

## Ultra pure water production by a small local water treatment plant suitable for high quality urea solution AUS 32 defined by DIN 70070

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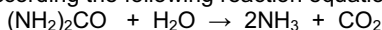
**Abstract:** A small local water treatment plant for high quality urea solution AUS 32 defined by DIN 70070 is created. The technological parameters of this plant are researched during the ultra pure water production used as a solvent for urea solution AUS 32. A very salty under-ground water was treated to get a deionized ultra pure water with specific electric conductivity  $\chi \leq 1 \mu\text{S/cm}$ . It was used as a solvent during the automotive grade urea solution (AUS 32) production. The product obtained was according to quality requirements of DIN 70070. The role and use of deionized ultra pure water addressed to quality assurance guidance document (QAGD) is discussed.

**Key words:** small water treatment plant, deionizer, urea solution

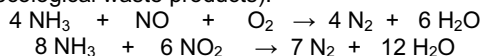
### INTRODUCTION

After evaluation different concepts to meet the Euro IV (10/2006) and Euro V (10/2009) standards for heavy-duty vehicles, the automotive industry has decided to use the Selective catalic reduction (SCR)-technology with AUS 32[1] as a reducing agent for most of these vehicles. The SCR-technology has demonstrated the capability to reduce nitrogen oxides emissions from heavy-duty tracks in test cycles and in on the real road operation. The combination of fuel-economy-optimised engines with low particulates and SCR is able to meet the Euro IV and Euro V regulation. The mentioned above technology with AUS 32 as a reducing agent has already been applied successfully to stationary applications, waste incinerators, power plants and to mobile Diesel engines in application such as ships and locomotives.

The basic chemical reaction [1] of the urea SCR-process is as follows: AUS 32 is injected into the hot exhaust gas and is hydrolysed above approx.180 °C forming ammonia (NH<sub>3</sub>) according the following reaction equation:



At the homogeneous extruded, base metal SCR catalyst the following reaction takes place to convert nitrogen oxides with NH<sub>3</sub> from the hydrolysis reaction to nitrogen and water (very positive ecological waste products).



### EXPERIMENTAL

A small local water treatment technology plant was created. (Fig.1). This paper describes the technological scheme, its components and recommended production parameters in producing a ultra pure deionized water to get 32.5% aqueous urea solution AUS 32 DIN 70070 grade, to preserve the quality of the solution from production via storage, handling and distribution to the end-user. A water as a solvent plays very important role. It takes a great deal to environmental protection aspects also. Its purity deals all participants in the distribution chain, producers of AUS 32, logistics partners, public filling and truck stations.

The water treatment plant is designed as a combination of different water technology methods (percolation; ion-exchange softening; reverse osmosis; ion-exchange deionization) The technological scheme is created on the base of raw water properties and our preliminary researches [2+4]. It is a suitable decision, because of relatively high salinity of this underground water.

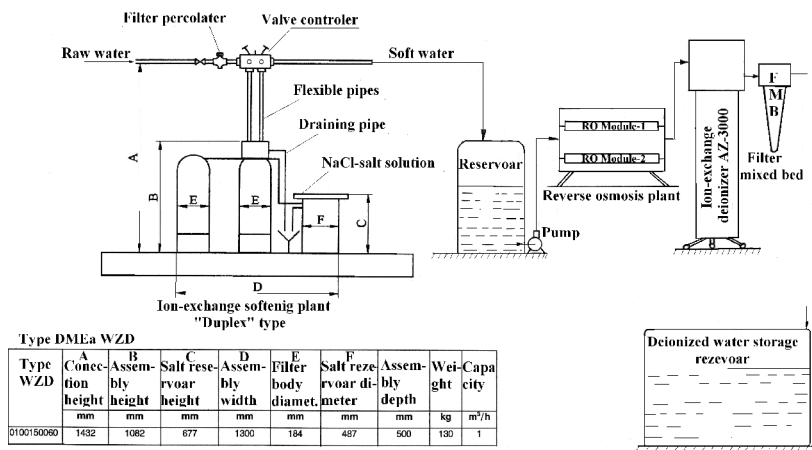


Fig 1. A technological scheme for ultra pure deionized water production used as a solvent for 32.5% aqueous urea solution AUS 32 DIN 70070 grade

