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Method and plant for continuous production of granular USP nitrogen and phosphate type fertilizers and products on their basis

Sebastian Schab, Piotr Rusek

Łukasiewicz Fertilizer Research Center

Purpose of work:

Development of a new continuous method for production of granular USP (urea-superphosphate) nitrogen and phosphate fertilizers and products based on it, in a continuous manner.

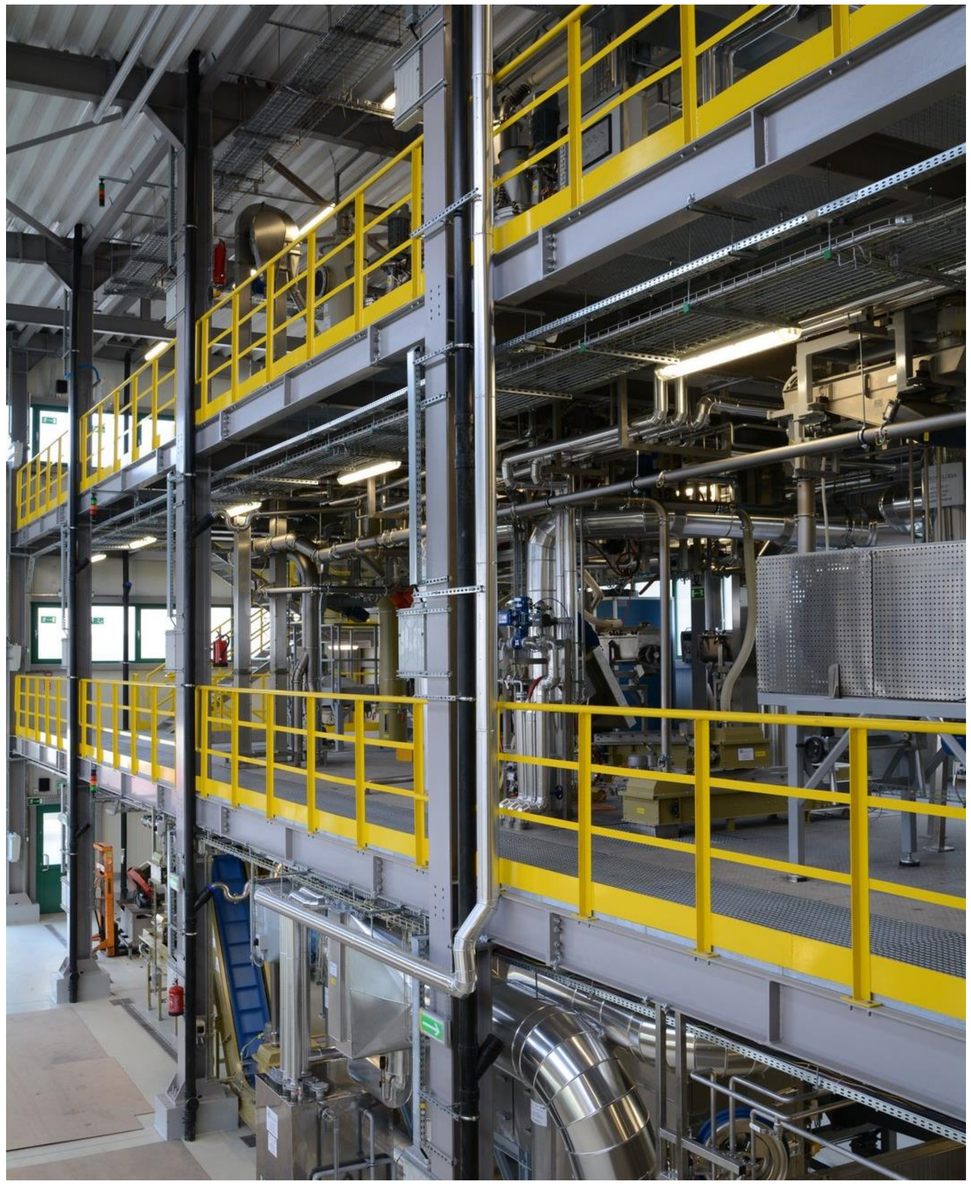
USP – urea superphoshate

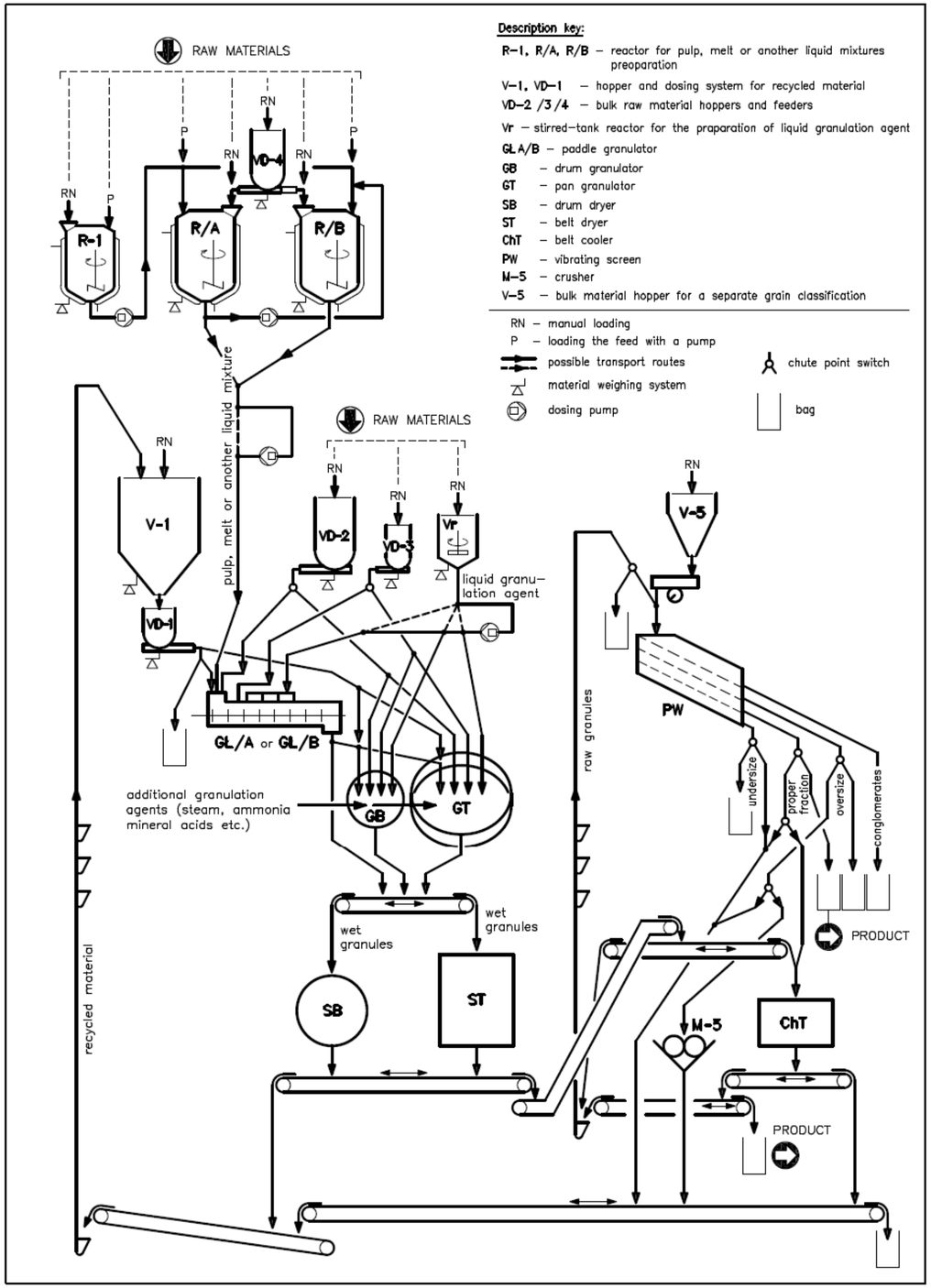
EU fertilizer type: "NP fertilizer with calcium and sulfur"

NP (CaO; SO_3) 20-10 (12;19)

