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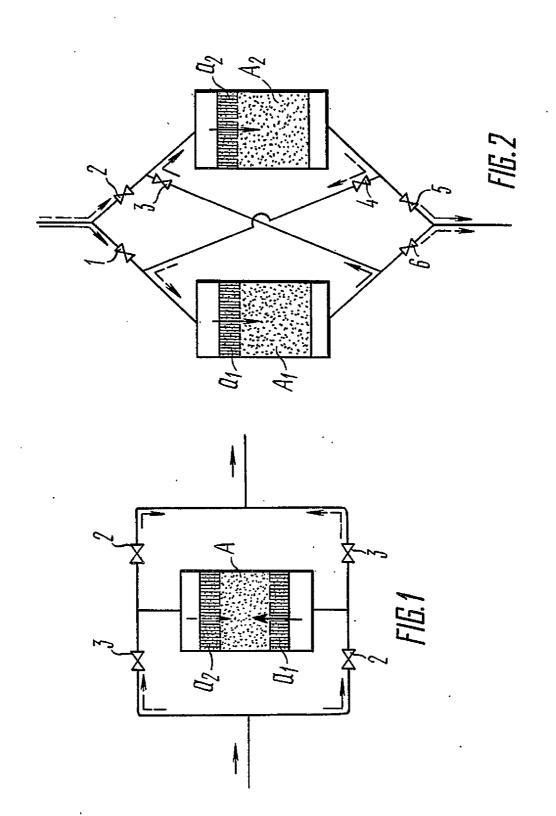
 Method of producing sulphur trioxide
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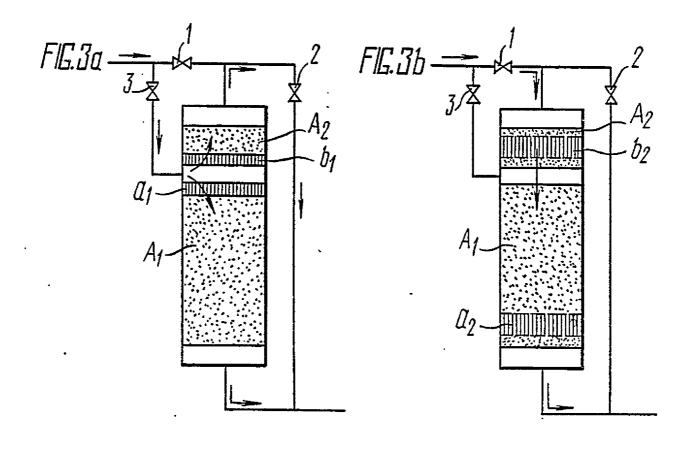
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METHOD OF PRODUCING SULPHUR TRIOXIDE

The present invention relates to processing sulphurous gases and more particularly to a method of producing sulphur trioxide.

Known in the art are methods of producing sulphur triox-5 ide by oxidation of sulphur dioxide with subsequent production of sulphuric acid. These methods are performed in contact apparatus with several (most frequently with 4 or 5) adiabatic beds of a catalyst and with built-in or separate heat-exchangers between the beds. The initial gas enters the 10 contact apparatus at a temperature of 20-100°C and is heated in heat-exchangers by the reacted gas up to the temperature of the beginning of the reaction on the first catalyst bed equal to 390-440°C. The most intensive exidation takes place on the first bed where the degree of conversion reaches 15 0.7-0.8 and the gas leaving the catalyst has a temperature of about 600°C. The temperature conditions in the apparatus on all catalyst beds is kept constant. The known methods are used for oxidizing gases with a sulphur dioxide content of 7.5-12 vol. %, these gases being obtained either by burning elemental 20 sulphur or by reasting sulphur-containing ores. ("Spravochnik

sernokislotchika" published in 1971, "Khimiya" Moscow, p.481).

Implementation of such known methods requires complex contact apparatus. The known methods also make possible the production of sulphur trioxide from waste gases, for instance, in non-ferrous metallurgy, with the sulphur dioxide content in them of 3.5-7.0 vol.%, the surface area of heat-exchangers in contact apparatus being considerably increased.