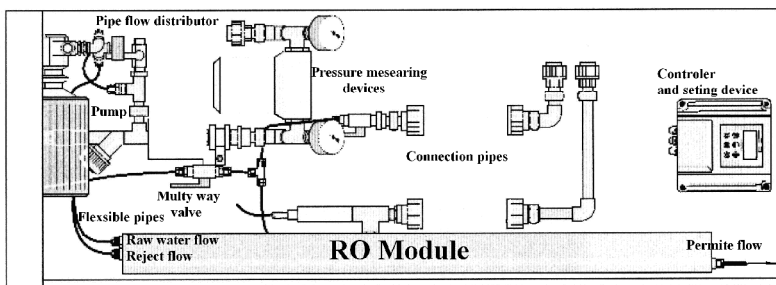
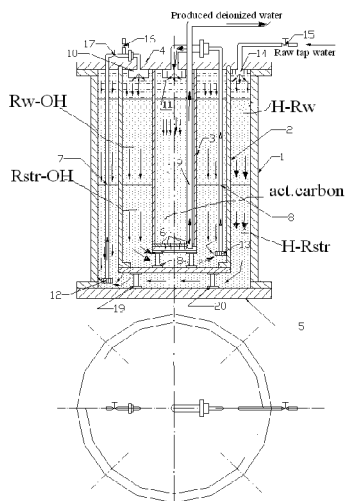


Fig. 2. Reverse osmosis module included into the technological scheme for ultra pure deionized water production

Because of very serious probability to obtain a dangerous scaling phenomenon on the reverse osmosis membrane surfaces, a first module on the raw water treated line is a softening module. It is an automatically controlled duplex type ion-exchange softening plant (Fig.1). After that, the water flow is treated by reverse osmosis module and then is finally conditioned by a filtration trough multi process mixed-functioning module (H-cation exchange / OH-anionexchange; adsorption processes). This module has a specific design [4] ( Fig. 3)



**Fig.3.** Design of multi process ion-exchange and sorption small water conditioning device: 1- outside body; 2, 3- inside bodies; 4- upper cover; 5- bottom; 6, 10+14- scatter nozzles; 15+17- valves; 18+20- bearings; H-R<sub>w</sub>- weak acid cation-exchanger (hydrogen ion form); H-R<sub>str</sub>- strong acid cation-exchanger hydrogen ion form); R<sub>w</sub>-OH- weak base anion-exchanger (hydroxyl ion form); R<sub>str</sub>-OH- strong base anion-exchanger (hydroxyl ion form) [4,5]

It's known that conventionally used small water treatment deionization systems include separate ion-exchange cartridges [6-8]. Each of them has to be regularly regenerated for continued use. Purchasing ready made replacement cartridges supplied by specialized dealers requires a subscription to their department. Our present task by these experimental technological research is to create and evaluate a multi cellular small water treatment ion-exchange and sorption device with a simple, cost-effective and user friendly design, to be used for production of water-solvent for AUS 32. The design enables a cheap regeneration of exhausted ion-exchange and sorption beds to be made locally by the user.

The device consists of three separated vertical cells which could be loaded by different ion-exchange and sorption materials (Fig.3). It produces secondary grade purified water in accordance with standards [1,9,10] from a potable source for small industrial applications. By this work was established that it is very suitable for a production of a ultra pure water-solvent to get 32.5% aqueous urea solution (AUS 32 DIN 70070 grade). The capacity was up to 200 dm<sup>3</sup>/h.

A major technological contribution of our design is a multi cell filtration body (Fig.3). During the water deionizing experiments, we loaded the three chambers as follows:

- layer of weak acid cation-exchange resin (Dowex MWC-2) H-R<sub>w</sub> (hydrogen form) and a layer of strong acid cation-exchange resin (Dowex MSC-1) H-R<sub>str</sub> (hydrogen form) in the chamber between walls 1 and 2;
- layer of weak base anion-exchange resin (Dowex MWA-1) R<sub>w</sub>-OH (hydroxyl form) and a layer of strong base anion-exchange resins (alter-native: Dowex MSA-1, Dowex MSA-2, Amberlite IRA-938) R<sub>str</sub>-OH (hydroxyl form) in the chamber between walls (2) and (3). This chamber was loaded with polyreticular [11], true porous [12] ion-exchange resins to be able to reduce humus natural organic impurities from the water treated [13];

- layer of active carbon sorption material in the central chamber.

A very important role has weak base anion-exchange resin (Dowex MWA-1). Because of the sorption-desorption ability of this resin to natural humus insoluble substances. It protects the strong base anion exchange resin against an irreversible poisoning of the ion-exchange functionality [14]. The device's features and price are minimized, but the technological behaviour of the device is very good. It produces secondary and third grade deionized water in accordance with BDS ISO 3696 [9]; ASTM D 1193-99 [10]; DIN 70070 [1] (Table 1) from a potable source for laboratory and other small industrial applications.