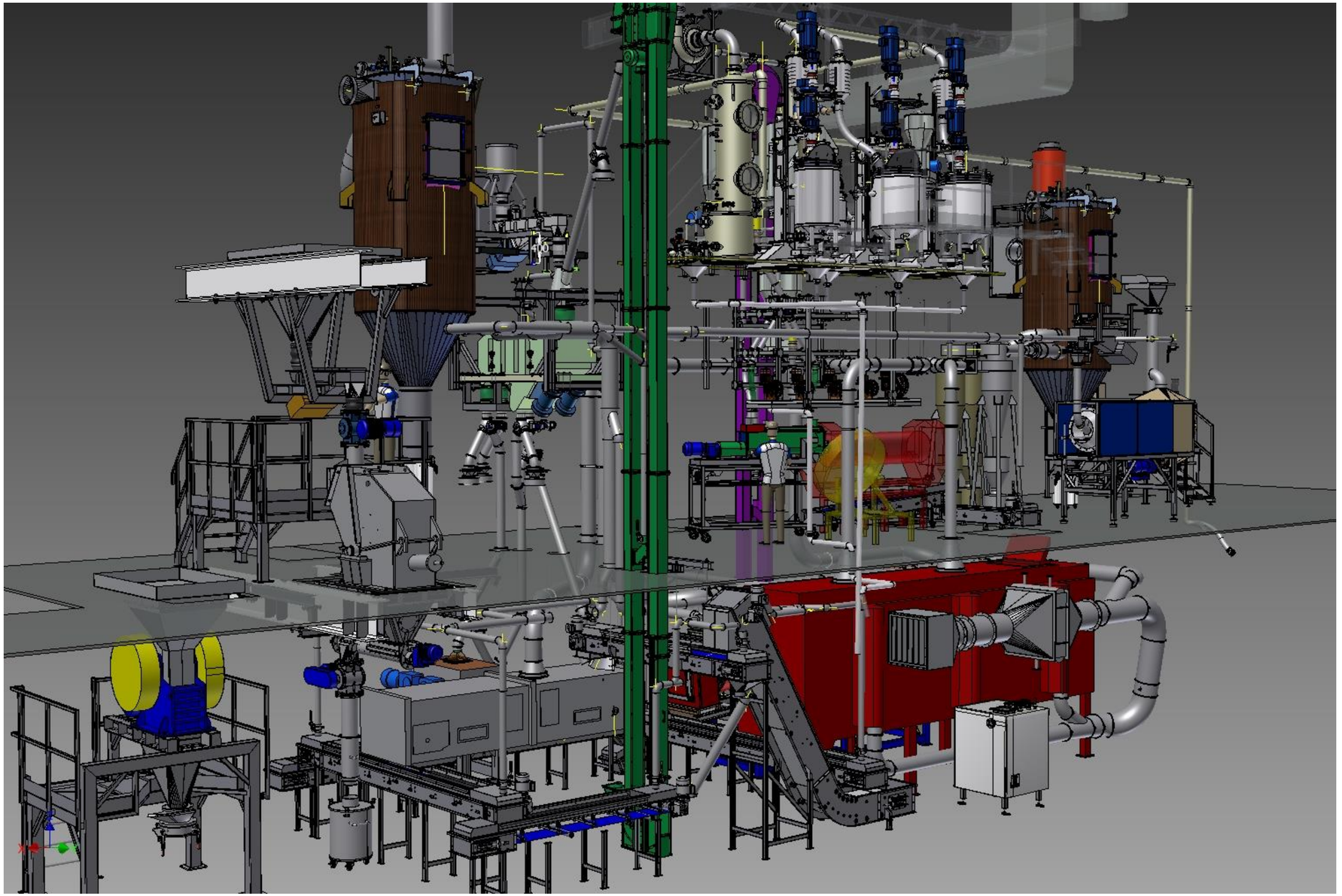
the main components of the fertilizer are: urea, ammonium sulphate, calcium phosphates, calcium sulphate