For exidation of gases with sulphur diexide content less than 3.5 vol.% constant heat supply is required, for 30 which purpose mazut or natural gas is used as a fuel.

Gases with sulphur dioxide content less than 2.0-2.5 vol. are mostly not processed, since the process becomes uneconomical and the gases are discharged into the etmosphere, thus contaminating and poisoning the environment.

Gases containing more than 12 vol.% of sulphur dioxide are not processed on conventional catalysts because of high and prolonged overheating of the catalyst in the reaction zone (above 650° C).

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It is partically impossible to process by the known methods, without recourse to special measures, waste gases with a variable content of sulphur dioxide in non-ferrous metallurgy when the content varies during short periods of time. It follows from the above that the known methods are technologically complicated and do not allow the processing of gases with low, variable, and high content of sulphur dioxide within a wide range of concentrations both constant and variable with time.

The present invention provides a method of producing sulphur trioxide by oxidizing sulphur dioxide, the initial reaction mixture comprising from 0.6 to 15% by volume sulphur dioxide, comprising introducing the said initial reaction mixture having a temperature of from 20 to 200°C into a stationary catalyst bed, part of the said catalyst bed having a maximum temperature of from 350 to 600°C, and either changing the direction of reaction mixture admission every 10 to 200 minutes by reversing the direction of the inflow of initial reaction mixture in the catalyst bed or varying the temperature of the initial reaction mixture cyclically before the catalyst bed from a minimum of 20 to 200°C to a maximum of 350 to 600°C over a period of 10 to 200 minutes.

The method according to the invention of producing sulphur trioxide will have high technical and economic characteristics and make it possible to process gases having a composition either constant or varying with time with a sulphur dioxide content of 0.6 to 15.0 vol.%.

Carrying out the method according to the invention allows for using simple and cheap contact apparatus of enhanced reliability and decreasing their metal capacity 10-20 times as compared with the known ones. In addition, it becomes possible to process gases with a sulphur dioxide content in a wide range of concentrations both constant and varying with time. Thus, for instance, gases containing 0.6 - 2.5 vol.% of sulphur dioxide, usually discharged into the atmosphere,

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can be processed without additional heat supply. It is expedient to carry out such method of oxidation at any low initial temperatures when the sulphur dioxide content in the reaction mixture varies with time from 0.6 to 5% or from 5 2 to 9 vol.%.

Here and hereafter the term "initial reaction mixture" is used to denote the gas at the input of the contact apparatus and at the input to the reaction zone, including the unreacted sulphur dioxide; the term "reaction mixture" is used to denote gases containing both sulphur dioxide and sulphur trioxide, residing in the regions of the beginning and end of the chemical reaction; the term "reacted reaction mixture" is used to denote gases at the outlet from the reaction zone and contact apparatus and containing, predominantly, sulphur trioxide.

The method is preferably accomplished in the following way. An initial reaction mixture with sulphur dioxide content of 1.0 or 10 vol.% is fed at 20°C onto the catalyst heated up to 500°C. The temperature of the mixture rises because of direct contact with the catalyst and at 350°C the reaction of oxidation begins. The part of the catalyst located at the input of the initial reaction mixture is cooled down to the temperature of the mixture, thus playing the role of a regenerative hest-exchanger. As a result of such mechanism of heat transfer along the catalyst bed, a moving heat front (reaction zone) arises whose temperature profile is determined by the initial concentration of sulphur dioxide and other parameters.

In order to keep the moving heat front of the reaction 30 within the catalyst bed, it is necessary to reverse periodically the direction of the movement of the initial reaction mixture during, say, 60 minutes. The cooling of that part of the catalyst begins which is adjacent to the input of the initial reaction mixture and the heat front reverses its direction.

Appropriate choice of a number of technological parameters (linear velocity, switching-over time, etc.) eliminates prolonged overheating of the catalyst when gases with a high content of sulphur dioxide (up to 15 vol.%) are processed and ensures an intensive course of the chemical reaction when processing gases with sulphur dioxide content as low as 0.6 to 2.5 vol.%.

