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(54) PROCESS FOR MANUFACTURING POTASSIUM NITRATE FERTILIZER AND OTHER METAL NITRATES

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Related U.S. Application Data

(63) Continuation-in-part of application No. 09/100,994, filed on Jun. 22, 1998, now patented.

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(51) **Int. Cl.**[7] **C01D 9/00**

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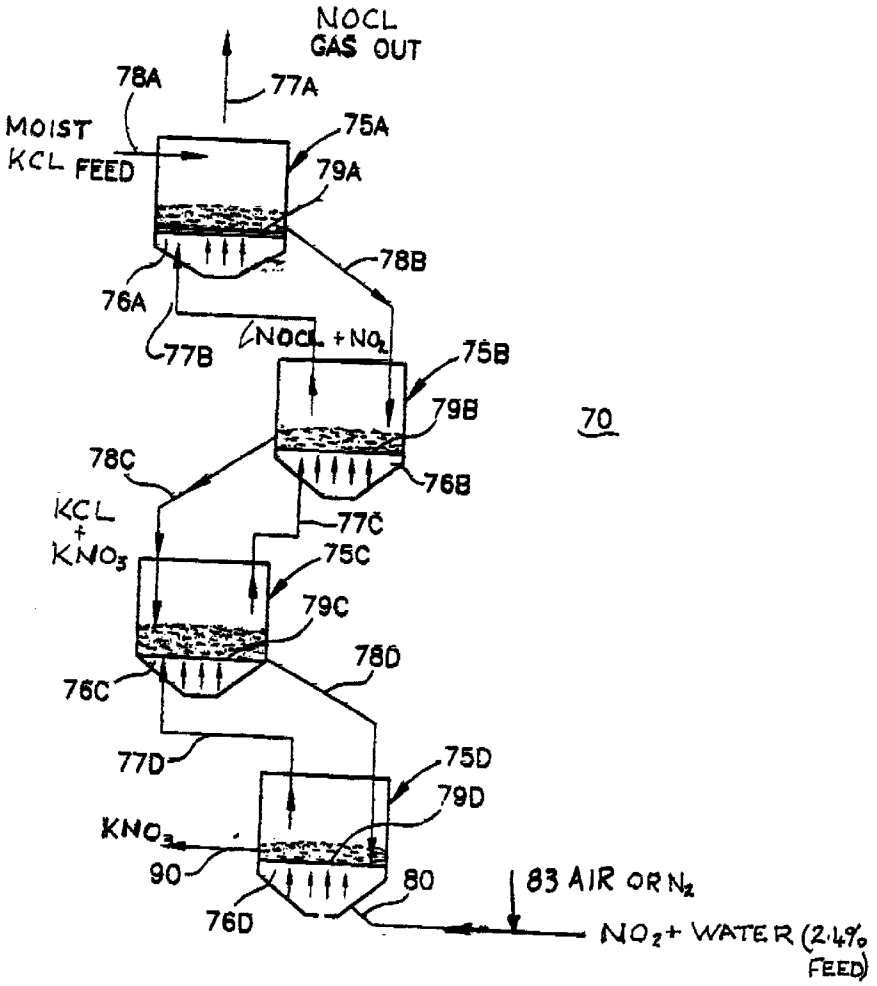
(57) **ABSTRACT**

A process for producing potassium nitrate and other metal nitrates from the chlorides, sulfates, oxides of these metals. The process uses nitrogen dioxide as a true fluidizing medium in shallow beds of the aforementioned solids at moderately elevated temperatures in a continuous counter current process to convert the metal chlorides, sulfates, and oxides, into metal nitrates and effluent gas and water vapor. The process may be carried out in a series of true fluidized beds arranged in a vertical configuration so that the solids flow downward due to the fluidized process and the nitrogen dioxide gas flows counter currently in an upward direction producing pure metal nitrates at the bottom and nitrosyl chloride gas and/or water vapor at the top.



