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MATERIAL BALANCES FOR OBTAINING SODIUM NITRITE

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ABSTRACT

Sodium nitrite can be obtained in two ways: by reacting sodium sulfate with calcium nitrite or nitrate, calcium nitrite, formed after the absorption of nitrous gases with lime milk. Study of mutual solubility of the quaternary water system of nitrites, nitrates and sulfates of sodium and calcium and its constituent ternary systems at 25⁰C. Their isothermal solubility diagrams have been set up.

KEYWORDS: *Binary And Ternary Water Systems, Eutonic Solutions Of Facet Ternary Systems, Thermostat, Figurative Points, Four-Water Calcium Nitrate, Isothermal Method.*

INTRODUCTION

The main consumers of sodium nitrite-nitrate salts are machine-tool, metallurgical, paper, rubber, textile, pharmaceutical, food industry, construction industry, medicine and agriculture.

One of the possible ways to obtain sodium nitrate and nitrite is the conversion of sodium sulfate in an aqueous medium with calcium nitrate and nitrite.

The technological basis of these processes is based on the physicochemical properties of quaternary systems $2\text{Na}^+, \text{Ca}^{2+} // \text{SO}_4^{2-}, 2\text{NO}_3^- - \text{H}_2\text{O}$; $2\text{Na}^+, \text{Ca}^{2+} // \text{SO}_4^{2-}, 2\text{NO}_2^- - \text{H}_2\text{O}$ and their constituent ternary systems. There is no information on these quaternary systems in the literature. In this regard, for the physicochemical substantiation of the process of obtaining sodium nitrate and nitrite by conversion of calcium nitrate and calcium sulfate, the solubility in the above quaternary systems and individual components of ternary systems at 25⁰C was studied [1].

Sodium nitrite is obtained in two ways: According to the first option, the nitrite-nitrate solution of calcium formed during alkaline absorption is first neutralized with nitric acid (up to pH 5 -

5.5). Then the solution is evaporated and calcium nitrite is crystallized with further separation from the mother liquor of calcium nitrate-nitrite (Fig. 1).

Solid calcium nitrite and sodium sulfate are sent for exchange decomposition, which is carried out in a reactor with a circulating sodium nitrite solution. According to the second variant, the solution of calcium nitrite-nitrates, formed during the absorption of nitrous gases by circulating solutions with active CaO, reacts at a stoichiometric ratio with sodium sulfate. After that, the obtained nitrite - sodium nitrate solution is separated on the filter from the gypsum. The resulting solution is subjected to evaporation and crystallization with the precipitation of sodium nitrite into the solid phase. After separation by centrifugation of the finished product – sodium nitrite, the mother liquor of sodium nitrite is returned to the evaporation stage and processed into a solid product [2].

Also, according to the first option, solid one aqueous calcium nitrite obtained according to the scheme (Fig. 1) and ten aqueous sodium sulfates, respectively, are dissolved in solvents (item 1a and item 1b), (Fig. 1). The prepared solution is sent for exchange decomposition (Fig. 1), which is carried out in a steel reactor (item 1). The conversion process takes about 1 hour. Gypsum is separated from the solution on a drum vacuum filter, and the solution (item 2) is sent for evaporation (item 6) to the first evaporator (item 6). The incoming liquor with a content of 26.75% NaNO_2 , 0.71% CaSO_4 is evaporated at 100°C to a sodium nitrite concentration of 61.71% NaNO_2 , 0.030% CaSO_4 (Fig. 1).

The precipitated gypsum precipitate is filtered (item 2^{II}). The separated liquor is sent through the collector (item 4^{II}) using a centrifugal pump (item 5^{II}) to the crystallizer (item 7) where NaNO_2 crystals are precipitated when the solution is cooled up to 10°C . The crystalline product after separation on the filter (item 2^{III}) contains 95-99% NaNO_2 . The resulting uterine liquor A₃ (containing 43.16 NaNO_2 , 0.046% CaSO_4) from (item 4^{III}) is sent to (item 4) using a centrifugal pump (item 5^{III}), where it is mixed with the uterine liquor formed after conversion. The wet sodium nitrite precipitate is dried at $100\text{-}110^\circ\text{C}$ on a drum dryer (item 8).

