace having a bustle type of air exhaust pipe, namely, an exhaust pipe which extends around the furnace and communicates at spaced points along its length with the furnace hearth and is connected at one of its ends to a suction device, when the dampers at the spaced points are adjusted so that the suction pressures at the spaced points of the hearth are about equal, the air velocity is so low that the suction air stream cannot carry all of the oxide and it will settle in the pipe.

The principal object of this invention, therefore, is to provide an improved furnace of the above type which is operable to produce more oxide of higher purity.

A further object of this invention is to provide a furnace of the above type with means for independently regulating the rate of air flow at each hearth section without affecting the air flow at any other hearth section so that optimum conditions can be obtained at all of the hearth sections.

Another object of this invention is to provide a furnace of the above type with an individual exhaust tube for each hearth section, and a draft or suction tube which is connected to the majority of the exhaust tubes at a position such that these exhaust tubes are all of substantially the same length.

Further objects, features and advantages of this invention will be come apparent from a consideration of the following description, the appended claims and the accompanying drawing in which:

Figure 1 is a fragmentary vertical sectional view of the furnace of this invention;

Fig. 2 is an enlarged fragmentary sectional view looking along the line 2—2 in Fig. 1;

Fig. 3 is a plan view of the furnace of this invention, with some parts broken away and other parts shown in section for the purpose of clarity;

Fig. 4 is an enlarged transverse sectional view looking along the line 4—4 in Fig. 3;

Fig. 5 is a sectional view looking along the line 5—5 in Fig. 3;

Fig. 6 is a horizontal sectional view of the distributor box in the furnace of this invention;

Fig. 7 is a sectional view looking along the line 7—7 in Fig. 4; and

Fig. 8 is a fragmentary sectional view looking along the line 8—8 in Fig. 3.

With reference to the drawing, the furnace of this invention, indicated generally at **10**, is illustrated in Fig. 1 as including a main frame **12** which supports an annular hood or furnace shell **14** at a position above a supporting floor surface **16**. The hood **14** has tubular inner and outer walls **18** and **20** arranged in a concentric relation and connected by a top wall **22** of substantially annular shape.

An annular hearth **24**, of substantially trough-shape in transverse section, is located within the lower portion of the hood **14**. As shown in Fig. 2, the width of the hearth **24** is less than the width of the hood **14** so that spaces **26** and **28** are provided between the hearth and the inner and outer hood walls **18** and **20**, respectively. The hearth **24** is mounted on a supporting I-beam **30** which is in turn carried on a ring gear **32** rotatably driven by any suitable means and supported on rollers **34**.

A pair of spaced partitions **23** and **25**, which extend substantially radially of the hood **14**, project upwardly from positions adjacent the top side of the hearth **24** so as to divide the furnace into a small feeding zone **27** and an elongated heating zone **29** which extends from the partition **25** in a counterclockwise direction, as viewed in Fig. 3, to the partition **23**.

Below its top wall **22**, the hood **14** is provided with a refractory arch **36** which extends the complete length of the heating zone **29** and forms the top wall of a heating chamber **40** disposed between the hearth member **24** and the arch **36**. A plurality of heating elements **42** extend substantially radially of the hood **14** through the chamber **40** and are positioned adjacent their ends in refractory blocks **44** supported on plates **46** carried by the inner and outer side walls **18** and **20** of the hood **14**. Each heating element **42** is of the silicon carbide electrical resistance type such as the Globar elements manufactured by the Carborundum Company and is supported at its ends by metal clips (not shown) which are supported on the furnace main frame and function as the electrical contacts for the heating elements.

At substantially equally spaced locations in the heating zone **29**, hollow exhaust port members **48** are supported on the outer plate **46**. Each member **48** is positioned between two groups of heating elements **42** and extends from a point above the hearth to the outer side wall **20** of the hood **14**. At the wall **20**, each port member **48** communicates with the horizontal portion **50** of an exhaust tube **52** which extends upwardly from the hood **14**. At its upper end, each tube **52** is connected to a downwardly extending pipe **54** on a distributor box **58**

located above the hood 14 so that its axis conicides with the vertical axis 56 of rotation of the hearth 24.

