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CALS-TECHNOLOGIES IN SYNTHESIS OF MULTIASSORTMENTAL MANUFACTURING FOR PHOSPHORUS SLUDGE UTILIZATION

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Abstract: CALS-projects of sodium phosphite and hypophosphite technologies – products of phosphorus sludge utilization (one of the main wastes of phosphoric industry) were developed. CALS-technology of the flexible two-grocery production was developed based on the theory of the flexible scheme synthesis.

Keywords: multiassortmental manufacturing, CALS-technologies, phosphorus sludge, sodium phosphite, sodium hypophosphite

Recovery of phosphorus sludge formed in large amounts upon phosphoric production is considered. The corresponding task was formulated as development of a closed-circuit technological process at which phosphorus sludge will re-enter processing for the maximal extraction of useful products from its composition [1].

At the first stage the nomenclature of the phosphorus-containing compounds entering the complex phosphorus sludge processing flow sheet (circuit) is examined. In the circuit four target products are considered: sodium hypophosphite, sodium phosphite and obtained through its further processing dibasic lead phosphite and phosphorous acid (Fig. 1).

Phosphorus sludge recovery processes were developed using the high end computer support system – CALS-technologies (Continuous Acquisition and Life cycle Support – continuous information support of life cycle of a product) [2]. The CALS concept is based on the complex of uniform information models, standardization of ways of access to the information and its correct interpretation in accordance with international standards. Thus uniform ways of process control and interaction of all participants of development are provided. A key idea of CALS concept is increasing of product life cycle due to increase of efficiency of control of the information on a product. CALS task is transformation of a product life cycle into highly automated process through re-structuring of its component business-processes [3].

Within the CALS concept two basic circuits of phosphorus sludge processing have been considered: with sodium phosphite [4] and sodium hypophosphite [5] as the final products. The yielded circuits were implemented in the CALS-project. In the CALS-project the typical computer structure of initial data for designing is used. It includes the following subcategories [6]: general data on technology (01); characteristics of the executed research and experimental work (02); feasibility study of a recommended production method (03); a patent card (04); characteristics of feedstock, auxiliary materials (05); physical and chemical constants and properties of initial, intermediate, and final products (06); chemical, physicochemical bases and basic production flow sheet (07); operating technological parameters of production process (08); mass balance of production process (09); characteristics of by-products and solid waste (10); mathematical description of technological processes and devices (11); data for calculation, designing, and choice of basic production equipment (12); recommendations for process automation (13); analytical control of production process (14); methods and technological parameters of purification from chemical and industrial pollutants (15); safety data sheet including fire and explosion hazard data, fire fighting procedures, health hazard data, and regulatory information (16); list of reports and recommended reading on the considered technology (17).

Based on the offered typical structure of initial data for designing, the CALS-project for sodium phosphite production technology has been developed. The block diagram in Fig. 2 includes a preparatory stage and 4 basic production stages: phosphorus sludge decomposition in the reactor, filtering of a mineral part, correction of solution density, and neutralization of excess of alkali in solution:

