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40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

PRESENTED BY

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40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

- By neutralising ammonia gas (NH_3) with concentrated (55 - 60 %) nitric acid (HNO_3) an 70-75 % ammonium nitrate (NH_4NO_3) solution is produced.

- The excess water is evaporated across the vacuum evaporators and the resulted melt with 99.5 % ammonium nitrate is used to make solid ammonium nitrate by spraying it in small beads across a prill tower or in granules in a rotating drum granulator. After drying cooling and coating these prills or granules are typical AN or CAN products in commerce.

- The evaporated water from the first stage evaporator containing small amounts of ammonia (0.2 - 0.9 g/l) and ammonium nitrate (1.2 - 2.5 g/l) is condensed and the resulted diluted process condensate is send to the nitric acid plant.

- The evaporated water from the second stage evaporator with higher amounts of ammonia (0.6 - 10 g/l) and ammonium nitrate (3 - 15 g/l), some times more, is condensed and the resulted concentrated condensates together with other nitrogen contaminated effluents, in many existing fertiliser factories, are discharged to the environment.

- By comparing with the river or lake water the discharged contaminated process condensates have very high cation and anion charges which could be 20 to 35 times higher.

- The loss of nitrogen products within the discharged condensates and the other waste effluents represents 0.3 to 1.0 % , some times higher, of the total nitrogen production yield.

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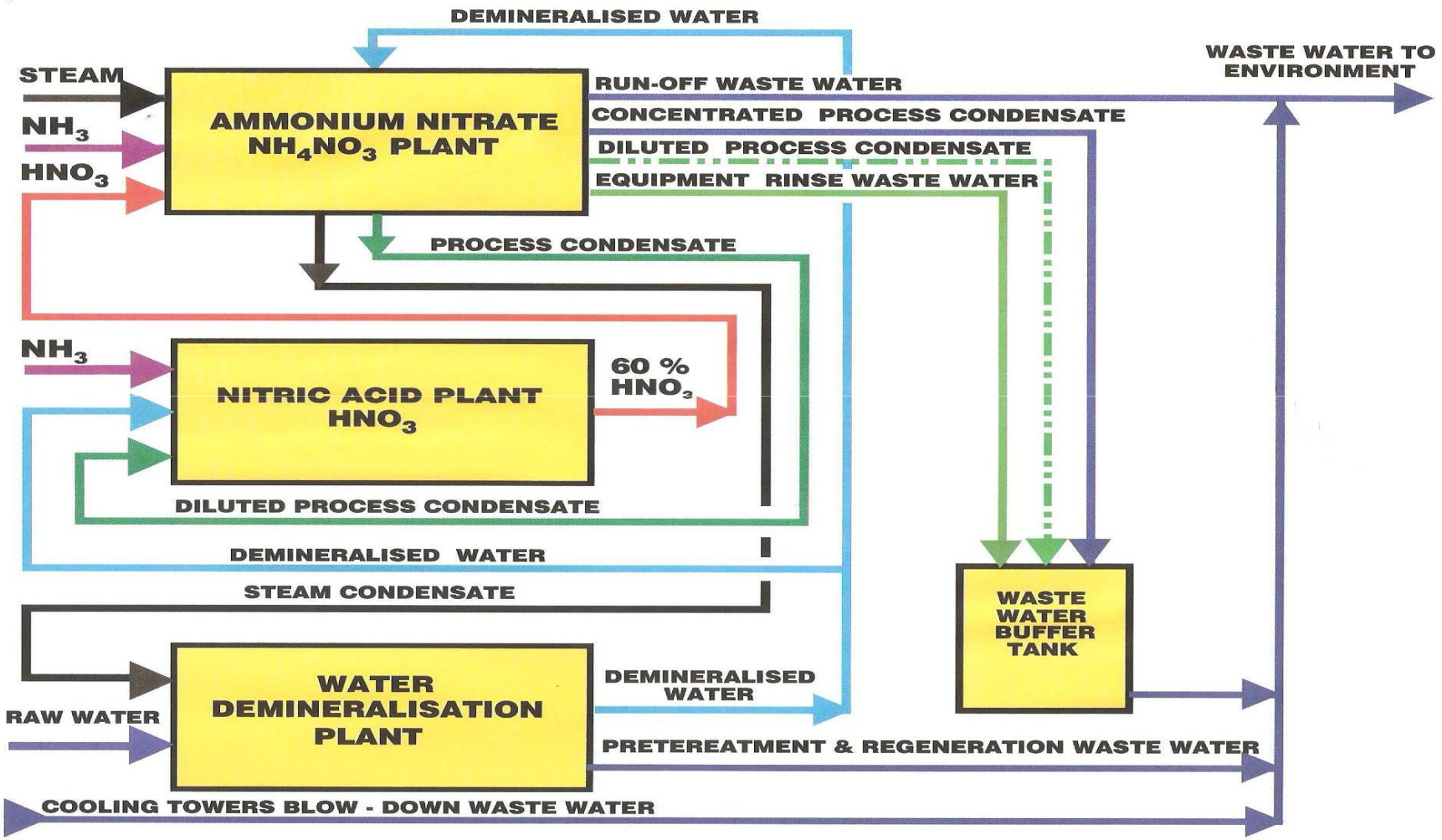

AMMONIUM NITRATE FACTORY WASTE EFFLUENTS DISCHARGED TO THE ENVIRONMENT

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

- The environmental protection rules limits the concentration of the nitrogen products discharged within the contaminated waste effluents to 2 - 3 mg/l ammonium (NH_4) and 25 - 30 mg/l nitrate (NO_3) ions.

- To reach such very low required nitrogen (N) limits from the highly contaminated discharged waste effluents the involved treatment process should be highly effective with a nitrogen removal efficiency higher than 95 %.

- Completely closed treatment processes, with recycling of all material involved back to the process plants and with zero or minimal discharge of supplementary waste effluents to the environment have to be used.

- For the treatment and recovery of the ammonium nitrate plant contaminated waste effluents the following processes has been investigated

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- **Biological Nitrification/Denitrification :** Due to the low nitrogen removal efficiency and the high operation costs the biological treatment process cannot be used.

- **Steam Stripping:** can remove only ammonia NH_3 and NH_4 salts from the waste effluent which was first alcalinised at pH of 10 - 11with NaOH or Ca(OH)2 . After stripping and the neutralising of the stripped effluent with H_2SO_4 or HCL the treated effluent contains the rest of NH4 as $(NH_4)_2SO_4$ and all nitrates as $NaNO_3$ or $Ca(NO_3)_2$. Due to the high energy and operation cost this treatment process cannot be recommended.

- **Reverse Osmosis (RO):** The discharged waste effluent with NH_3 and NH_4NO_3 is first neutralised with concentrated nitric acid to a pH value of 4 – 4.5 and then desalinated accros the RO membranes. For large flow-rates the neutralising process could be difficult. The discharged NH_4NO_3 concentrate product with a strength of 4 – 6 % only , is not sufficient for a profitable recoveryare. So far the RO process has only been operated in pilot plants and there are no industrial scale installations this process cannot be used.

- **Evaporation** is an effective treatment process especially for the concentrated process condensates that have low flow-rates (2-15 m^3/h) and high levels of NH_3 (5 - 35 g/l) and NH_4NO_3, some times even more. Due to the high steam and cooling water consumption, which are directly proportional to the amount of water to be evaporated, the operating cost for evaporation can be very high. To reduce the steam and cooling water consumption multi stage vacuum evaporators or vacuum evaporators with mechanical vapour recompression (MVR) have to be used.

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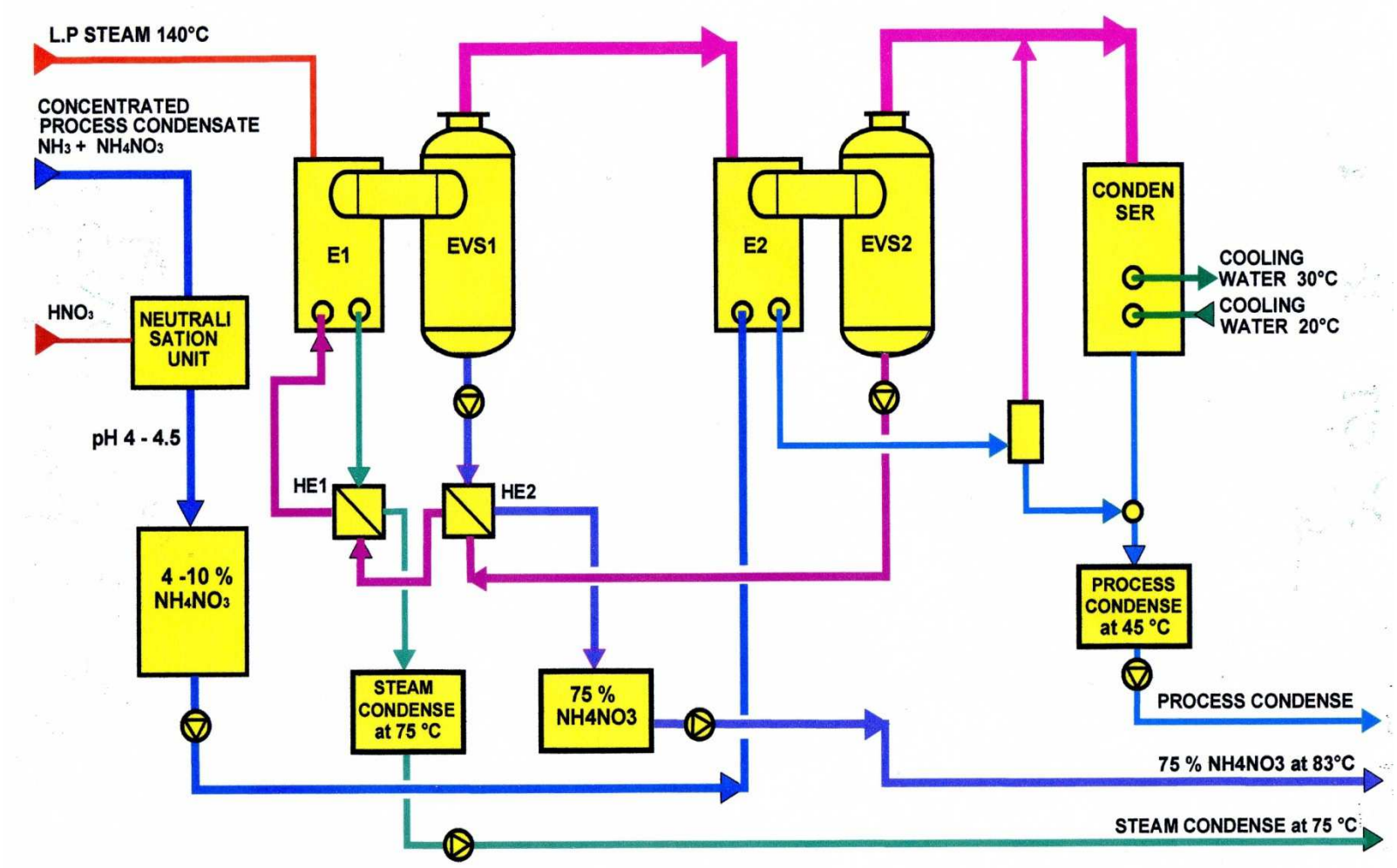

