

REPORT

# Emissions and formation of degradation products in amine-based carbon capture plants

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# Introduction

Application of amine solvents in carbon capture plants will result in potential atmospheric emissions of the solvent-amine and its degradation products. From a health perspective, degradation products to consider are nitrosamines and nitramines due to their carcinogenic properties.

Solvent degradation in amine-based carbon capture plants can take place in the absorber/desorber via thermal degradation or oxidative degradation as a result of chemical reactions with the impurities present in the flue gas or from the impurities caused by corrosion of the carbon capture plant materials.

The chemical composition of the emissions depends on the flue gas, the plant operation conditions, and the applied solvent. Monoethanolamine (MEA) is the benchmark solvent applied in amine-based carbon capture plants. Other amines and mixtures of amines can be applied, depending on the requirements for the amine's thermal stability and capacity for CO<sub>2</sub> absorption.

Emissions from the absorber tower occur both as gas and aerosol particle emissions<sup>1</sup>, depending on the solvent amine's volatility and the occurrence of ultrafine particles in the flue gas entering the absorber.

## Compound groups formed in the solvent

### Ammonia NH<sub>3</sub>

Ammonia has been found to constitute the main component emitted from MEA-based carbon capture plants<sup>2,3</sup>. In one study, it was found to constitute 67% of the lost MEA in terms of moles of produced NH<sub>3</sub> relative to moles of lost MEA<sup>3</sup>. Ammonia is continuously formed as an oxidative degradation product of MEA. Hence, the O<sub>2</sub> concentration in the flue gas has been found to increase the ammonia emissions because of oxidative degradation<sup>2</sup>. The presence of metal ions in the solvent, such as iron, has been observed to increase ammonia emissions through the catalytic formation of ammonia<sup>2</sup>. Because of its high volatility, ammonia emissions primarily occur in the gas phase.

### MEA

MEA has also been shown to make up a significant contribution to the overall emissions<sup>1,3</sup>, and has been observed to show emission behaviour with strong variations over time, possibly because of particle/mist formation<sup>1</sup>.

The emissions are strongly influenced by the flue gas composition, the solvent type and the plant operation conditions, e.g. the temperature of the lean amine (the amine solution circulated from the desorber back to the absorber) and the extent of corrosion, Figure 1. Water and acid wash are usually applied after the absorber to reduce emissions from the absorber tower<sup>3,4</sup>. Furthermore, the emissions are influenced by the CO<sub>2</sub> concentration in the flue gas, which influences the solvent pH<sup>4</sup>.

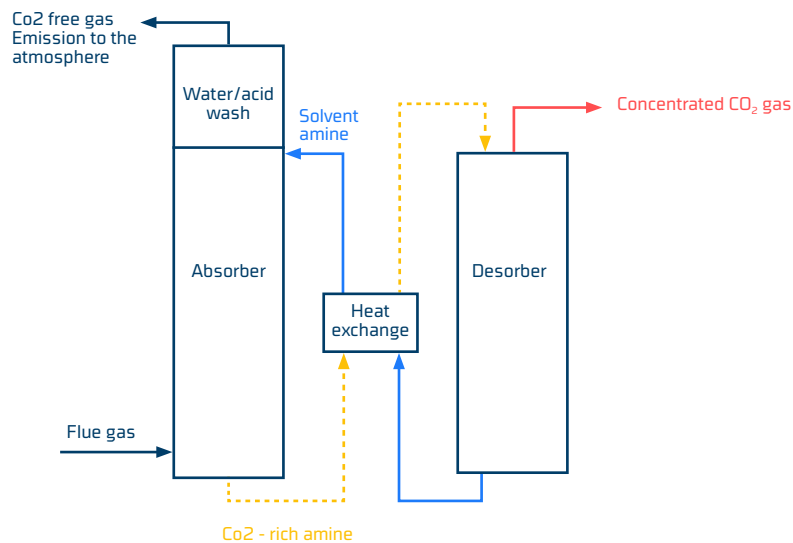


Figure 1. Simplified illustration of an amine-based carbon capture plant. The CO<sub>2</sub>-containing flue gas enters the absorber, where the amine solvent captures the CO<sub>2</sub>. The CO<sub>2</sub>-free gas leaves the absorber tower and is emitted into the atmosphere. The CO<sub>2</sub>-rich solvent circulates to the desorber, where it is heated to release a concentrated stream of CO<sub>2</sub>. The lean solvent amine recirculates to the absorber tower.

