REPORT

Degassing barge tanks from low vapour pressure products

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1 Introduction

1.1 Background

Inland tanker vessels transport a variety of liquids for industry. After cargo has been unloaded, small amounts of liquid and vapour product remains in the cargo tanks. This is often released to the atmosphere to avoid contamination of the next cargo. With hazardous cargoes, this so-called degassing of the tanks is harmful for humans and the environment. The governments of the CDNI[1] countries pursuit the incorporation of restrictions to degassing of barges in the CDNI treaty in order to limit harmful emissions to the air.

The CDNI/G[2] working group prepares this gradual incorporation in the CDNI treaty. In addition, a steering committee was established to advice. Members of this Steering committee Gaseous Residues of liquid cargo in inland Tanker Shipping (GRTS) represents a wide number of stakeholders.

Late June 2016 the CDNI Contracting Parties Conference, CPC, finished a first full draft of regulations concerning the handling of liquid cargo gaseous residues and conducts a public consultation from 15 July to 15 September 2016 [1].

This first full draft text includes in Annex VI Part I, II and III, a list of 37 products, presented as UN product codes, for which degassing is prescribed. The GRTS questions whether or not degassing serves a useful purpose for the low vapour pressure products included in these tables. To support the discussion on this concern FuelsEurope and CEFIC, both represented in the GRTS, asked Royal HaskoningDHV to study degassing of low vapour pressure products. The current document reports the results of this study and will be used by the GRTS in their preparation of a response to the public consultation conducted by the CPC.

1.2 Questions to answer

This report answers the following questions:

- What are the volumes of liquid remains in cargo tanks and in what condition are these remains present?

- For which products, ranked on basis of vapour pressure, will a degassing operation remove all liquid remains within a reasonable period of time?

Answering these questions requires understanding of parameters 'vapour pressure' and 'evaporation rate', 'degassing rate' and the behaviour of mixtures. This report starts with a discussion on these parameters.

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[1] CDNI treaty: Convention on the collection, deposit and reception of waste produced during navigation on the Rhine and inland waterways
[2] CDNI/G: Group of national experts ad hoc

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2 Degassing rate: theoretical approach

2.1 Degassing rate

In controlled degassing, the vapours are sucked from the tanks and treated (see *Figure 1*).
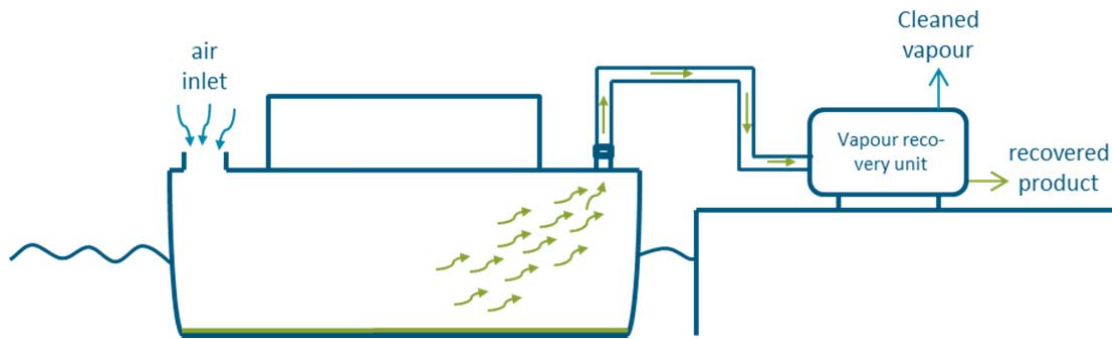
Figure 1: Schematic of controlled degassing

When the fresh air mixes completely with the vapour in the tanks and no liquid is present in the tanks, the course of the vapour concentration (C) in time (t) depends on the total volume of the tanks (V_{tanks}) and the volumetric rate of degassing ($Q_{degassing}$):

$$\frac{dC}{dt} = -\frac{Q_{degassing}}{V_{tanks}} C(t)$$

and:

$$C(t) = C_0\, e^{-\frac{Q_{degassing}}{V_{tanks}}t}$$

This results in a typical graph as shown in *Figure 2*.