Optimal conditions for the implementation of the technological process:

Concentration of calcium nitrite solution, %.....	80-90%
Conversion temperature of calcium nitrite with sodium sulfate, $^\circ\text{C}$	25-30
Concentration of sodium nitrite circulating solution, %.....	30-45
T:J ratio in slurry with gypsum sediment	1: (2,5÷3,5)
Duration of conversion of calcium nitrite with sodium sulfate, min.....	60-120
Evaporator temperature, $^\circ\text{C}$	100-110
Crystallizer temperature, $^\circ\text{C}$	10 -25
Temperature when drying wet sodium nitrite in a drum dryer, $^\circ\text{C}$	100-110

The shop for producing sodium nitrite according to the second option from nitrogen oxides, milk of lime and sodium sulfate consists of three sections: alkaline absorption, conversion and evaporation (Fig. 1). Nitrous gases containing about 2-4% nitrogen oxides pass through the absorption towers (item 1). The absorption tower is irrigated with milk of lime (100-140 g/l CaO) from the collector (item 2) with a nitrite-nitrate solution coming out of the collector (item 3). Nitrite-nitrate liquor from the absorption tower is sent to the conversion through the collector (item 3) by a centrifugal pump (item 4). After the absorption tower, nitrous gases are directed to a scrubber (item 15) and then removed to the atmosphere. Nitrite-nitrate lye obtained as a result of alkaline absorption contains about 75-95% nitrite and 5-25% calcium nitrate [3].

Solutions of nitrite and calcium nitrate are sent for exchange decomposition, which is carried out in a steel reactor (item 5) heated by steam. Crushed sodium sulfate or its solution in the form of weak hot liquors containing sodium nitrite-nitrate is gradually introduced into the reactor.

To accelerate the conversion process and prevent clogging of the reactor with calcium sulfate precipitate formed as a result of the exchange reaction between Na_2SO_4 and $\text{Ca}(\text{NO}_2)_2$, compressed air is supplied to the lower part of the apparatus or the solution is stirred with a mechanical stirrer.

The conversion process takes about 1 hour. The gypsum is separated from the solution on fabric filters; the ratio T:J = 2.5-3:1, the concentration of NaNO_2 and NaNO_3 in the liquid phase is 14-34.0% and 1.5-4.5%, respectively. This suspension is filtered from $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ on a drum vacuum filter (item 7) and sent to the evaporation. Gypsum, containing after filtration 10.2-13.0% NaNO_2 and 1.02-1.30% NaNO_3 , is washed in a repulperator with rinsing water from item 15, while the total content of NaNO_2 and NaNO_3 in the sludge is reduced to 0.05- 2%. Calcium sulfate removed from the filter (item 7) can be used for technical purposes.

Evaporation of solutions of nitrite and sodium nitrate Po (containing 22.14% NaNO_2 , 2.44% NaNO_3 and 0.56% CaSO_4) is carried out in two-vessel evaporators [3].

Vertical evaporators with internal or external heating chambers can be used. On the first evaporator (item 11), the incoming liquor with the Po content is evaporated at 100-110°C to the concentration of sodium nitrite P1 (containing 51.66% NaNO_2 ; 5.74% NaNO_3 and 0.03% CaSO_4). The precipitated gypsum precipitate is filtered (item 7). To avoid crystallization of NaNO_2 , the temperature in the tank (item 9) is maintained within the range of 90-105°C using steam heating. The separated liquor is sent through the collector (item 9) using a centrifugal pump (item 10) to the second evaporator, where it is evaporated to form a slurry containing 76% sodium nitrite and 8.0% sodium nitrate. A hot solution of nitrite and sodium nitrate is sent to the crystallizer (item 12) Here, when the solution is cooled to 21°C, crystals of NaNO_2 are isolated.

The crystalline product after separation on the filter (item 7) from the uterine liquor contains 95-99% NaNO_2 . The resulting uterine liquor from (item 9^{III}) is sent to, conversion or inversion (item 9) using a centrifugal pump (item 10) to obtain sodium nitrate. The wet sodium nitrite precipitate is dried at 100-110°C in a drum dryer (item 13).

To obtain purer sodium nitrite, crystalline sodium nitrite after the first crystallization is usually washed with cold water, dissolved in steam condensate and subjected to crystallization. A concentrated NaNO_2 solution obtained by dissolving salt in a steam condensate is filtered off on

a filter and sent to a crystallizer. After separation from the mother liquor on the filter, crystalline sodium nitrite is dried with hot air (105-110°C) in a rotating drum [4,5].