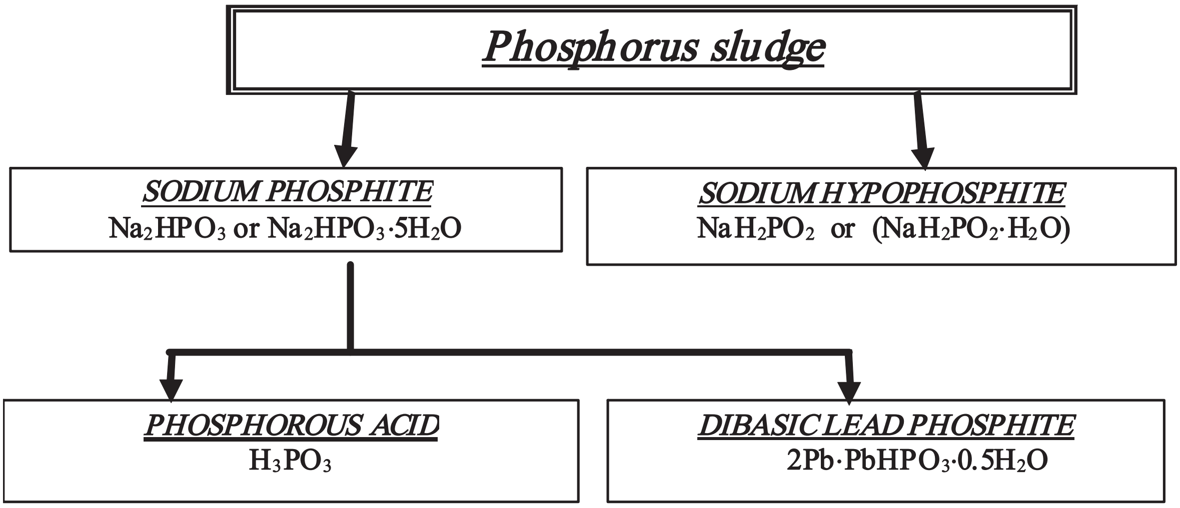

Fig. 1. Phosphorus sludge utilization multiassortment process scheme

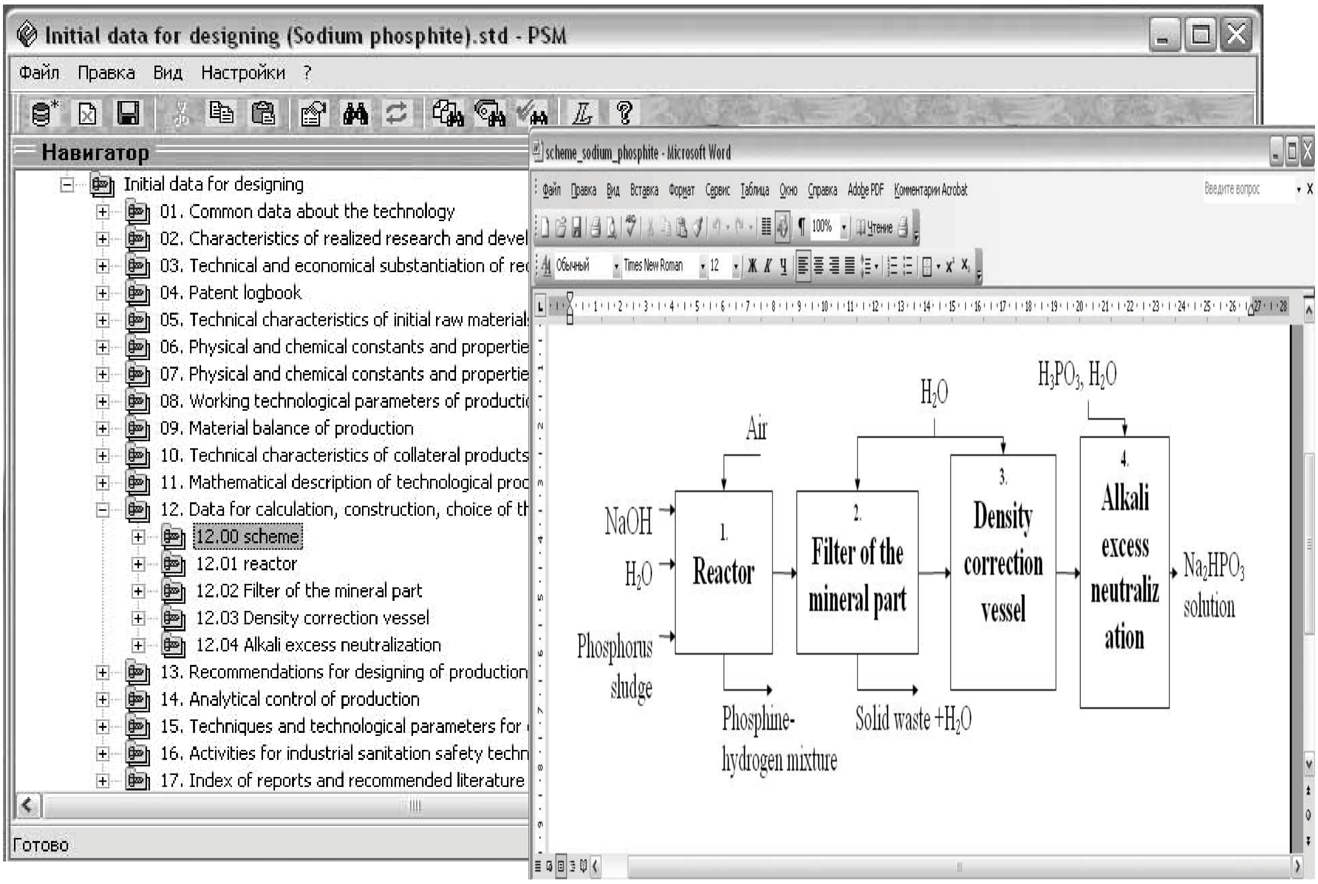

Fig. 2. Element of the CALS-project "Flow diagram of sodium phosphite production"

1. The Preparatory stage. The phosphorus sludge is classified in a grinder to particles size optimal for interaction with sodium alkali (NaOH) and then a solution is prepared. Simultaneously alkali solution is prepared through diluting alkali liquor to concentration NaOH = 31 % using water as a solvent. All stages of the corresponding technological block with characteristics of the equipment as well as additional information are put in the CALS-project and are used by both technology developers, and engineering personnel (analysts, *etc.*).

2. Decomposition of phosphorus sludge in a reactor. In the reactor (1) NaOH solution is fed and

simultaneously phosphorus sludge is charged. The reaction is conducted at the temperature of 373 K. Upon interaction of phosphorus sludge with sodium alkali from the reactor the phosphine-hydrogen mix leaves. The obtained product solution is directed to the next stage (2). The corresponding technological block is included in the CALS-project with all necessary characteristics of the used equipment.

3. Filtering of a mineral part. The solution directed from reactor (1) to vessel (2) is filtered. A deposit remaining on the filter is a mineral part of phosphorus sludge and is used as a mineral fertilizer. The solution passed through filters (2) enters the next stage (3). Drawings of the filter, input, and output parameters together with other major characteristics are included in the CALS-project.