The method eliminates the use of heat-exchangers or additional heat sources for heating the initial reaction 10 mixture since the catalyst bed itself acts as regenerative heat-exchangers, heating the initial reaction mixture and cooling the reacted mixture. A temperature regime close to a theoretical one is established thus ensuring a high degree of sulphur dioxide conversion into trioxide in one 15 catalyst bed.

The second variant of realizing the method . can be performed in a catalyst bed when the temperature reaction mixture periodically changes from 20-200°. to 350-600°C. If the reaction mixture entering 20 the catalyst bed has a high temperature (for instance, 420°C), the temperature profile is formed at the initial part of the catalyst bed where the chemical transformation takes place. A decrease of the temperature of the mixture to minimum values (for instance, 20°C) cools down the catalyst adjac-25 ent to the input. A heat front is formed in the bed which moves to the output and chemical transformation takes place in this front. ... When the heat front is close to the output of the reaction mixture from the catalyst bed, the temperature of the reaction mixture is raised again 30 up to maximum and the region of high temperatures is formed on the initial part of the catalyst bed where chemical transformation takes place. Then the initial temperature is lowered again and in the catalyst bed a heat front re-appears instead of that which has just left the bed. Such a method of oxidiz-35 ing sulphur dioxide in the catalyst bed favours the formation

of a temperature profile close, on the average, to theoretical thus ensuring a degree of conversion of sulphur dioxide into trioxide up to 0.98 in one catalyst bed.

The invention will be further described, by way of example only, with reference to the accompanying drawings, in which Figures 1,2 and 3 illustrate respective embodiments of the method of the invention.

Figure 1 shows the oxidation of the initial 10 reaction mixture in one catalyst bed. The initial reaction mixture with a sulphur dioxide content of 3.5 vol.% is fed at 20°C on the catalyst A pre-heated up to 500°C; the direction of the delivery is shown by solid arrows. In this case gates 2 are opened and valves 3 15 Direct contact with the catalyst raises the are closed. temperature of the initial reaction mixture up to 350°C, which corresponds to the temperature of the beginning of the reaction. As a result, a heat front a arises along the catalyst bed; in 60 minutes this front occupies the position a2. Ahead of the heat front, which moves in 20 the direction of filtration, a zone of the catalyst appears cooled to a temperature of the initial reaction mixture (20°C). In 60 minutes valves 2 and 3 are switched over simultaneously and the initial reaction mixture 25 reverses the direction of its movement (dashed arrow), The reaction front ap moves in an opposite direction and

in 60 minutes occupies the position a₁. After this a complete cycle equal to 120 minutes repeats which provides a continuous output of the reacted reaction mixture from the catalyst bed.

It is seen from Figure 1 that, upon moving the heat from "a" between positions a₁ and a₂, there are catalyst zones ahead and behind the front which do not participate in the chemical reaction but play the role of regenerative heat-exchangers heating the initial reaction mixture with a temperature of 20°C to the temperature corresponding to the beginning of the reaction and cooling the reacted reaction mixture at the expense of transfer of the reaction heat to the catalyst cooled to 20°C.