Since the hearth 24 is rotated in a counterclockwise direction as viewed in Fig. 3, the exhaust tubes 52 are provided with consecutive letter suffix designations from *a–n*, inclusive also in a counterclockwise direction about the hearth, to facilitate reference hereinafter to individual tubes. All of the exhaust tubes 52*c–n*, inclusive, are of substantially the same length and are connected by the pipes 54 to the distributor box 58 which forms a part of an upwardly extending draft tube connected to any suitable fan or other air suction means for exerting substantial upward drafts in the tubes 52*c–n*. The box 58 has a cone shape bottom 59 to facilitate a smooth upward flow of volatilized oxide from the tubes 52.

As shown in Fig. 4, each exhaust tube 52 has a damper assembly 60 disposed in a vertical portion 62 thereof adjacent the horizontal portion 50. Each damper assembly 60 includes a plate 64 mounted for movement about a horizontal shaft 66 to different adjusted positions. Each damper assembly 60 is operable to control the amount of air and volatilized oxide which flows upwardly in the tube 52 in which the assembly 60 is installed.

To maintain the porthole members 48 clear of solidified oxide, a porthole cleaning mechanism 70 is mounted adjacent each porthole member 48. Each mechanism 70 is mounted on a supporting bracket 72 secured to the outer wall 20 of the hood 14, and includes a pneumatic piston and cylinder assembly 74 consisting of a horizontal cylinder 76 secured to the bracket 72 and a piston rod 78 which extends from the cylinder 76 toward the porthole member 48. On its terminal end, the piston rod 78 carries a circular plate 80 located within the horizontal portion 50 of the adjacent tube 52 and provided with pointed blades 82. The piston rod 78 extends through an opening in a vertical back door 84, for the exhaust tube portion 50, which can be opened for manual cleaning of the portion 50 and maintenance of the mechanism 70.

On operation of the cylinder assembly 74 to extend the piston rod 78, which is shown in its retracted position in Fig. 4, the plate 80 is moved to a position adjacent to but slightly inwardly of the inner end 85 of the exhaust port member 48. In this position of the plate 80 the blades 82 project out of the member 48 so that substantially all of the solidified oxide in the member 48 has been pushed back onto the hearth 24 to maintain the passage 86 through the member 48 clear for the travel of gaseous products therethrough.

In the use of the furnace 10, the hearth 24 is filled with sand 88, and technical grade oxide, which is in powder form, is fed from storage bins to the upper end of a feed hopper 90 located above the hearth in the feeding zone 27. As shown in Figs. 5 and 8, the zone 27 has a top wall 91 supported on horizontal beams 93 which are a part of the main frame 12. The oxide in the hopper 90 is discharged from the lower end 94 thereof by a feed roll 98 which is driven by a motor 116 and extends radially across the hearth 24. The roll 98 is generally cylindrical in shape and has grooves 99 in its outer surface. On rotation of the roll 98, each groove 99 fills with oxide in the hopper 90 and then deposits this oxide onto the upper end of an inclined feed plate 100 positioned below the hopper 90. The feed plate 100 is suspended from the frame 12 by rods 101 and is formed with small downwardly extending grooves 102 which maintain the form of the oxide issuing from the feed roll 98 as a wide stream and deposit the oxide on the hearth 24. This bed of oxide then travels with the rotating hearth 24 in a counterclockwise direction as viewed in Fig. 3 so that after it travels under the partition 25 it is exposed to the heat from the heating elements 42. The heat in the chamber 40 quickly brings the temperature of the oxide to a volatilizing temperature.

The oxide bed on the hearth 24 which is in a position opposite the first exhaust tube, indicated at 52*a* in Fig. 1, is in a heated condition such that some volatilized oxide is present. This volatilized oxide is drawn upwardly through the exhaust tube 52*a* which communicates with a draft tube 104 which is also connected to the draft tube 52*b* opposite the next porthole member 48. The draft tube 104 also communicates with an exhaust duct 106 which terminates at its lower end in the feeding zone 27 so that any dust in the zone 27 is drawn upwardly through the duct 106 to the draft tube 104 where it is mixed with the oxide fumes drawn upwardly through the exhaust tubes 52*a* and 52*b*. In the draft tube 104 the temperature of the oxide and air is lowered to about 180°–200° F. and the resulting solid or crystalline oxide is conveyed by the air to a secondary baghouse where it is fed into a conveyor mechanism which terminates in an upright feeder tube 105 (Figs. 3 and 5) located in the feeding zone 27. The tube 105 deposits this oxide, which is of a lower purity than desired, onto the hearth 24 for travel into the heating zone 29. Removal of the dust from the feeding zone 27 through the duct 106 prevents any of the dust from being drawn into the heating zone 29 and adversely affecting the purity of the volatilized oxide therein.

