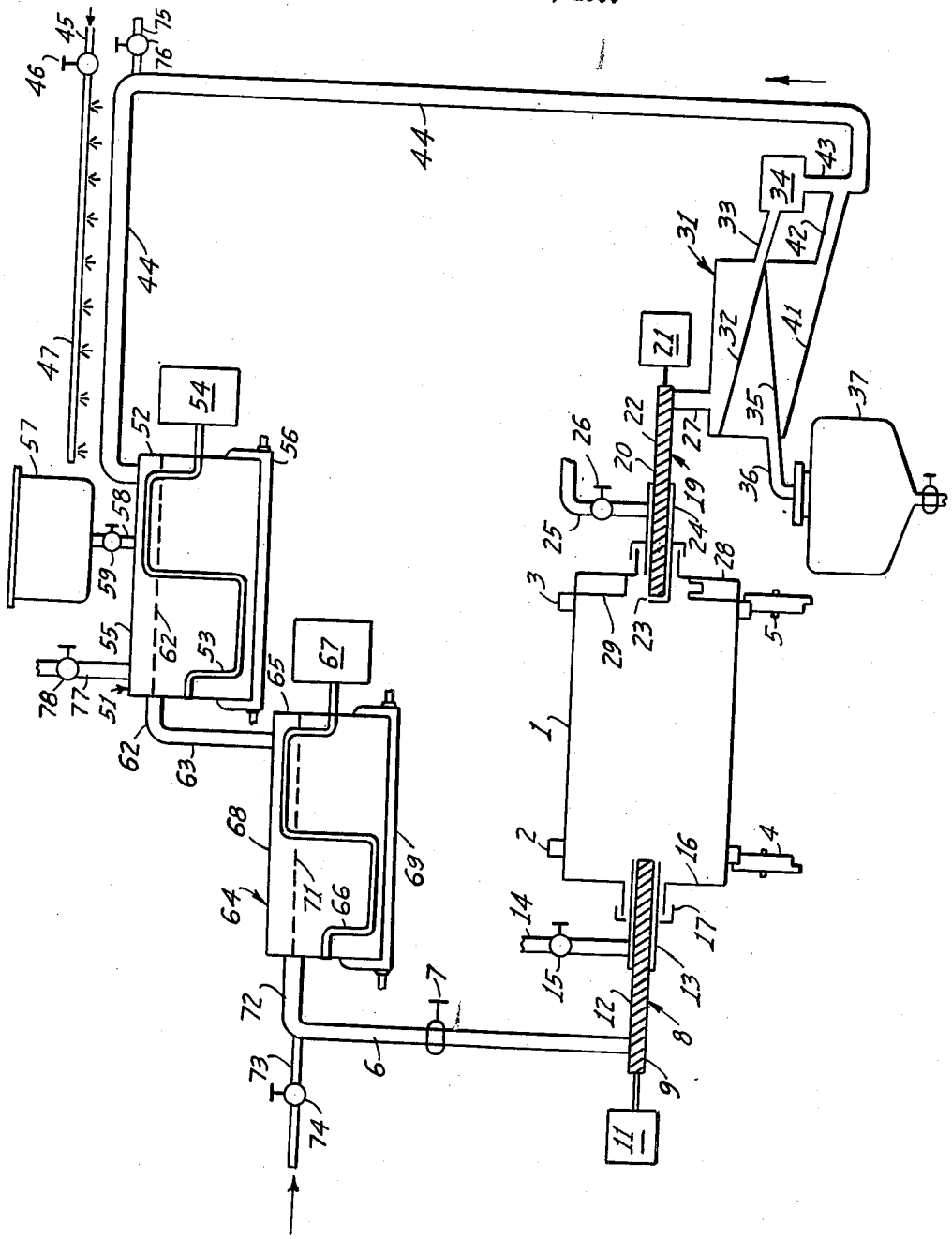
Aug. 3, 1954

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2,685,500

PROCESS FOR GENERATING SODIUM MONOXIDE

Filed March 29, 1950



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Patented Aug. 3, 1954

2,685,500

UNITED STATES PATENT OFFICE

2,685,500

PROCESS FOR GENERATING SODIUM MONOXIDE

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Application March 29, 1950, Serial No. 152,623

10 Claims. (Cl. 23—184)

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This invention is a new and useful process for generating sodium monoxide and, inter alia, for generating sodium peroxide from the sodium monoxide so formed.