As shown by toxicological studies of the Republican Center for State Sanitary and Epidemiological Supervision, synthesized sodium nitrite complies with GOST 19906-74 (toxicological conclusion is attached). The proposed technology for the production of sodium nitrite (Appendix 1) has been tested on the model plant of Chirchik JSC "Elektrkimyosanoat". A pilot batch of sodium nitrite has been produced, the following main technological parameters of its production have been worked out and established (Appendix 2). Laboratory regulations and test report are attached (Appendices 2 and 3). Optimal technological parameters for the production of sodium nitrite.

Concentration of milk of lime, g/l.....	100-140
NO:NO ₂ ratio, mol	(60-70):(40-30)
Duration of irrigation of nitrous gases, min...	30
Absorption tower temperature, °C.....	30-45
Conversion temperature of calcium nitrite with sodium sulfate, °C.....	25-30
Concentration of sodium nitrite circulating solution, %.	25-50
T:J ratio in pulp with gypsum sediment, mol	1: (2,5-3,5)
Calcium nitrite: sodium sulfate ratio at conversion, mol	1:1
Duration of conversion of calcium nitrite with sodium sulfate, min	60
Evaporator temperature, °C.....	100-110
Crystallizer temperature, °C.....	10-25
Temperature when drying wet sodium nitrite in a drum dryer, °C.....	100-110

Thus, not on the basis of the studies carried out, a generalized technological scheme and initial data (Appendix 4) for obtaining sodium and calcium nitrite from limestone, mirabilite and nitrous gases have been proposed.