When the degassing starts with a vapour concentration of C_0 and ends at the Acceptable Vent Free Level (AVFL; C_{AVFL}), the total time for degassing t_{AVFL} is:

$$t_{AVFL} = -LN\left(\frac{C_{AVFL}}{C_0}\right)C_0\,\frac{V_{tanks}}{Q_{degassing}}$$

<u>Example</u>
Degassing a 5,000 m³ barge discharged form gasoline at a degassing rate of 3,000 m³/h. Initial concentration at start is 500 g/m³ (about 60% of saturation vapour pressure at 10°C). The AVFL is 3.5 g/m³. The total degassing time in this example is 8,2 hours. Figure 2 shows the concentration as function of time.
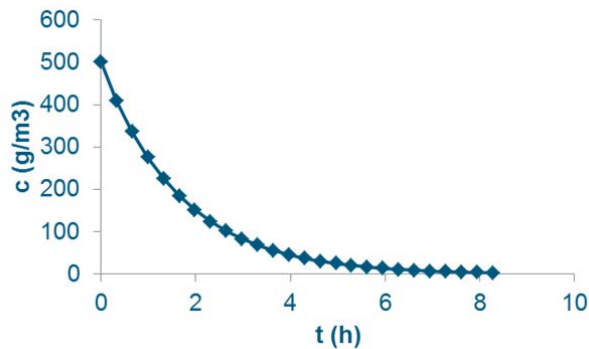
Figure 2: Example of theoretical degassing rate for gasoline; vapour concentration as function of time.

2.2 The effect of remaining liquid on the degassing

The theoretical degassing rate as described in section 2.1 is based on tanks free of liquid remains. It is known that in practice certain amounts of liquid product remains in the tanks. Commonly, it is assumed that this liquid evaporates during the first phase of degassing, resulting in a constant concentration in this first phase. In other words: evaporating liquid replenished the vapour removed until the moment all liquid is evaporated.

An underlying assumption is that the evaporation rate is sufficiently high to evaporate all liquid during the degassing. Key parameters determining the evaporation rate are:

- Vapour pressure   Physical property of substance; high vapour pressure = more volatile
- Temperature   Temperature of the liquid to evaporate
- Evaporating surface   Is the volume of liquid spread over a thin layer or in a well?
- Surface effects   Stagnant air layer or continuously mixed air / vapour above surface?

Finally the evaporation time of all liquid also largely depends on the amount of remaining liquid and the evaporating surface area.

2.3 Remaining liquid

Most barges are equipped with double-hull tanks. Single-hull tanks are phasing down and will be out of service end of 2018 [14]. Each tank is equipped with a lowered section in the bottom of each tank where liquid collects. This well (see *Figure 3*) contains approximately 5 – 10 litres of liquid product, depending on the ambient temperature, product vapour pressure and product viscosity [14].

Figure 3: Photo from a well in bottom of double hull tank in barge (Photo provided with memo [14])

A 'typical' barge for calculation purposes is considered:

- 10 equally sized separate tanks;

- Tank dimensions (W x L x H): 3 m x 20 m x 5 m = 300 m^3;

- All tanks in series flushed with air during degassing (air from one tank to the next).

In some cases, liquid from piping flows back in tanks after uncoupling. This can be up to 15 litres. An effective stripping[3] is part of normal operation. However, stripping procedures do not prescribe a final result that needs to be achieved, so in day-to-day practice stripping results differs case by case. ESO claims a negligible difference in amounts of liquid remains between RVS, mild steel and coated tanks [14].

The bottom of the tanks (and the wells) and the liquid residues are expected to be at or near the temperature of the water surrounding the barge. This is a rather constant temperature of about 10°C.

Concluding, we assume a total amount of liquid in the 'typical' barge to be 50 – 150 litres. It should be noted that some operators of (experimental) degassing installations claim higher volumes of liquid remains. These operators therefore re-strip the tanks right before degassing since removal of liquid is less expensive then evaporative removal by degassing. ESO also recommends re-stripping before degassing [14].

2.4   Predicting the rate of evaporation

Predicting the rate of evaporation is known to be difficult. An empirical (and theoretical) correlation to estimate the maximum evaporation rate under equilibrium conditions based on kinetic theory is the Knudsen equation [i.a. 6]. This is only valid when the pressure is near the vapour pressure and consequently at atmospheric pressure a liquid temperature near the boiling point. This is not valid for evaporation at temperatures well below the boiling point.