Flow diagram of semi-pilot plant at Research Centre at Ł-INS

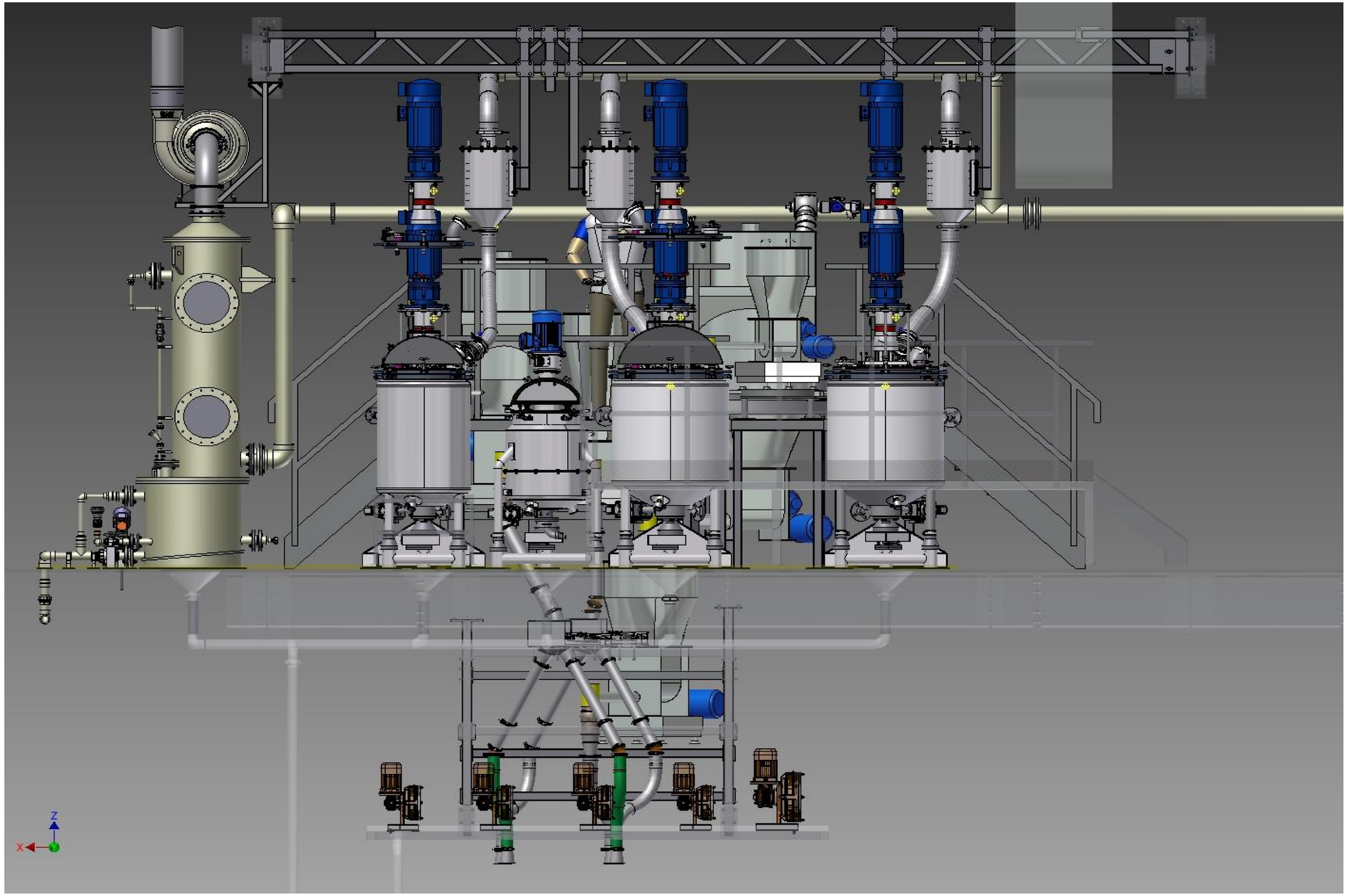


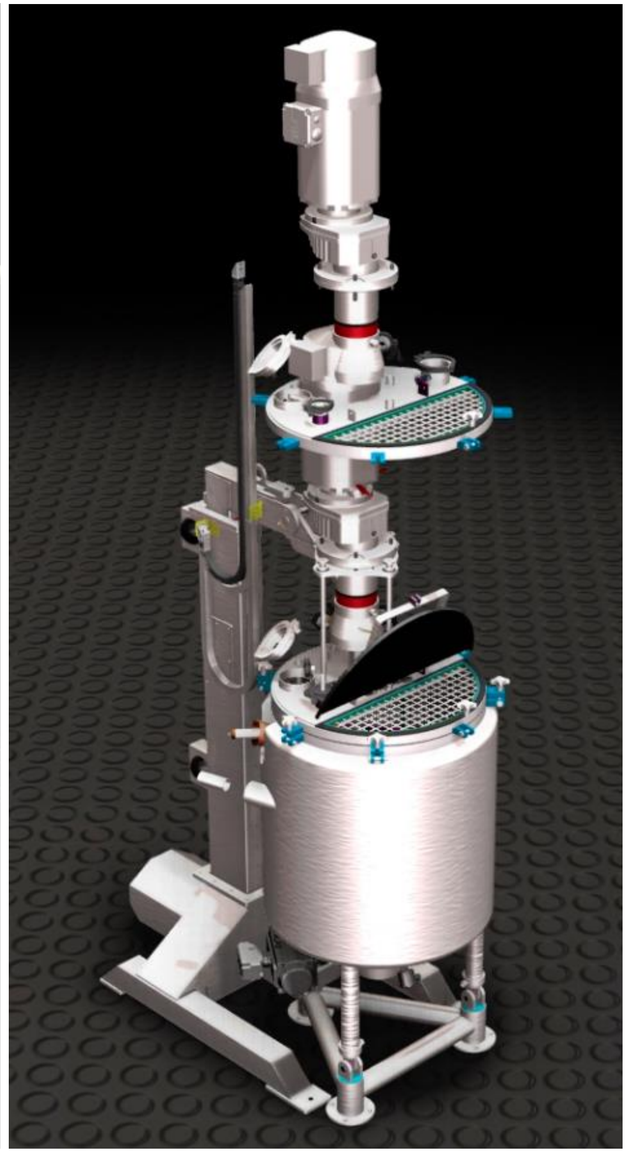


Semi pilot plant – spatial view



A system of reactors for melts and slurries production

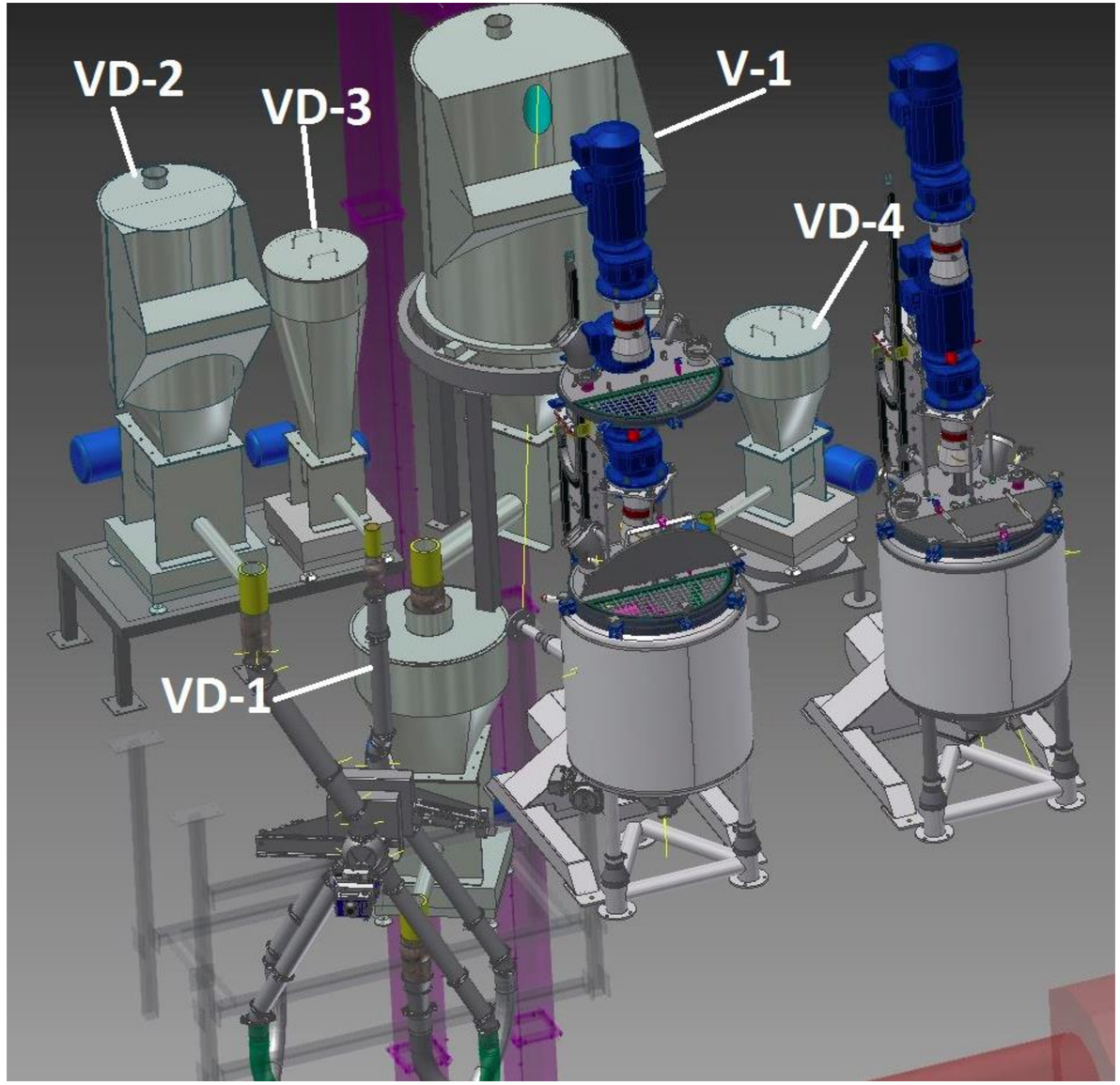




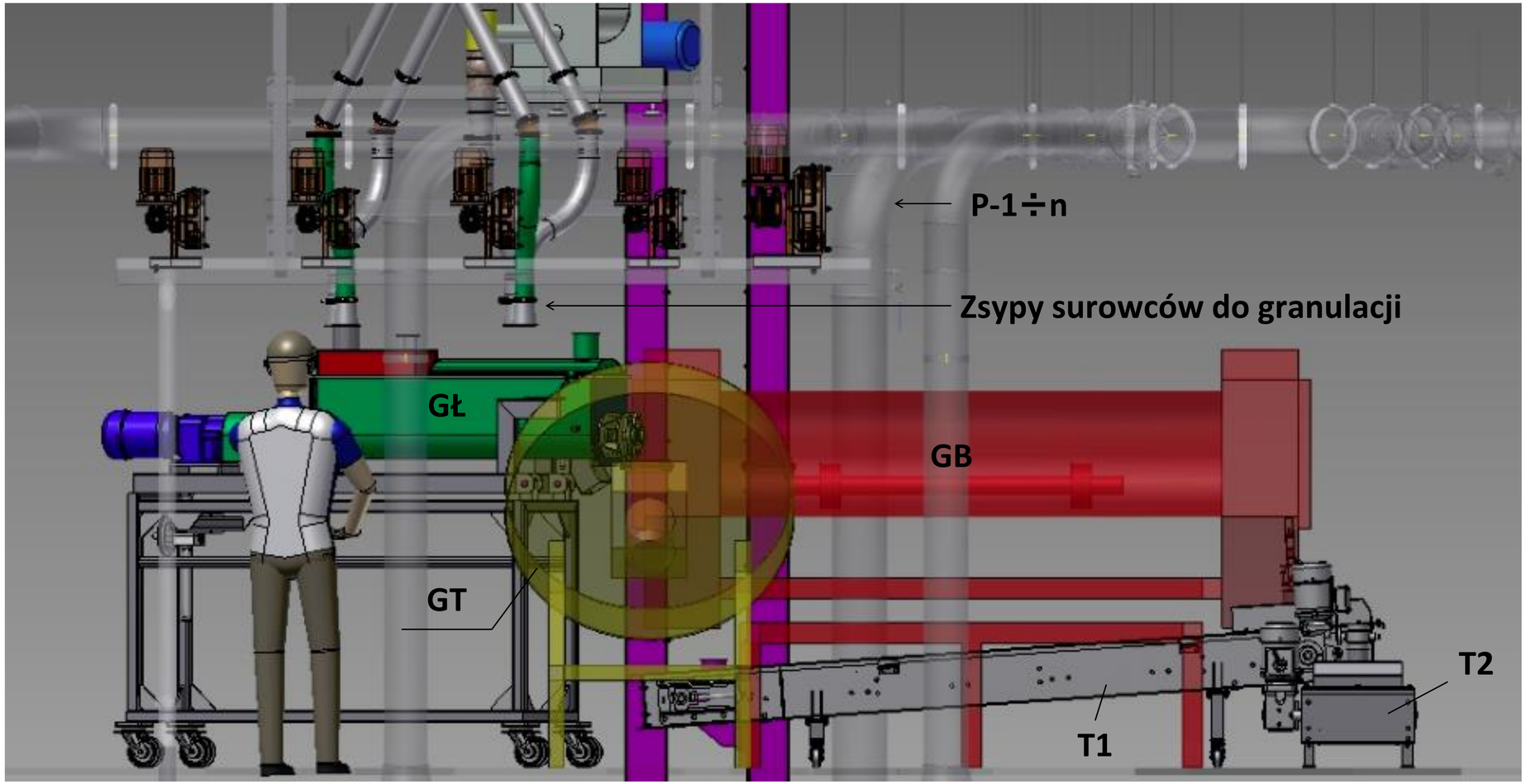
A system of reactors for melts and slurries production