4. Correction of sodium phosphite solution density. The obtained sodium phosphite solution is diluted with water to a necessary concentration. The ratio of components, temperature, and characteristics of the equipment are included in the CALS-project.

5. Alkali excess neutralization of. After stage (3) sodium phosphite enters stage (4) where neutralization of excess of sodium alkali (NaOH) with phosphorous acid (H_3PO_3) solution is carried out.

Finally sodium phosphite goes to the packing stage. Types of packing and its characteristics are included in the CALS-project. The database of the CALS-project also contains the basic documents on sodium phosphite production: certificates, process regulations, characteristics of final products, performance characteristics of the used equipment, *etc*.

Similarly to production of sodium phosphate, the typical structure of the CALS-project has been developed for production of sodium hypophosphite. The corresponding developed block diagram shown in Fig. 3 includes a preparatory stage and 9 basic production stages. The scheme includes the following blocks:

1. The Preparatory stage. Phosphorus sludge with the content of phosphorus 30–50 %, in a liquid state, heated to the temperature of 343 K, is pumped to storage tanks (receivers). For prevention of phosphorus sludge stratification in storage tanks the latter are fitted with agitators. The dosage of phosphorus sludge from the tanks is determined by water forcing.

2. Preparation of calcium hydroxide suspension (5) and sodium hydroxide (4) is conducted in two parallel tanks - mixers of suspension (40 m[3] volume each) heated with