TWO STAGE VACUUM EVAPORATION PLANT

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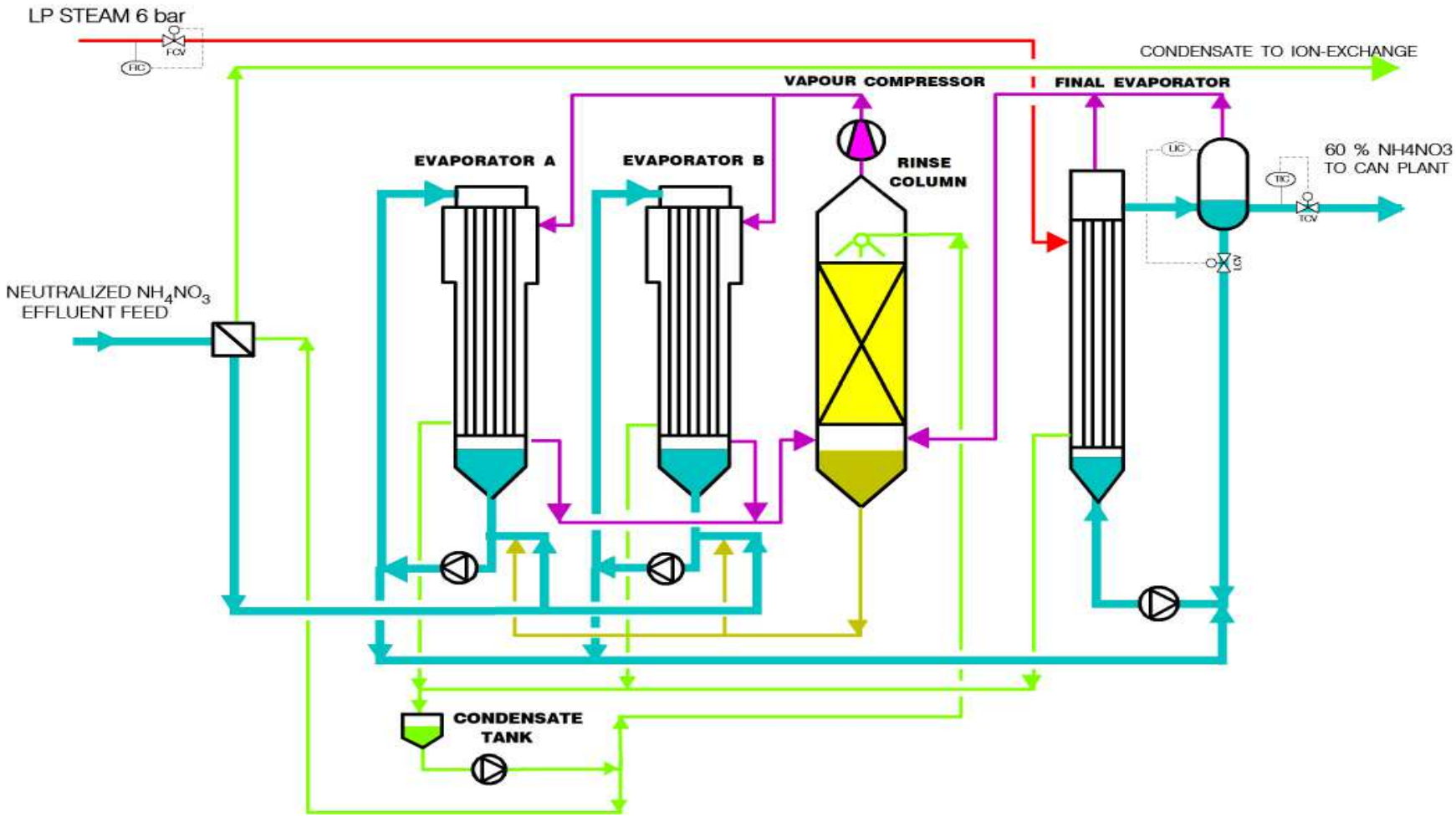

THREE STAGE **VACUUM EVAPORATION PLANT WITH** MECHANICAL VAPOUR RECOMPRESSION (MVR)

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

THE ION-EXCHANGE PROCESS represents the most effective and economical fertiliser contaminated waste effluents treatment and recovery process.

LOADING

For demineralisation the contaminated process condensate flows first through a strong acidic cation resin where NH_4 ions are removed and then across a medium basic anion resin which removes the NO_3 ions. For the two reactions with the major ions of such effluents the following equations can be written

CATION RESIN (R-H)

$$NH_4NO_3 + R\text{-}H \;\text{<==>}\; R\text{-}NH_4 + HNO_3$$

$$NH_3 + R\text{-}H \;\text{<==>}\; R\text{-}NH_4$$

ANION RESIN (R-OH)

$$HNO_3 + R\text{-}OH \;\text{<==>}\; R\text{-}NO_3 + H_2O$$

REGENERATION

During regeneration, the exhausted resins are converted back into their original state according to the following equations

CATION RESIN

$$R\text{-}NH_4 + HNO_3 \;\text{<==>}\; NH_4NO_3 + R\text{-}H$$

ANION RESIN

$$R\text{-}NO_3 + NH_4OH \;\text{<==>}\; NH_4NO_3 + R\text{-}OH$$

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SPECIAL ION-EXCHANGE SYSTEMS

- Due to the high amount of nitrogen products discharged within the fertiliser waste effluents such as 30 to 150 kg/h NH_3 and 60 to 450 kg/h NH_4NO_3 the standard ion-exchange systems cannot be used.

- These amounts in kg represents very large cation and anion charges such as 2'500 to 14'450 eq/h NH_4 and 1'000 to 6'000 eq/h NO_3

- Because of these high ionic charges the ion - exchange system should be capable, each hour, to perform, regeneration of large amounts of ion - exchange resins such as 3 to 15 m^3 of cation resin and 2 to 7 m^3 anion resin, some times higher.

- To achieve such requirements, since 1960 special water treatment companies such as Chemical Separation (Chem-Seps) in USA, Degremont in France, Bran & Lübbe in Germany. Iprochim and Ipran in Romania, Christ and Arionex in Switzerland, have developed and installed: **Special Continuous or Short Cycle Non-continuous Ion - Exchange Systems** which are capable to regenerate hourly large amounts of ion-exchange resins.

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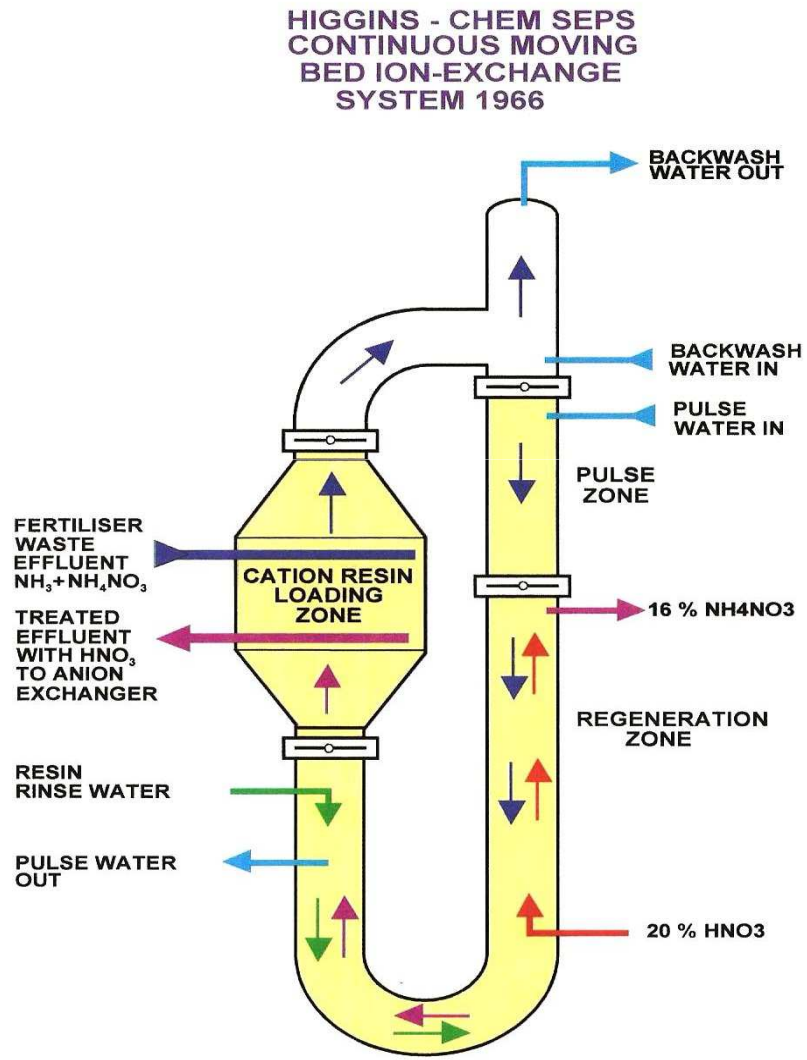