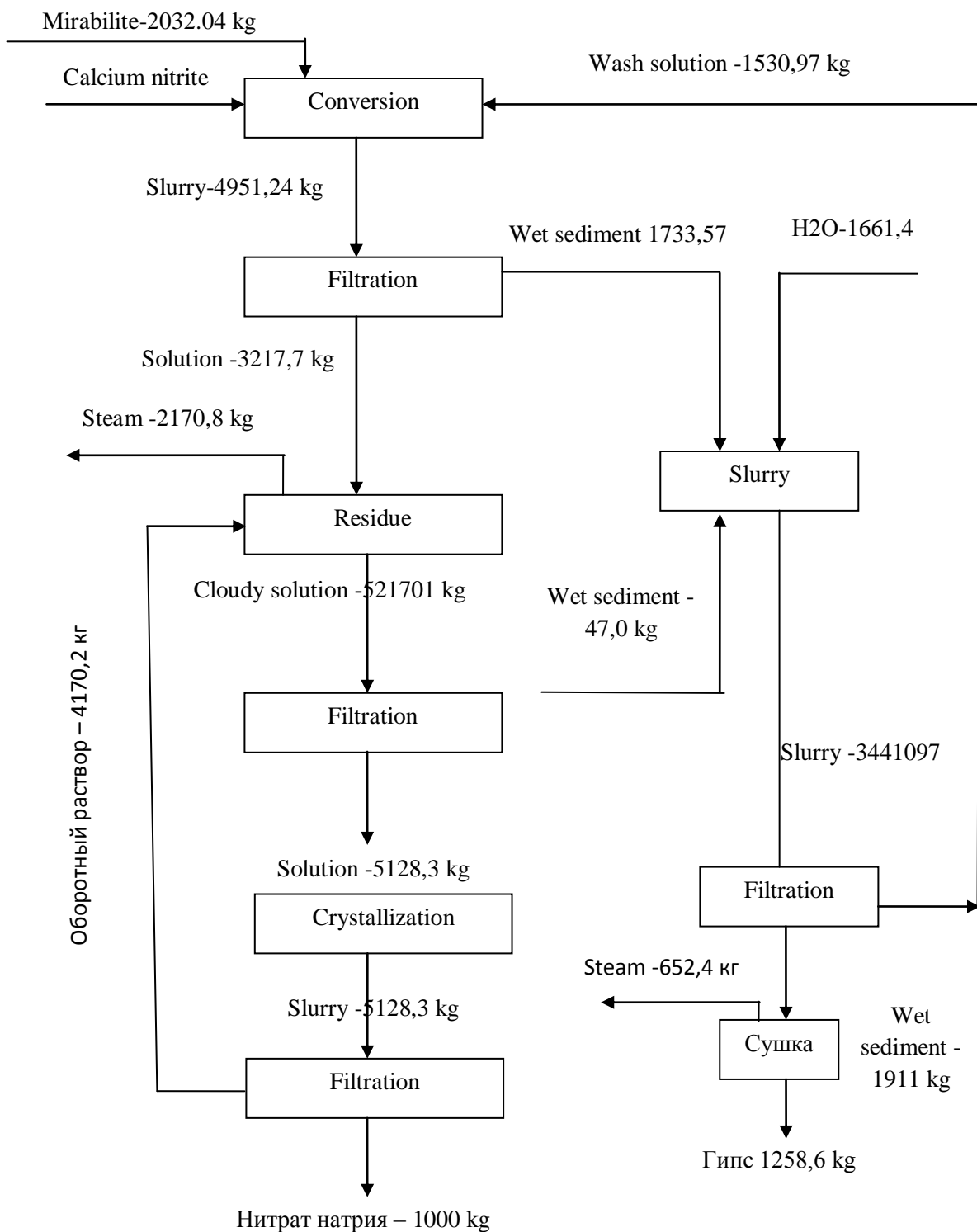


Figure 1. Material balance of obtaining sodium nitrite form calcium nitrite

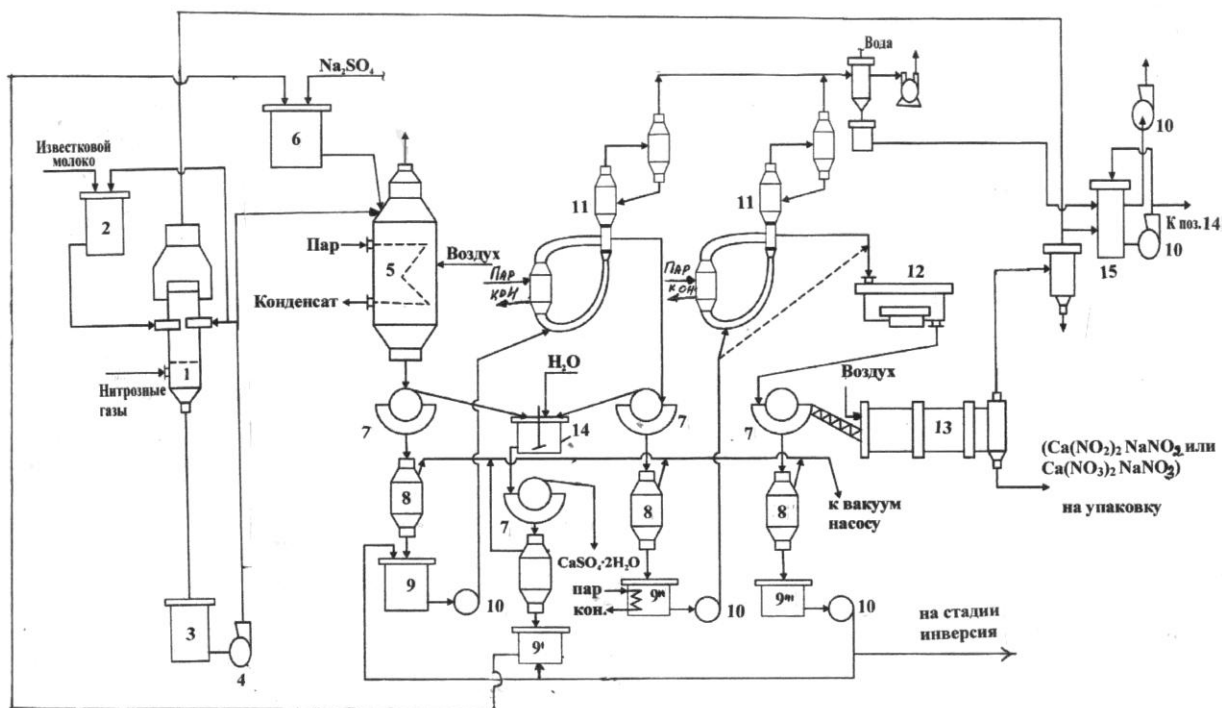


Рис.5.10. Технологическая схема производства нитрита натрия

1-абсорбционная башня; 2,3,6,9-сборники; 4,10 - центробежные насосы; 5-реактор; 7-барабанные фильтры; 8-резервуар; 11-выпарные аппараты; 12-кристаллизатор; 13-барабанная сушилка; 14-регулятор; 15-скруббер.