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Fig. 2 illustrates the oxidation scheme which is realized when the temperature of the reaction mixture changes from 20-200° to 350-600°C during 10-200 minutes. The initial reaction mixture is fed into the catalyst bed in one direc-5 tion. In the catalyst divided into two equal parts A and A2 the heat front of the reaction periodically moves from position a1 to a2 and then according to the scheme a2-a1-a2--a1-a2-a1 etc. The movement of the heat front is performed by alternating switch over of the valves 1-6. The reacted 10 reaction mixture is removed from the catalyst bed in the direction pointed by the arrows. For instance, the initial reaction mixture with a sulphur dioxide content of 9 vol.% and a temperature of 70°C is delivered to the catalyst A_1 pre--heated to 500°C in the direction indicated by solid arrows. The heat front thus arising begins to move from the position a, to a. The gates 1, 3 and 5 are opened whereas 2, 4 and 6 are closed. In 100 minutes (duration of semi-cycle) the reaction mixture with a temperature 500°C passes into a non-heated part Ao of the bed. At this instant the 1, 3 gates become closed, the gate 2 becomes opened and the initial reaction mixture with a temperature of 70°C is fed into the part A, of the catalyst bed. When the gates 1, 3 are completely closed and gate 2 is completely opened, the gates 4, 5 and 6 begin to operate simultaneously. The gate 5 becomes closed and gates 4, 6 become opened; the reacted reaction mixture from the part A2 of the bed is fed into part A1 and removed from the catalyst (dashed arrows). When the heat front passes from the position a to the position a2 and vice versa both in upper and low zones of the parts A_1 and A_2 , the temperature in the zones rises from 70°C to maximum 600°C and lowers from 600°C to 70°C. The mechanism of the movement of the heat front along the catalyst bed with an adternating formation of hot and cold zones of the catalyst, which play the role of heat--exchangers, is similar to that shown in Fig. 1. The successive switching over of the gates 1-6 ensures a continuous

movement of the heat front according to the scheme $a_1-a_2-a_1-a_2$ and so on in one direction and, consequently, a continuous removal of the reacted reaction mixture from the catalyst bed.

. Fig. 3 illustrates one more scheme of oxidation which 5 is also realized upon changing the temperature of the mixture from 20-200° to 350-600°C during 10-200 minutes. The initial reaction mixture is fed into a catalyst bed, divided into two unequal parts A1 and A2, periodically in two directions. The part A2 of the bed serves as a "primer" of the main bed A4; 10 the temperature of the mixture entering the part A2 is periodically changed. For instance, an initial reaction mixture with a sulphur dioxide content of 0.6 vol.% at a temperature 200°C is fed on the catalyst A_1 and A_2 pre-heated up to 500°C in the direction shown by the arrows (Fig. 3a). In each part 15 A_1 and A_2 two heat fronts arise (a_1 and b_1) which begin to move in opposite directions. The gate 4 is closed and the gate 3 is opened; the gate 2 is opened incompletely thus controlling a slow movement of the heat front b_1 with respect to the movement of a. In 60 minutes the heat fronts occupy 20 positions a2 and b2 (Fig. 3b) after which the gates 2 and 3 become closed and the gate 1 becomes opened and the initial reaction mixture is delivered in the direction shown by arrows. Sulphur dioxide contained in the initial reaction mixture oxidizes in the reaction zone b2. Since no sulphur dioxide 25 enters zone az, the reaction in said zone does not take place and the heat front a2 removes from the bed. In 10 minutes the heat front b2 occupies the position shown in Fig. 3a and splits into two heat fronts a and b . In this position the gates 1, 2 and 3 are switched over and the cycle, whose 30 duration is 70 minutes, repeats. In front of the part A, of the catalyst bed, where the mixture enters the catalyst, the temperature of the catalyst is periodically (each 70 minutes) changed from 200° to 350-600°C. Successive switching

over of the gates 1, 2 and 3 provides a pulse arising and 35 damping of the heat front a₁ which ensures a continuous removal

of the reacted reaction mixture from the catalyst bed.

The part A₂ of the catalyst periodically acts as a "primer".

Example 1

An initial reaction mixture formed during combustion of sulphur and consisting of 10.5 vol.% of sulphur dioxide, 10.5 vol.% of oxygen, and 79 vol.% of nitrogen is fed into a contact apparatus with one adiabatic catalyst bed (Fig. 1). The composition of the catalyst is as follows (wt.%):

V₂O₅ 6-9 K₂O 10-16 SO₃ 8-12 Support SiO₂ the balance.

The temperature of the initial reaction mixture is 20°C; the conditional contact time about 6 sec. Prior to introduction of the initial reaction mixture, the bed is heated to 500°C.