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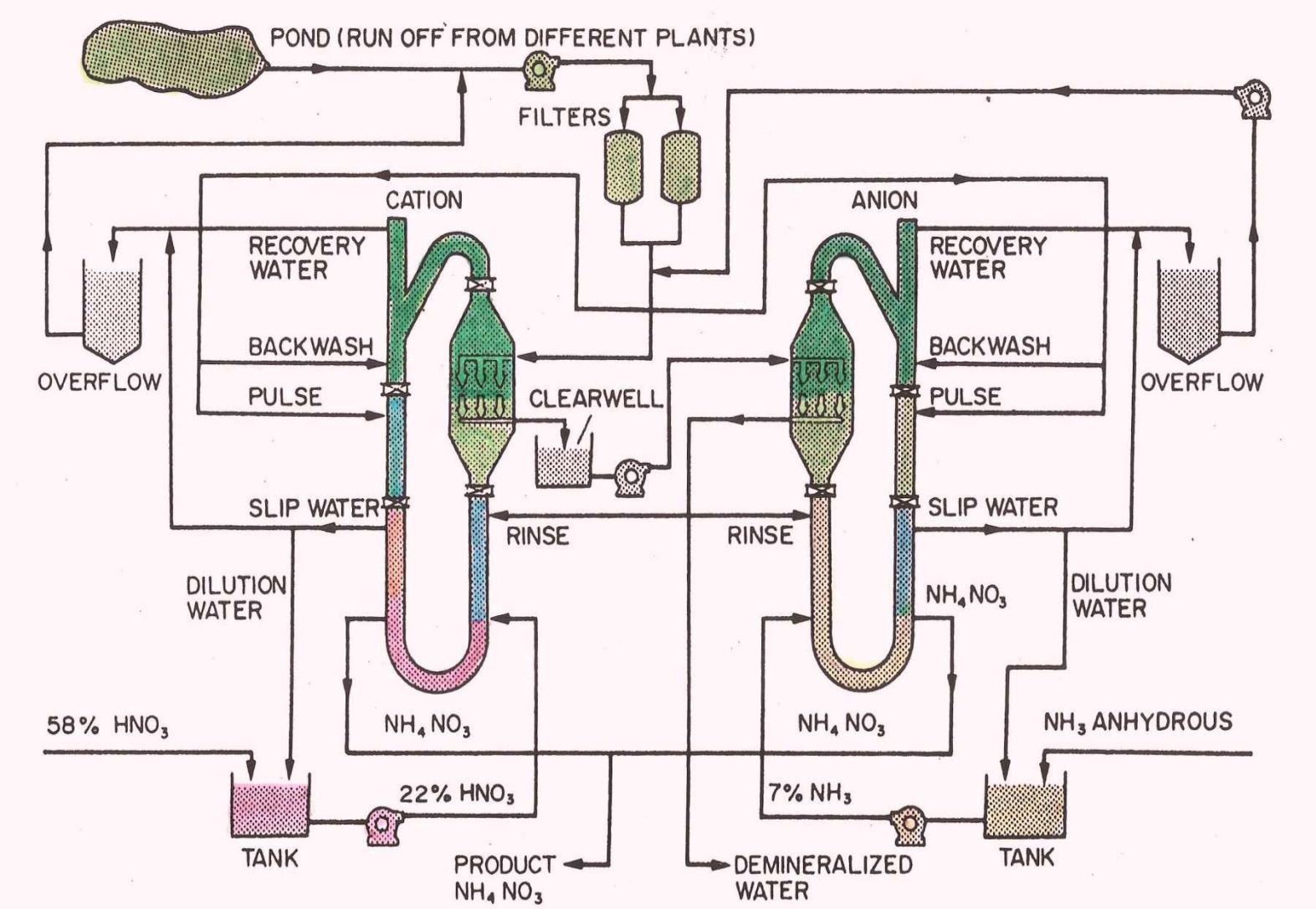
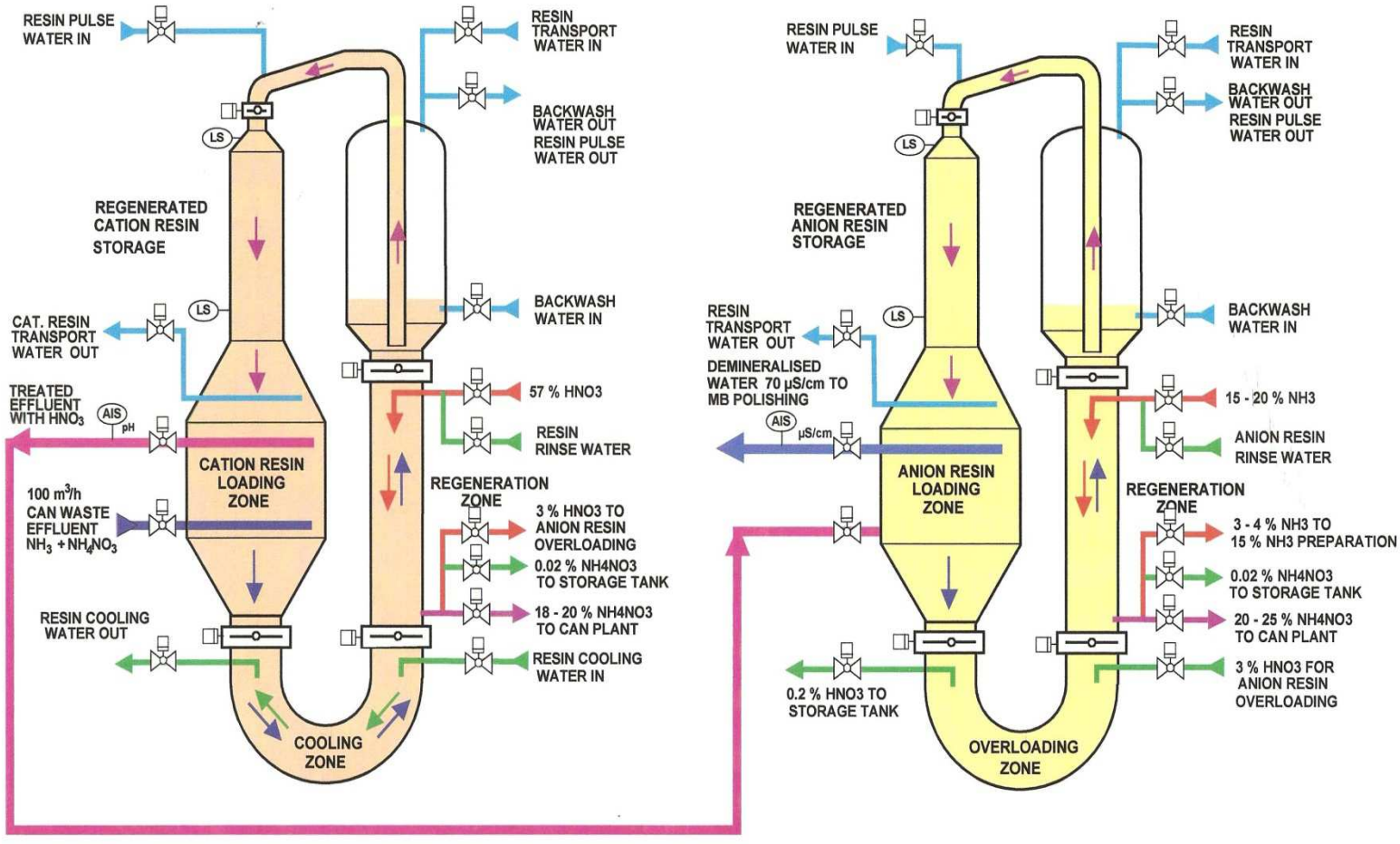


CHEM-SEPS (HIGGINS) CONTINUOUS MOVING BED ION-EXCHANGE FERTILISER WASTE WATER TREATMENT AND RECOVERY PLANT AT CF INDUSTRIES -TENESSEE USA - 1966

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

IPROCHIM -ARION NONCONTINUOUS SHORT CYCLE MOVING BED ION EXCHANGE SYSTEM AT THE FERILISER COMPLEX DOLJ-CHIM - ROMANIA 1976