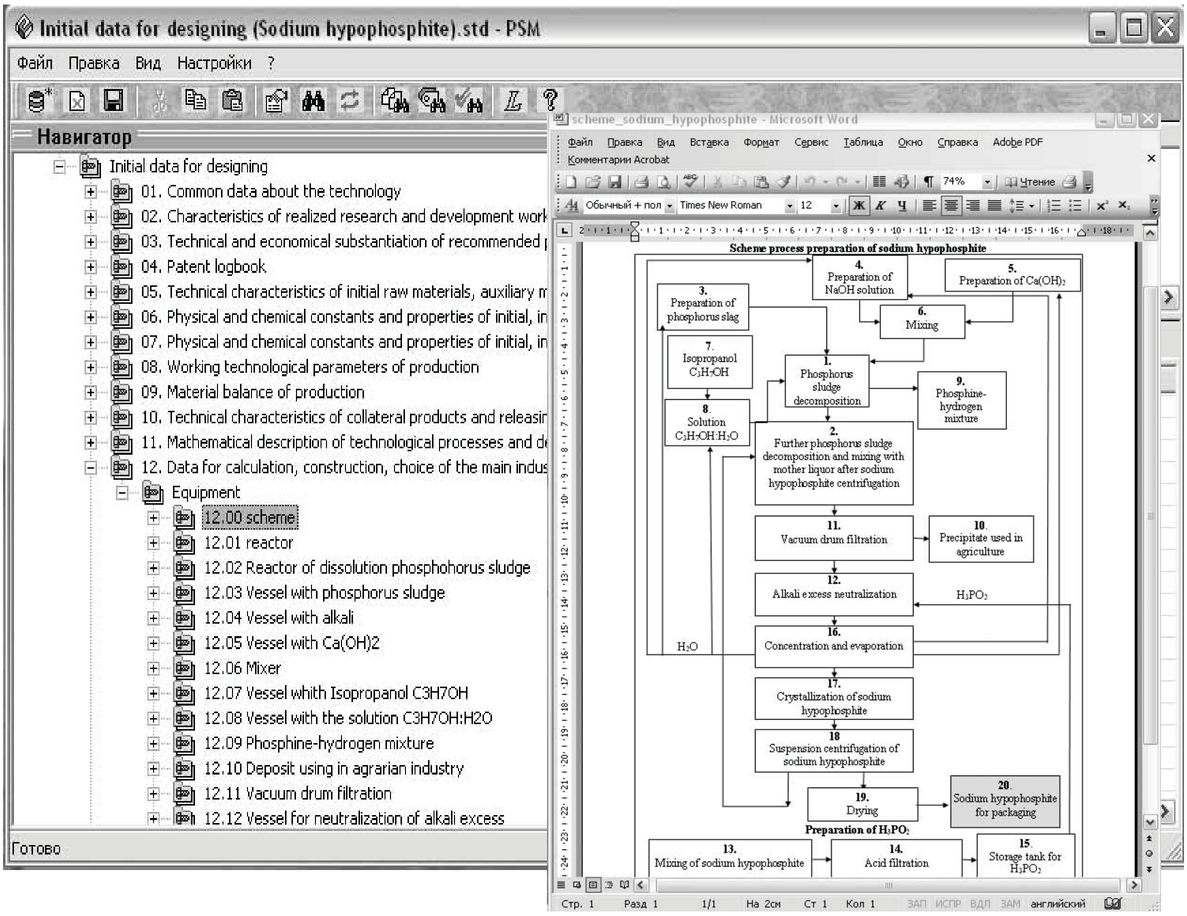

Fig. 3. Element of the CALS-project "Flow diagram of sodium hypophosphite production"

external pipe coil to the temperature of 773 K. Sodium hydroxide is pumped from intermediate storehouse to receiver tank with an impeller pump. When the required amount of sodium hydroxide is fed, the agitator is started and charging of calcium oxide hydrate (slaked lime) begins. The dosage of 12 tones (one batch) takes one hour. Then at constant stirring 12 m^3 of water is added and for 8 h this mix is agitated in the receiver tank (6). Preparation of suspension proceeds with intense reaction and foaming. After eight-hour stirring the mix is considered ready for application.

3. Decomposition of phosphorus sludge in a reactor (1). The phosphorus sludge is loaded into reactor from position (3) along with the obtained solution from the mixer (6). After some time the solution of isopropanol (7) is fed to the reactor (1). This is necessary for fuller extraction of phosphorus from phosphorus sludge. The reaction is conducted at the temperature of 358–363 K. Upon interaction of phosphorus sludge with sodium alkali from the reactor the phosphine-hydrogen mix leaves. The obtained product solution is directed to the next stage (2).

4. Further phosphorus sludge decomposition in an additional reactor (2): from the reactor (1) the obtained mix is directed to reactor (2) where it is mixed with a mother solution after sodium hypophosphite centrifuging. After the end of the reaction, the solution from the additional reactor (2) enters vacuum filters (11).