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[3] *The main pump is not able to remove up the last residues. With stripping the last liquid residues are removed.*

2.4.1 Film model based correlations

There is scientific literature on estimation methods of evaporation rates of organic liquids. This concerns mostly studies of spills of chemicals [e.g. 9] or oil spills [e.g. 11]. This is commonly based on the well-known 'double film model' [9]. Often, only mass transfer in the gaseous phase is modelled.
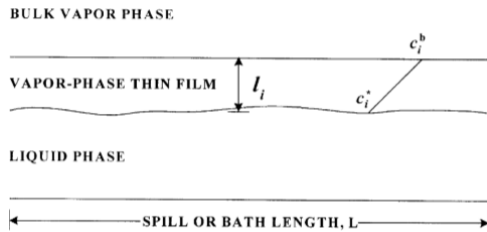

Figure 4: Schematic of liquid evaporation through vapour-phase film [9]

The general equation is (nomenclature deviates from *Figure 4*):

$$J = \frac{D_g}{l_g}\left(C_g^* - C_g\right) = K_g\left(C_g^* - C_g\right)$$

J	: Mass flow rate / evaporation rate	(kg s^{-1} m^{-2})
Dg	: Diffusivity of component in air	(m^2 s^{-1})
l$_g$	: Gas phase film thickness	(m)
C$_g$*	: Equilibrium concentration in air	(kg m^{-3})
C$_g$*	: Concentration in air	(kg m^{-3})
K$_g$	: Mass transfer coefficient	(m s^{-1})

When assuming an ideal gas, the equilibrium concentration can be expressed in terms of the ideal gas. In this way the evaporation rate is expressed as function of the vapour pressure:

$$J = K_g \frac{P_V(T)\ M}{R\ T}$$

P$_V$(T)	: Vapour pressure component(function of T)	(Pa)
M	: Molar mass of component	(g mol^{-1})
R	: Universal gas constant	(J mol^{-1} K^{-1})
T	: Temperature liquid	(K)

Since evaporation is an endothermic process, the evaporation will result in a temperature decrease of the liquid. For this reason, the above mass balance must be coupled with a heat balance. A further explanation of this is out of the scope of this study, however, coupling of mass and heat balance results in an equation of K$_g$ as function of the wind speed (u), air density (ρ), the dynamic viscosity of air (v), the equivalent diameter (d) of the evaporating liquid pool and the diffusion coefficient and the component (D_g):

$$K_g = f\left(u^a, d^b, D_g^c, v^d\right)$$

Often this is expressed in dimensionless numbers: Sherwood (Sh; dimensionless mass transfer), Reynolds (Re; fluid flow) and Schmidt (Sc; diffusivity) in the form:

$$Sh = K \cdot Re^p \cdot Sc^q$$

with K, p and q being constant that are fitted on experimental data.

Heymes et al. compared in 2013 values of the exponents a – d from various studies and concludes that these vary widely [10], resulting in significant differences.

2.4.2 Empirical correlations

A common equation to estimate the evaporation from a liquid pool is the equation from the American Environmental Protection Agency (EPA) [12]. This is (converted to metric units):

$$E = \frac{0.1288 \cdot A \cdot P \cdot M^{2/3} \cdot u^{0.78}}{T}$$

E : Evaporation rate (kg/min)
A : Evaporation area liquid (m^2)
P : Vapour pressure of liquid (kPa)
M : Molar mass of liquid (g/mol)
u : Air flow speed above liquid (m/s)
T : Liquid temperature (K)

A simple correlation was develop by Mackey et al. (and discussed in [13]):

$$E = 4.07 \cdot 10^{-10} \cdot M \cdot P$$

When we plot the EPA-correlation (see *Figure 5*) and the correlation by Mackey, we see consistent evaporation rates for some substances and significant differences for most others. Differences are discussed on more detail in section 2.4.3.
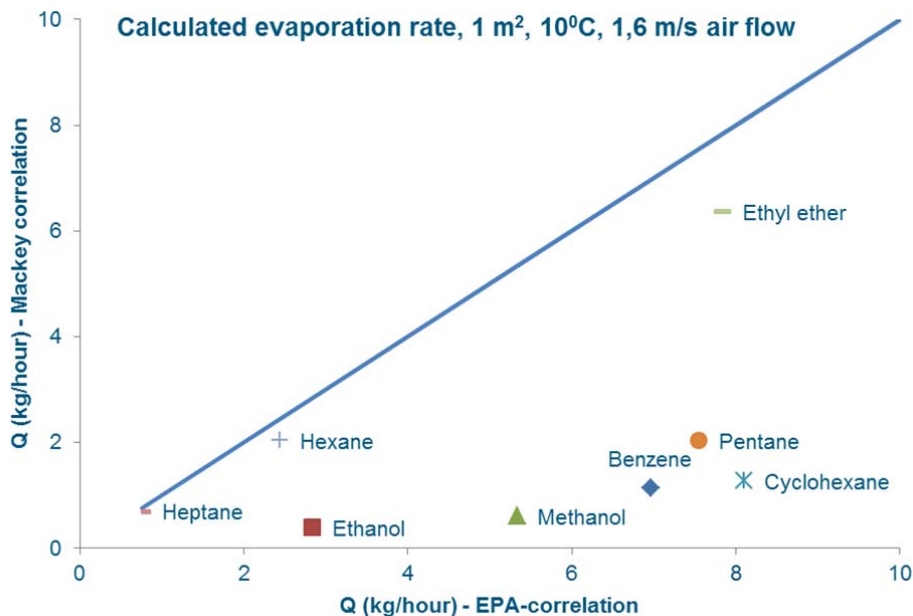