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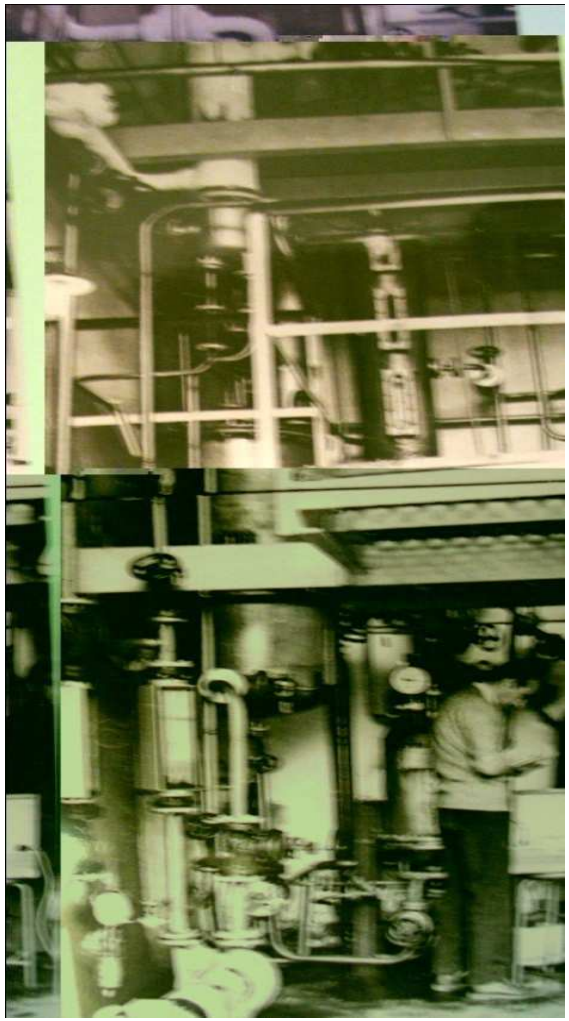
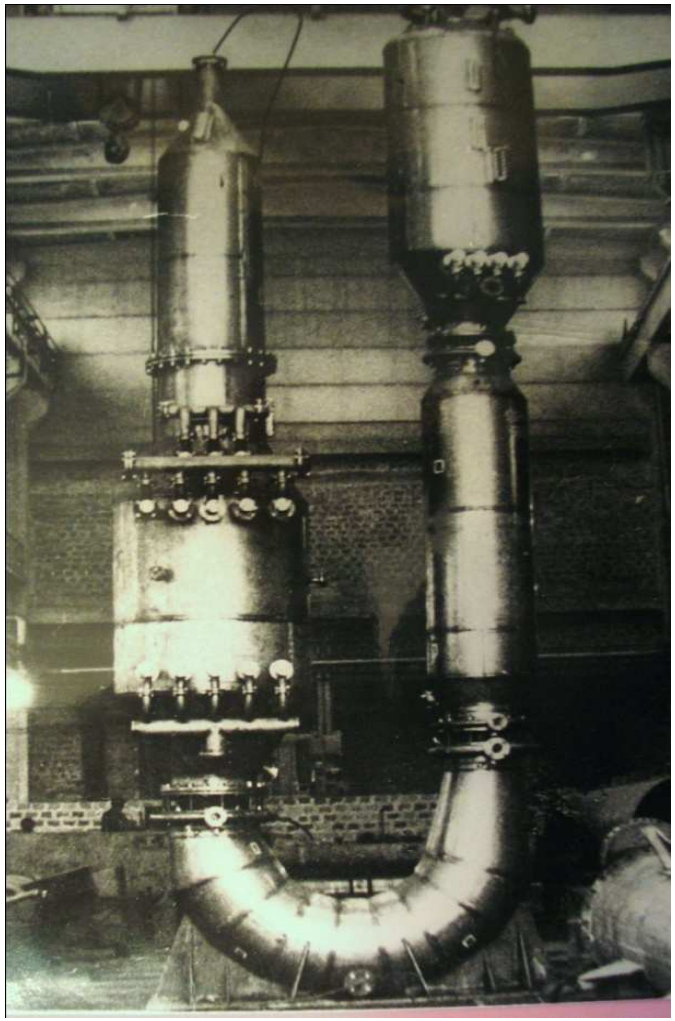


Pilot Plant 6m^3 /h Start-up 1968



Cation Resin Moving Bed Short Cycle Ion-Exchange System -100 m^3/h Start-up 1976

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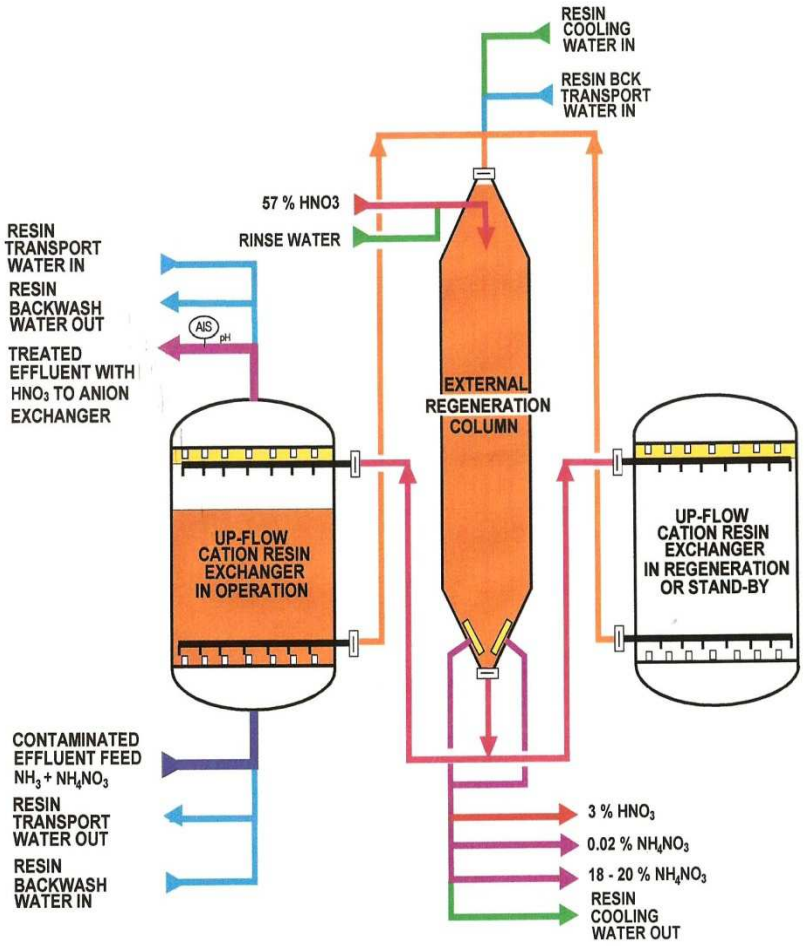
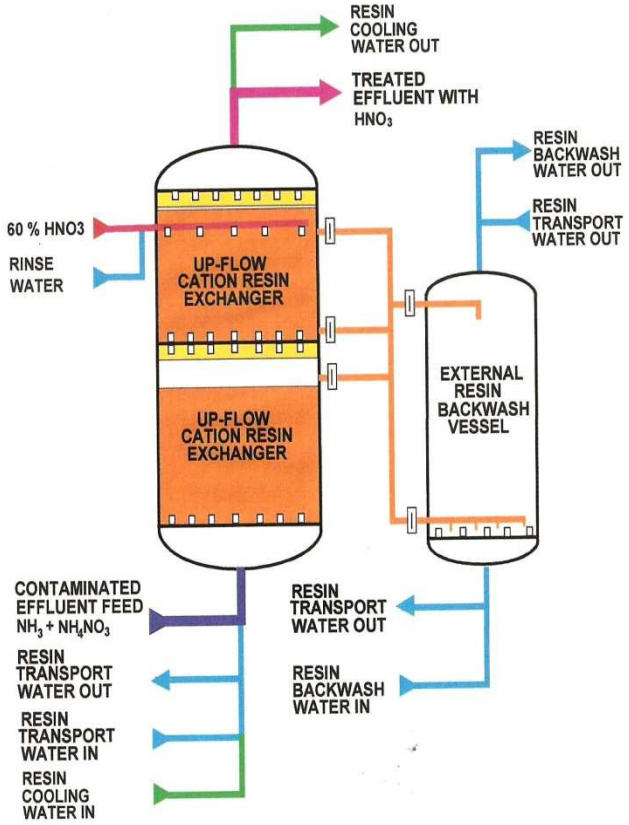
ARION- CHRIST SHORT-CYCLE
ION-EXCHANGE SYSTEM WITH
EXTERNAL REGENERATION
1980



ARIONEX FERTAREX
UP-FLOW COMPACT BED SHORT-CYCLE
ION-EXCHANGE SYSTEM WITH INTERNAL
RESIN REGENERATION
1986/2003



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CHRIST- ARION SHORT CYCLE UP-FLOW COMPACTED RESIN BED ION-EXCHANGE SYSTEM WITH EXTERNAL RESIN REGENERATION - 250 m^3/h AT THE FERTILISER COMPLEX KUTINA PETROKEMJIA - CROATIA START-UP 1982

CATION RESIN EXTERNAL REGENERATION COLUMN

CATION RESIN UP-FLOW COMPACTED BED ION- EXCHANGER - 250 m^3/h

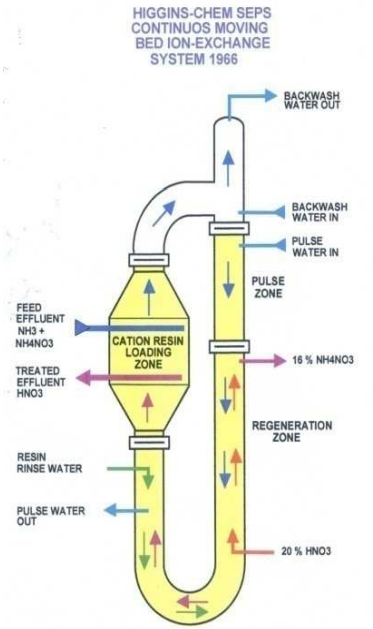
40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

THE FERTAREX SHORT- CYCLE UP- FLOW COMPACTED RESIN BED ION - EXCHANGE SYSTEM WITH INTERNAL REGENERATION AT THE FERTILISER COMPLEX PETI NITROGEN - HUNGARY START UP 1987

