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Nitrosamine formation in the CESAR1 solvent

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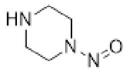
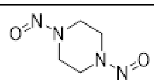
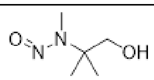
Abstract

The CESAR1 solvent is a non-proprietary blend of 2-amino-2-methylpropanol (AMP) and piperazine (PZ), that has been extensively studied as a good candidate for post-combustion CO_2 capture. It offers lower energy demand, more flexibility of operation and higher chemical stability than the earlier benchmark solvent, 30wt% ethanolamine (*aq.*) [1]. The Horizon Europe project AURORA aims to close the remaining knowledge gaps associated with post-combustion CO_2 capture using the CESAR1 solvent and a core topic in that work is deepening the understanding of CESAR1 solvent stability and the consequences of CESAR1 degradation.

Nitrosamines may form during post-combustion CO_2 capture (PCC) using amine solvent, particularly if the flue gas contains NO_2. Both AMP and PZ have been extensively studied in the PCC context and their respective nitrosamines and nitramines are well known, and frequently also studied during the CO_2 capture operation with CESAR1. The known and studied nitrosamines and nitramines of AMP and PZ are listed in Table 1. None of these compounds originate from any of the degradation compounds of AMP and PZ, but directly from the fresh solvent itself. A recent publication from the HEU AURORA project identified 48 degradation compounds that may form during oxidative and thermal CESAR1 degradation, of which 15 were identified and quantified for the first time [2]. It is very probable that some of these newly identified degradation compounds may also contribute to nitrosamine formation in the solvent.

Table 1: Nitrosamines known to form during PCC operation with the CESAR1 solvent.

Abbreviaton	Name	Structure	CAS number
MNPZ	Mononitrosopiperazine		5632-47-3
DNPZ	Dinitrosopiperazine		140-79-4
NMAMP	Nitroso-*N*-methyl-2-amino-2-methylpropanol		27646-81-7

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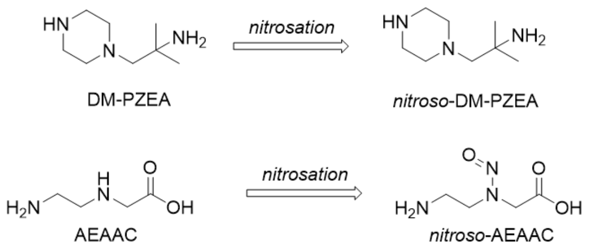
Secondary amines, such as PZ, may readily form stable nitrosamines in the presence of a nitrosation agent like nitrogen dioxide (NO_2) from the flue gas, or possibly nitrite (NO_2^-) formed during oxidation of the amines. Four different CESAR1 samples that had undergone extensive pilot testing in a series of campaigns and projects at TCM were analysed for their concentration of the three known CESAR1 specific nitrosamines NMAMP, MNPZ and DNPZ, as well as for their total nitrosamine (TONO) concentration. The measured concentrations shown in Table 2 indicate that up to 16% of the nitrosamines contained in degraded CESAR1 are not accounted for counting only these three known nitrosamines.

Table 2: Specific nitrosamine and TONO concentrations of four different CESAR1 pilot samples.

Sample	MNPZ	NMAMP	DNPZ	TONO	TONO unaccounted for
	[mmol/kg]				
A	6.7	0.4	0.001	8.2	14%
B	11.1	0.6	0.003	13.9	16%
C	27.3	1.6	0.02	29.1	1%
D	9.6	1.4	< LOQ	12.8	14%

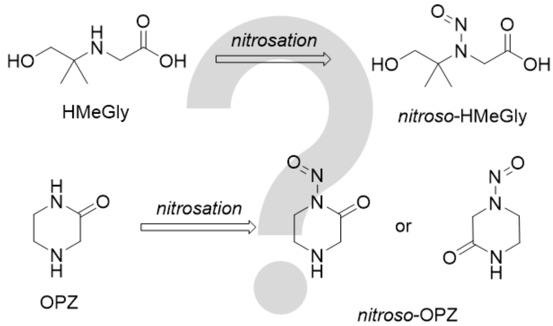
Among the ten most abundant degradation compounds in a CESAR1 solvent sample from Technology Centre Mongstad (TCM), five are secondary amines [3]. It is likely that some, if not all of these also form nitrosamines in the process. We have already confirmed the existence of two new nitrosamines formed from DM-PZEA and AEAAC, in a solvent sample from pilot-scale PCC operation. The nitrosamines and the educts are shown in Scheme 1. None of these have been quantified yet, but analytical methods will be set up for their quantification in solvent samples from HEU AURORA pilot campaigns at SINTEF's Tiller pilot plant, and TCM.

Scheme 1: Nitrosamines formed from CESAR1 degradation compounds, which have been identified in degraded CESAR1.



New analytical methods on LC-MSMS are being developed and will give new insights to the degradation compounds that may or may not form during PCC with CESAR1. We will start by looking for likely nitrosamine products, that are suggested based on the abundance of their corresponding secondary amine degradation compound. Among the more likely candidates to also form nitrosamines during PCC operation are HMeGly, a newly identified degradation compound of CESAR1, and the known PZ oxidation product OPZ, both shown in Scheme 2.

Scheme 2: Hypothetical nitrosamines that are likely to form during solvent degradation of CESAR1 in the PCC process.



DMOZD is an intermediate product of AMP carbamate polymerisation, and present in significant concentrations in used CESAR1. Being a secondary amine, DMOZD could in theory be able to form stable nitrosamines according to Scheme 3, but the steric hindrance in the molecule indicates that this reaction is less likely to take place. Analysis of the solvent samples from pilot scale may determine whether this product is formed or not.

Scheme 3: A more unlikely, but possible nitrosamine that could form during CESAR1 degradation.

At PCCC-8 we aim to present these new nitrosamine compounds, including quantification of nitrosamines found in solvent samples after the completion of the ongoing CESAR1 test campaign at Tiller in Trondheim, and possibly also new samples from the upcoming CESAR1 campaign at TCM, both in Norway. The results and their implications for the safety of operation and the environment will be discussed.

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