Figure 5: Calculated evaporation according EPA-correlation and the correlation by Mackey

2.4.3 Comparison of experimental and calculated values

A recent study [13] comparing various correlations (both film model based as well as empirical) to predict evaporation rates from pools confirms the wide spread in outcomes of these correlations. *Figure 6* shows the results of this comparison and demonstrates that even the best correlations show deviations of up to 100%. Explorative calculations we performed with various correlations showed similar differences between correlations.

It turns out that a specific correlation based on a set of experimental data that fits that particular set of data, does not fit experimental data for the same substance under different experimental data. Knowing that most researches apply similar experimental set-ups (a kind of wind tunnel with a cup or box with liquid that is weighted before and after a period of time), demonstrates that experimental conditions are important. We believe the conditions in a ship being degassed deviate significantly from experimental conditions in lab experiments and differ a lot more than the conditions differ between several lab set-ups. From this we conclude that the calculation of theoretical evaporation rates is of little value.

There is no straight forward correlation between the evaporation rates and boiling points of solvents. For chemically related hydrocarbons volatility decreases with increasing boiling point. Hydrocarbons that form hydrogen bonds (e.g. alcohols) are less volatile than other hydrocarbons with the same boiling points [4]. Smallwood [3] states "there is no satisfactory method of calculating the rate of evaporation of a solvent, since it depends on the equipment in which evaporation takes place as well as a number of properties of the solvent".
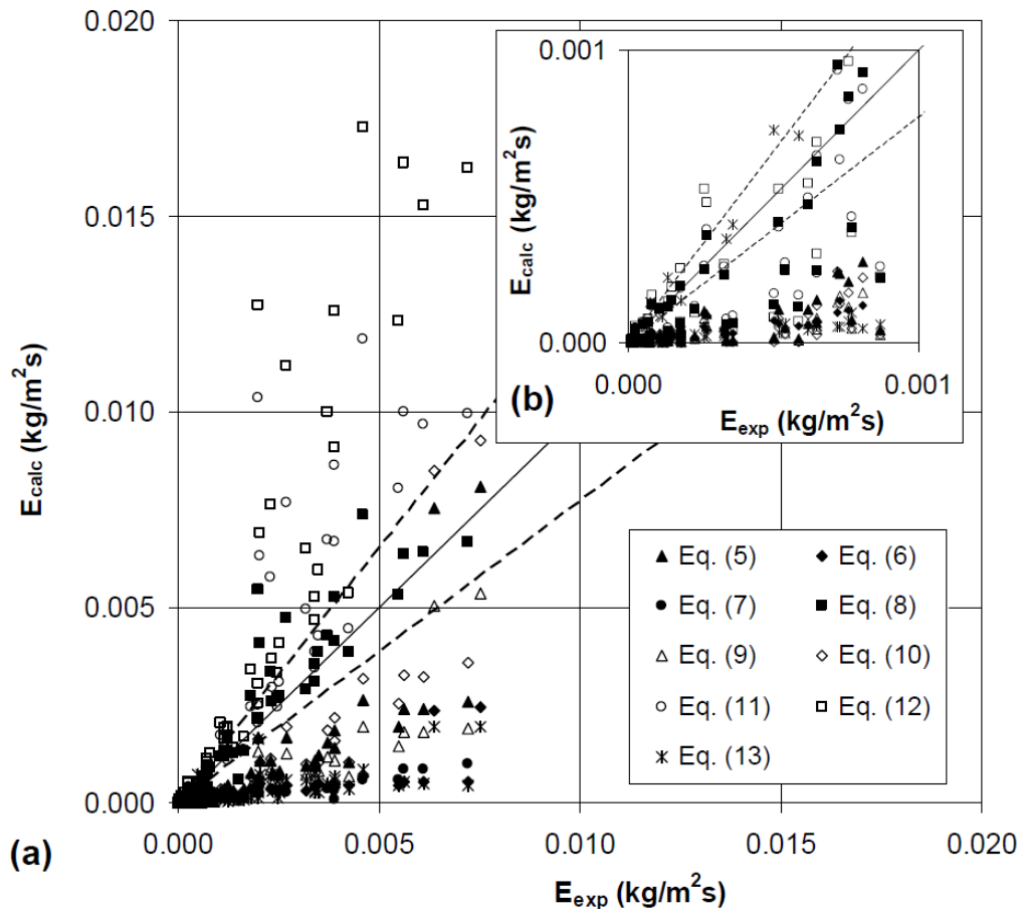
Figure 6: Comparison of calculated versus experimental evaporation rates based on various correlations and experimental data sets. The dotted lines show the +/- 30% deviation. (b) is an enlargement of the lower values part of (a). The equations 5 – 13 are proposed by various authors and differ in form and complexity. Figure taken from Rubbico et al. (2016) [13]