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

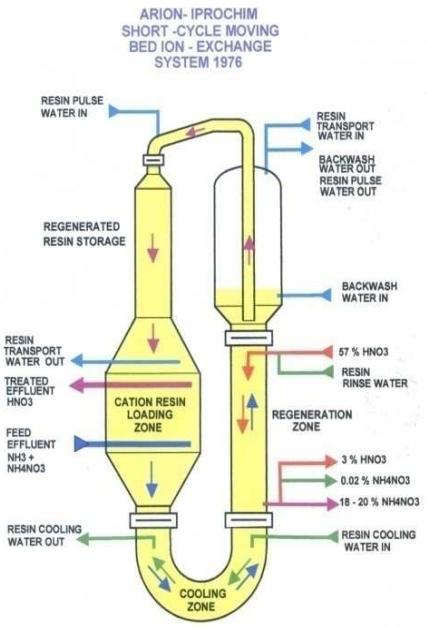
SIMPLIFYING EVOLUTION OF SEVERAL SPECIAL ION-EXCHANGE FERTILISER WASTE WATER TREATMENT SYSTEMS

HIGGINS CHEM-SEPS



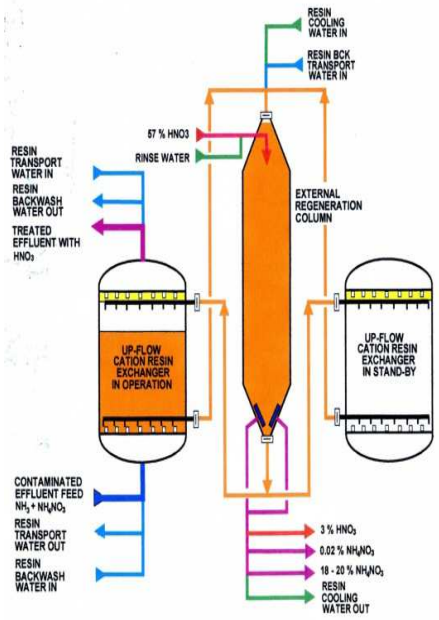
1966

ARION IPROCHIM



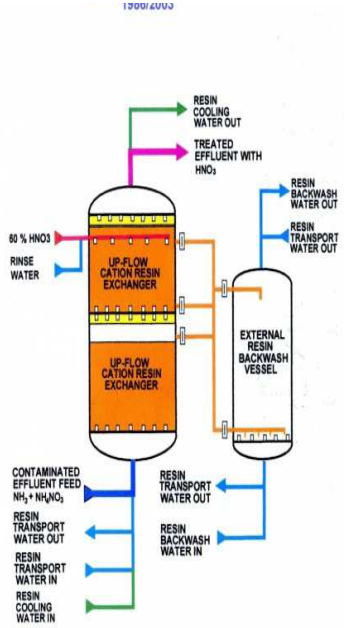
1976

ARION BRAN & LUEBBE/CHRIST



1982

ARIONEX FERTAREX



1987 - 2010

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

- The ARIONEX FERTAREX short cycle up-fow compacted resin bed ion-exchange system with external resin regeneration, is the most effective and economical fertiliser waste effluent treatment and recovery system. Its effectiveness results from the followin features and benefits:

- It is simple, intensive, robust, reliable and safe ion-exchange treatment and recovery process with a removal efficiency higher then 99.7 %

- Due to the use of 55 - 60 % HNO_3 and 15-20 % NH_3 short regeneration time of 35 to 50 minutes for large volumes of cation 3 to 15 m^3 and anion resins 2 to 7 m^3 are achieved.

- As result short loading cycle time of less 120 minutes may be performed which make it possible to remove large amounts of nitrogen products such as 30 to 150 kg/h NH_4 and 50 to 450 kg/h NO_3 , some time higher.

- Safe system for the cation resin regeneration with concentrated HNO_3 due to the use of special cation resins, the cooling of the cation resin regeneration system and the short contact time of less 4 minutes between the cation resin and nitric acid.

- The demineralised water leaving the ion-exchange system is of high quality with conductivity value of less 0.1 µS/cm

- Due to the selective cation and anion regeneration effluent fractioning process highly concentrated (18 to 25 %) NH_4NO_3 solution can be recovered

- In the Nitrogen Fertilizer Chemical Complexes regenerants for the ion-exchange resins, such as nitric acid and ammonia are readily available at site with low cost.

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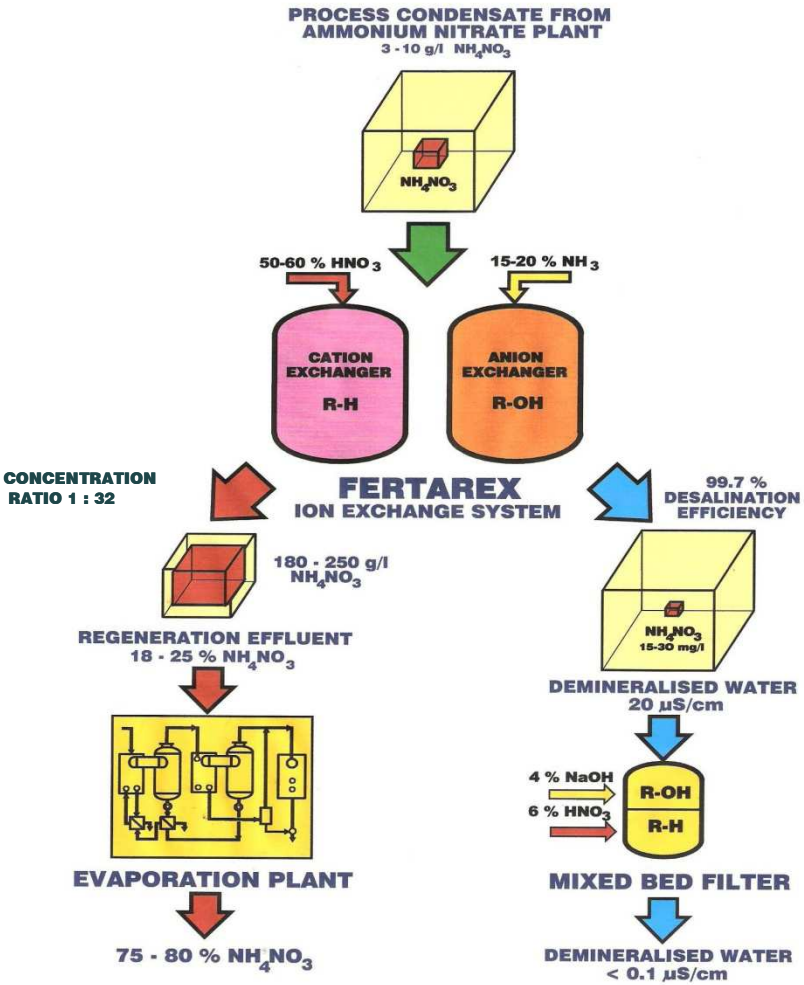
CONTAMINATED FERTILIZER WASTE WATER TREATMENT BY THE FERTAREX ION-EXCHANGE SYSTEM AND EVAPORATION



40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

- From the ammonium nitrate plants continuously concentrated and diluted process condensates together with other discontinuously contaminated effluents are discharged.

- All nitrogen products discharged within the contaminated waste effluents has to be recovered as 75 to 80 % ammonium nitrate solution which is recycled to the AN or CAN process plant.

- As result the final treatment process step for the fertiliser waste effluents treatment and recovery should be the Evaporation.

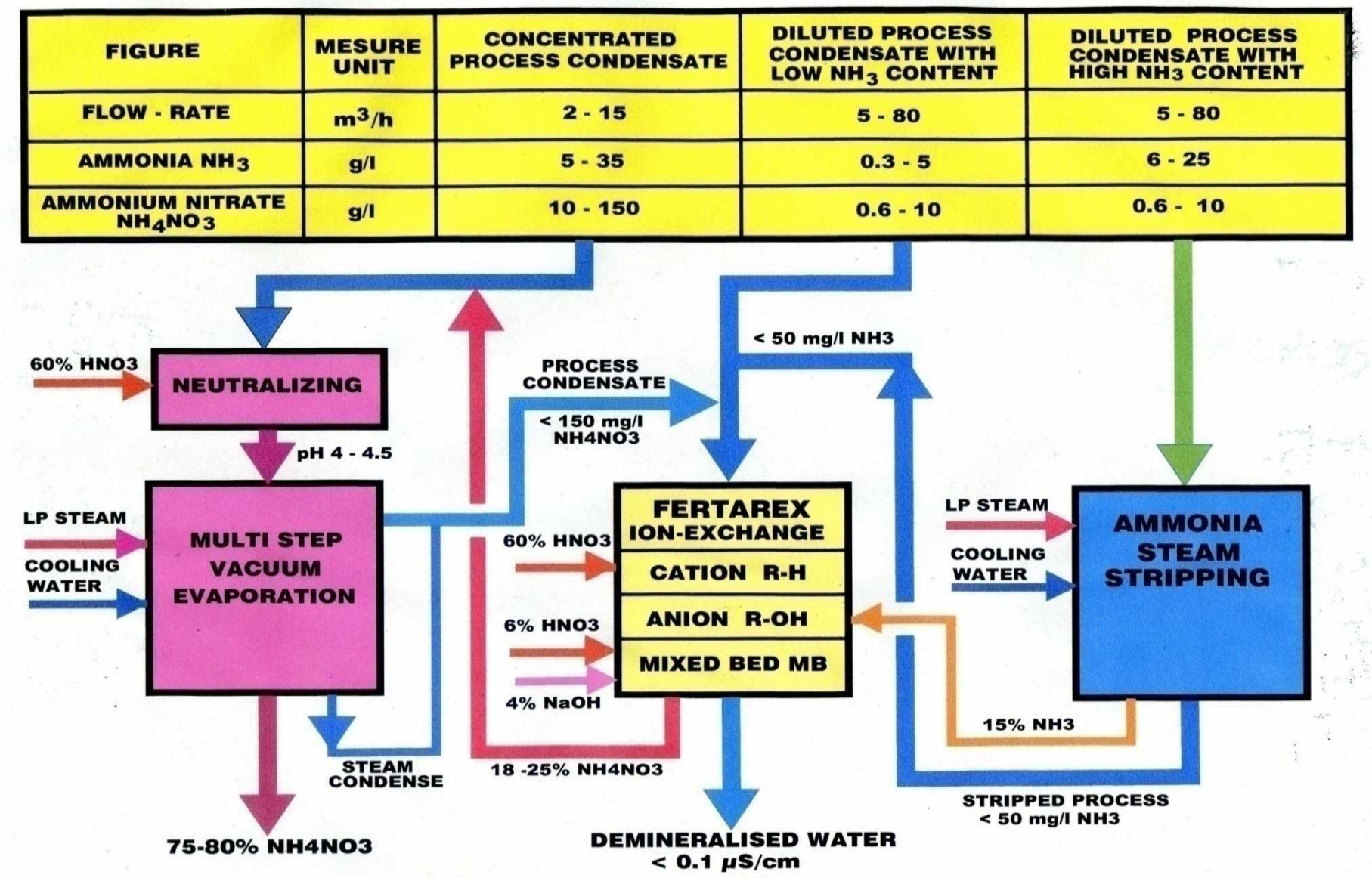
- To reduce the energy and utilities consumption of the evaporation process the amount of water to be evaporated has to be reduced at a minimum by using of a desalination process such as the Fertarex ion-exchange.