In this example the direction of movement of the reaction mixture flow (Fig. 1) is reversed by interchanging the input and output of the mixture in 40 minutes. In next 40 minutes the direction of movement of the initial reaction mixture is reversed again, and so on. Duration of one cycle is 80 minutes. An average degree of sulphur dioxide conversion into trioxide is 98.3% per cycle which is equivalent to operation in stationary regime of a contact apparatus with five adiabatic catalyst beds with intermediate heat-exchangers.

Example 2

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An initial reaction mixture formed during calcination of pyrite and consisting of 7.5 vol.% of sulphur dioxide, 10.5 vol.% of oxygen, and 82 vol.% of nitrogen is delivered into a contact apparatus with one adiabatic catalyst bed (Fig. 1). The composition of the catalyst is similar to that described in Example 1. The temperature of the initial reaction mixture is 150°C; the conditional contact time is 5 sec. Prior to introduction of the initial reaction mixture,

the bed is heated up to 550°C. The direction of movement of the reaction mixture is reversed each 55 minutes; a cycle duration is 110 minutes. The degree of sulphur dioxide conversion into trioxide is 98.5% per cycle.

Example 3

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An initial reaction mixture formed during combustion of sulphur and consisting of 12 vol.% of sulphur dioxide, 11 vol.% of oxygen, and 78 vol.% of nitrogen is delivered into a contact apparatus with one adiabatic catalyst bed (Fig. 1). The composition of the catalyst is similar to that described in Example 1. The temperature of the initial reaction mixture is 20°C; the conditional contact time about 6 sec. Prior to introduction of the initial reaction mixture, the catalyst bed is heated up to 500°C. Each 30 minutes the direction of the movement of the reaction mixture is reversed; cycle duration is 60 minutes. An average degree of sulphur dioxide conversion into trioxide is 98.1% per cycle.

Example 4

The procedure is similar to that described in Example 1. Conditional contact time is 7 sec; cycle duration 200 minutes. An average conversion degree is 98.0% per cycle.

Example 5

The procedure is similar to that described in Example 1. The conditional contact time is 5 sec; cycle duration 10 minutes. An average conversion degree is 98.5% per cycle.

Example 6

The procedure is similar to that described in Example 1. The composition of the initial reaction mixture is 0.6 vol.% of sulphur dioxide, 5 vol.% of oxygen, and 94.4 vol.% of nitrogen, said mixture being fed into a contact apparatus with one catalyst bed. Cycle duration is 60 minutes; an average conversion degree is 99.3% per cycle.

Example 7

The initial reaction mixture and the catalyst are similar to those described in Example 1. The catalyst bed is divided into two equal parts; the mode of the catalyst

action is presented in description of Fig. 2. In this example the temperature of the reaction mixture is changed in front of the catalyst bed and its parts A, and A, (Fig. 2). The total amount of the catalyst in both parts corresponds to a conditional contact time 8 sec. The part A_1 of the bed is pre-heated up to 500° C. The temperature of the initial reaction mixture at the input of part A₄ is 20°C. The reaction mixture, after passing through parts A_1 and A_2 , leaves the latter and the apparatus at a temperature which changes smoothly from 10 20 to 350°C during 55 minutes (semi-cycle); then the gates are switched over in the succession given in description of Fig. 2. In 35 minutes after the beginning of the semicycle the temperature of the reaction mixture at the input to the upper part A2 of the bed attains 250°C, in the next 10 minutes it attains 280°C, and in another 5 minutes - 320°C; towards the end of the semi-cycle (55 minutes) the temperature attains 350°C. After switching over the gates in 55 minutes the initial reaction mixture is fed to the part A_2 of the bed in the direction shown by dashed 20 arrows. In the next 55 minutes the heat front from the position a, passes completely into the position a, and the cycle with duration 110 minutes is repeated. An average conversion degree is 98.4% per cycle which is equivalent to the operation of a contact apparatus with five successively 25 located catalyst beds and intermediate heat-exchangers.