The oxide bed on the hearth 24 opposite the third exhaust tube 52*c* is in the desired sticky state. Consequently, the volatilized oxide withdrawn from the hearth through the exhaust tube 52*c* is of a higher quality than that withdrawn through the tubes 52*a* and 52*b*. This quality is maintained by adjusting the damper assembly 60 for the tube 52*c* so that optimum conditions are maintained on the oxide bed on the section of the hearth 24 adjacent the exhaust tube 52*c*. By virtue of the connection of all of the exhaust tubes 52*c–n* to the draft tube 58 at a single point, an adjustment of the damper assembly 60 for the tube 52*c* is divided between all of the other exhaust tubes so that the amount of exhaust air at any one hearth section is not affected to any great extent. In other words, if the damper assembly 60 for the tube 52*c* is adjusted to decrease the exhaust through the tube 52*c*, the corresponding total exhaust increase for the other tubes 52*d–n* is equally divided between the tubes so that the effect on any one tube is minimized. The adjustment of the damper assembly 60 controls the amount of air which flows upwardly through the space 26 opposite the tube 52*c* so that the inner rim of the hearth is cooled and the volume of air traveling across the hearth 24 is sufficient to maintain the oxide bed in the desired state. Air also travels upwardly through the space 28 adjacent the exhaust tube 52*c* primarily for cooling the outer rim of the hearth. The amount of air which travels upwardly through the space 28 corresponding to each tube 52 is controlled by a pair of dampers 112 and 113 of manually adjustable type carried by the hood 14. Each damper 112 is located in the outer wall 20 adjacent the lower end of the space 28, and each damper 113 is located in a wall which closes the lower end of the space 28.

Adjustment of the dampers 112 and/or 113 is effective to control the ratio of air admitted to the inner side of the corresponding hearth section to the air admitted to the outer side of the hearth section. The restriction of the amount of air permitted to enter the space 28 insures an upward travel of air through the space 26, so that air sweeps across the hearth in sufficient quantity to convey the sublimed oxide into the porthole members 48. By adjusting a corresponding pair of dampers 64 and 112 or 113, the air at the sides of the corresponding hearth section is regulated so that the inner and outer sides of the MoO_3 bed on the hearth are cooled and solidify so that they form barriers to prevent any liquid portions of the bed from running off the hearth, and the desired volume of air is provided for sweeping the sublimed MoO_3 from the hearth. Any spillage of oxide off the bed is noticeable

at the dampers 113 and either or both dampers 112 and 113 are adjusted at each hearth section to provide the required air. Observation openings are also provided in the furnace wall 20 at positions horizontally aligned with the oxide bed and some air also enters the furnace at these openings. All of the exhaust tubes 52*c* to 52*n*, inclusive, communicate with the distributor box 58 which is part of a draft tube connected to a suitable supply of suction air. The oxide in the tubes 52*c* to *n*, inclusive, is of high quality and after cooling to a solidifying temperature is further cooled and then conducted to the primary baghouse for collection and subsequent densifying and packaging.

The residue remaining on the hearth after a complete revolution of the hearth is removed by a tailing screw 114 (Fig. 5) which extends transversely of the hearth 24 in the feeding zone 27 at a position between the partition 23 and the feed roll 98. As a result of the continuous operation of the screw 114, all of the residue on each portion of the hearth is removed before additional oxide is deposited thereon by the feed roll 98. The dust resulting from operation of the screw 114 is removed from the zone 27 by the duct 106. A chain 115 extends about sprockets 117 and 119 on the tailing screw 114 and the feed roll 98, respectively, to provide for driving of the screw 114 by the motor 116. The residue removed from the hearth by the tailing screw 114 falls from the screw into a hopper 120 located below one end of the screw. This residue is equal to about 30–40% of the original oxide charge.

The porthole cleaning mechanisms 70 are operated automatically by any suitable control mechanism so that each cylinder assembly 74 is operated at predetermined intervals of time to extend the piston rod 78 so that the disk 80 and blades 82 maintain the porthole passage 86 free of solid oxide which would interfere with the air flow across the corresponding hearth section.