The invention will be fully understood from the following description read in conjunction with the drawing, which is a diagrammatic showing of apparatus in which the invention may be carried into effect.

We have found that sodium reacts rapidly with sodium peroxide to form sodium monoxide at approximately 250° C. and above. In the first stage of our invention we maintain a mass of sodium peroxide in the state of continuous movement and/or agitation, and at a temperature above the melting point of sodium but below the said temperature at which sodium reacts rapidly with sodium peroxide. We preferably maintain a temperature in the said mass between 130 and 200° C. The mass of sodium peroxide is maintained in an atmosphere substantially free from oxygen and inert with respect to sodium peroxide and/or sodium. To this mass while so maintained in an inert atmosphere, we add sodium to the extent only of a minor part by weight of the sodium peroxide, and normally to the extent only of from 1–10% by weight of the said peroxide. The amount of the sodium added is preferably from 3–8% by weight of the sodium peroxide. At this temperature and under these conditions the sodium does not react with peroxide or otherwise, but disseminates itself uniformly through the finely divided sodium peroxide, superficially wetting the individual particles of peroxide. The sodium peroxide preferably is below 20 mesh.

In the next step of our process, the sodium peroxide carrying the said sodium, uniformly disseminated throughout, is elevated to a temperature at which reaction takes place between the said sodium and the said sodium peroxide, resulting in the formation of sodium monoxide. This reaction takes place at practical rates at 260° C. and at higher temperatures is almost instantaneous. While the reaction takes place almost instantly at higher temperatures, it is still preferable to exclude the presence of any oxidizing gas at this point, and we therefore preferably maintain the reactants at this stage in an atmosphere substantially free from oxygen and inert with respect to these materials.

These two steps are preferably carried out continuously by the continuous or intermittent addition of sodium and sodium peroxide to a mass of sodium peroxide in a condition of motion or

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agitation and by the continuous abstraction, from said mass, of sodium peroxide wetted with sodium, which is continuously introduced into a second mass of sodium peroxide and sodium undergoing reaction to sodium monoxide and held at a temperature at which the sodium and sodium peroxide react to form sodium monoxide.

While the two steps hereinbefore described may be used to produce sodium monoxide as the end product, they are preferably used as steps in the manufacture of sodium peroxide, and for this purpose a part of the sodium monoxide formed in the second stage is continuously abstracted and preferably added continuously to a third maintained mass of sodium peroxide coated with sodium monoxide, which sodium monoxide is undergoing oxidation to the peroxide. For this purpose the third mass is maintained at a temperature between 250 and 490° C. and preferably from 350–425° C., and at this temperature is continuously contacted with a dry oxygen-containing gas, which may be either dry air or air which has been enriched in oxygen or relatively pure oxygen. The pressure of the atmosphere in contact with this mass is preferably slightly above atmospheric, although higher pressures may be employed and will in fact accelerate the oxidation reaction.

We prefer a cyclic process of oxidation in which sodium peroxide is continuously abstracted from the said third mass and in part returned with intermediate cooling to the said first mass. The product of the process is of commercial quality running over 96% Na_2O_2.

Referring to the drawing 1 designates a reactor of the rotary kiln type. The reactor is provided with circular rims 2 and 3 carried by flanged wheels, such as 4 and 5. Some of the wheels are driven by suitable means (not shown) to impart a gradual turning movement to the reactor. Provision (not shown) is also made for heating the reactor to keep it at the desired operating temperature of from 400 to 430° C. Material may be introduced into the reactor from pipe 6 controlled by valve 7 discharging into worm conveyor 8 consisting of worm 9 driven by any suitable means diagrammatically indicated by 11 and housed in pipe 12 which extends into the interior of the reactor. Pipe 12 is in turn surrounded by pipe 13. Pipes 12 and 13 collectively form an annular duct extending into the interior of the reactor connected to pipe 14 controlled by valve 15. The space between the exterior of pipe 13, which is stationary, and end 16 of reactor 1, which is rotatable, is sealed by