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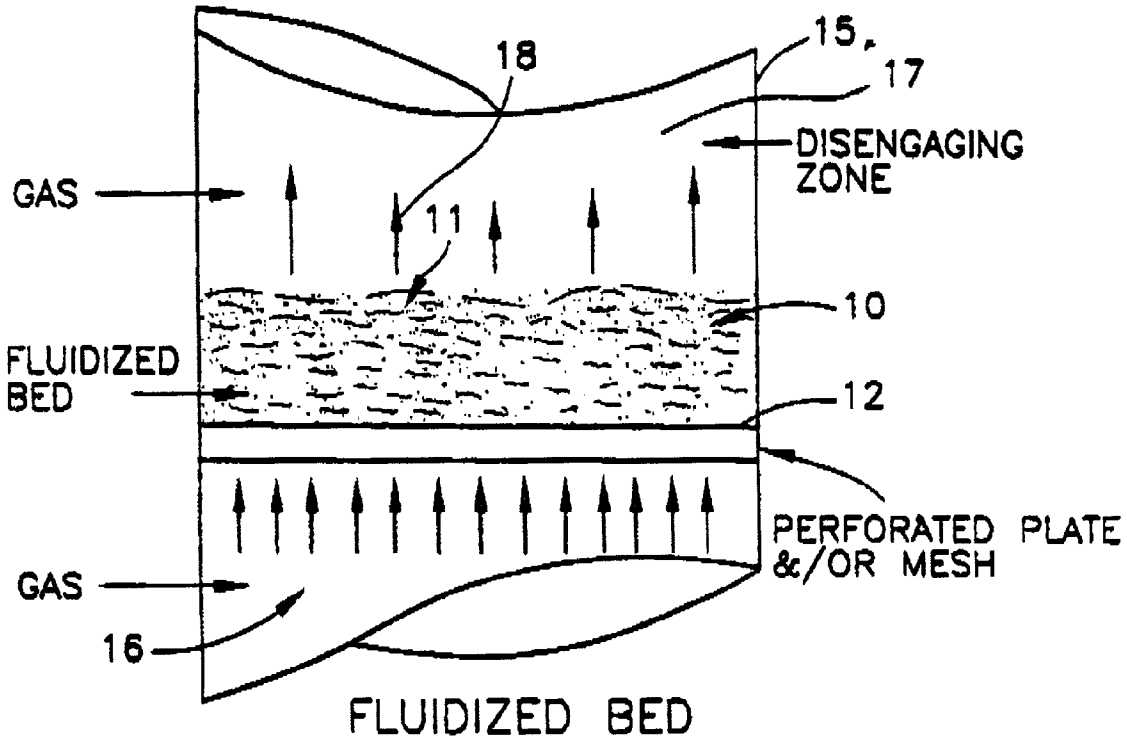