It will be understood that the specific construction of the improved sublimed oxide furnace herein disclosed and described is presented for purposes of explanation and illustration, and is not intended to indicate limits of the invention, the scope of which is defined by the following claims.

What is claimed is:

1. In a furnace for producing high purity MoO_3 from lower purity MoO_3 which includes a horizontal annular hearth mounted for rotation about a substantially vertical axis, means operatively associated with the furnace for feeding MoO_3 onto said hearth, a hood arranged in a covering relation with said hearth, and heating means above the hearth for volatilizing MoO_3 thereon; a plurality of exhaust tubes connected to peripherally spaced portions of the hood at positions adjacent said hearth for withdrawing said volatilized MoO_3 therefrom, draft tube means connected to said exhaust tubes at a single point remote from said hood for providing drafts therein for withdrawing said volatilized MoO_3 from said furnace, means for supplying air to said hearth, and means operatively associated with each of said exhaust tubes for regulating and adjusting the rate of flow of material therein.

2. In a furnace for producing high purity MoO_3 from lower purity MoO_3, said furnace including a horizontal annular hearth mounted for rotation in one direction about a substantially vertical axis, means for feeding said lower purity MoO_3 to one portion of said hearth, an annular hood having horizontally spaced inner and outer side walls which extend downwardly to positions horizontally spaced from the inner and outer sides, respectively, of the hearth so as to provide spaces for the upward travel of air on the inner and outer sides of the said hearth, heating means in the hood above the hearth, hollow exhaust port members mounted on peripherally spaced portions of said hood at positions above said hearth, each of said port members extending horizontally from a point above the hearth to the outer side wall of said hood, the improvement comprising a plurality of exhaust tubes corresponding to said exhaust port members, each one of the majority of said exhaust tubes communicating with the corresponding exhaust port member and extending upwardly therefrom to a single point above said hood, a draft tube connected to the upper ends of the exhaust tubes at said single point for providing upwardly traveling drafts therein, the remaining ones of said exhaust tubes being located adjacent said feeding means on the side thereof in the direction of rotation of said hearth, separate draft means connected to said remaining exhaust tubes, and separate damper means in each of said exhaust tubes for adjusting the draft therein to obtain optimum draft conditions at the hearth section corresponding thereto.

3. In a furnace for producing high purity MoO_3 from lower purity MoO_3 which includes a horizontal annular hearth mounted for rotation about a substantially vertical axis and having an inner side and an outer side, means operatively associated with the furnace for feeding MoO_3 onto said hearth, an annular hood arranged in a covering relation with said hearth and spaced from said sides, and heating means above the hearth for volatilizing MoO_3 thereon; a plurality of exhaust tubes connected to peripherally spaced portions of the hood at positions adjacent said hearth for withdrawing said volatilized MoO_3 therefrom, draft tube means connected to said exhaust tubes at a single point remote from said hood for providing drafts therein for withdrawing said volatilized MoO_3 from said furnace and for drawing air into said furnace through the spaces between said hearth sides and said hood, and individually adjustable damper means in each of said exhaust tubes.

4. In a furnace for producing high purity MoO_3 from lower purity MoO_3, said furnace including a horizontal annular hearth mounted for rotation in one direction about a substantially vertical axis, means for feeding said lower purity MoO_3 to one portion of said hearth, an annular hood having horizontally spaced inner and outer side walls which extend downwardly to positions horizontally spaced from the inner and outer sides, respectively, of the hearth so as to provide spaces for the upward travel of air on the inner and outer sides of said hearth, and heating means in the hood above the hearth; a plurality of exhaust tubes mounted on peripherally spaced portions of said hood, the majority of said exhaust tubes being of substantially the same length and extending upwardly from said hood to a single point above the hood, a draft tube connected to the upper ends of the exhaust tubes at said point for providing upwardly traveling drafts therein, and separate damper means in each of said exhaust tubes for adjusting the draft therein to obtain optimum draft condtions at the hearth section corresponding thereto.

