

# MEASUREMENT OF THE CONCENTRATION OF NH<sub>3</sub> AND CO<sub>2</sub> IN WET GAS IN THE CRUDE SODA PRODUCTION PROCESS BY THE SOLVAY METHOD

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## INTRODUCTION

The method of producing soda ash was created in the early 60s of the nineteenth century when two Belgian brothers Ernest and Alfred Solvay developed an improved way of synthetic soda production.

For the production of soda (sodium carbonate) limestone (calcium carbonate) and rock salt (sodium chloride) are used. In practice, it is necessary to use additives and auxiliaries (coke, ammonia as a catalyst).

The Solvay method is based on various processes such as calcination of the limestone, absorption of ammonia by brine and CO<sub>2</sub> saturation (carbonization) of the resulting solution, separation of sodium bicarbonate NaHCO<sub>3</sub> crystals and its calcination, and recovery of ammonia from the liquid after crystallization of bicarbonate using lime milk.

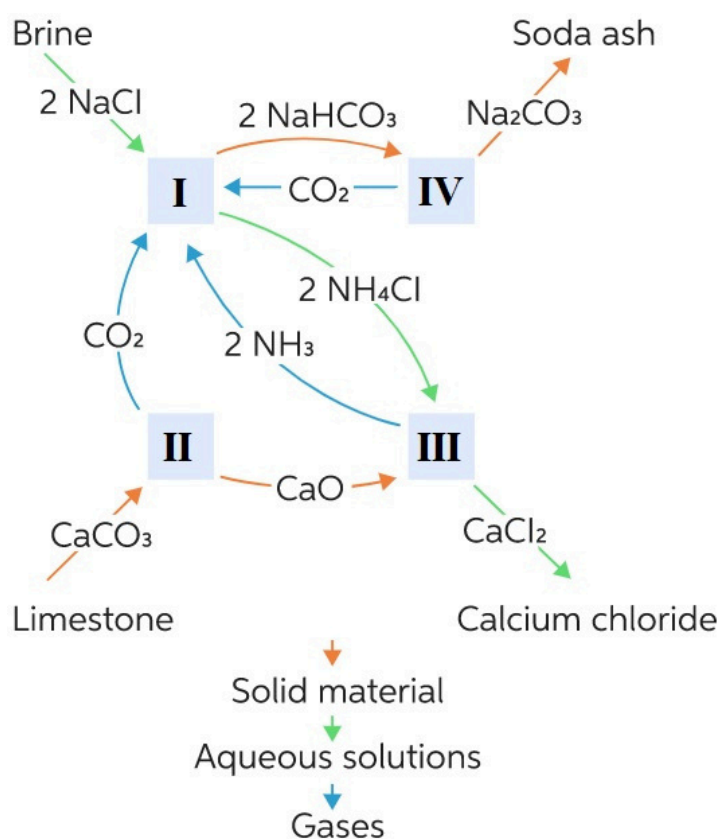


Fig. 1. Chemical reactions the Solvay method

# A REAL CHALLENGE

Optimization of the process should lead to an increased productivity and, at the same time, a reduced consumption of auxiliary materials, thus generating additional profit.

For this purpose, it is necessary to precisely and constantly measure the concentration of both CO<sub>2</sub> and NH<sub>3</sub> in the process gas.

Such measurement represents a pioneering engineering challenge due to the high and variable concentration of ammonia in the gas (>10%) with the simultaneous presence of water at the saturation point and presence of dissolved ammonium, carbonate and chloride ions, which crystallize at temperatures below 55°C.