2.5   Evaporation numbers

In section 2.4.3 we concluded that the rate of evaporation cannot be predicted adequately by correlations. For this reason, evaporation rates are commonly expressed relatively against the time of evaporation of diethyl ether (commonly referred to as just 'ether') or butyl acetate (BuAc) under the same circumstances and conditions. These relative evaporation rates are called 'evaporation numbers'. The definitions are (note that the ratios are opposite!) [4]:

$$E_{ether} = \frac{t_{test\ solvent}}{t_{ether}}$$

$$E_{BuAc} = \frac{t_{BuAc,90\%}}{t_{test\ solvent,90\%}}$$

Where E is the relative evaporation rate and the t's are the evaporation times (full evaporation for ether; 90% of evaporation for BuAc). Evaporation numbers are typically determined at 20°C.

The approximate relationship between both is [3]: $E_{BuAc} = \dfrac{15}{E_{ether}}$

Example: the evaporation number (ether = 1) of benzene is 2.6 [3]. When ether evaporates at 0.75 g/s/m², benzene evaporates under the same circumstances at 0.75 / 2.6 = 0.29 g/s/m².

Table 1 provides vapour pressures and evaporation numbers of all pure (no mixtures) substances that are included in the draft of regulations concerning the handling of liquid cargo gaseous residues that is available for public consultation from 15 July to 15 September 2016 [1].It can be seen that many substances have a evaporation number larger than benzene (a substance often considered in degassing).

It should be noted that some UN-numbers are mixtures of isomers. There are:

- UN1216   Iso-octens   isomers with significantly different vapour pressures (not in *Table 1*);

- UN1307   Xylenes   mixture of ortho-, meta-, and para-xyleen (p-xylene in *Table 1*).

Het mixtures of hydrocarbons such as UN1268 could be considered as a mixture of mainly linear alkanes. For this reason, the alkanes butane (C4), pentane (C5), hexane (C6), heptane (C7), octane (C8) and nonane (C9) are included in *Table 1*. The vapour pressures are calculated with the empirical Antoine equation, based on mainly [7] and [8]. The evaporation numbers are mainly taken from [3]. Since Antoine's equation is valid over a certain temperature range, vapour pressures outside this range are missing. Butyl acetate is included in the table since this product is used as reference for evaporation numbers.

Table 1: Vapour pressures (calculated, Antoine equation) and evaporation numbers of selected substances at 20°C

Substance	UN-number	Molar weight	Boiling point	Vap. Press.	Evaporation number	
		[g/mol]	[°C]	[kPa]	(ether = 1)	(BuAc=1)
benzene	1114	78,114	80,09	10,01	2,60	
ethylbenzene	1175	106,167	136,21	1,01	8,80	0,84
ethanol	1170	46,069	78,65	5,86	8,30	2,40
Methanol	1230	32,042	64,54	13,00	6,30	4,10
acetone	1090	58	56,07	24,71	1,80	5,60
cyclohexane	1145	84,161	80,78	10,34	3,40	5,60
formaldehyde	1198	30,026	-19,1	-		
MTBE	2398	88,1482	55,05	27,25		
iso-propyl amine	1221	59,111	31,78	63,67		
p-xylene	1307	106,167	138,38	-	13,50	0,76
ethyl acrylate	1917	100,1158	99,85	3,92		
methyl acrylate	1919	86,0892	79,85	9,14		