stuffing box 17. At the other end 28 of the reactor, worm conveyor 19 extends to a point within the reactor. This consists of a worm 20 driven by any suitable means diagrammatically indicated by 21 housed in pipe 22 which extends to a point within the reactor and terminates in trough 23. Pipe 22 is surrounded by pipe 24. Pipes 22 and 24 collectively form an annular space extending into the interior of the reactor communicating with pipe 25 controlled by valve 26. The conveyor 18 discharges into pipe 27. End 28 of reactor 1 carries lifts 29 so that when the reactor is being rotated, solid material in the reactor will be lifted and discharged into the open top of trough 23, and can thereby be removed from the reactor by operating the worm conveyor 19. Pipe 27 discharges into vibrating screens 31. Of these the topmost screen 32 is of about 20 mesh and the material retained on this screen is diverted through pipe 33 to grinder diagrammatically indicated by 34. Material passing through screen 32 is discharged onto screen 35, which is of about 40 mesh, and the material retained on screen 35, which will be approximately 20–40 mesh, is discharged through chute 36 into tank 37. Material passing through screen 35 which will be finer than 40 mesh is caught in pan 41 and discharged thence through chute 42 into pipe 43. The grinder 34 is operated to yield a product which will pass 40 mesh, and the combined products from grinder 34 and pan 41 are picked up and transported by enclosed conveyor 44, which may be of the so-called Redler type. Provision is made for cooling material transported by this conveyor, diagrammatically indicated by pipe 45 controlled by valve 46 discharging through shower head 47, by which coolant may be distributed over the outer surface of the conveyor housing. The conveyor 44 discharges into mixer 51. This consists of tank 52 carrying mixer bar 53 driven by any suitable means diagrammatically indicated by 54 and provided with cover 55. The contents of the mixer may be held at any desired temperature by indirect heat exchange with liquid circulating in jacket 56. Sodium may be introduced to mixer 51, either continuously or intermittently, from tank 57 through pipe 58 controlled by valve 59. Any excess of material in mixer 51 over that necessary to maintain the level indicated by line 61 will overflow through side outlet 62 and chute 63 into mixer 64. This consists of tank 65 carrying mixer bar 66 driven by any suitable means diagrammatically indicated by 67. The tank is provided with cover 68 and the contents of the tank may be held at any desired temperature by indirect heat exchange with a liquid circulating in jacket 69. Material supplied to mixer 64 over that necessary to maintain the level indicated by line 71 will overflow through side outlet 72 into pipe 6. An inert gas, such as nitrogen, may be introduced through pipe 73 controlled by valve 74 to maintain an inert atmosphere in mixers 51 and 64 passing thence counter-current to incoming material in conveyor 44 and discharging from the system through pipe 75 controlled by valve 76. Material may be introduced to mixer 51 through pipe 77 controlled by valve 78.

In starting the process nitrogen is fed through pipe 73 and exhausted through pipe 75 until an inert atmosphere is present in mixers 51 and 64, and in the horizontal portion of conveyor 44. Dry air is fed into reactor 1 through pipe 25 until all moist air is displaced through pipe 14. Then sodium peroxide is fed into reactor 51 until the level reaches 62, whereupon the peroxide overflows until the level reaches line 71 in reactor 64 and then the level builds up in reactor 1 to one-third to one-half of the depth of the reactor. The feeding of sodium peroxide is discontinued when 51, 64 and 1 have reached normal levels indicated above. During this period the temperature in mixer 51 is brought to 130–225° C., preferably 130–200° C., by circulation of a suitable hot liquid in jacket 56; the temperature in mixer 64 is brought to at least 260° C., preferably between 260 and 400° C., by circulation of a suitable liquid in jacket 69; the temperature in reactor 1 is brought to 250°–490° C., preferably 400–430° C., by indirect oil or gas firing. The auxiliary equipment including conveyor 19, screen 31, grinder 34, conveyor 43, and cooling system 47, are then started up and normal rates of flow established in the system.