FIG. 1

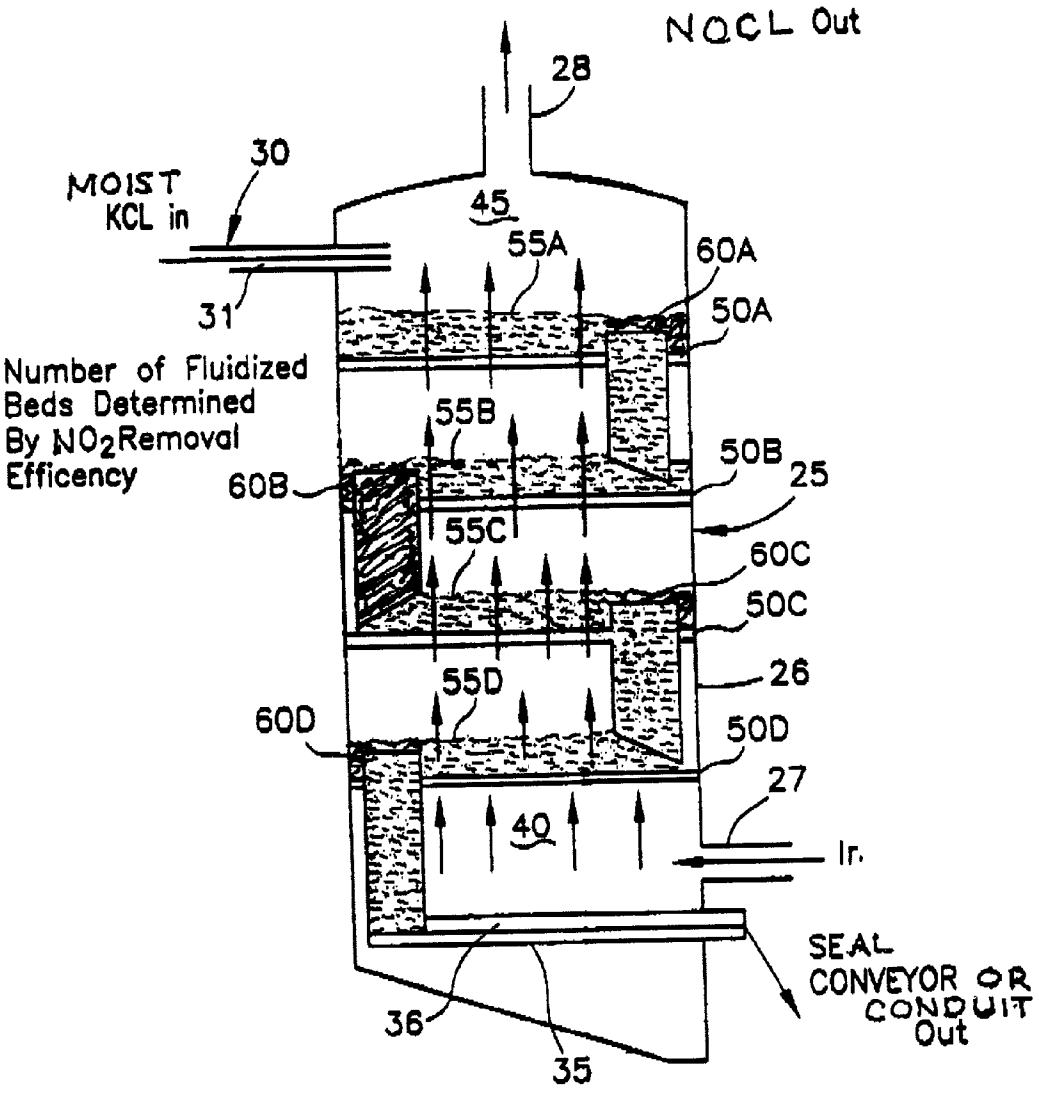

FIG. 2

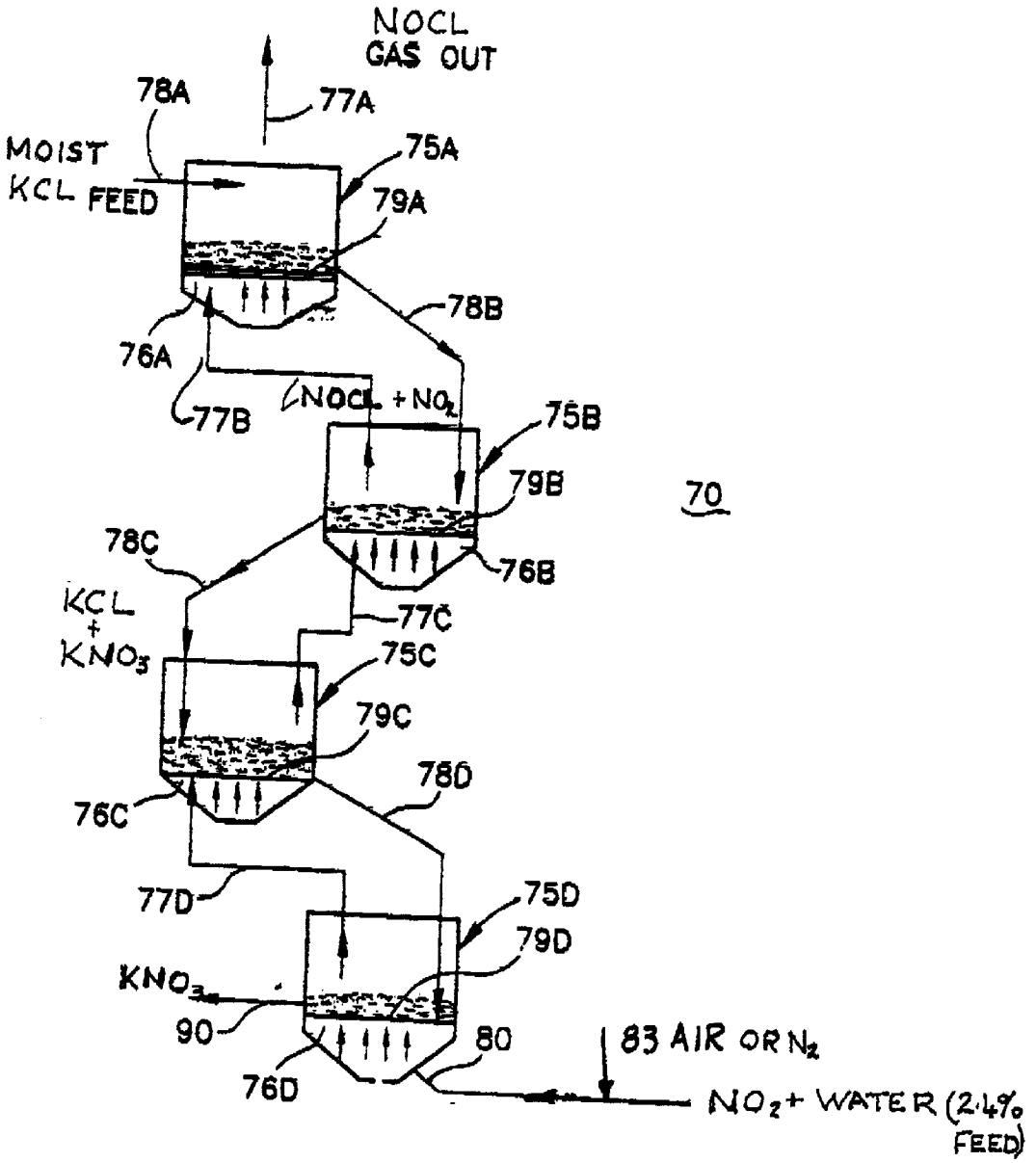

FIG. 3

PROCESS FOR MANUFACTURING POTASSIUM NITRATE FERTILIZER AND OTHER METAL NITRATES

RELATED APPLICATIONS

[0001] This is a continuation-in-part to application Ser. No. 09/100,994 filed Jun. 28, 1998.

FIELD OF THE INVENTION

[0002] This invention relates to a process for the reaction of water moistened particulate metal chlorides, sulfates or oxides with nitrogen dioxide gas in a fluidized state, with the production of a solid having a composition composed of the metal and the nitrate ion. More particularly, the invention relates to the production of potassium nitrate or calcium nitrate and nitrosyl chloride gas (depending on the composition of the particulate) using nitrogen dioxide gas and water moistened particulate potassium chloride or calcium sulfate in an energy efficient process using counter current flow and fluidized bed technology. The unique aspects of this process is that it permits the reaction to take place at a rapid rate at moderately elevated temperatures while retaining essentially the same crystal size as the original potassium chloride or calcium sulfate. The counter current aspects of the invention permit the production of potassium nitrate or calcium nitrate and nitrosyl chloride gas (depending on the solid) essentially free of nitrogen dioxide.