- When the ammonia content within the contaminated effluent is higher than approx. 6 g/l NH_3 an ammonia steam stripping process in front of the Fertarex is reccomended .

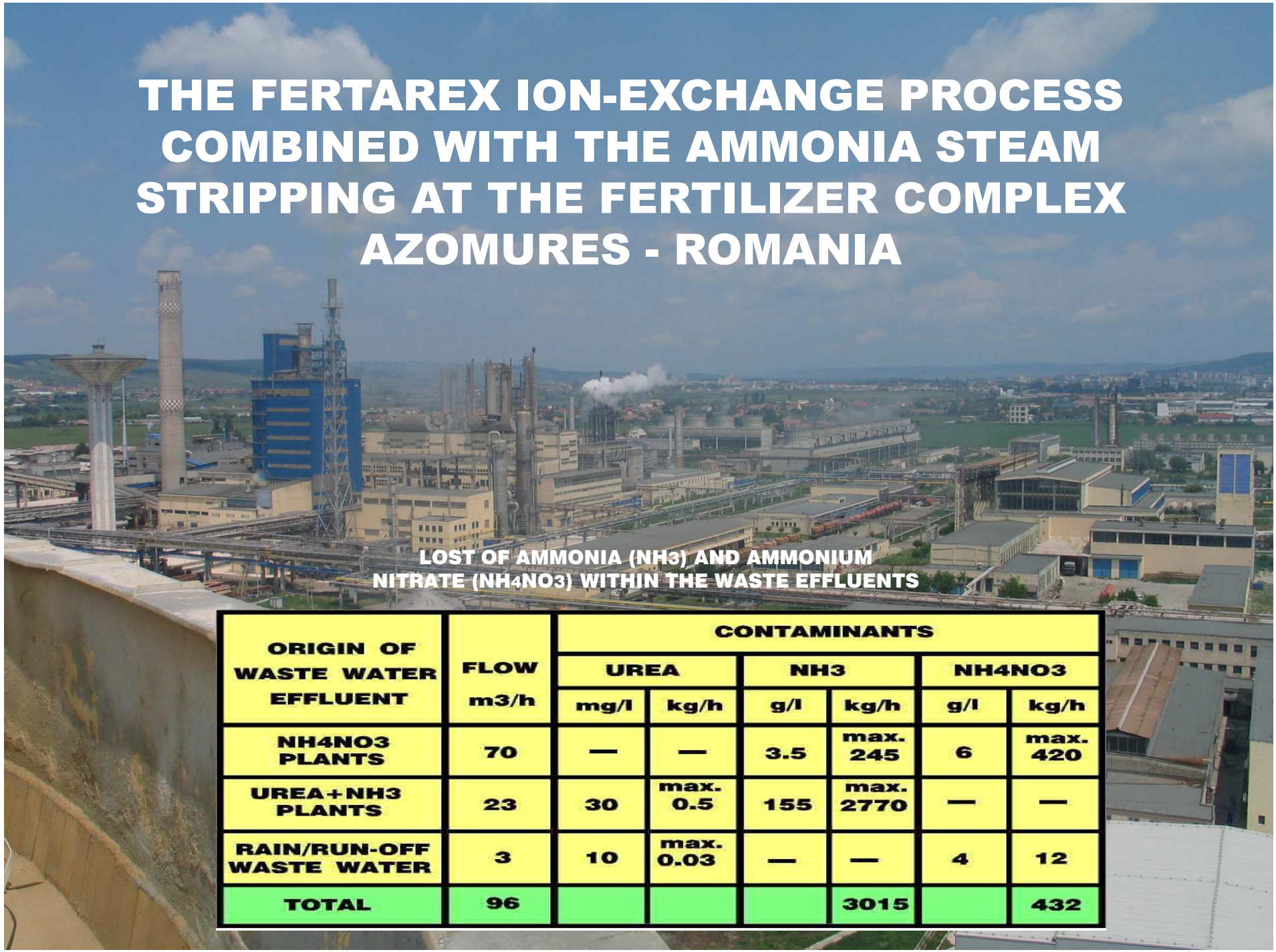
- For treatment and recovery the process and steam condensates from the evaporation plant are recycled to the Fertarex ion-exchange plant.

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

FIGURE	MESURE UNIT	CONCENTRATED PROCESS CONDENSATE	DILUTED PROCESS CONDENSATE WITH LOW NH_3 CONTENT	DILUTED PROCESS CONDENSATE WITH HIGH NH_3 CONTENT
FLOW - RATE	m^3/h	2 - 15	5 - 80	5 - 80
AMMONIA NH_3	g/l	5 - 35	0.3 - 5	6 - 25
AMMONIUM NITRATE NH_4NO_3	g/l	10 - 150	0.6 - 10	0.6 - 10



THE FERTAREX ION-EXCHANGE PROCESS COMBINED WITH THE AMMONIA STEAM STRIPPING AT THE FERTILIZER COMPLEX AZOMURES - ROMANIA



LOST OF AMMONIA (NH_3) AND AMMONIUM NITRATE (NH_4NO_3) WITHIN THE WASTE EFFLUENTS

ORIGIN OF WASTE WATER EFFLUENT	FLOW m3/h	CONTAMINANTS					
		UREA		NH3		NH4NO3	
		mg/l	kg/h	g/l	kg/h	g/l	kg/h
NH4NO3 PLANTS	70	—	—	3.5	max. 245	6	max. 420
UREA+NH3 PLANTS	23	30	max. 0.5	155	max. 2770	—	—
RAIN/RUN-OFF WASTE WATER	3	10	max. 0.03	—	—	4	12
TOTAL	96				3015		432