Substance	UN-number	Molar weight [g/mol]	Boiling point [⁰C]	Vap. Press. [kPa]	Evaporation number (ether = 1)	(BuAc=1)
isopropylbenzene	1918	120,1916	151,85	0,37		
isobutyraldehyde	2045	72,1057	62,85	17,49		
styrene	2055	104,1491	145,85	0,62	16,00	
n-Butyl acrylate	2348	128,169	146,85	0,54		
iso-Butyl acrylate	2527	128,169	132,05	-		
acetic acid	2789	60,05196	117,9	1,57		
butane		58,123	-0,49	-		
pentane		72,15	36,07	56,56	1,00	13,00
hexane		86,177	68,73	16,18	1,40	8,40
heptane		100,204	98,42	4,73	3,00	3,30
octane		114,231	125,67	-		1,23
nonane		128,258	150,82	-		
ethyl ether		74,123	34,44	58,60	1,00	28,00
butyl acetate		116,16	125,97	-	11,80	1,00

Figure 7: Vapour pressures (calculated) and evaporation numbers of selected substances (plotted data from Table 1)
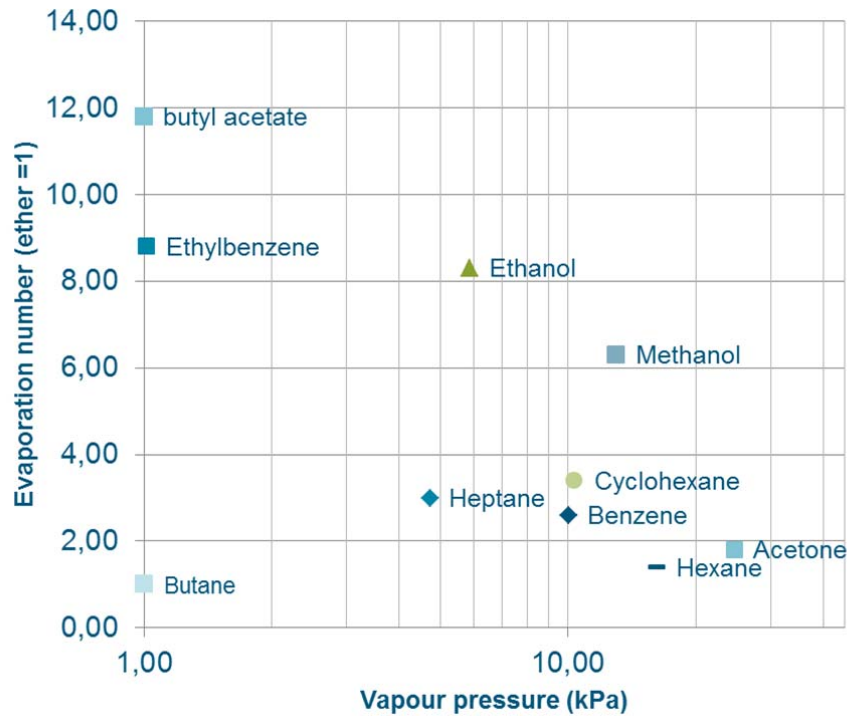
Plotting the evaporation numbers as function of the logarithm of the vapour pressure value results in a poor correlation; linear regression results in a coefficient of determination (this is R^2) of about 0.40. The fit becomes better when only certain groups of products (for example: alcohol or linear alkanes) are considered. However, this is of little value for the general correlation we are looking for. Finally, evaporation numbers are not available for all substances.

3 Which products are 'degasable'?

From chapter 0 we learned that the actual evaporation rate cannot be predicted based only on parameters that can be determined during a degassing operation. Only trends and order-of magnitude evaporations rates can be determined. For most substances we consider, the order-of-magnitude evaporation rate will be in the range of 1 – 10 kg/h. This is based on explorative calculations (see also *Figure 5*) and some experimental values from literature. The evaporation of the remaining liquid of 50 – 150 litres will easily take longer than a typical degassing takes. This endorses the before mentioned recommendation to start a degassing with re-stripping. Moreover, it also demonstrates that less volatile products could be considered as 'not degasable'.