## RESULTS AND DISCUSSION

Technological project calculations were carried out and then a technological scheme for ultra pure deionized water production used as a solvent for 32.5% aqueous urea solution AUS 32 DIN 70070 grade was realized. It is shown on Fig.1 with all main technological components. The water treatment plant includes the created new type small ion-exchange water deionizer [4,5] (fig.3, 4).



**Фиг.4.** Визуален изглед на цилиндричен еднокорпусен двукамерен малогабаритен „Дейонизатор AZ 2000M“ за четиристепенна йонообменна обработка на водата при подобрена обтикаемост на йонитните пълнежи и потопени в слоя дренажни сонди отвеждащи потоците [4,5]

A pilot experiments were carried out in two alternative ways: with one or with two FMB modules. The R-H and R-PH ion-exchange modules and FMB modules were loaded with polyreticular [10] ion-exchange resins to reduce humus and natural organic impurities from the treated water [11]. The specific electrical conductivity of the water produced was measured by a flow type conductance measuring apparatus equipped with two sensors. When the capacity of the device is exhausted, its regeneration is recovered by treatment of ion-exchange chambers with reagent regeneration solutions as follows (see Fig.3): a solution of 5÷8% hydrochloric acid is passed through the exhausted ion-exchange R-H beds; a solution of 5÷6% sodium hydroxide is passed through the ion-exchange R-OH

beds; the FMB module is filled with a mixture of a strong acid cation-exchanger and a strong base anion-exchanger (type MB), preliminarily regenerated into H<sup>+</sup> and OH<sup>-</sup> ion forms, respectively ;

The reagent and washing flows pass through the filter under their gravitation ability (see Fig.3).The presence of an operator, control or monitoring are not necessary during this process

The results obtained are presented in Table 1.

**Table 1.** Results from experimental pilot study

№	Parameters	Purity			Purity									
		By BDS ISO 3696			With one FMB, bed vol. I					With two FMB, bed vol.				
		I	II	III	10	20	30	40	50	10	20	30	40	50
1	pH at 25°C	Not appl.	Not appl.	5+ 7,5	6,6	6,4	6,3	6,5	6,7	6,8	6,9	6,8	6,5	6,7
2	Spec.electric conductivity, μs/cm <sup>3</sup>	0,1	1	5	0,93	0,89	0,91	0,90	0,96	0,52	0,49	0,50	0,58	0,69
3	Permanganate oxygen demand,* mgO <sub>2</sub> / dm <sup>3</sup>	Not appl.	0,08	0,4	0,08	0,07	0,07	0,06	0,07	0,06	0,06	0,07	0,06	0,06
4	Absorption at 254 nm cuvette – 1 cm	0,001	0,01	Not appl	0,02	0,02	0,02	0,02	0,02	0,01	0,01	0,01	0,01	0,01
5	Dry residue at 110°C, mg/kg	Not appl.	1	2	1,2	1,1	1,1	1,1	1,3	0,8	0,7	0,8	0,9	0,9
6	Si O <sub>2</sub> μg/kg	10	20	Not appl	14	16	15	16	12	8	9	17	19	11

\*Permanganate demand of the water produced was measured by samples taken from the flow after the final adsorption module charged with active carbon

The results in Table 1 show that the properties of the water produced by our small water deionization device comply with the requirements for water with primary or secondary degree of purity (BDS ISO 3696,1993;ASTM 1193, 1999; DIN 70070)[8,9]. Therefore, it was concluded that the product can be used as a solvent of urea to produce AUS 32; for usual laboratory needs, as well as for more precise laboratory analyses both for laboratory and small industrial purposes.

## CONCLUSIONS

1. A small local water treatment plant for high quality urea solution AUS 32 defined by DIN 700070 is created. The technological parameters of this plant are researched during the ultra pure water production used as a solvent for urea solution AUS 32.

2. The device`s features and price are minimized, but the technological behaviour of the device is very good. It produces secondary and third grade deionized water in accordance with BDS ISO 3696;ASTM D 1193-99]; DIN 70070 from a potable source for laboratory and other small industrial applications.

3. The cost of the device is much cheaper than the market available deionizers, but the operating expenses are much lower and the device allows easy control of the processes, technological maintenance and repair, as well as much longer technological life.

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**Докладът е рецензиран.**