Examle 8

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The initial data are similar to those given in Examples 7 and 1. The difference consists in that switching over of the gates in 55 minutes are re-distribution of the entering initial reaction mixture between the catalyst parts A_1 and A_2 (see Fig. 2) are performed when heat fronts a_1 and a_2 are successively placed in the middle of the parts A_1 and A_2 . The succession of switching over the gates is the same as in description of Fig. 2. A semi-cycle (55 minutes) begins when the heat front a_1 occupies the position in the

middle of A_1 and the initial reaction mixture at a temperature of 20°C is fed in the direction shown by solid arrows (Fig. 2). The heat front passes through the upper part A_2 of the bed with the same temperatures as those described in Example 7 and in 55 minutes it occupies the position a_2 in the middle of A_2 . The gates are switched over and the initial reaction mixture at 20°C is delivered into the upper part A_2 of the bed in the direction shown by dashed arrows (Fig. 2). In next 55 minutes, i.e. in 110 minutes of the complete cycle, the heat front a_2 occupies the position a_1 (in the middle of A_1) and the cycle is repeated. During each semi-cycle the reacted reaction mixture is removed successively from the parts A_1 and A_2 of the bed and from the apparatus, the temperature of said mixture being smoothly changed from 20 to 350°C.

Example 9

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The example is similar to Example 7. The initial reaction mixture consisting of 0.6 vol.% of sulphur dioxide, 15 vol.% of oxygen and 84 vol.% of nitrogen is successively delivered to the parts A_1 and A_2 of the bed (Fig. 2) at a temperature of 200°C. Cycle duration is 75 minutes. From cycle to cycle the temperature changes smoothly from 200° to 600°C and lowers down to 200°C at the inputs of the mixture into the parts A_1 and A_2 . An average conversion degree is 99.4% per cycle.

Example 10

The Example is similar to Example 7. The reaction mixture with a sulphur dioxide content varying in time from 0.6 to 7 vol.% and oxygen content 9-10 vol.% is successively delivered to the parts A₁ and A₂ of the catalyst bed (Fig. 2) at 100°C. Cycle duration is 80 minutes. An average conversion degree is 98.7% per cycle.

Example 11

An initial reaction mixture formed during combustion of sulphur and consisting of 12 vol.% of sulphur dioxide,

9 vol. % of oxygen, and 79 vol. % of nitrogen is fed into a contact apparatus with a "primer" as is shown in Fig. 3. The catalyst is pre-heated to 500°C, conditional contact time 9 sec. The temperature of the initial reaction mixture is 20°C. The initial reaction mixture is delivered both to the part A_1 and A_2 (Fig. 3a) for 170 minutes after which the heat fronts a1 and b1 occupy the positions a2 and b2. During next 30 minutes the initial reaction mixture is fed into the bed A_1 through a "primer" A_2 as is shown in Fig. 3b. During this period of time the heat front b, occupies the position a_1 and b_1 whereas the heat front a_2 is removed from the part A_1 . When the heat front b_2 is split, in the upper part of the bed A and in the lower part of the bed A the temperature at the moment of switching over of the gate is 20°C, in next 15 minutes - 180°C, in 5 minutes - 350°C, and in 30 minutes - 600°C. At this moment the gates are switched over again in the same succession which is given in description of Fig. 3. The initial reaction mixture is fed at 20°C into a layer between the parts A_1 and A_2 of the catalyst bed. The system occupies the position shown in Fig. 3a which corresponds to the cycle duration 200 minutes. An average conversion degree is 98.1% per cycle which is equivalent to the operation of a contact apparatus with five adiabatic

Example 12

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The Example is similar to Example 11. The initial reaction mixture is delivered on the catalyst at 200°C. Cycle duration is 150 minutes. An average conversion degree is 98.2%.

catalyst beds and with intermediate heat-exchangers.

Example 13

The Example is similar to Example 11. The initial reaction mixture containing 1.5 vol.% of sulphur dioxide, 15 vol.% of oxygen, and 83.5 vol.% of nitrogen are processed at a cycle duration of 130 minutes. An average conversion degree is 99.3% per cycle.