Fig. 4. Crystallizing salts in the pipeline

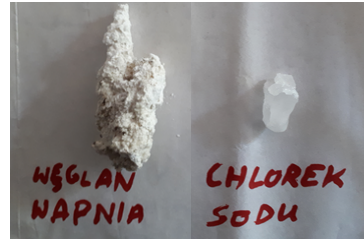


Fig. 5. Salts crystals

PRÓBA	ZASADOWOŚĆ BEZPOŚREDNIA	MLCaH	ZAWARTOŚĆ CO <sub>2</sub>	JEDNOSTKA
Próba 019/03.10.2018	30,12	31,23	4,51	mval/20 cm <sup>3</sup>
Próba 020/03.10.2018	12,51	14,15	nie wykryto	mval/20 cm <sup>3</sup>
Próba 021/03.10.2018	10,98	11,99	nie wykryto	mval/20 cm <sup>3</sup>
Próba 022/03.10.2018	30,55	31,64	nie wykryto	mval/20 cm <sup>3</sup>
Próba 023/03.10.2018	11,54	13,85	nie wykryto	mval/20 cm <sup>3</sup>
METODYKA	Zgodnie z instrukcją MB-09/Lab CRD- pkt 1.	Metoda wyliczeniowa zgodnie z MB-09/Lab CRD pkt 2	Zgodnie z instrukcją MB-09/Lab CRD- pkt 3	-

Fig. 2. Analysis of condensate taken from different samples in the conditioning system

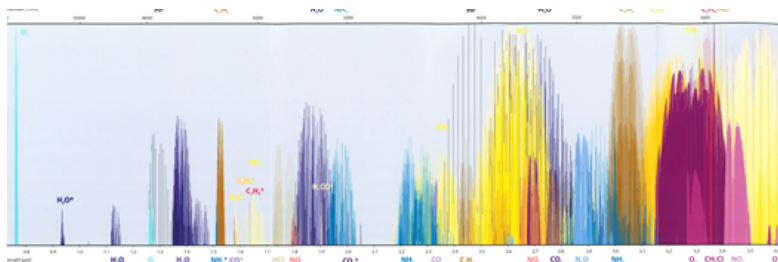


Fig. 3. Exemplary near-infrared absorption spectrum

The salt present in the sample, which crystals block the free flow of the gas from the process pipeline to the analyzer is a real problem. The salt must be removed from the sample in an appropriately designed and constructed system (where extraction, transport and conditioning of the sample take place). At the same time, the desalination process must not change the composition of the gas before it flows through the NH<sub>3</sub>/CO<sub>2</sub> analyser.

An additional challenge is the proper selection of the measurement method and the design of the analytical system. Commonly used absorption spectroscopy requires the so-called extraction of the sample (i.e. taking it from the process pipe and transporting it to the analyzer without disturbing the composition or loss of the measured components).

Another difficulty to overcome is the fact that NDIR analyzers (using near-infrared absorption) are used to measure ammonia in dry gases. In turn, the measurement of ammonia in wet (H<sub>2</sub>O saturated) gases is carried out, according to accepted practice, using laser spectroscopy. At this point, it is worth mentioning that the aforementioned method of measurement in wet gases has been tested only at ammonia concentrations from single ppm to 1%. Therefore, higher concentrations of ammonia in wet gases have not been

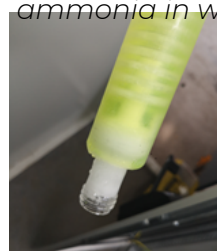


Fig. 6. NaCl in radiator exchange

implemented so far and require the use of innovative solutions and R&D work to select the IR spectrum wavelength, where the measurement of NH<sub>3</sub> will not be disturbed by other components present in the gas.

Experienced analytical process engineers, in collaboration with the R&D department, have selected an NH<sub>3</sub> measurement method based on hot extraction. A sample of the measured gas is kept continuously at temperatures above the crystallization and condensation point, from the sample probe to a specially designed heated measuring cell of a laser spectroscopic analyzer. The analyzer selected for this purpose has pre-programmed unique method, which allows precise measurement of both high and low concentrations of ammonia, ignoring other components and impurities present in the gas.