## Influence of the lean amine temperature on MEA emissions

Several studies have shown that the introduction of H<sub>2</sub>SO<sub>4</sub> or soot particles to the flue gas entering the absorber results in increased MEA emissions due to condensation of MEA onto the particles<sup>4,5,6,7</sup>. Khakharia et al.<sup>4</sup> demonstrated that the MEA emissions at the standard operating temperature of 40°C of the lean amine were dominated by particle-bound MEA, with H<sub>2</sub>SO<sub>4</sub> particle concentrations of 10<sup>7</sup>-10<sup>8</sup> particles/cm<sup>3</sup> and a CO<sub>2</sub> concentration of 13 vol% in the flue gas. A decrease in aerosol MEA emissions was observed at increased lean amine temperature. At further increased temperatures in the range of 70-80°C, the MEA emissions primarily consisted of volatile MEA<sup>4</sup>. This was explained by reduced condensation of amine onto particles since the saturation vapour pressure increases at increased lean amine temperatures. The particle MEA concentrations in these measurements decreased more than the gas-phase MEA emissions increased at increased lean amine temperature. Hence, increasing the lean amine temperature from 40°C may reduce aerosol emissions of amines and, as such, the overall MEA emissions.

Lombardo et al.<sup>5</sup> made similar tests of varying the lean amine temperature at CO<sub>2</sub> concentrations of 3.7, 8.0, and 12.7 vol% with particle concentrations between 5.4 • 10<sup>5</sup> - 9.5 • 10<sup>5</sup> particles/cm<sup>3</sup>. They demonstrated a reduction in MEA emissions at increased lean amine temperatures at CO<sub>2</sub> concentrations of 3.7 and 8.0 vol%. With a CO<sub>2</sub> concentration of 12.7 vol% in the flue gas, there was no clear trend in the reduction of MEA emission. However, when the lean amine temperature was decreased to 30°C, a clear increase in MEA was measured.

Mertens et al. (2012)<sup>6</sup>, did a similar test by reducing the lean amine temperature from 40 to 30°C in an absorber operated with a solvent mixture of 2-amino-2methyl-1-propanol (AMP) and piperazine (PZ). The reduction in lean amine temperature caused increased AMP and PZ emissions after the washing section. The particle and CO<sub>2</sub> concentration in the flue gas were not measured in this study. However, the observed increase in concentration was explained by a reduced temperature difference between the flue gas entering the water wash and the flue gas leaving the water wash, which did not ensure efficient condensation of water.

Contrary to these studies, Yi et al.<sup>8</sup> reported increasing emissions of the solvent amines with increasing lean amine temperature in a solvent mixture of AMP and MEA. The emissions were measured in the presence of NaCl particles. Yi et al. reported dominant emissions of gas-phase solvent amines, as AMP is significantly more volatile than MEA. The reported temperature profile in the absorber is different from the temperature profiles reported in studies with MEA only.

The results by Khakharia et al.<sup>4</sup> and Lombardo et al.<sup>5</sup> indicate that the MEA emissions strongly depend on the lean amine temperature, the flue gas aerosol concentration, the CO<sub>2</sub> concentrations in the flue gas and the pH of the lean solvent. It should be noted that the described trends only apply when particles are present in the flue gas. The studies highlight the importance of understanding the aerosol emissions from carbon capture plants and how the aerosol emissions are influenced by various operating parameters of the plant.

## Influence of water wash temperature on MEA and NH<sub>3</sub> emissions

Mertens et al.<sup>2</sup> tested the influence of water wash temperature on the ammonia and MEA emissions, i.e. the influence of temperature of the scrubber water for removal of pollutants to the atmosphere. They found that a reduction in water wash temperature from 50 to 35°C reduced the ammonia emissions temporarily and did not show any clear effect on MEA emissions. The temporary ammonia decrease was explained by the time it takes for a new equilibrium to be installed between the volatile ammonia and the low amount of dissolved ammonia in the washing water. After a short time, the ammonia emissions returned to the concentration before the temperature change<sup>2</sup>. Mertens et al. also showed that one step of water wash essentially had no effect on ammonia emissions, whereas it had a strong reducing effect (around 50%) on MEA emissions. Since it is known that water wash is not efficiently removing sub-micron aerosols, Mertens et al. suggested that the water wash primarily removed volatile MEA<sup>2</sup>.