## 3.1 Vapour pressure as cut-off parameter?

Since evaporation rate estimations turned out to be unreliable and actual evaporation rates highly depend on actual conditions and circumstances, a limiting value will be rather arbitrary. We propose to fall back to the most fundamental volatility parameter: the vapour pressure. The vapour pressure is known for all products, even for mixtures in most cases.

Qualitatively we can say about cargo tanks emptied from:

- products with a high vapour pressure <u>can</u> be cleaned and brought below the AVFL by degassing;
- products with a low vapour pressure <u>cannot</u> be cleaned and brought below the AVFL by degassing.

To sort products in 'high vapour pressure' and 'low vapour pressure', we need a limiting value or a limiting range of vapour pressure. *Figure 8* shows the vapour pressures of the products in *Table 1*.

Figure 8: Vapour pressures of selected products
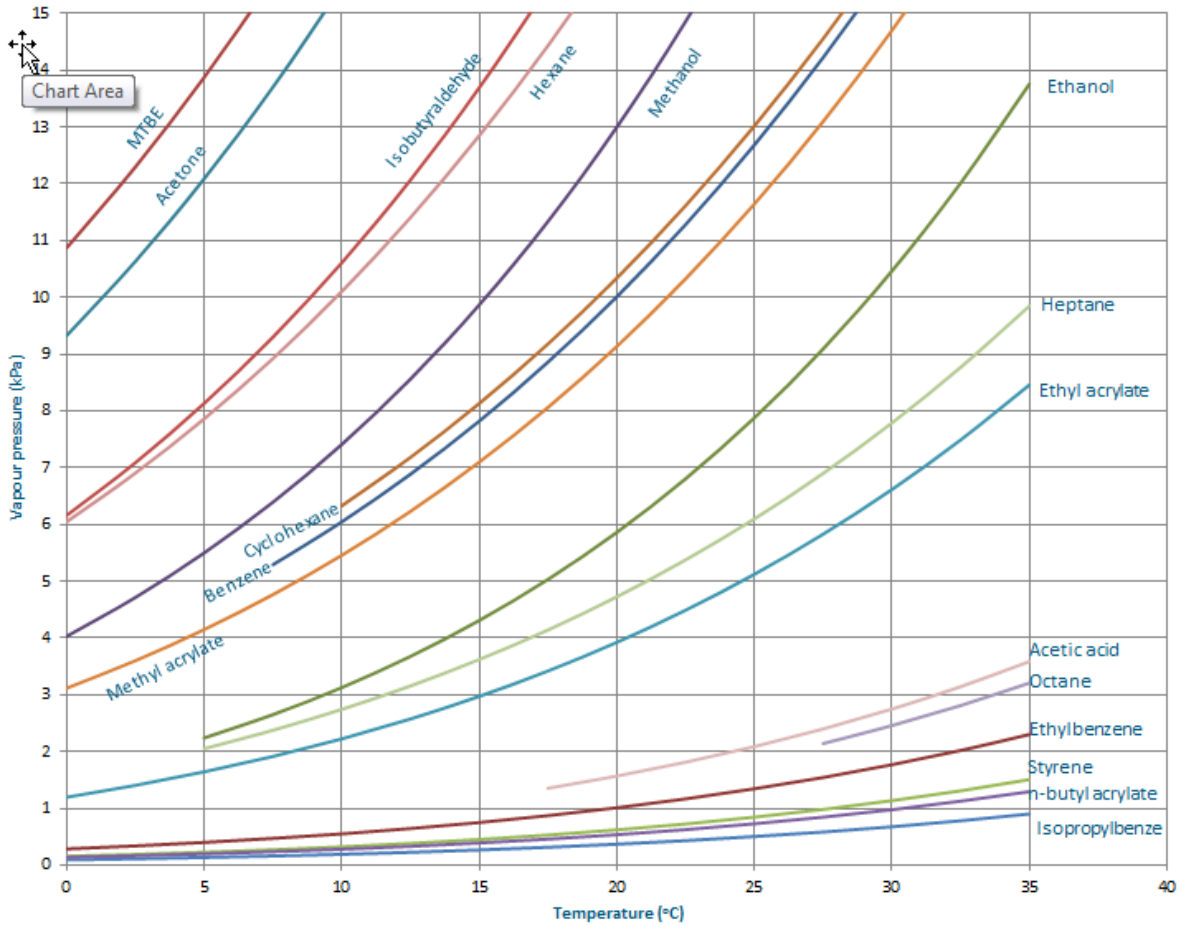
To determine limiting values, we propose to follow on from MARPOL (International Convention for the Prevention of Pollution from Ships; www.imo.org) for seagoing vessels that states that cargo residues from substances with a vapour pressure of > 5 kPa at 20°C may be removed from a cargo tank by ventilation.