5. Filtering in drum-type vacuum filters. The solution obtained from the reactor (2) is filtered off in drum-type vacuum filters (11). The deposit formed at filtering is collected and used as fertilizer in agriculture. The solution passed through the vacuum filters (11) enters the neutralizer (12).

6. Sodium alkali excess neutralization. After passing the drum-type vacuum filters a solution consisting of NaH_2PO_2 (8 %), Na_2HPO_3 (9 %), and $CaHPO_3$ (25 %) enters the neutralizer (12). Neutralization of excess of sodium alkali is afforded by dilution with hypophosphorous acid which is stored in vessel (15).

7. Hypophosphorous acid (H_3PO_2) preparation. The product solution (calcium hypophosphite) with concentration of 12 % obtained at stage 5 is mixed with oxalic acid. The obtained solution is filtered (14) and the formed hypophosphorous acid is stored in vessel (15) and is used as required in the neutralization stage (12).

8. Sodium hypophosphite concentrating. Sodium hypophosphite is concentrated (16) by evaporation for further fine filtration which is carried out in a vessel (17). The formed sodium alkali and sodium hypophosphite are directed to recycling.

9. Sodium hypophosphite crystallization (17) and suspension (18) centrifuging. After crystallization (17) the suspension goes to a centrifuge. The formed mother solution goes to recycling through an additional reactor (2). After centrifuging (18) sodium hypophosphite is dried (19) to remove excessive moisture from the final product.

After the production cycle is finished, the product goes for packing (20). The operation mode and constructional characteristics for each stage are included in the corresponding sections of the CALS-project. The CALS-project also contains marketing analysis results. It is shown, that the considered products are in great demand. Sodium phosphite is one of the most scarce salts of phosphorus. It is widely used: in electroplating, as a reagent for synthesis of dibasic lead phosphite – the best stabilizer of PVC-compositions and also as a reducer in inorganic syntheses [4]. Sodium hypophosphite is used as a reducer at depositing nickel, cobalt, and tin coatings on metals and plastic; as antioxidant preventing discoloration of alkyd resins upon their preparation *etc.* [5].

As the considered processes have many related attributes, we were faced with the task of their association in uniform production unit for sodium phosphite and sodium hypophosphite. For optimum development of two-product production, the theory of synthesis of flexible multiassortment chemical engineering systems (Fig. 4) developed by our group has been used [7, 8].

For similar multiassortment schemes four levels of system analysis are considered: nomenclature, productional-technological, organizational-technological, and organizational-productional.

An attribute of the top (4-th) technological level is a separate shop as a complex cybernetic system. Associated problems are: stabilization of material and information streams between aggregated sections; distribution of raw material, power, and manpower resources. An attribute of the 3-rd - organizational level is the aggregated section. Associated problems are: optimization of equipment arrangement and minimization of a production cycle.

The bottom (1-st) level is the nomenclature level. Its characteristic attributes are: a single product or one technological stage. The primary goals are: expansion of a set of available grades for this one product or variation of available capacity of a technological stage. Functioning of the bottom (1-st) level is provided by technological flexibility which is determined by the possibility of carrying out several technological tasks on the existing equipment using flexible scheme adaptable for production of a given product (under the nomenclature) or with insignificant expenses on the equipment readjustment (washing, pipelines fitting, *etc*).

The second technological level is of greatest interest to us. Its characteristic attribute is multiassortment technology. Associated problems are: optimum use of intermediate products and common initial reagents; using of elements of flexibility with the purpose of assortment expansion; variation of capacity of the whole technological process.