A system of reactors for melts and slurries

A system of dosing raw material



Granulation unit



GŁ - paddle ganulator

GT -pan granulator

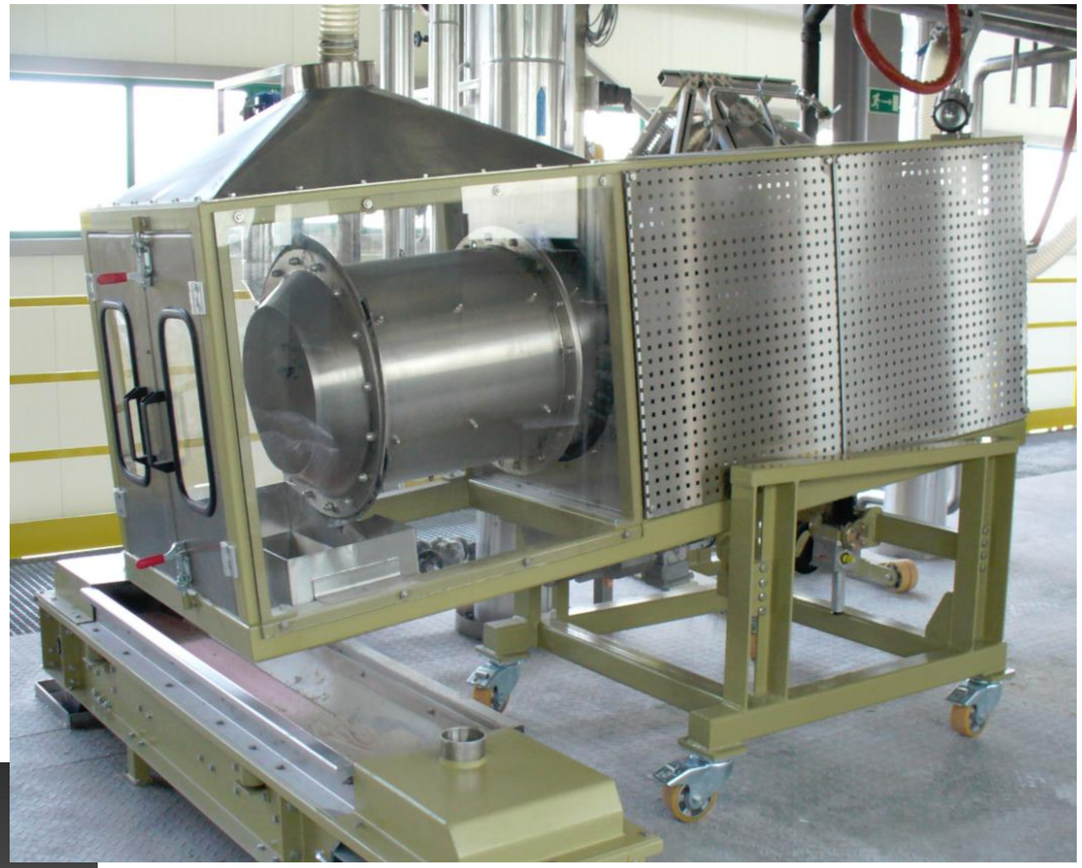
GB - drum granulator

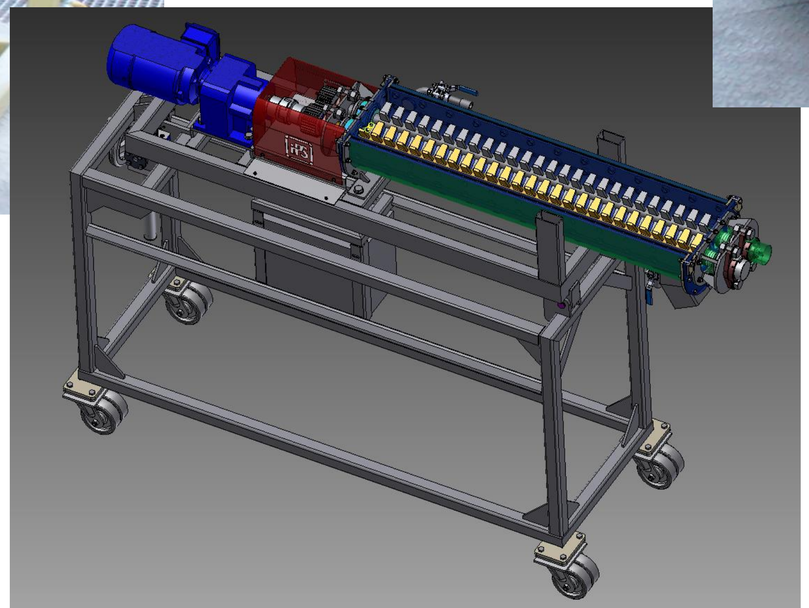
P-1÷n - pumping system

T1, T2 - belt conveyors

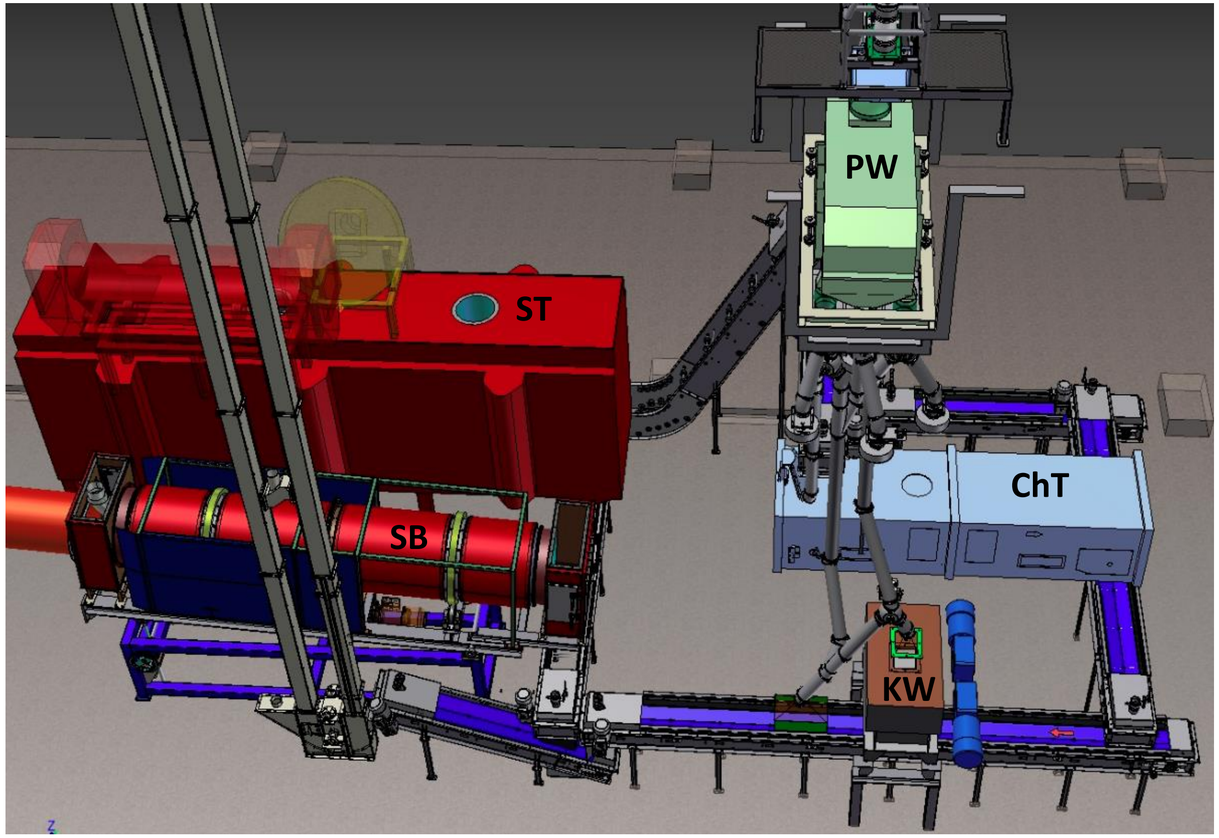
Granulation unit

Granulation unit





Drying unit



ST - belt dryer
SB - drum dryer

ChT - belt cooler
KW - roller crusher

PW - vibrating screen

Drying unit - belt dryer

- 3 belt decks 5 m long and working width 70 cm,
- drying air from a steam heater,
- the flow of drying air distributed in a labyrinth through successive decks, starting from the lower one.

Drying unit – drum dryer

- a drum with a diameter of 70 cm and a length of 4 m,
- paddle system designed by our team,
- drying with air from a steam heater or natural gas fumes,
- optional co-current and counter-current drying air flow,
- adjustable rotation speed and tilt of the drum.