Example 14

An initial reaction mixture formed upon combustion of sulphur and containing 15 vol.% of sulphur dioxide, 11 vol.% of oxygen, and 74% of nitrogen is fed into a contact apparatus 5 with one catalyst bed. Any scheme shown in Figs 1, 2 and 3 can be realized. The temperature of the initial reaction mixture at first stage is 50°C; conditional contact time - 5 sec. Prior to introduction of the initial reaction mixture, the catalyst bed is pre-heated to 500°C. An average degree . 10 of sulphur dioxide conversion into trioxide at this stage is 94%, cycle duration 110 minutes. Such a conversion degree is relatively low and additional oxidation of the non-oxidized sulphur dioxide is required. For this purpose, the reaction mixture obtained is delivered to the first absorption stage after which the reaction mixture containing 0.75 vol.% of 15 sulphur dioxide, 3.6 vol.% of oxygen, and nitrogen - the balance is fed into the second catalyst bed at 60°C and conditional contact time 5 sec. Said second catalyst bed is the second stage of contacting. There the reaction mixture 20 is oxidized up to a high conversion degree (99.8%), the duration of the cycle being 80 minutes. The scheme of the contact apparatus may be similar to that chosen for the first stage or be any one of those shown in Figs 1, 2 or 3. The catalyst for the first and second stage is similar to that used in 25 Example 1.

Example 15

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An initial reaction mixture formed upon burning sulphur in oxygen saturated air and consisting of 15 vol.% of sulphur dioxide, 12 vol.% of oxygen, and 73 vol.% of nitrogen is fed to the first stage of contacting. The rest conditions are similar to those described in Example 14. After first stage the conversion degree is 94.2% and the duration of the cycle 160 minutes. The reaction mixture after the first absorption stage goes to the second stage of contacting with a sulphur dioxide content 0.886 vol.%, 4.07 vol.% of oxygen, and

nitrogen - the balance. Cycle duration is 80 minutes; the total conversion degree 99.87%.

Example 16

The Example is similar to Example 1. The difference consists in that a sulphur dioxide content in the reaction mixture changes with time at random from 0.6 to 5%, whereas an oxygen content remains almost constant and equals 9%. Cycle duration is 80 minutes, an average conversion degree 98.6%.

10 Example 17

The Example is similar to Example 7. The difference consists in that a sulphur dioxide content in the initial reaction mixture changes with time from 2 to 9 vol.% whereas an oxygen content remains almost constant and equals 12 vol.%.

Cycle duration is 90 minutes; an average conversion degree 98.2%.

Example 18

The Example is similar to Example 14. The difference resides in that sulphur dioxide content is 11.5% oxygen content 20 is 9.5%, and nitrogen is the balance. The conversion degree of sulphur dioxide at first stage of contacting is 94.6%; after second stage the conversion degree is 99.9%.

The present invention may find application in the manufacture of sulphuric acid.

CLAIMS:

- 1. A method of producing sulphur trioxide by oxidizing sulphur dioxide, the initial reaction mixture comprising from 0.6 to 15% by volume sulphur dioxide,
- comprising introducing the said initial reaction mixture having a temperature of from 20 to 200°C into a stationary catalyst bed, part of the said catalyst bed having a maximum temperature of from 350 to 600°C, and either changing the direction of reaction mixture admission
- 10 every 10 to 200 minutes by reversing the direction of the inflow of the initial reaction mixture in the catalyst bed or varying the temperature of the initial reaction mixture cyclically before the catalyst bed from a minimum of 20 to 200°C to a maximum of 350 to 600°C over a period of 10 to 200 minutes.
 - 2. A method as claimed in Claim 1, wherein the sulphur dioxide content in the reaction mixture varies with time from 0.6 to 5% by volume.
- A method as claimed in Claim 1, wherein the sulphur
 dioxide content in the reaction mixture varies with time
 from 2 to 9% by volume.
 - 4. A method according to Claim 1 of producing sulphur trioxide, substantially as herein described with reference

to Figure 1, Figure 2 or Figure 3 of the accompanying drawings.

- 5. A method according to Claim 1 of producing sulphur trioxide, substantially as herein described in any of5 the foregoing Examples.
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