5. In a furnace for producing high purity MoO_3 from lower purity MoO_3 which includes a horizontal annular hearth mounted for rotation about a substantially vertical axis and having an inner side and an outer side, means operatively associated with the furnace for feeding MoO_3 onto said hearth, an annular hood arranged in a covering relation with said hearth and spaced from said sides, and heating means above the hearth for volatilizing MoO_3 thereon; a plurality of exhaust tubes connected to peripherally spaced portions of the hood at positions adjacent said hearth for withdrawing said volatilized MoO_3 therefrom, draft tube means connected to said exhaust tubes at a single point remote from said hood for providing drafts therein for withdrawing said volatilized MoO_3 from said furnace and for drawing air into said furnace through the spaces between said hearth sides and said hood, individually adjustable damper means on said hood for controlling the air admitted to the outer side of said hearth for thereby controlling the ratio of the amounts of air admitted to the inner and outer sides of

said hearth, and individually adjustable damper means in each of said exhaust tubes.

6. In a furnace for producing high purity MoO_3 from lower purity MoO_3, said furnace including a horizontal annular hearth mounted for rotation in one direction about a substantially vertical axis, a hood positioned over said hearth and having horizontally spaced inner and outer side walls which extend downwardly to positions on opposite sides of the hearth, partition means in the hood extending downwardly to a position adjacent the hearth and dividing the interior of the hood into a feeding section and a heating section, means for feeding MoO_3 onto the hearth in said feeding section, said heating section having an inlet end and an outlet end, and heating means in said heating section for continuously heating MoO_3 traveling on said hearth from said inlet end to said outlet end; the improvement comprising providing means for admitting air to the portion of said hearth in said heating section, exhaust tube members mounted on and communicating with the interior of said hood at substantially equally spaced positions in said heating section, a draft tube connected to the majority of said exhaust tubes at a single point located such that said majority are all of substantially the same length, and adjustable means on said exhaust tubes for regulating the flow of material therethrough.

7. In a furnace for producing high purity MoO_3 from lower purity MoO_3, said furnace including a horizontal annular hearth mounted for rotation in one direction about a substantially vertical axis, a hood positioned over said hearth and having horizontally spaced inner and outer side walls which extend downwardly to positions on opposite sides of the hearth, partition means in the hood extending downwardly to a position adjacent the hearth and dividing the interior of the hood into a feeding section and a heating section, means for feeding MoO_3 onto the hearth in said feeding section, said heating section having an inlet end and an outlet end, and heating means in said heating section for continuously heating MoO_3 traveling on said hearth from said inlet end to said outlet end; the improvement comprising providing means for admitting air to the portion of the said hearth in said heating section, exhaust tube members mounted on and communicating with the interior of said hood at substantially equally spaced positions in said heating section, first draft tube means connected to some of said exhaust tubes adjacent said inlet end, means communicating said first

draft tube means with said feeding section for removing dust therefrom, second draft tube means connected to the remainder of said exhaust tubes at a single point located such that said remainder are all of substantially the same length, and adjustable means on said exhaust tubes for regulatnig the flow of material therethrough.

8. In a furnace for producing high purity MoO_3 from lower purity MoO_3, said furnace including a horizontal annular hearth mounted for rotation in one direction about a substantially vertical axis, means for feeding said lower purity MoO_3 to one portion of said hearth, an annular hood having horizontally spaced inner and outer side walls which extend downwardly to positions horizontally spaced from the inner and outer sides, respectively, of the hearth so as to provide spaces for the upward travel of air on the inner and outer sides of the said hearth, heating means in the hood above the hearth, hollow exhaust port members mounted on peripherally spaced portions of said hood at positions above said hearth, each of said port members extending horizontally from a point above the hearth to the outer side wall of said hood; the improvement comprising a plurality of exhaust tubes corresponding to said exhaust port members, each one of the majority of said exhaust tubes communicating with the corresponding exhaust port member and extending upwardly therefrom to a single point above said hood, a draft tube connected to the upper ends of the exhaust tubes at said single point for providing upwardly traveling drafts therein, the remaining ones of said exhaust tubes being located adjacent said feeding means on the side thereof in the direction of rotation of said hearth, separate draft means connected to said remaining exhaust tubes, adjustable damper means for controlling the amount of air admitted to the space on the outer side of said hearth and thereby controlling the ratio of the amounts of air admitted to the inner and outer sides of said hearth, and separate damper means in each of said exhaust tubes for adjusting the draft therein to obtain optimum draft conditions at the hearth section corresponding thereto.

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