# Formation of Nitrosamines and Nitramines

## Primary Amines

Nitrosamines and nitramines are formed from a reaction between the solvent amine and NO<sub>x</sub> originating from the flue gas. Increased NO<sub>x</sub> concentrations have been observed to cause higher nitrosamine and nitramine concentrations in the solvent for amines such as MEA<sup>3</sup>, morpholine<sup>9</sup>, as well as other amines<sup>10</sup>.

Primary amines like MEA do not form stable nitrosamines, as nitrosamines derived from primary amines decompose rapidly after formation. Hence nitrosamines formed in the MEA solvent are generated from secondary amines that are formed from the degradation of MEA, such as diethanolamine (DEA), N-(2-hydroxyethyl) glycine (HeGly) and hydroxyethyl-ethylenediamine (HEEDA)<sup>11</sup>. The resulting nitrosamines formed from these three degradation products are N-nitrosodiethanolamine (NDELA), N-nitroso(2-hydroxyethyl)glycine (NO-HeGly) and N-methyl, N-nitroso-methanamine (NDMA), respectively. These have all been detected in MEA lean solvent<sup>3</sup>.

## Secondary, Tertiary and Steric Hindered Amines

Tertiary amines like MDEA (methyldiethanolamine) and steric hindered amines like AMP (aminomethylpropanol) are also applied as solvents for carbon capture. Because of their tertiary structure or steric hindrance, they do not form the carbamate ion in the presence of CO<sub>2</sub>, like primary and secondary amines. The carbamate ion has a catalysing effect on the nitrosation reaction, i.e. the formation of nitrosamines. Instead, CO<sub>2</sub> is captured by bicarbonate formation, and tertiary amines are therefore expected to result in a lower yield of nitrosamines and nitramines. However, the reaction rate for CO<sub>2</sub> capture of tertiary amines is also generally slower, and the capacity to absorb CO<sub>2</sub> is therefore reduced for tertiary amines relative to primary and secondary amines, and the addition of a secondary amine like piperazine (PZ) is therefore typically necessary to enhance the reaction rate of CO<sub>2</sub> capture with tertiary amines. However, piperazine is a secondary amine with two amino groups, which forms stable nitrosamines. In a laboratory study of desorber conditions, it has been shown that the nitrosamine mononitrosopiperazine (MNPZ) is formed in the reaction between piperazine and NO<sub>x</sub>. The yield of dinitrosopiperazine (DNPZ), i.e., nitrosation at both amino groups, was negligible since [MNPZ] << [PZ]. The study showed that PZ accounted for almost all the nitrosation. MNPZ can be degraded in the desorber by increasing the regeneration temperature to, e.g. 150°C. However, tertiary amines themselves are less thermally stable<sup>11</sup>.

## Aldehydes

Aldehydes such as formaldehyde and acetaldehyde are formed in the oxidative degradation of MEA like ammonia, and have been measured as part of the volatile emissions after the absorber tower. Formaldehyde and acetaldehyde can react further to form carboxylic acids, which are non-volatile. Carboxylic acids can react further to other non-volatile compounds like heat-stable salts<sup>12</sup>.

## Alkyl amines

Short-chained alkylamines like methylamine (MA) and dimethylamine (DMA) are possible degradation products from MEA and other amines. Because of their volatile nature, they are likely to be emitted from the absorber tower if formed in the absorber/desorber process. They are primarily of concern because they can undergo nitrosation in the atmosphere upon emission<sup>13</sup>.

## Amides

Amides like formamide and acetamide can be formed in the oxidative degradation of MEA and other solvent amines. They are possibly formed by a reaction between formaldehyde and the solvent amine in the presence of oxygen<sup>14</sup>. Various other larger amides have been identified as solvent degradation products from MEA<sup>15</sup>.

# Flue gas composition and its impact on the emissions

## Influence of aerosols in the flue gas

Emissions from the absorber tower occur both in the form of gases and aerosols. Apart from the particles that may already be present in the stack gas introduced into the absorber, aerosols can be formed in the absorber tower by two different mechanisms: 1) homogeneous nucleation, which is based on gas-condensation processes and requires supersaturation of the gas 2) heterogeneous

nucleation where vapours condensate on pre-existing condensation nuclei, which can occur when the gas species reach the saturation vapour pressure. Heterogeneous nucleation is most likely the dominant aerosol formation mechanism in the absorber tower since the incoming flue gas often has a high number concentration of submicron aerosols (condensation nuclei)<sup>1,4</sup>.