Raw materials used in a research

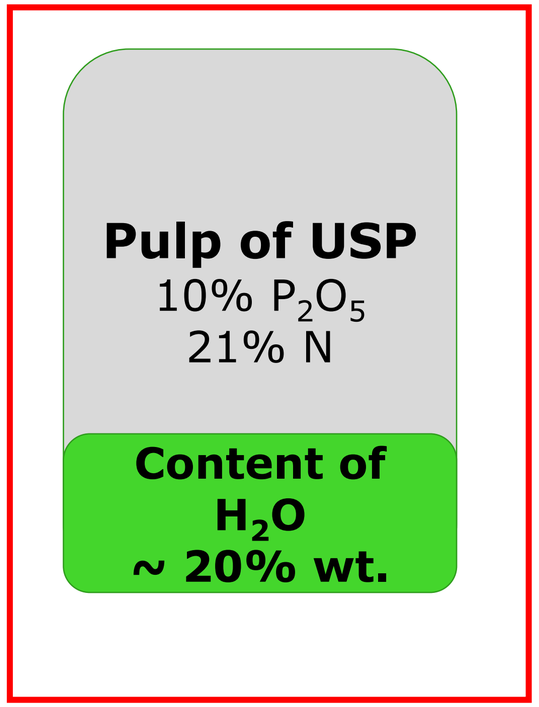
Pulp of USP:

- **Phosphate raw material (Algerian phosphate rock)**
  - **P_2O_5 tot. – 30,18% wt.**
  - **CaO tot. – 48,49% wt.**

- **Urea – urea prill (from G.A. Z.A. „Puławy" S.A.)**
  - **N tot. – min. 46, 0% wt.**
  - **biuret – max. 1,2% wt.**
  - **% H_2O – max. 0,3% wt.**

- **Sulfuric acid**
  - **concentration 94,0%**

- **Water**



Raw materials used in a research

Other raw materials and additives:

- **Magnesite**
  - **MgO tot. – 30-44%**

- **Ammonia (from G.A. Z.A. „Puławy” S.A.)**

- **SBO - antifoam agent**

(a mixture of dioctyl sodium sulfosuccinate, water and octyl alcohol)

- **Water**

Chemistry of the process

Three desirable reactions can be distinguished during the USP manufacturing process:

$$H_2SO_4 + CO(NH_2)_2 \longrightarrow CO(NH_2)_2 \cdot H_2SO_4 \quad (1)$$

$$CO(NH_2)_2 + CO(NH_2)_2 \cdot H_2SO_4 \longrightarrow 2CO(NH_2)_2 \cdot H_2SO_4 \quad (2)$$

$$Ca_{10}(PO_4)_6F_2 + 6[H_2SO_4 + 3{,}6CO(NH_2)_2] + 3H_2O \longrightarrow 3Ca(H_2PO_4)_2 \cdot H_2O + CaF_2 + 5{,}4[CaSO_4 \cdot 4CO(NH_2)_2 + 0{,}6[CaSO_4] \quad (3)$$

Semi-technical trials – verification tests

The process parameters in the granulation tests were as follows:

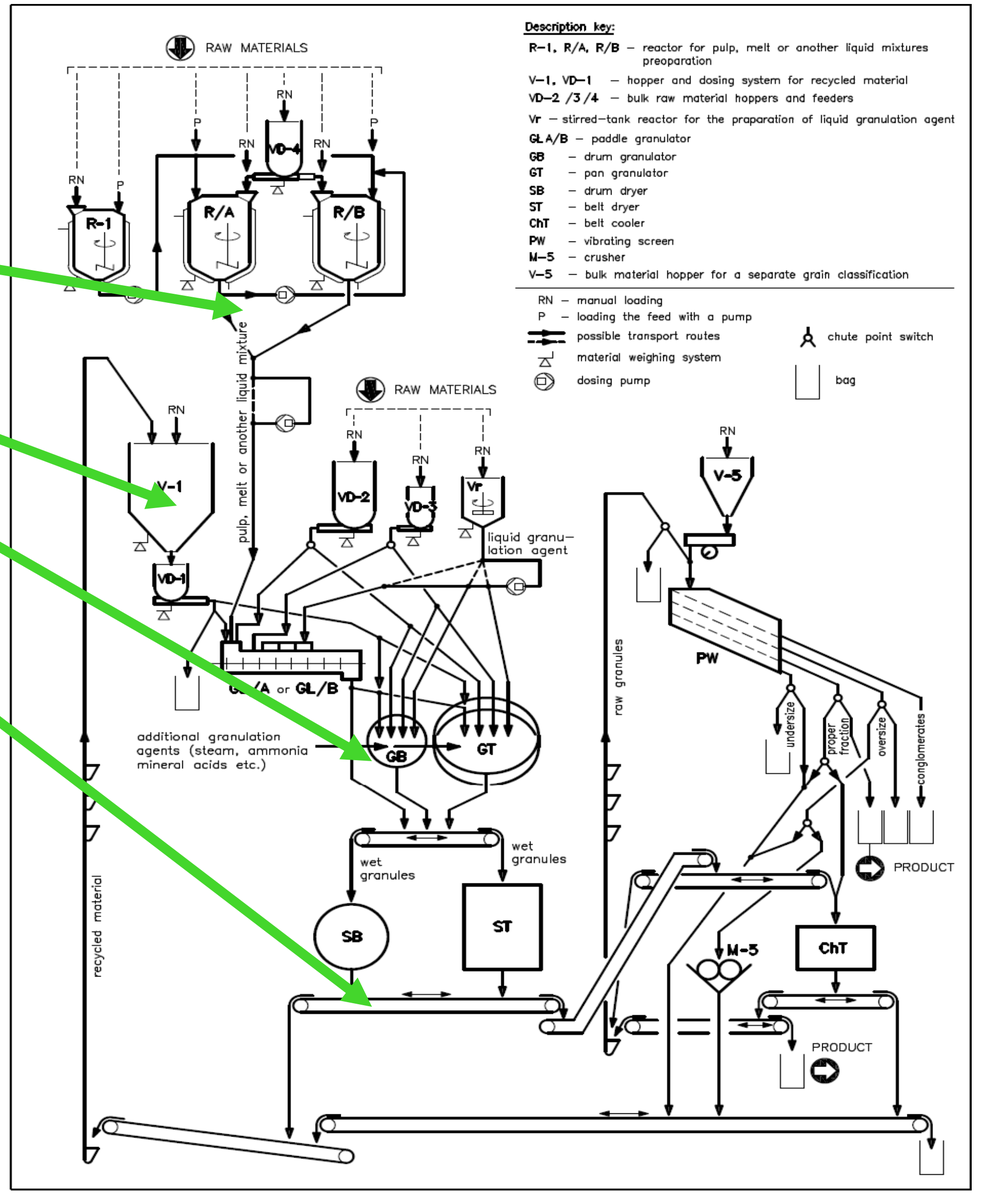
- granulation pulp temperature 75-90°C
- recycle temperature: 60-70°C
- the temperature in the granulation unit: 55-70°C
- product temperature after drying: 85-95°C

The tests carried out to produce a urea superphosphate fertilizer were aimed at:

i) assessment of the transport and granulation properties of the pulps,

ii) determination of technological and process parameters,

iii) assessment of product physicochemical properties.



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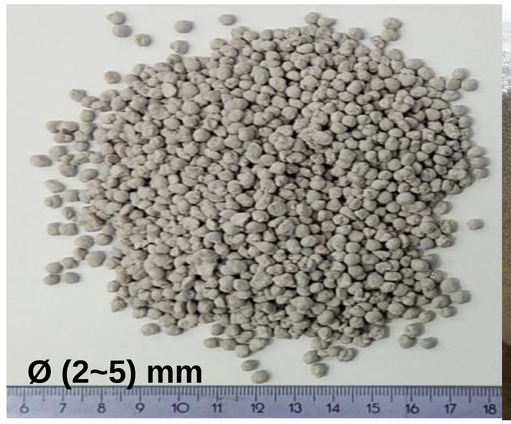