This approach was applied to development of the block diagram of flexible two-product scheme of sodium hypophosphite and sodium phosphite synthesis. The developed production is the basic unit for the whole

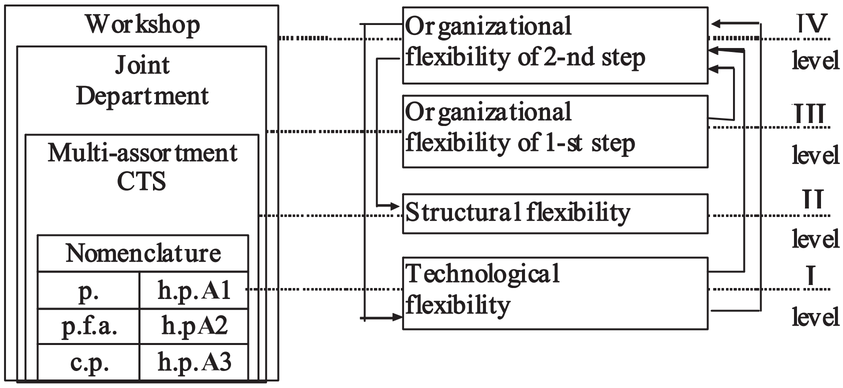

Fig. 4. Hierarchical structure of flexible chemical engineering systems synthesis

complex of phosphorus sludge processing (Fig. 1). Incorporated into this complex individual production processes for dibasic lead phosphite and phosphorous acid employ sodium phosphite obtained using flexible scheme as raw material.

To substantiate the reasonability of uniting the two processes in one flexible scheme it is necessary to carry out the analysis of existing individual production processes with the purpose of specifying groups of technologies which are suitable for organizing by a flexible principle. The first stage of this analysis is decomposition of the considered product assortment using hierarchical approach based on two basic attributes: technological and chemical similarity.

Each of the pointed attributes has its gradation level. Thus, technological similarity is subdivided into similarity of raw material preparation methods (dissolution, filtration, crushing, etc.), production methods (type of transformation of raw material to a main product, uniformity of technological operations, and the used equipment), and packing methods. Chemical similarity is determined, first of all, by substances' belonging to the same class (acid, base, salt, ether, *etc.*) inside which class sublevels are isolated on the basis of physical and chemical properties of substances. For example, salts are classified according to the character of the anion (acid residue) – nitrates, sulphates, phosphates, *etc.*

The analyzed production processes for sodium hypophosphite and sodium phosphite meet both attributes of the theory of flexible chemical engineering systems as they have both technological and chemical similarity. This allowed us to carry out synthesis of the flexible two-product scheme (Fig. 5).

The developed optimal scheme includes 23 blocks: 9 combined blocks containing operations used in both production processes for sodium phosphite and sodium hypophosphite (solid line); 3 blocks applied only to production of sodium phosphite (dot line); 11 blocks relating only to production of sodium hypophosphite (dashed line).

For transferring from one product to another 2 flexible switching units are included in the scheme: Flexible Unit of Switching-1 (FUS-1) and Flexible Unit of Switching-2 (FUS-2). The unit FUS-1 is responsible for streams switching: either directing sodium alkali from the position (4) directly to reactor (1) in production of sodium phosphite, or to position (6) for mixing with lime hydrate in the production of sodium hypophosphite. After the reactor (1) in case of sodium phosphite synthesis in the unit FUS-2 switching to position (10) is suggested for filtering of the obtained reaction mixture, or switching to position (2) for phosphorus sludge further decomposition and mixing with mother liquor in case of sodium hypophosphite synthesis. Flexible switching units allow producing sodium hypophosphite and sodium phosphite at minimal controlling acts.

The developed flexible scheme (Fig. 5) is included in the CALS-project (Fig. 6) with all operation characteristics, drawings of the used equipment, etc. Each element of equipment included in CALS information system has one of three identification attributes: unit used only for production of sodium phosphite; unit used for production of sodium hypophosphite; unit which will be probably used for production of sodium hypophosphite and sodium phosphite. In the pilot CALS-project drawings of all apparatuses included into the block diagram (Fig. 6) are given. If necessary the drawing of the element of interest can be viewed separately in subsection 12 "Data for calculation, designing, and industrial application". For example, subsection 12.01 (Reactor) contains drawings of a reactor, maintenance instruction, certificates of conformity, *etc.*

Development of the design documentation was carried out using the specialized computerized designing software «AutoCAD». For data storage convenience and searching time reduction some big drawings and block diagrams as well as large MS Word documents have been transformed into PDF (Fig. 6).