Sulphuric acid ( $H_2SO_4$ ) aerosols have been observed by Mertens et al.<sup>1</sup> to be present in the flue gas as ultrafine particles that will grow through the absorber column taking up water and solvent amine. Generally, ultrafine aerosol particles in the flue gas, such as salts and soot particles, act as condensation nuclei and lead to enhanced aerosol emissions from the absorber tower. Hence, the reduction of fine and ultrafine particles in the flue gas that enters the absorber tower can reduce the final emissions from the absorber tower of aerosol particles, which may contain amines and their degradation products. Mertens et al. conducted particle measurements after the absorber tower (no water wash) and found that the majority of the particles by number were below  $1\mu m$ . However, the highest water and amine content was found to be present in particle sizes from  $0.5-2\mu m$ . Hence, the reduction of these particle sizes will result in the highest decrease in aerosol-borne amine emissions, while the reduction of ultrafine particles after the absorber tower will not have a strong effect on amine emission reductions<sup>1</sup>.

## Metals

For MEA, it has been shown that cobber ions in the solvent catalyse the formation of nitrosamines. This effect increases with increasing oxygen concentration. The effect is expected for primary amines with hydroxyl or oxygenated functional groups like MEA but not for secondary amines, tertiary amines, primary amines with steric hindrance and amines without an oxygen-containing functional group<sup>16</sup>.  $Fe^{2+}$  and  $Fe^{3+}$  can also catalyse the oxidation of MEA<sup>16,17</sup>. In general, ions of stainless-steel metals like Fe, Ni and Cr catalyse the oxidation of piperazine and MEA with a greater effect on MEA<sup>18</sup>.

## $NO_x$ , $SO_2$ and $SO_3$

Increased  $NO_x$  concentrations in the flue gas have been observed to result in higher nitrosamine yields for several amines like MEA<sup>3</sup> and morpholine<sup>9</sup>, among others<sup>10</sup>.

$SO_2$  is likely to have an inhibiting effect on the formation of nitrosamines. This has been observed for the amine morpholine<sup>19</sup>.

$SO_3$  can act as a precursor for aerosol particle formation in the absorber<sup>1</sup>. Higher aerosol particle emissions are expected to result in higher emissions of particle-bound amines and degradation products<sup>7</sup>.

# Degradation products measured in various solvents

**Piperazine (PZ)** As mentioned, MNPZ has been measured in the solvent in a mixture of PZ with MDEA, with a negligible yield of DNPZ<sup>11</sup>. The nitrosamine formation for the secondary amine PZ is at least one order of magnitude higher relative to the primary amine MEA and the tertiary amine MDEA<sup>20</sup>. Ethylenediamine, formyl amides and formic acid have all been detected as degradation products from PZ<sup>18</sup>.



**Methyldiethanolamine (MDEA)** The nitrosamine N-nitrosodiethanolamine (NDELA) is a possible degradation product formed from MDEA. Furthermore, the amino acid bicine has been determined as a degradation product from MDEA, and diethanolamine (DEA) has been found to make up a large fraction of the solvent degradation products from MDEA<sup>21,22</sup>.

Diethanol formamide, ethanol methanol formamide as well as dimethanol formamide have been suggested as the atmospheric degradation products from MDEA<sup>23</sup>.

Solvent mixtures of tertiary amines or sterically hindered amines with PZ are expected to give the highest nitrosamine yield<sup>11</sup>.

**Monoisopropanolamine (MIPA)** MIPA is a primary amine which forms unstable nitrosamines. The nitrosamine formation is therefore likely to stem from secondary amines formed in the degradation of MIPA, as mentioned for MEA<sup>3,24</sup>.

From the thermal degradation of MIPA, 5-methyl-2-oxazolanone<sup>25,26</sup> and 1-(2-Hydroxypropyl)-5-methyl-2-imidazolidinone<sup>25</sup> are expected products.

**2-amino-2methyl-1-propanol (AMP)** Acetone, 2,4-lutidine and 4,4-dimethyl-2-oxazolidinone have been observed as the main products formed from oxidative degradation<sup>27</sup>.

All compounds mentioned in this article are listed in Table 1 with their emission limit (if available) and classification.

## Conclusions

This article describes the emissions from amine-based carbon capture plants applying various solvents. Besides the type of amine applied, other factors like the composition of the flue gas, e.g. the particle concentration and the CO<sub>2</sub> loading, and the plant operation parameters have a large influence on the emissions. Furthermore, it is important to understand how these parameters influence the gas- and particle emissions from carbon capture plants.