## THE SOLUTION – A HYBRID SYSTEM, “HOT” FOR NH<sub>3</sub> AND “COLD” FOR CO<sub>2</sub>



Fig. 7-10. Elements of the "cold" CO<sub>2</sub> analysis system

CO<sub>2</sub> measurement is carried out "cold", by rapid cooling of the hot gas sample to a temperature of 4°C and removing salts and all impurities potentially interfering with the measurement. A dedicated sample conditioning system has been designed and built to cool, desalinate and filtrate contaminants. In result a trouble-free and accurate measurement of carbon dioxide with the NDIR analyzer is ensured. The selectivity of the method was also experimentally tested and the compensation co-efficients were adjusted. Properly selected solvent, added continuously during the cooling of the sample, allows it to desalinate it without losing the components measured in the gas

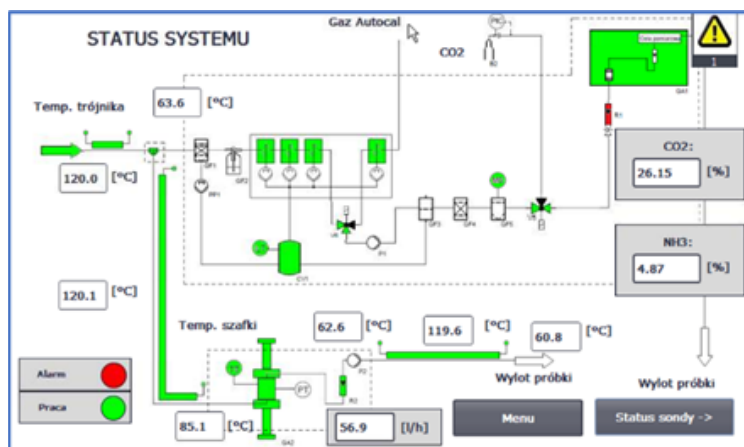


Fig. 11. Explanatory diagram of the analytical system



Fig. 12. Analytical system cabinet

# THE MOST IMPORTANT FEATURES OF THE "COLD AND HOT" HYBRID SYSTEM

- An individually configured analyser with reliable CO<sub>2</sub> measurement thanks to a specially designed sample conditioning system;
- Possibility of an automatic calibration of the zero point of the CO<sub>2</sub> analyzer;
- Proven desalination and filtration system;
- Precise temperature control of the gas sample and implemented self-diagnosis of the system;
- Dedicated and individually designed method of the TDL<sub>S</sub> laser analyzer for NH<sub>3</sub> measurement;
- 8 cm long measuring cell capable of NH<sub>3</sub> measurement up to 20%.

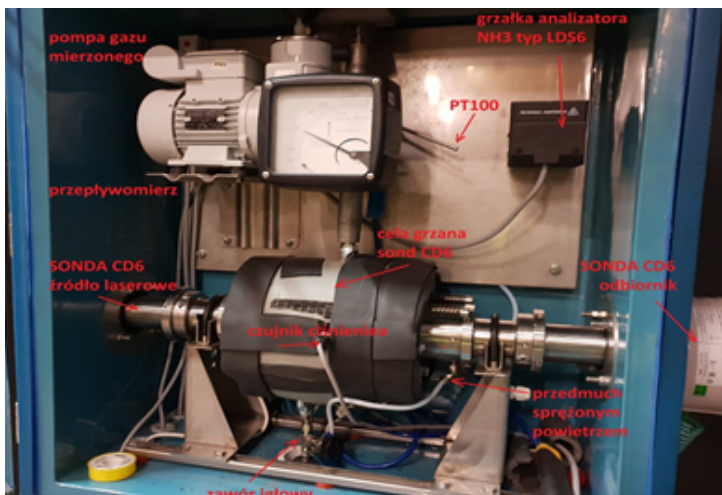


Fig. 13. Elements of the "hot" NH<sub>3</sub> analysis system



Fig. 14. Conditioned cabinet with a built-in laser spectroscopy - NH<sub>3</sub> analysis