Then sodium is fed continuously or intermittently through pipe 58 to the extent of 1–10%, preferably 3–8%, of the feed of sodium peroxide into reactor 51 from conveyor 44. By operation of the mixer bar 53 the sodium is thoroughly incorporated with the sodium peroxide at a temperature between 130 and 225° C., preferably between 130–200° C. At this temperature the sodium and sodium peroxide do not react and the sodium wets the particles of sodium peroxide superficially, disseminating itself uniformly throughout the mass. As sodium peroxide and sodium continue to be fed into mixer 51, the excess overflows through pipe 63 into reactor 64, in which it is kept in a state of continuous agitation by mixer bar 66 at a temperature above 260° C., preferably between 260 and 400° C. This causes reaction to take place between the sodium peroxide and the sodium on the surface, with the resultant formation of a superficial layer or coating of sodium monoxide. As more sodium peroxide wetted with sodium is fed into this reactor and each particle is converted to monoxide to a depth approximately equivalent stoichiometrically to the percent of sodium originally on the surface, the product overflows through side outlet 72 into pipe 6 and is transported by worm conveyor 8 into reactor 1. The heat of reaction in the conversion step is sufficient to raise the temperature to 300–450° C. This temperature rise is advantageous since it reduces heat requirements necessary in the next succeeding oxidation stage.

The contents of reactor 1 are held at a temperature between 250 and 490° C., preferably between 400 and 430° C. Dry oxidizing gas such as air, air enriched with oxygen, or pure oxygen is introduced through pipe 25, the residue thereof passing out of the system through pipe 14. The amount of oxygen so introduced is slightly greater stoichiometrically than the sodium monoxide simultaneously introduced, with the resulting conversion of the entire material to sodium peroxide of commercial quality (above 96% Na_2O_2). The retention time in reactor 1 is comparatively short, one-half to one and one-half hours, depending on percentage sodium addition, since only the superficial layer of monoxide on the surface must be oxidized.

The product passes through screen 31 where the oversize material is scalped off by screen 32 ground in grinder 34 and returned to mixer 51 together with undersize material passing through screen 35. The desired product is retained by screen 35 and passes into storage bin 37. We

prefer to use 20 mesh screen for 32 and 40 mesh screen for 35 but other screen sizes may be chosen. The above conditions are maintained and the system operated continuously until it becomes necessary to shut down for maintenance.

The foregoing description is for purposes of illustration and not of limitation and it is therefore our intention that the invention be limited only by the appended claims or their equivalent wherein we have endeavored to claim broadly all inherent novelty.

We claim:

1. In the process of generating particles of sodium peroxide by the successive formation of a superficial layer of sodium monoxide followed by the oxidation of such layer of sodium monoxide with oxygen to sodium peroxide, the steps of mixing sodium with a larger amount by weight of sodium peroxide at a temperature above the melting point of sodium and below the temperature of reaction between sodium and sodium peroxide in an atmosphere substantially free from oxygen and inert with respect to sodium and sodium peroxide, thereby forming a film of sodium upon the particles of sodium peroxide and thereafter, while maintaining thereon an atmosphere substantially free from oxygen and inert with respect to sodium and sodium peroxide, elevating said sodium peroxide carrying said superficial film of metallic sodium to a temperature at which reaction takes place between said sodium and said sodium peroxide, thereby forming a superficial layer of sodium monoxide upon said particles of sodium peroxide.

2. Process according to claim 1 in which the said sodium mixed with said sodium peroxide is from 1–10% by weight of said sodium peroxide.

3. In the process of generating particles of sodium peroxide by the successive formation of a superficial layer of sodium monoxide followed by the oxidation of such layer of sodium monoxide with oxygen to sodium peroxide, the steps of mixing sodium with a larger amount by weight of sodium peroxide at a temperature above the melting point of sodium and below 250° C. in an atmosphere substantially free from oxygen and inert with respect to sodium and sodium peroxide, thereby forming a film of sodium upon the particles of sodium peroxide and thereafter, while maintaining thereon an atmosphere substantially free from oxygen and inert with respect to sodium and sodium peroxide, elevating said sodium peroxide carrying said superficial film of metallic sodium to a temperature above 260° C. at which reaction takes place between said sodium and said sodium peroxide, thereby forming a superficial layer of sodium monoxide upon said particles of sodium peroxide.

4. Process according to claim 3 in which the said sodium mixed with said sodium peroxide is from 1–10% by weight of said sodium peroxide.