**Table 1: List of solvents and possible degradation products, and their respective emission limits and classifications.**

Name	Cas	Emission Limit DK (mg/Nm <sup>3</sup> )	Air quality (a) Emission Limit (e)	Harmonised class. ECHA	IARC class
<b>Amines and N-containing compounds</b>					
MEA (monoethanolamine)	141-43-5	5	10 µg/m <sup>3</sup> (a) b-værdi 10 µg/m <sup>3</sup> (a) (NIPH)	Acute Tox. 4, H302, H312, H332 Skin Corr. 1B, H314	
DEA (diethanolamine)	111-42-2	5		Acute Tox. 4, H302 Skin Irrit. 2, H315 Eye Dam. 1, H318 STOT RE 2, H373 Repr. 2, H361*	2B
TEA (triethanolamine)	102-71-6	5		Acute Tox. 4, H302* Skin Irrit. 2, H315* Eye Dam. 1, H318* STOT RE 2, H373* Repr. 2, H361*	3
MDEA	105-59-9			Eye irrit. 2, H319	
MIPA	78-96-6			Skin Corr. 1B, H314	
AMP	124-68-5			Skin Irrit. 2, H315 Eye irrit. 2, H319 Aquatic Chronic 3, H412	
Piperazine	110-85-0		5 µg/m <sup>3</sup> (a) (NIPH)	Skin Corr. 1B, H314* Skin Sens. 1, H317* Resp. Sens. 1, H334* Repr. 2, H361fd*	
1-methylpiperazine	109-01-3			Acute Tox. 4, H302, H312, H332* Skin Corr. 1B, H314* Skin Sens. 1, H317* Eye Dam. 1, H318* Acute Tox. 2, H330* Acute Tox. 3, H331*	
1,4-dimethylpiperazine	106-58-1			Acute Tox. 4, H302* Skin Corr. 1B, H314* Acute Tox. 3, H311* Aquatic Chronic 2, H412*	
2-dimethylaminoethanol	108-01-0			Acute Tox. 4, H302, H312, H332 Skin Corr. 1B, H314	
2-methylaminoethanol	109-83-1			Acute Tox. 4, H302, H312 Skin Corr. 1B, H314	
Ethylenediamine	107-15-3			Acute Tox. 4, H302, H312 Skin Corr. 1B, H314 Skin Sens. 1, H317 Resp. Sens. 1, H334	
Bicine	150-25-4			Skin Irrit. 2, H315* Eye Irrit. 2, H319* STOT SE 3, H335*	
Ammonia	7664-41-7	500		Flam. Gas 2, H221 Skin Corr. 1B, H314 Acute Tox. 3, H331 Aquatic Acute 1, H400	

Name	Cas	Emission Limit DK (mg/Nm <sup>3</sup> )	Air quality (a) Emission Limit (e)	Harmonised class. ECHA	IARC class
<b>Amides</b>					
Formamide	75-12-7		10 µg/m <sup>3</sup> (a) b-værdi	Repr. 1B, H360D	
Acetamide	60-35-5		20 mg/m <sup>3</sup> (e) (le)	Carc. 2, H351	2B
Diethanolformamid	25209-66-9			Skin Irrit. 2, H315* Eye irrit. 2, H319* STOT SE 3, H335*	
2-piperazinone	629-162-6			Skin Irrit. 2, H315* Skin Sens. 1, H317* Eye irrit. 2, H319* STOT SE 3, H335*	
5-methyl-2-oxazolanone	1072-70-4			Eye Dam. 1, H318*	
1-(2-Hydroxypropyl)-5-methyl-2-imidazolidinone	6497-75-2			Not listed in the ECHA C&L database	
<b>Aldehydes, ketones, acids</b>					
Formaldehyde	50-00-0	2.5	20 mg/m <sup>3</sup> (e) (le)	Acute Tox. 3, H301, H311, H331 Skin Corr. 1B, H314 Skin Sens. 1, H317 Muta. 2, H341 Carc. 1B, H350	1
Acetaldehyde	75-07-0	2.5	20 mg/m <sup>3</sup> (e) (le)	Flam. Liq. 1, H224 Eye Irrit. 2, H319 STOT SE 3, H335 Muta. 2, H341 Carc. 1B, H350	2B
Acetone	67-64-1	300		Flam. Liq. 2, H225 Eye Irrit. 2, H319 STOT SE 3, H336	
Formic acid	64-18-6			Skin Corr. 1A, H314	
<b>Nitrosamines</b>					
NDELA	1116-54-7		0,3 ng/m <sup>3</sup> (a) (NIPH)	Carc. 1B, H350	2B
NO-HeGly	80556-89-4			Not listed in the ECHA C&L database	
NDMA	62-75-9		0,1 µg/m <sup>3</sup> (a) b-værdi	Acute Tox. 3, H301 Acute Tox. 2, H330 Carc. 1B, H350 STOT RE 1, H372 Aquatic Chronic 2, H411	2A
MNPZ	5632-47-3			Skin Corr. 1B, H314* Skin Sens. 1, H317* Resp. Sens. 1, H334* Carc. 2, H351* Repr. 2, H361*	
DNPZ	140-79-4			Acute Tox. 3, H301* Skin Irrit. 2, H315* Eye Irrit. 2A, H319* STOT SE 3, H335* Carc. 1B, H350*	
N-Nitrosomethylethanolamine	26921-68-6			Muta. 2, H341* Carc. 1B, H350*	