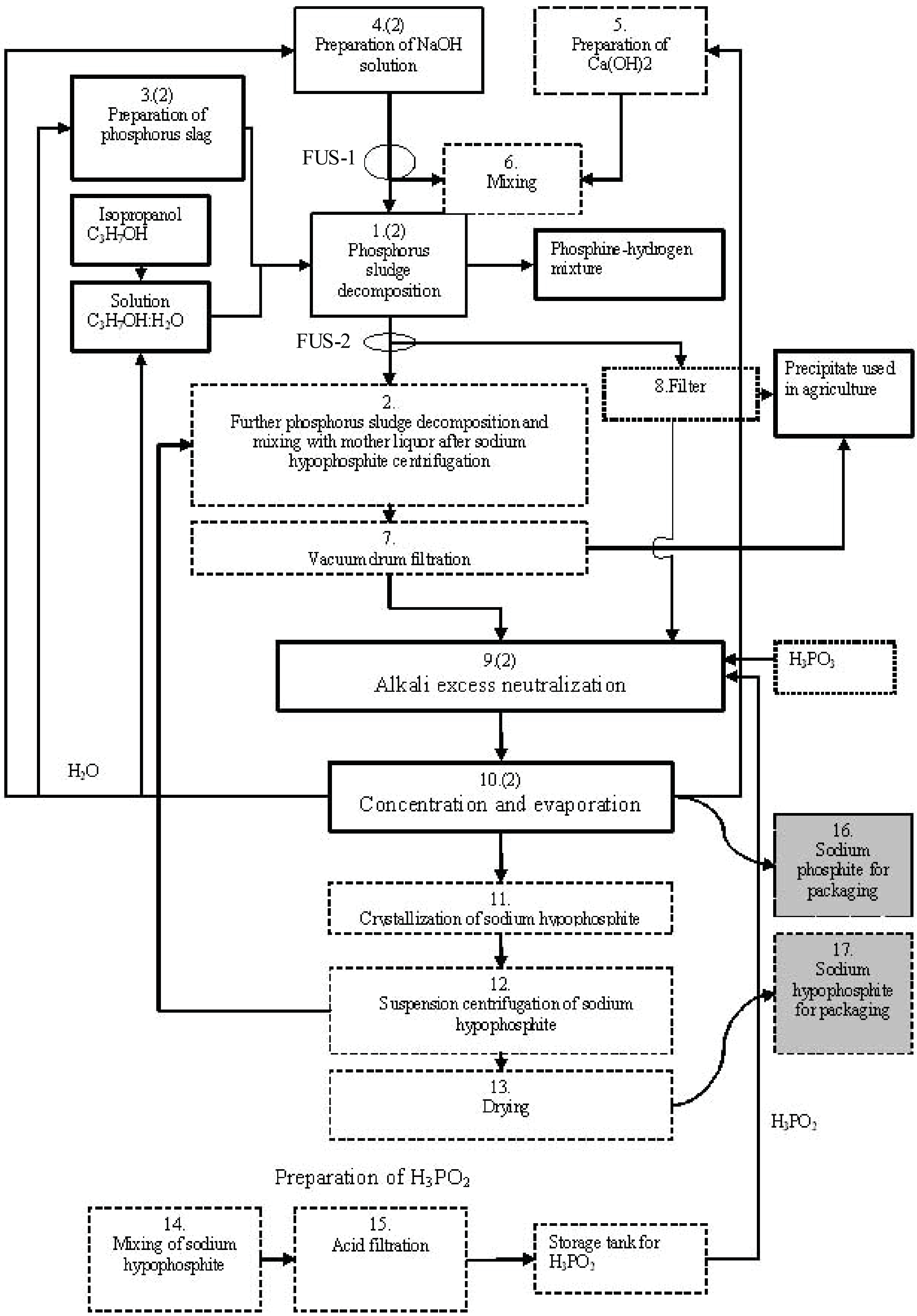

Fig. 5. Flexible scheme of sodium hypophosphite and sodium phosphite production

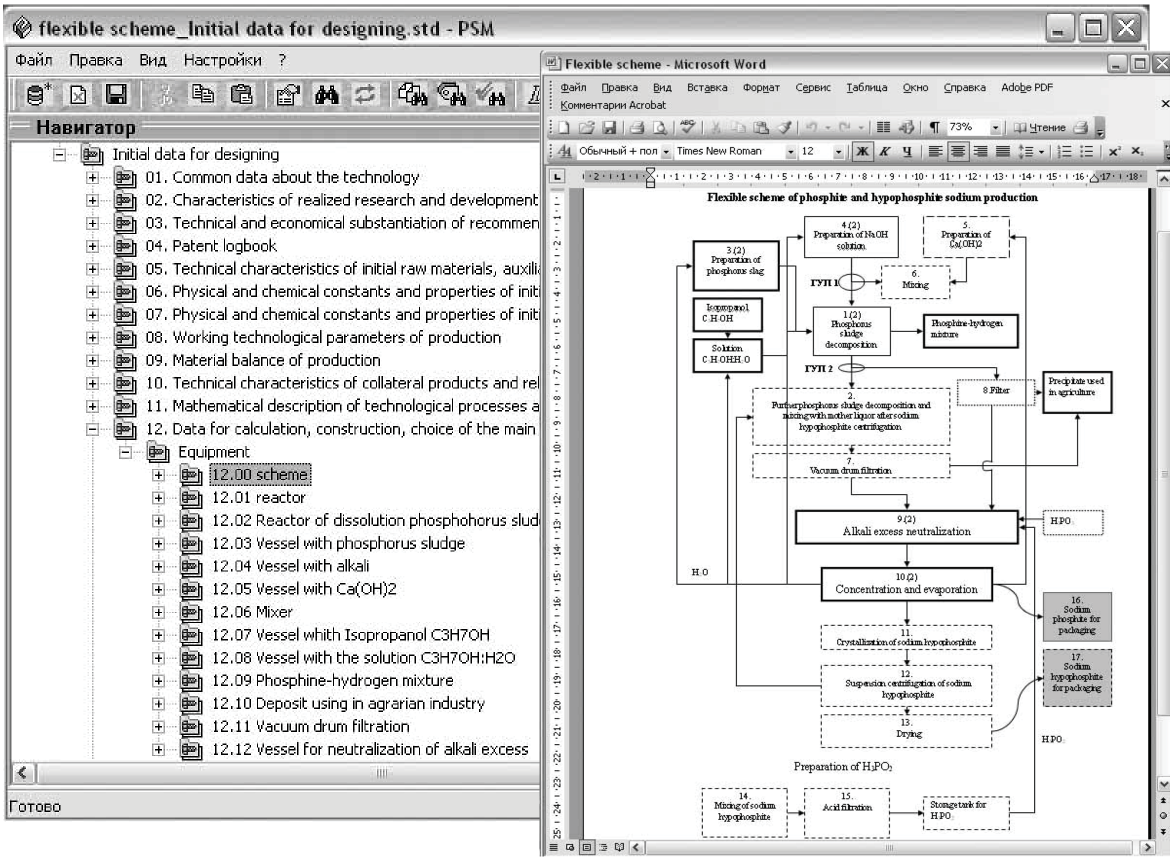

Fig. 6. An element of the CALS-project "Initial data for designing"
(the flexible scheme of sodium hypophosphite and sodium phosphite production)

The modern level of innovative production development is intimately connected with CALS-technologies, *i.e.* with the use of uniform information space at all stages of the product life cycle - from designing and utilization to disposal (recovery). Introduction of information CALS-technologies for sodium phosphite and sodium hypophosphite flexible production designing allows not only to obtain both salts with high characteristics, but also to provide full post-sale support including documentation in the electronic form.

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CALS-ТЕХНОЛОГІЇ СИНТЕЗУ МУЛЬТИСОРТИМЕНТНОГО ВИРОБНИЦТВА ДЛЯ УТИЛІЗАЦІЇ ФОСФОРНОГО ШЛАМУ

Анотація*. Для утилізації фосфорного шламу (основного відпаду виробництва фосфатної кислоти) розроблений інформаційний проект гнучкої технології одержання фосфіту і гіпофосфіту натрію. Проект створений на основі інформаційних CALS-технологій.*

Ключові слова*: мультиасортиментне виробництво, CALS-технології, фосфорний шлам, фосфіт натрію, гіпофосфіт натрію.*