Figure 5.10. Technological scheme for the production of sodium nitrite 1-absorption tower; 2,3,6,9-collections; 4,10-centrifugal pumps; 5-reactor; 7-drum filters; 8-reservoir; 11-evaporators; 12-crystallizer; 13-drum dryer; 14-regulator; 15-scrubber.

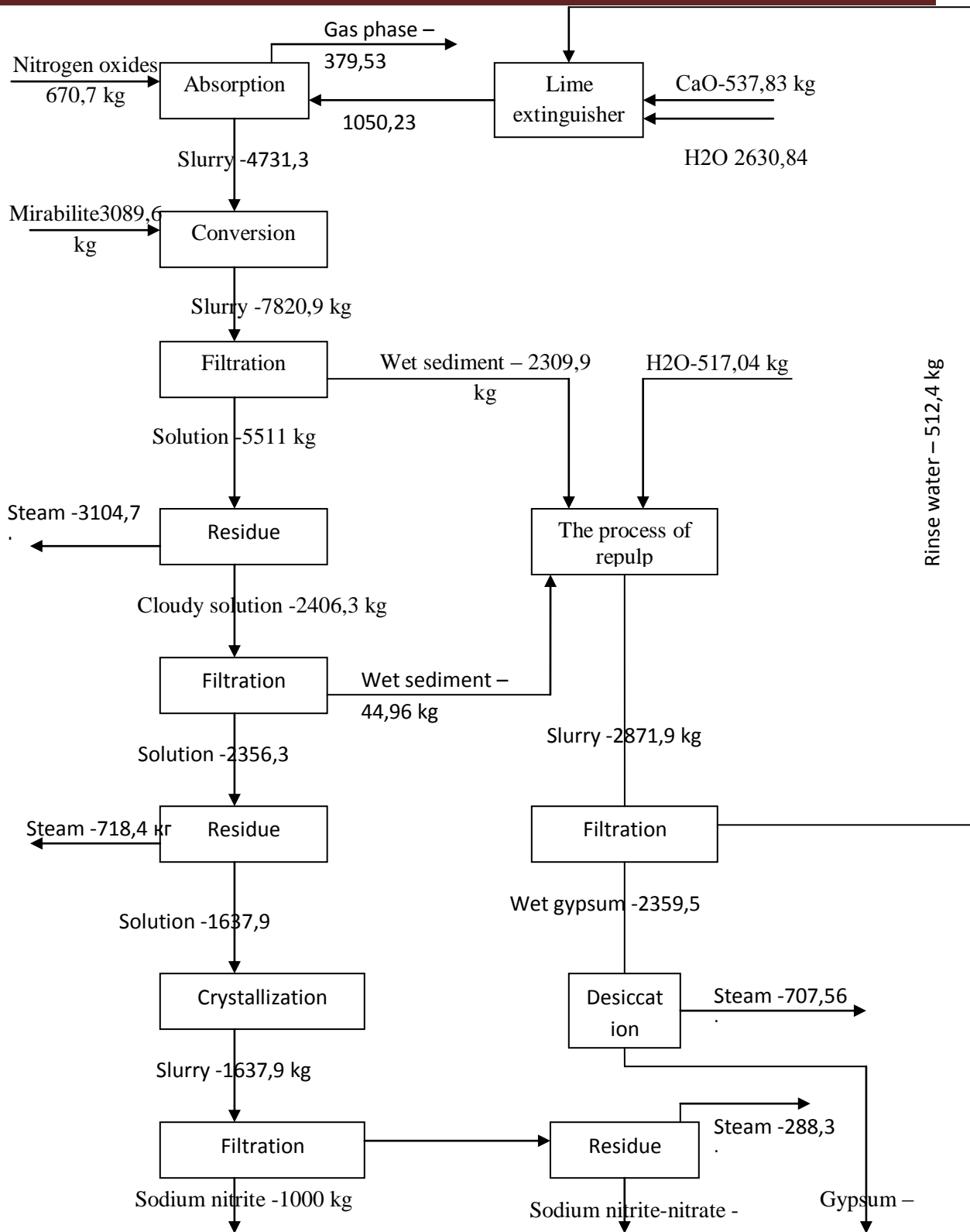


Figure 2. Material balance of obtaining sodium nitrite from nitrogen oxides, lime and mirabilite.

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