When we apply this limiting value to the products in *Table 1* and *Figure 8*, we can extract a list of products that are 'degasable' or 'not degasable'. This is shown in *Table 2*.

Table 2: Vapour pressures at 20^0C and 'degasibility' based on limiting vapour pressure value of 5 kPa at 20^0C

Substance	UN-number	Molar weight [g/mol]	Boiling point [^{0}C]	P_{VAP} @20^{0}C [kPa]	"Degasable?"
benzene	1114	78,11	80,1	10,0	yes
ethylbenzene	1175	106,17	136,2	1,0	no
ethanol	1170	46,07	78,7	5,9	yes
methanol	1230	32,04	64,5	13,0	yes
acetone	1090	58,08	56,1	24,7	yes
cyclohexane	1145	84,16	80,8	10,3	yes
formaldehyde	1198	30,03	-19,1	> 100	yes
MTBE	2398	88,15	55,1	27,2	yes
iso-propyl amine	1221	59,11	31,8	63,7	yes
p-xylene	1307	106,17	138,4	< 2	no
ethyl acrylate	1917	100,12	99,9	3,9	no
methyl acrylate	1919	86,09	79,9	9,1	yes
isopropylbenzene	1918	120,19	151,9	0,4	no
isobutyraldehyde	2045	72,11	62,9	17,5	yes
styrene	2055	104,15	145,9	0,6	no
n-Butyl acrylate	2348	128,17	146,9	0,5	no
iso-Butyl acrylate	2527	128,17	132,1	≈ 0,5	no
acetic acid	2789	60,05	117,9	1,6	no
butane		58,12	-0,5	> 150	yes
pentane		72,15	36,1	56,6	yes
hexane		86,18	68,7	16,2	yes
heptane		100,20	98,4	4,7	no
octane		114,23	125,7	< 0,1	no
nonane		128,26	150,8	< 0,1	no
ethyl ether		74,12	34,4	58,6	yes
butyl acetate		116,16	126,0	< 2	no

3.2   Vapour pressures of mixtures

The vapour pressure of a mixture is the sum of the partial vapour pressure of the mixtures components. The total vapour pressure can be estimated by Raoult's law that states that the partial vapour pressures are equal to the mole fractions multiplied with the pure component vapour pressures. It is important to understand that the evaporation rate of a mixture is not constant in time: the vapour is first richer in components with the higher vapour pressures and later the vapour is richer in the components with lower vapour pressures.

A mixture with a vapour pressure below 5 kPa can consist of components with vapour pressures above and below 5 kPa. The individual components can therefore partly be classified as 'degasable' and partly 'not degasable'. Whether or not the mixture is 'degasable' in practical circumstances depends on the composition and how wide the range of vapour pressures of the individual components is. A mixture with a low fraction of a component with a high vapour pressure (for example octane; $P_{VAP} \approx 1.5$ kPa at 20°C) and high fractions of components with high vapour pressures (for example pentane and hexane; $P_{VAP} \approx 55$ and 15 kPa at 20°C) will have a total vapour pressure above 5 kPa and be considered 'degasable', while during degassing the high vapour pressure components will not evaporate and remain as liquid residue.

4   Conclusions

When a barge is degassed, liquid will remain in the cargo tanks. This is estimated to be 50 – 150 litres for a typical 3,000 m^3 barge with 10 tanks. This liquid is mainly contained in 'wells' present in each of the tanks.

Evaporation periods cannot be estimated on basis of physical properties of the products and degassing circumstances and conditions. Existing correlations to estimate result in large deviations from experimental evaporation rates. However, very rough calculations show that the evaporation of the remaining 50 – 150 litres will take easily longer than a typical degassing takes. For this reason, we recommend to start a degassing with re-stripping. But even after re-stripping, less volatile products could be considered as 'not degasable'.

Given the above, a limiting value for 'degasability' of products is difficult. Therefore we propose to fall back to the vapour pressure. Picking a limiting value is for the same reasons rather arbitrary, though a value in the range 1 – 10 kPa seems reasonable. We propose the follow on from MARPOL (International Convention for the Prevention of Pollution from Ships) for seagoing vessels that states that products with vapour pressures over 5 kPa at 20°C can be removed from cargo tanks by ventilation. It is recommend to apply this criteria also to mixture of products.

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