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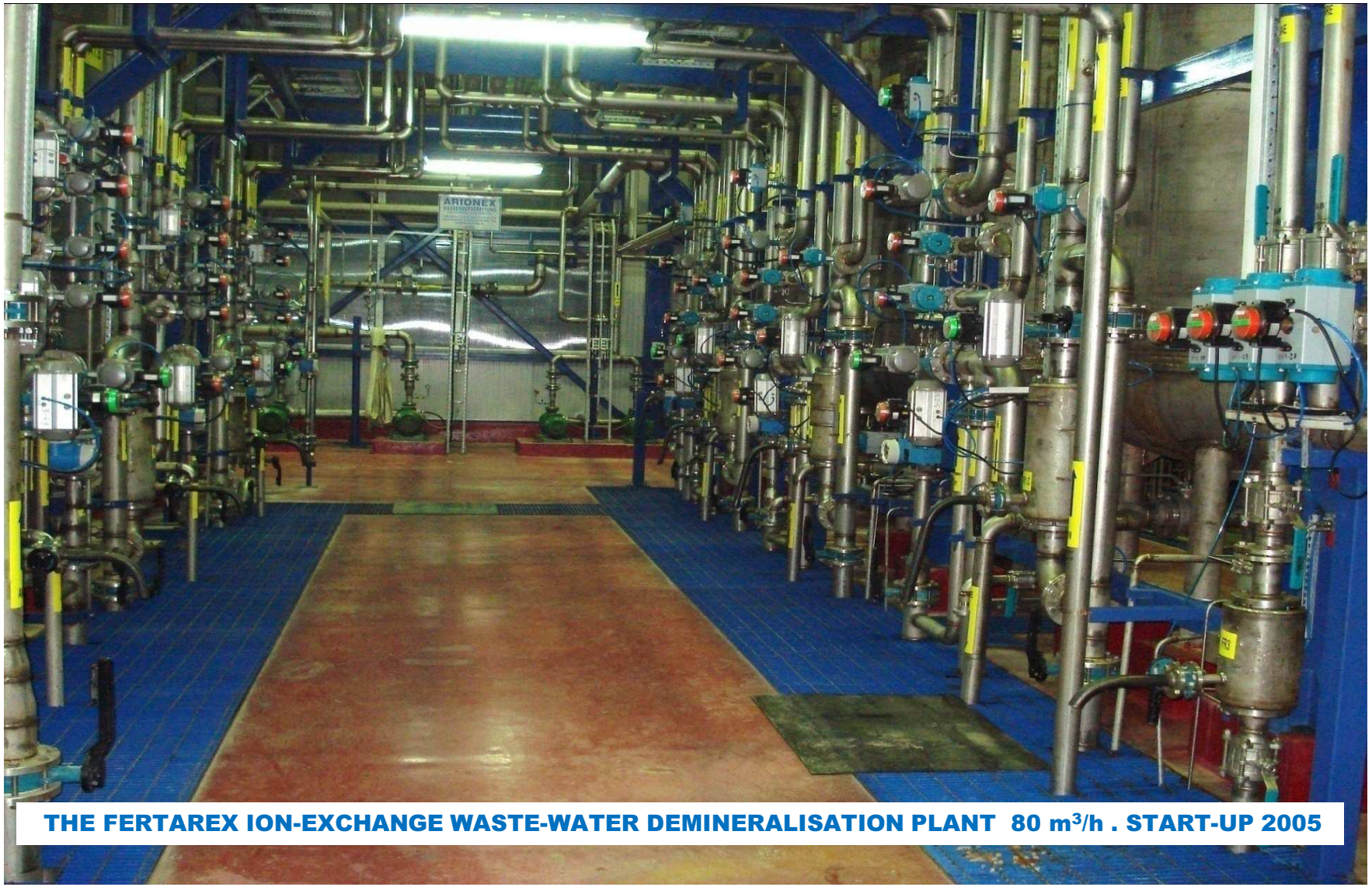
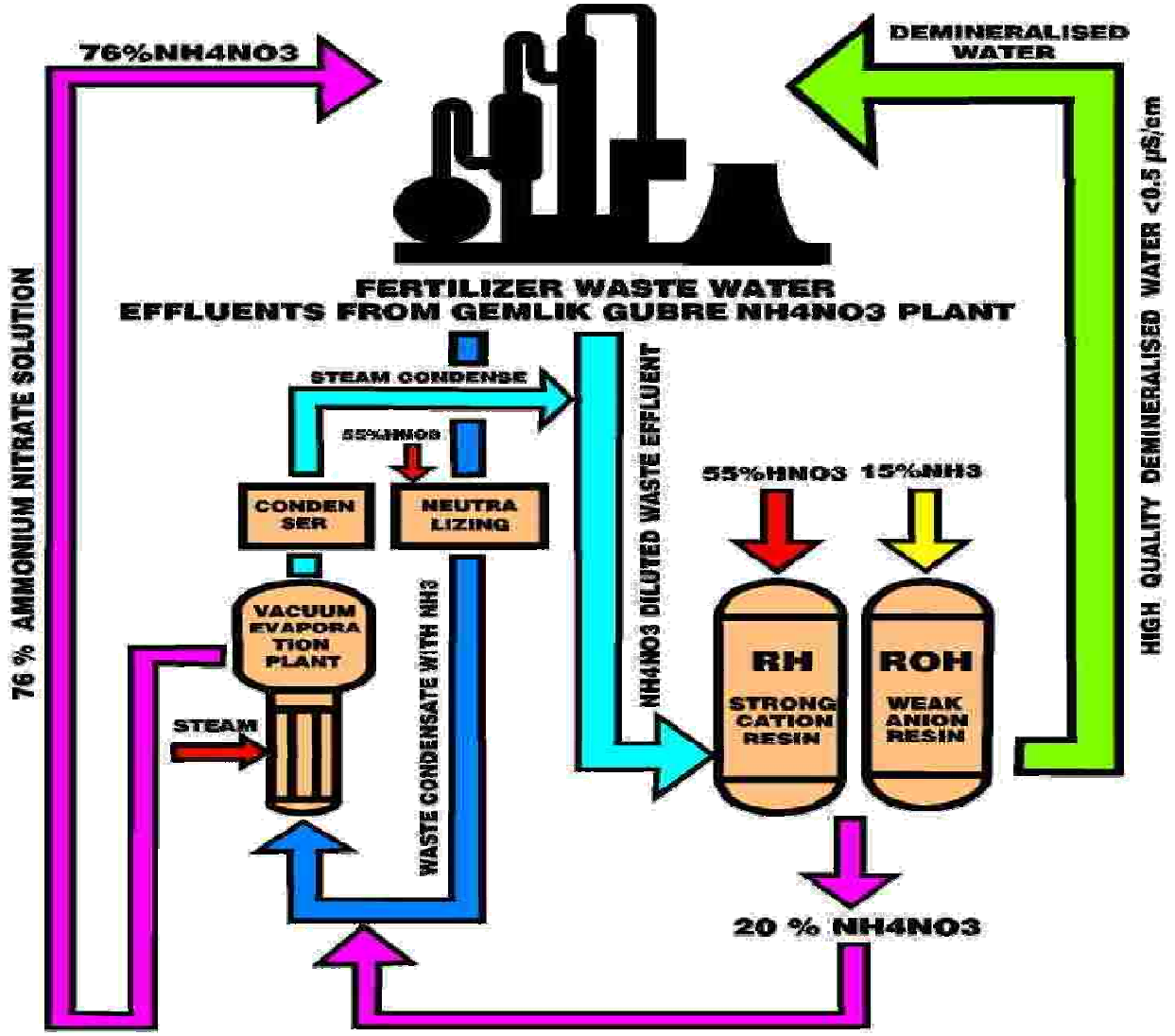


THE FERTAREX ION-EXCHANGE WASTE-WATER DEMINERALISATION PLANT 80 m^3/h . START-UP 2005

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

THE LOCAL CONTROL PANELS

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS



THE FERTAREX ION-EXCHANGE PROCESS COMBINED WITH EVAPORATION AT THE FERTILISER COMPLEX GEMLIK-GRUEBE - TURKEY

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THE FERTAREX ION-EXCHANGE DEMINERALISATION PLANT 60 m^3/h

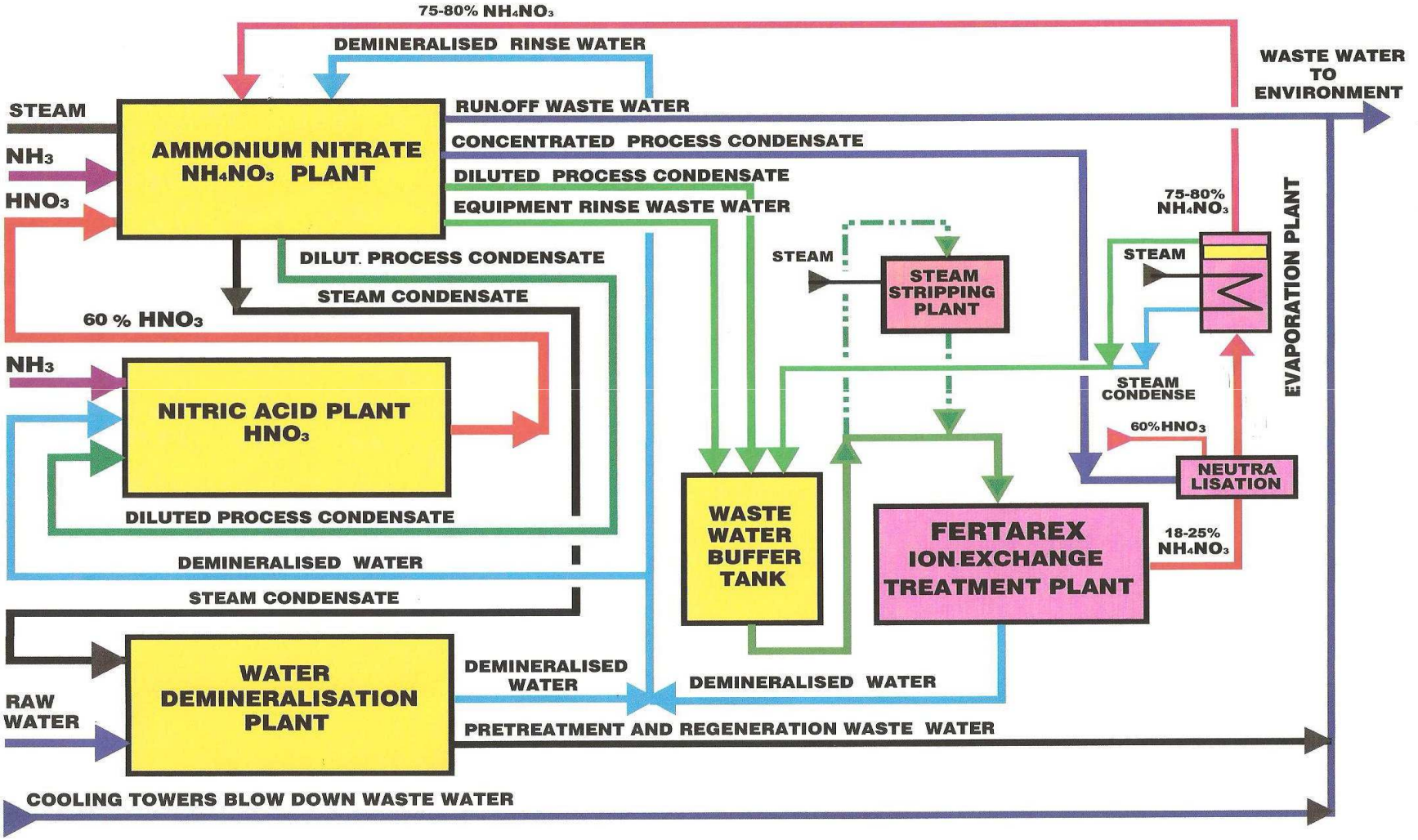
40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