Product and process

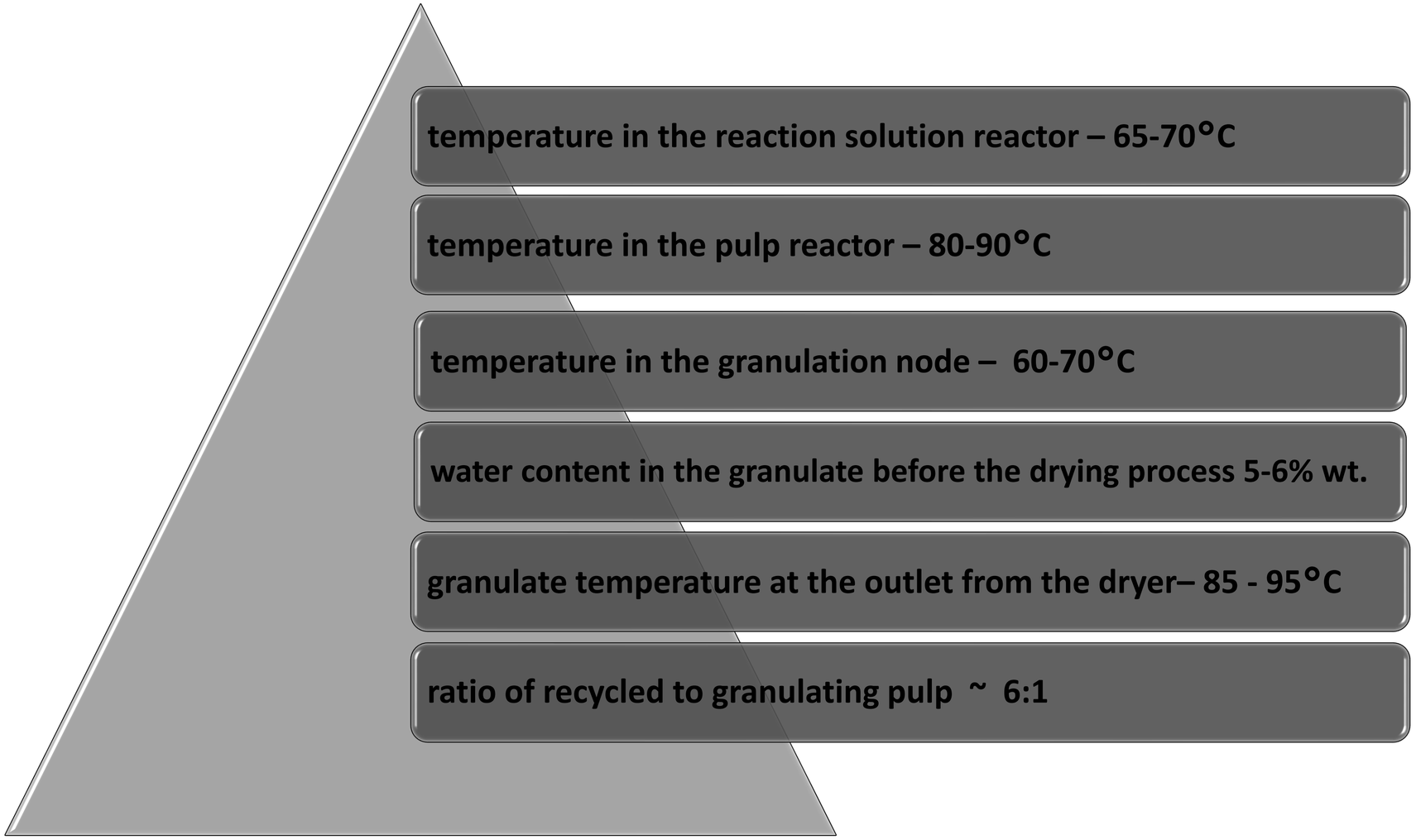
Research and analysis results

Table 1. Physicochemical analysis results of USP average sample prepared in tests on semi-technical scale.

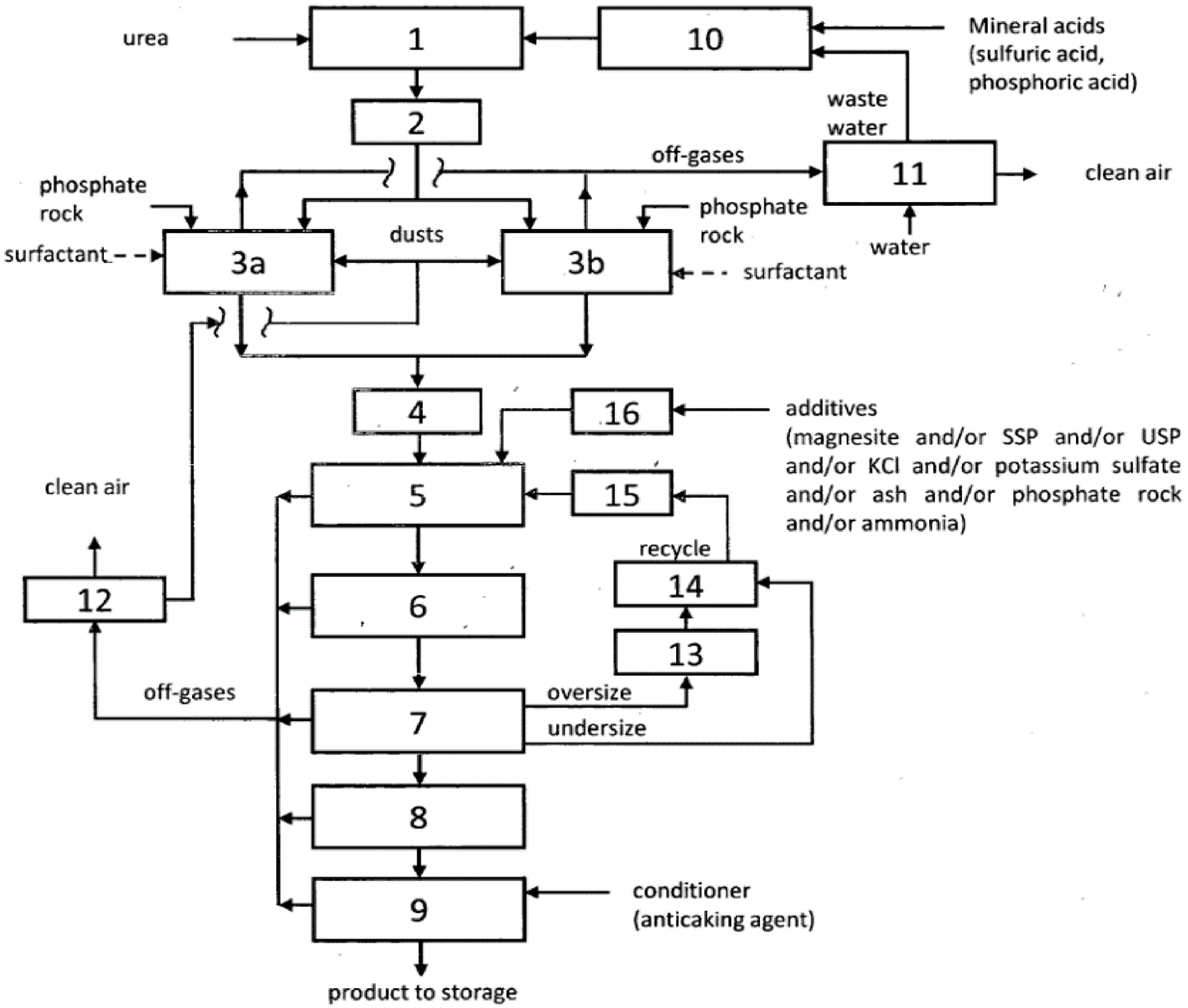
Total wt%	N	N_{NH3}	P_2O_5	$P_2O_{5\ (ws+c)}$	$P_2O_{5\ (ws)}$	CaO	SO_4	H_2O	Crush strength, kG/granule	Median diameter, mm
USP	20.15	0.89	9.16	7.61	5.52	12.31	23.43	1.77	4.5	3.81

$N_{tot.}$	20.15	$P_2O_{5\ tot.}$	9.16	$P_2O_{5\ (ws+c)}$	7.61

Main parameters of the technological process



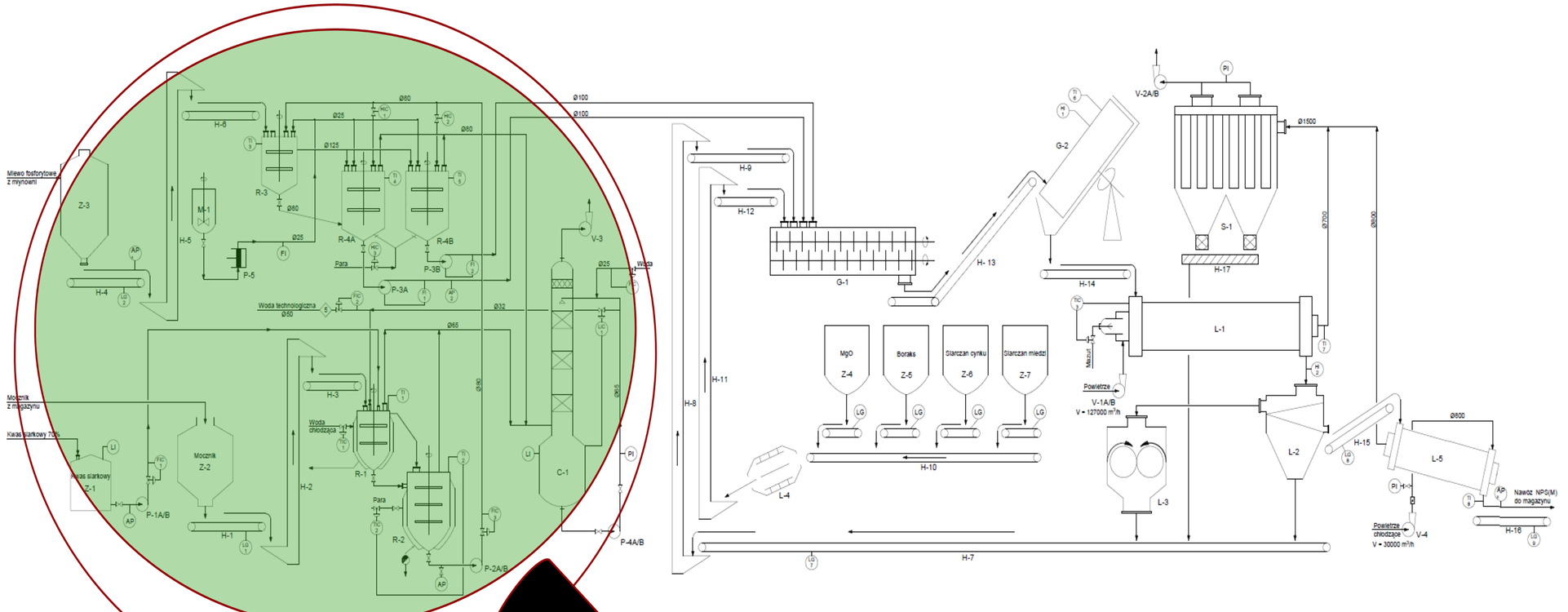
Block diagram



An industrial plant presented on the block diagram, consists of the following components:

- reactor-mixer 1 for the production of reactant solution,
- reactant solution dispenser 2,
- pulp reactors 3a,b,
- pulp dispenser 4,
- granulator set 5 consisting of a granulator-mixer and a plate granulator,
- rotary dryer 6 supplied co-currently with heated air,
– a set of sieves 7,
- cooler 8,
– conditioning drum 9,
- acid thinner/mixer 10,
- wet-treatment unit: scrubber 11,
- dry treatment unit: cyclone 12,
- oversized material mill 13,
- recycle buffer tank 14,
- recycle dispenser 15,
- additional raw materials dispenser 16.