BACKGROUND OF THE INVENTION

[0003] Potassium is one of three essential elements (N.P.K.) in the life cycle of all plants. Fertilizers therefore generally contain all three in one form or another. Potassium, however is generally present as a chloride since it is the most readily available, least expensive potassium compound. For many crops (e.g., citrus, tobacco) a fertilizer containing small amounts of chlorides is toxic. Thus, there is created a sizable demand for manufactured potassium nitrate as a non-chloride source of potassium. However, it must be produced at a relatively low cost to compete with existing processes such as that produced from natural deposits. The use of potassium nitrate as a fertilizer was first suggested by Glauber in 1655. A few years later its value was discussed by Digby in what is said to be the earliest known record of the actual use of fertilizers as distinct from decaying organic matter. The world supply of potassium nitrate was formerly derived from incrustations on the soils around habitations in tropical countries, chiefly India, Sri Lanka, Mexico and Egypt. Its presence there is due to the decomposition of organic matter by nitrifying organisms in soils containing soluble potassium compounds.

[0004] Much of the potassium nitrate in commerce was formerly made by the "Conversion Process" in which sodium nitrate and potassium chloride undergo a double decomposition. This process depends on the wide variation in the solubility of potassium nitrate in hot and cold solutions.

$$KCl+NaNO_3 \acute{a} KNO_3+NaCl$$

[0005] This process has been displaced by more efficient operations as will be shown as follows:

RELATED ART

[0006] Potassium nitrate, otherwise known as saltpeter or nitrate of potash, is important in the production of fertilizers, explosives, glass, and numerous other industrial chemicals. It is one of the oldest known "industrial" chemicals. Potassium nitrate has been used on a large scale since around the year 1300, when the Chinese discovered that saltpeter could be combined with sulfur and charcoal to produce the common explosive known as black powder. The ever-growing demand for potassium nitrate for these and other such uses has resulted in a prolonged search for improved potassium nitrate production processes, and various methods have been invented to produce potassium nitrate. For example, large quantities of potassium nitrate are commercially produced by the reaction of potassium chloride with nitric acid in the presence of oxygen, yielding the following overall reaction:

$$2KCl+2HNO_3+1/2O_2 \rightarrow 2KNO_3+Cl_2+H_2O$$

[0007] The potassium chloride and nitric acid must be reacted at 100° C. to produce potassium nitrate, nitrosyl chloride and water as follows:

$$3KCl+4HNO_3 \rightarrow 3KNO_3+NOCl+Cl_2+2H_2O$$

[0008] The nitrosyl chloride is then oxidized to chlorine and nitrogen dioxide, NO_2, with nitric acid. See Chemical Process Industries, 4th Ed., Shreve and Brink, McGraw-Hill, Inc., New York (1977), pp. 272-273.

[0009] Smith et al, in U.S. Pat. No. 2,963,345, disclose a process for producing potassium nitrate, which involves agitating solid particulate potassium chloride with liquid nitrogen peroxide under anhydrous conditions at a temperature of 15° C.; excess nitrosyl chloride vapors produced by the reaction are continuously withdrawn to maintain the reaction. Potassium nitrate and unreacted potassium chloride are then separated by addition to a brine that contains dissolved potassium nitrate and potassium chloride; the brine solution is heated to about 85° C. to dissolve the potassium nitrate, but not the solid particles of potassium chloride. The solid particles of potassium chloride are then separated by filtration. Large volumes of potassium nitrate are also produced by the reaction of sodium nitrate with potassium chloride, the overall reaction being:

$$KCl+NaNO_3 \rightarrow KNO_3+NaCl$$

[0010] This process requires that potassium chloride be dissolved in a hot solution of sodium nitrate; upon heating, sodium chloride crystals are formed. The hot potassium nitrate solution is then run through the sodium chloride crystals forming at the bottom of the reaction vessel. However, a mixture of potassium nitrate and sodium chloride is formed, so additional processing operations are required to separate potassium nitrate.

[0011] Lehto, in U.S. Pat. No. 3,983, 222, discloses a continuous process for producing potassium nitrate, which includes the steps of extracting nitrate from aqueous solutions with an organic amine salt dissolved in an organic

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