5. The cyclic process of generating sodium peroxide which comprises mixing sodium with a larger amount by weight of sodium peroxide in a maintained mass at a temperature above the melting point of sodium and below the temperature of reaction between sodium and sodium peroxide, in an atmosphere substantially free from oxygen and inert with respect to sodium and sodium peroxide, thereby forming a film of sodium upon the particles of sodium peroxide in said mass, abstracting from said mass particles of sodium peroxide carrying a superficial film of metallic sodium and, while maintaining thereon an atmosphere substantially free from oxygen and inert with respect to sodium and sodium peroxide, elevating said particles to a temperature at which reaction takes place between said sodium and said sodium peroxide, thereby forming a superficial layer of sodium monoxide upon said particles of sodium peroxide, and thereafter introducing said particles of sodium peroxide carrying such superficial film of sodium monoxide into a maintained mass of such particles in a dry oxidizing atmosphere and at a temperature at which sodium monoxide oxidizes to sodium peroxide, maintaining the particles so introduced in said mass until the superficial film of sodium monoxide thereon has become substantially completely oxidized by such atmosphere to sodium peroxide, withdrawing particles of sodium peroxide from said last mentioned maintained mass, abstracting a part of the same and recirculating the remainder to the said first mentioned maintained mass.

6. Process according to claim 5 in which the particles of sodium peroxide withdrawn from said last mentioned maintained mass are screened, particles of a size retained on said screen are withdrawn and particles of a size passing through said screen are recirculated to said first mentioned maintained mass.

7. The cyclic process of generating sodium peroxide which comprises mixing sodium with a larger amount by weight of sodium peroxide in a maintained mass at a temperature above the melting point of sodium and below 250° C., in an atmosphere substantially free from oxygen and inert with respect to sodium and sodium peroxide, thereby forming a film of sodium upon the particles of sodium peroxide in said mass, abstracting from said mass particles of sodium peroxide carrying a superficial film of metallic sodium and, while maintaining thereon an atmosphere substantially free from oxygen and inert with respect to sodium and sodium peroxide, elevating said particles to a temperature above 260° C. at which reaction takes place between said sodium and said sodium peroxide, thereby forming a superficial layer of sodium monoxide upon said particles of sodium peroxide and thereafter introducing said particles of sodium peroxide carrying such superficial film of sodium monoxide into a maintained mass of such particles in a dry oxidizing atmosphere and at a temperature at which sodium monoxide oxidizes to sodium peroxide, maintaining the particles so introduced in said mass until the superficial film of sodium monoxide thereon has become substantially completely oxidized by such atmosphere to sodium peroxide, withdrawing the particles of sodium peroxide from said last mentioned maintained mass, abstracting a part of the same and recirculating the remainder to the said first mentioned maintained mass.

8. Process according to claim 7 in which the particles of sodium peroxide withdrawn from said last mentioned maintained mass are screened, particles of a size retained on said screen are withdrawn and particles of a size passing through said screen are recirculated to said first mentioned maintained mass.

9. A method for producing a product consisting essentially of particles having a sodium peroxide core and a relatively thin adherent outer layer of sodium monoxide said method comprising forming a thin layer of elemental sodium upon finely divided sodium peroxide particles undergoing agitation, the weight of said sodium layer on each peroxide particle not exceeding 5%

of the weight of the individual sodium peroxide particle treated, at a temperature above the melting point of sodium and below the temperature of reaction between sodium and sodium peroxide in an atmosphere substantially free from oxygen and inert with respect to the sodium and sodium peroxide components, heating the resulting mixture of individual sodium peroxide particles of sodium peroxide with adherent layers of sodium to a temperature at which a chemical reaction takes place between the sodium and said sodium peroxide, thereby forming the superficial adherent layer of sodium monoxide upon said particles of sodium peroxide.

10. A method for producing a product consisting essentially of particles having a sodium peroxide core and a relatively thin adherent outer layer of sodium monoxide said method comprising forming a thin layer of elemental sodium upon finely divided sodium peroxide particles undergoing agitation, the weight of said sodium layer on each peroxide particle not exceeding 5% of the weight of the individual sodium peroxide particle treated, at a temperature above the melting point of sodium and below the temperature of reaction between sodium and sodium peroxide in an atmosphere substantially free from oxygen and inert with respect to the sodium and

sodium peroxide components, heating the resulting mixture of individual sodium peroxide particles of sodium peroxide with adherent layers of sodium to a temperature at which a chemical reaction takes place between the sodium and said sodium peroxide, thereby forming the superficial adherent layer of sodium monoxide upon said particles of sodium peroxide, and thereafter subjecting said particles of sodium peroxide carrying such superficial layer of sodium monoxide to an oxidizing atmosphere and at a temperature at which sodium monoxide oxidizes to sodium peroxide to form thereby particles of sodium peroxide.

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