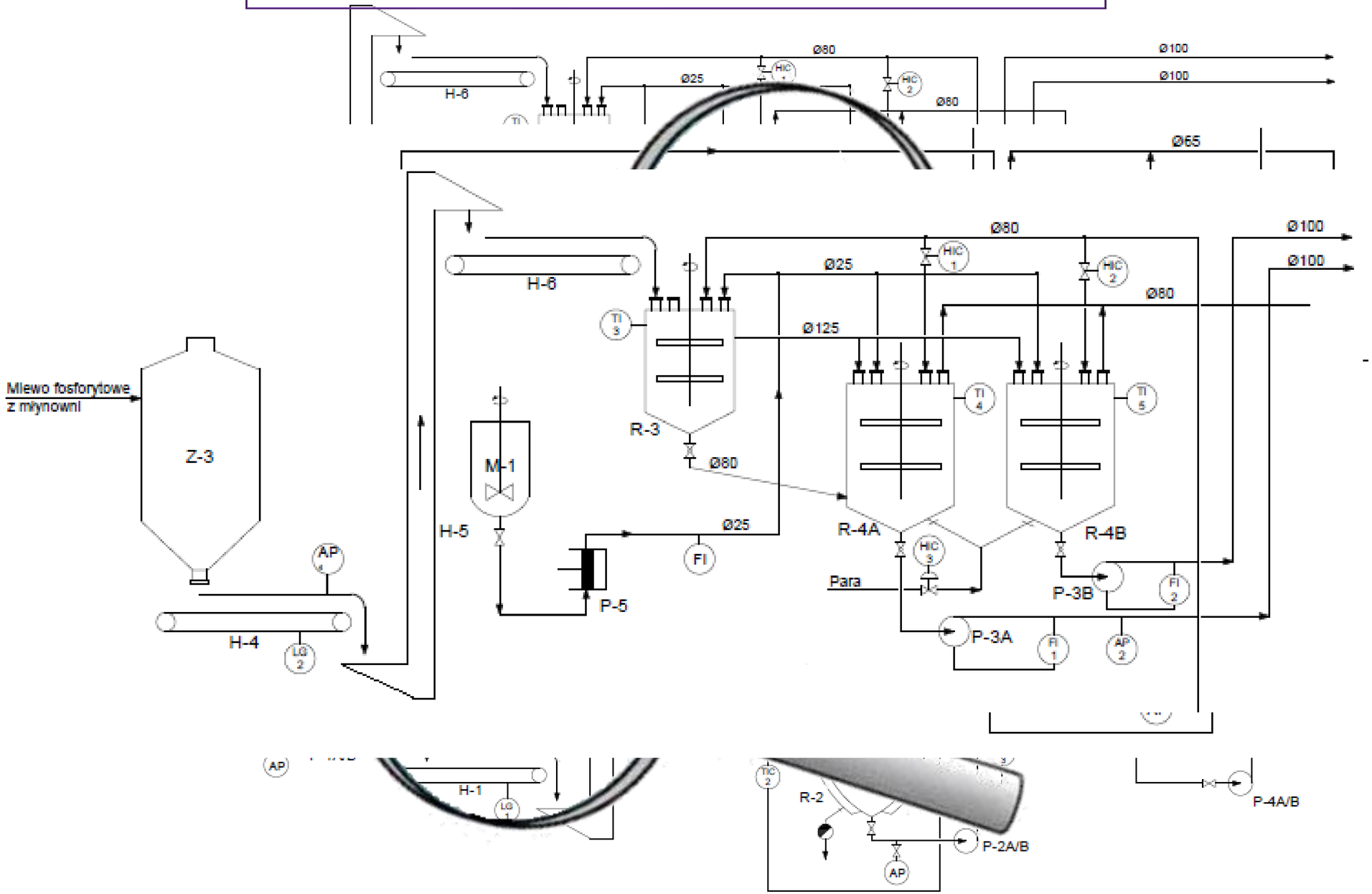
Process diagram



Ozn.	Nazwa aparatu/ urządzenia	Szt.	Charakterystyka
H-1	Przenośnik taśmowy dozujący mocznika	1	G=9000 kg/h
H-2	Przenośnik kubełkowy mocznika	1	G=9000 kg/h
H-3	Przenośnik taśmowy mocznika	1	G=9000 kg/h
H-4	Przenośnik srubowy mączki fosforytowej	1	G=7000 kg/h
H-5	Przenośnik kubełkowy mączki fosforytowej	1	G=7000 kg/h
H-6	Przenośnik taśmowy mączki fosforytowej	1	G=7000 kg/h
H-7	Przenośnik taśmowy zawrotu	1	G=60000 kg/h
H-8	Przenośnik kubełkowy zawrotu	1	G=60000 kg/h
H-9	Przenośnik dozujący zawrotu	1	G=60000 kg/h
H-10	Przenośnik taśmowy mikroelementów	1	G=1500 kg/h
H-11	Przenośnik kubełkowy mikroelementów	1	G=1500 kg/h
H-12	Przenośnik dozujący mikroelementów	1	G=1500 kg/h
H-13	Przenośnik taśmowy granulatu	1	G=85000 kg/h
H-14	Przenośnik taśmowy granulatu	1	G=85000 kg/h
H-15	Przenośnik taśmowy produktu	1	G=20000 kg/h
H-16	Przenośnik taśmowy produktu	1	G=20000 kg/h
H-17	Przenośnik śrubowy pyłów	1	G=1500 kg/h
V-1A/B	Wentylator powietrza do suszarki	2	V=127000 m^3/h
V-2A/B	Wentylator filtra workowego	2	V=165000 m^3/h
V-3	Wentylator kolumny absorpcyjnej	1	V=2000 m^3/h
V-4	Wentylator powietrza chłodzącego	1	V=30000 m^3/h
P-1A/B	Pompa kwasu siarkowego	2	V=5 m^3/h
P-2A/B	Pompa roztworu reakcyjnego	2	V=13 m^3/h

P-3A/B	Pom	2	V=17 m^3/h
P-4A/B	Pom	2	V=5 m^3/h
P-5A/B	Pomp	2	V=10 l/h
S-1	Filtr wor	1	F=2262m^2,n=960 szt.
L-1	Suszarka b	1	D=3,6 m L=27,4 m
L-2	Przesiewacz	1	G=76000 kg/h
L-3	Młyn walcowy	1	G=5000 kg/h
L-4	Mieszalnik mikroele	1	D=1,2 m L=3 m
L-5	Chłodnica granulatu		D=1,6 m L=10 m
G-1	Granulator łopatkowy		= 800mm L=2,68m
G-2	Granulator talerzowy		m
C-1	Kolumna absorpcyjna		mm H=10 m
M-1	Mieszalnik SBO		
R-1	Reaktor roztwarzania mocznika		
R-2	Reaktor roztworu reakcyjnego		
R-3	Reaktor wstępny (premikser)		
R-4A/B	Reaktor przygotowania pulpy		
Z-1	Zbiornik kwasu siarkowego		
Z-2	Zbiornik mocznika	1	
Z-3	Silos mlewa fosforytowego	1	V=
Z-4	Zbiornik magnezytu z przenośnikiem	1	V=20
Z-5	Zbiornik boraksu z przenośnikiem	1	V=10 m^3
Z-6	Zbiornik siarczanu cynku z przenośnikiem	1	V=10 m^3
Z-7	Zbiornik siarczanu miedzi z przenośnikiem	1	V=10 m^3