Name	Cas	Emission Limit DK (mg/Nm <sup>3</sup> )	Air quality (a) Emission Limit (e)	Harmonised class. ECHA	IARC class
<b>Alkylamines</b>					
Methylamine	74-89-5	100	20 mg/m <sup>3</sup> (e) (le)	Flam. Gas 1, H220 Skin Irrit. 2, H315 Eye Dam. 1, H318 Acute Tox. 4, H332 STOT SE 3, H335	
Dimethylamine	124-40-3	100		Flam. Gas 1, H220 Skin Irrit. 2, H315 Eye Dam. 1, H318 Acute Tox. 4, H332 STOT SE 3, H335	
Ethylamine	75-04-7	5	20 mg/m <sup>3</sup> (e) (le)	Flam. Gas 1, H220 Eye Irrit. 2, H319 STOT SE 3, H335	
Diethylamine	109-89-7	100		Flam. Liq. 2, H225 Acute Tox. 4, H302, H312, H332 Skin Corr. 1A, H314	
<b>Solvent-specific degradation products (MEA)</b>					
BHEOX	1871-89-2			Skin Sens. 1, H317* Skin Irrit. 2, H315* Eye Irrit. 2, H319*	
HEA	142-26-7			Skin Irrit. 2, H315* Eye Dam. 1, H318* STOT SE 3, H335*	
HEF	693-06-1			Skin Irrit. 2, H315* Eye Irrit. 2, H319* STOT SE 3, H335*	
HeGly	5835-28-9			Acute Tox. 4, H302* Skin Irrit. 2, H315* Eye Irrit. 2A, H319* STOT SE 3, H335*	
HEI	1615-14-1			Acute Tox. 4, H302, H312, H332* Skin Irrit. 2, H315* Eye Irrit. 2A, H319* STOT SE 3, H335*	
HEIA	3699-54-5			Skin Irrit. 2, H315* Eye Irrit. 2, H319* STOT SE 3, H335*	
HEPO	23936-04-1			Not listed in the ECHA C&L database	
OZD	497-25-6			Not listed in the ECHA C&L database	
MEA-Urea	15438-70-7			Not classified*	

### IARC classification

Group 1: Carcinogenic to humans

Group 2B: Possibly carcinogenic to humans

Group 3: Not classifiable as to its carcinogenicity to humans

### ECHA classification

\* Indicates that the classification is not harmonised but notified by several or some of the notifiers according to ECHA C&L database

H220 = Extremely flammable gas.

H221 = Flammable gas

H224 = Extremely flammable liquid and vapour.

H225 = Highly flammable liquid and vapour.

H301 = Toxic if swallowed.

H302 = Harmful if swallowed

H311 = Toxic in contact with skin.

H312 = Harmful in contact with skin

H314 = Causes severe skin burn and eye damage

H315 = Causes skin irritation

H317 = May cause an allergic reaction

H318 = Causes serious eye damage

H319 = Causes serious eye irritation.

H331 = Toxic if inhaled

H332 = Harmful if inhaled

H334 = May cause allergy or asthma symptoms or breathing difficulties if inhaled

H335 = May cause respiratory irritation.

H336 = May cause drowsiness or dizziness.

H341 = Suspected of causing genetic defects

H350 = May cause cancer

H351 = Suspected of causing cancer

H360D = May damage the unborn child

H361 = Suspected of damaging fertility or the unborn child. H361fd = Suspected of damaging fertility.  
Suspected of damaging the unborn child

H372 = Causes damage to organs

H373 = May cause damage to organs through prolonged or repeated exposure

H400 = Very toxic to aquatic life

H411 = Toxic to aquatic life with long-lasting effects

H412 = harmful to aquatic life with long-lasting effects

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