THE NEUTRALISATION AND THE VACUUM EVAPORATION PLANTS 10 m^3/h

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

MONITORING OF THE DEMINERALISED WATER CONDUCTIVITY AFTER THE MIXED BED

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS



COMBINED STEAM STRIPPING , FERTAREX ION-EXCHANGE AND EVAPORATION PROCESSES FOR THE TREATMENT AND RECOVERY OF AMMONIUM NITRATE PLANT CONTAMINATED WASTE EFFLUENTS

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

List of Arion Short Cycle Ion-Exchange Plans with 55 - 60 % nitric acid regeneration for the treatment and recovery of ammonia and ammonium nitrate contaminated waste effluents

Pos.	Type of treatment and recovery plant	Flow m^3/h	Client	Country	Ion-Exchange System	Chemicals for regeneration	Contractor	Engineering Company	Start-up
01	Ion Exchange	50	AMONIL Slobozia	Romania	Down Flow Co-Current	10% HNO_3 10 % NH_3	MICH Bucharest-RO	IPRAN Bucharest	1975
02	Ion Exchange	100	DOLJCHIM Craiova	Romania	Moving Bed Short-Cycle with external regeneration	55% HNO_3 15% NH_3	MICH Bucharest-RO	IPROCHIM Bucharest	1976
03	Ion Exchange	80	C.Ch.ARAD Arad	Romania	Moving Bed Short-Cycle with external regeneration	55% HNO_3 15% NH_3	MICH Bucharest-RO	IPROCHIM Bucharest	1977
04	Ion Exchange + Evaporation	200	INA-Petrokemija Kutina	Croatia	Short-Cycle, Up-flow with external regeneration.	56% HNO_3 18% NH_3	Kellog Amsterdam-NL	Bran+Lübbe D-Hamburg	1982
05	Fertarex Ion Exchange + MVR Evaporation	30	Peti-Nitrogen Varpalota	Hungary	Short - Cycle, Up-flow with internal regeneration	60 % HNO_3 23 % NH_3	Christ AG Aesch-CH	Christ AG Switzerland	1987
06	Ion Exchange	30	Deepack Fertilizer Bombay	India	Short - Cycle, Up-flow with internal regeneration	30 % H^2SO_4 18 % NH_3	Humphrey Glasgow - UK	ARIONEX Switzerland	1991
07	NH_3 Steam Stripping + Fertarex Ion Exchange	90	AZOMURE S.A. Târgu Mures	Romania	Short - Cycle, Up-flow with internal regeneration + Mixed Bed.	54 % HNO_3 15 % NH_3	AZOMURES Târgu-Mures Romania	ARIONEX Switzerland	2005
08	Fertarex Ion Exchange +Vacuum Evaporation	80	Gemlik Gübre Sanayii	Turkey	Short - Cycle, Up-flow + Mixed Bed	55 % HNO_3 15 % NH3	Yildirim Holding Turkey	ARIONEX Switzerland	2010
09	Neutralising and Storage system for 10% - 75%	10	Gemlik Gübre Sanayii	Turkey	10 m^3/h on-line neutralising .	55 % HNO_3		ARIONEX Switzerland	2010

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

CONCLUSION

- During the last 40 years the ARION SPECIAL ION - EXCHANGE SYSTEMS have been successfully used for the recovery of ammonia, ammonium nitrate and water from the contaminated waste effluents, mainly as process condensates, discharged from the ammonium nitrate plants .

- The recovery of the discharged nitrogen products as 75-80 % ammonium nitrate and of the water as high quality demineralised water may represents saving of several million euros per year.

- Along with noticeable economic advantages for ammonium nitrate producers there is additional benefit of the ARIONEX - FERTAREX zero discharge system recovering nitrogen products and protecting the environment.

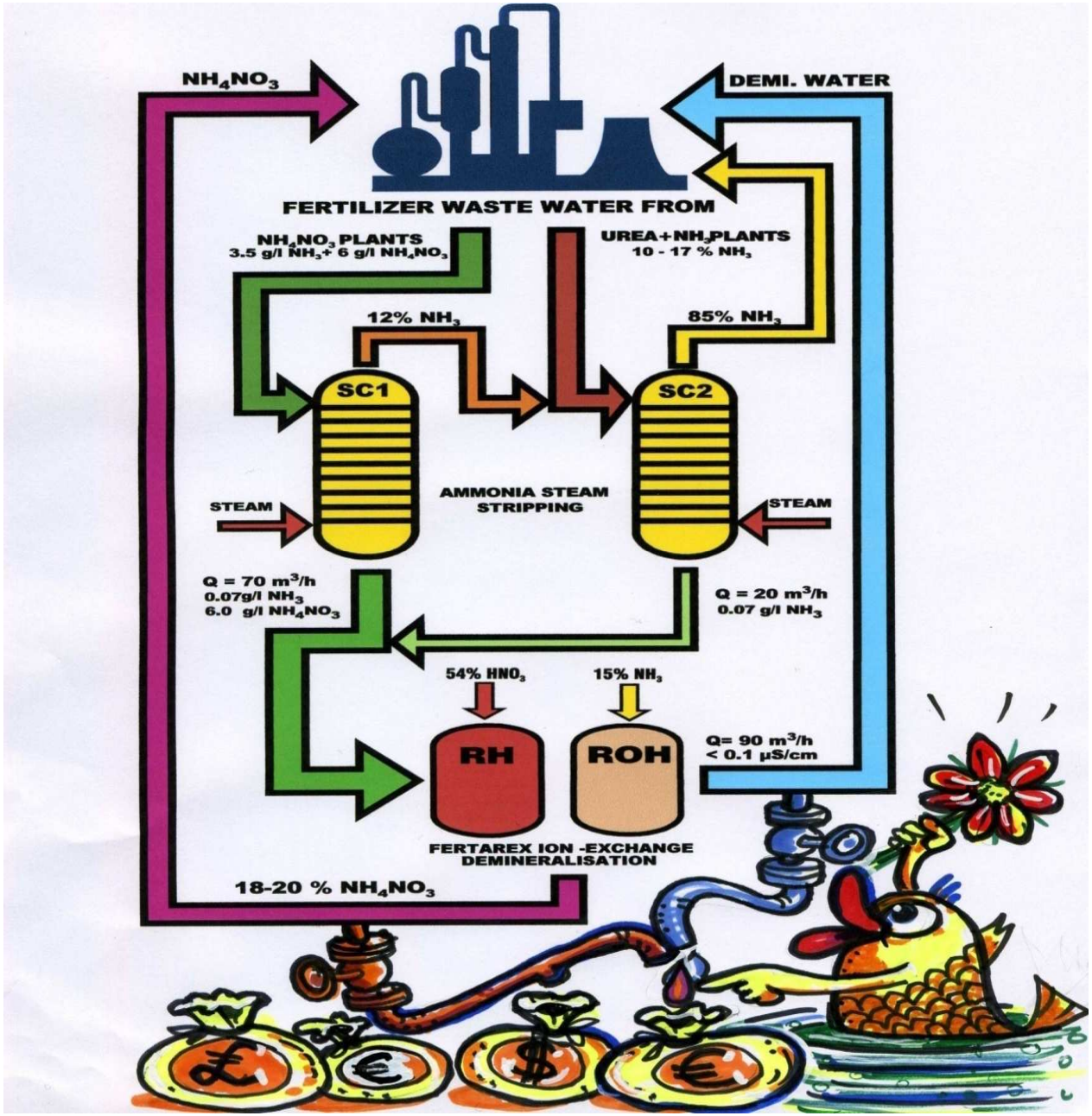
- Since 1976 six special Arion ion - Exchange industrial Treatment and Recovery Plants using regeneration of the special ion-exchange resins with concentrated nitric acid and ammonia have been installed in Europe and Asia. The first was commissioned in Romania in and the most recent in Turkey.

- In addition to the treatment of the waste effluents discharged from ammonium nitrate fertiliser plants, other projects involving the treatment by the Arion Fertarex Ion-Exchange System of contaminated effluents from ID ammonium nitrate plants for the mining and explosive industries are underway

- In spite of cation resin regeneration with concentrated nitric acid due to the special process safety measures over all these 40 years there have been no hazardous incidents of ammonium nitrate contaminated effluents treatment with the Arion-Special Ion-Exchange Systems

40 YEARS EXPERIENCE USING ION-EXCHANGE TO TREAT AND RECOVER CONTAMINATED PROCESS CONDENSATE FROM AMMONIUM NITRATE PLANTS

THE ARIONEX -SPECIAL ION-EXCHANGE SYSTEM FERTAREX IS A VERY EFFICIENT, ROBUST, RELIABLE AND SAFE TREATMENT AND RECOVERY PROCESS FOR THE DISCHARGED AMMONIA AND AMMONIUM NITRATE WITHIN THE CONTAMINATED WASTE EFFLUENTS FROM THE NITROGEN FERTILIZER PLANTS


NH_4NO_3
DEMI. WATER
FERTILIZER WASTE WATER FROM
NH_4NO_3 PLANTS
3.5 g/l NH_3+ 6 g/l NH_4NO_3
UREA+NH_3PLANTS
10 - 17 % NH_3
12% NH_3
85% NH_3
SC1
SC2
AMMONIA STEAM STRIPPING
STEAM
STEAM
Q = 70 m^3/h
0.07g/l NH_3
6.0 g/l NH_4NO_3
Q = 20 m^3/h
0.07 g/l NH_3
54% HNO_3
15% NH_3
RH
ROH
Q= 90 m^3/h
< 0.1 µS/cm
FERTAREX ION -EXCHANGE DEMINERALISATION
18-20 % NH_4NO_3

MANY THANKS FOR YOUR ATTENTION !!!!!