Process diagram



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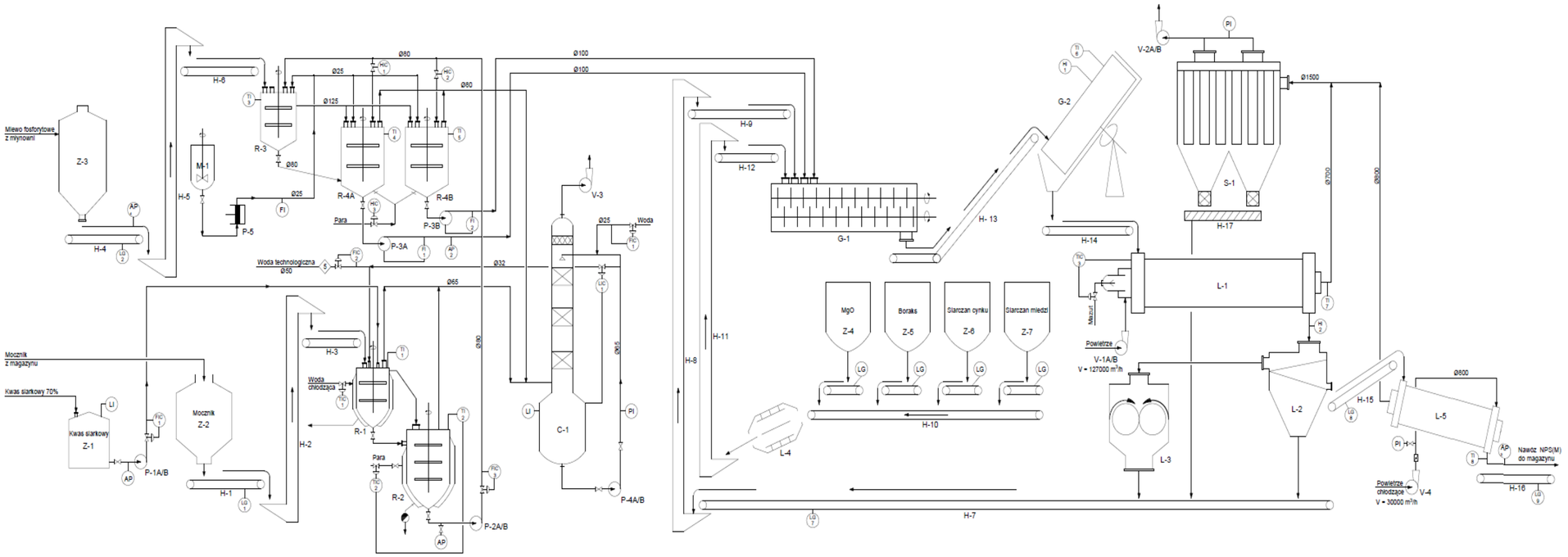

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L-3	Młyn walcowy	1	G=5000 kg/h
L-4	Mieszalnik mikroelementów nawozowych	1	D=1,2 m L=3 m
L-5	Chłodnica granulatu	1	D=1,6 m L=10 m
G-1	Granulator łopatkowy	1	D= 800mm L=2,68m
G-2	Granulator talerzowy	1	D=4 m
C-1	Kolumna absorpcyjna	1	D=650 mm H=10 m
M-1	Mieszalnik SBO	1	V=1 m^3
R-1	Reaktor roztwarzania mocznika	1	V=6,3 m^3
R-2	Reaktor roztworu reakcyjnego	1	V=20 m^3
R-3	Reaktor wstępny (premikser)	1	V=3,2 m^3
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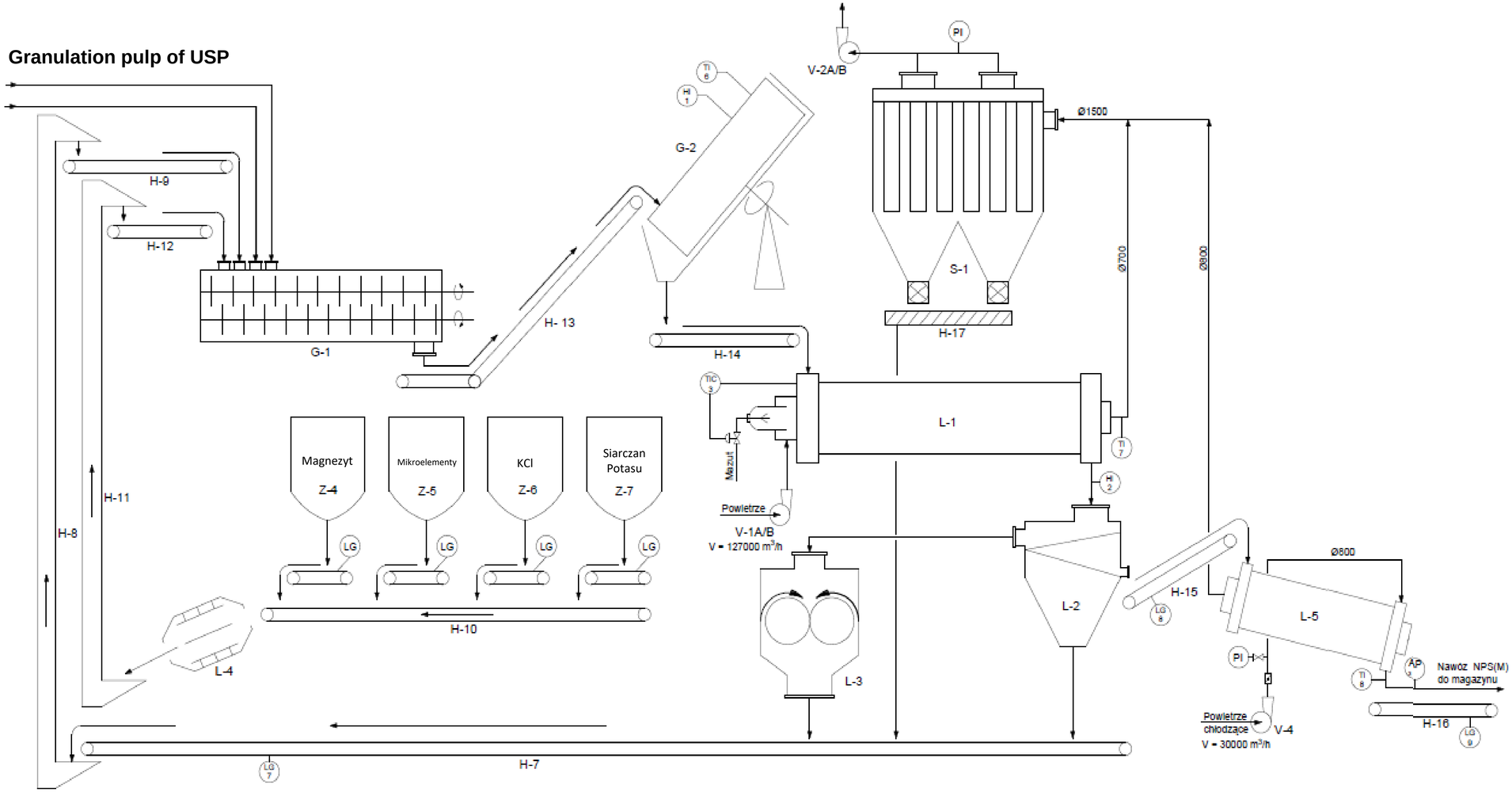
Process diagram

Granulation pulp of USP



Summary

The production process is feasible.

The main advantages of the presented method are:

- **ability to conduct the continuous production, avoiding the necessity of additional storage and additional operations in the production process, related to the aging,**
- **possibility to use sulphuric acid with lower concentration, e.g. recovered from various industries,**
- **reduced process security risks by using the reactant solution with lower concentration and by completed reaction of sulphuric acid with rock phosphate during preparation of the process pulp,**
- **mitigation of the risk of formation of substances harmful to plants by using reactive solution with lower concentration,**
- **in order to avoid dependence on the type of phosphorite, the use of two slurry reactors operating alternately, is preferred.**

it is advisable to use additives supporting the process, i.e. magnesite and ammonia, they increase the thermal resistance of granules during the drying process, especially at elevated temperatures (after exceeding the product temperature of 90°C at the dryer outlet, the strength of the granules drastically decreases and they become brittle, and in the further plastic and adhesive sequences).

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Summary

The main advantages of the presented method are:

- **the two-stage granulation method (granulator-mixer and flow granulator, i.e. drum granulator or pan granulator) allows to increase the share of the specific fraction of the product, which translates into reduced recycle and energy consumption of the process, as well as reduced conversion of urea in side reactions leading to to the formation of products harmful to plants and the retardation of phosphorus.**
- **it is advisable to use additives supporting the process, i.e. magnesite and ammonia, they increase the thermal resistance of granules during the drying process, especially at elevated temperatures (after exceeding the product temperature of 100°C at the dryer outlet, the strength of the granules drastically decreases and they become brittle, and later plastic and sticky).**

Thank you for attention!

Contact:

dr inż. Sebastian Schab

Łukasiewicz Research Network – New Chemical Syntheses Institute

e-mail: Sebastian.Schab@ins.lukasiewicz.gov.pl

tel. +48 81 473 14 72

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