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SAFETY OF ORGANIC SOLVENTS
IN WASTE TANKS

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TABLE 1. Flash Points and Flammability Limits

	Decane	Undecane	Dodecane	Tridecane	Tetradecane	TBP
Flash Point, °C	45.5	61.3	75.5	87.3	102.2	146
Lower Point, vol. %						
At Flash Point	0.772	0.704	0.648	0.596	0.554	0.999
At 80°C	0.751	0.694	0.645	0.600	0.564	1.056
At 100°C	0.739	0.682	0.635	0.590	0.555	1.039
Lower Limit, mg/L						
At Flash Point	42.0	40.1	38.6	37.2	35.7	77.4
At 80°C	36.9	37.4	37.9	38.2	38.6	97.1
At 100°C	34.3	34.8	35.3	35.5	35.9	90.4

The allowable concentration by weight in air decreases as the temperature increases according to the formula

$$L = 49 \frac{\text{mg}}{\text{l}} \times \frac{273.16}{273.16+t}$$

where t is the temperature in °C. This decrease corresponds to thermal expansion of the air and consequent reduction of oxygen content. Flammability limits are also lowered as the temperature increases according to the formula (Zabetakis 1965):

$$\frac{L_t}{L_{25}} = 1 - 0.000784 (t - 25).$$

Thus, at temperatures above the flash point, the allowable quantity of material in air is further reduced.

Mixtures of flammable gases will have a flammability limit related to the limit of each component (L_i) and its mole fraction (X_i). The limit of the mixture is given by LeChatelier's law (Coward and Jones 1952):

$$\frac{1}{L} = \frac{X_1}{L_1} + \frac{X_2}{L_2} + \dots + \frac{X_n}{L_n}.$$

It should be noted that the values of L_i should all be at the same temperature to obtain a value of L for that same temperature.

Addition of nonflammable gases (e.g. N_2 , CO_2 , H_2O) to the vapor phase will prevent combustion if the concentration is sufficiently high (Zabetakis 1965). Very little information is available on the effect of water vapor, but the effect of water on heptane flammability is graphically presented by

Zabetakis. He also presents the effect of CO₂ and N₂ on a single graph for all normal paraffin hydrocarbons higher than pentane, so the effect of water vapor on heptane should be similar to the effect on C₁₀ to C₁₄ NPH. With up to about 22 volume percent water vapor, there is little effect on the lower flammability limit. Between 22 and 33 percent the limit increases gradually, and above 33 percent there is no heptane concentration which is flammable., Antonik et al., (1972) report in a brief article that at 120°C the minimum water vapor content to suppress flammability of n-octane is 35%, while n-decane requires about 40% water vapor. Their work involves development and testing of a new system, and their measurements of lower limits for n-decane to n-tridecane disagree appreciably from accepted values. Therefore, their data are considered to be less reliable than the data of Zabetakis (1965). However, it is the only reference to measurements with water vapor and a hydrocarbon of C₁₀ or higher.

Other measures of flammability should not be confused with flash point or lower flammability limits. The autoignition temperature is the minimum temperature of a heated object which can cause flammability of a material. For C₁₀ to C₁₄ NPH the autoignition temperature is slightly above 200°C. The fire point is the lowest temperature at which a liquid will continue to burn when ignited. It is appreciably above the flash point since only the vapor phase burns at the flash point (Roberts and Quince 1973). Detonation limits are concentrations of fuel in air which can propagate a reaction at speeds above the speed of sound (Zabetakis 1965). At the lower flammability limit the flame propagates much more slowly, perhaps only 10 cm/sec (Harris 1967). The oxygen index is the minimum concentration of oxygen required to support combustion over a flammable liquid (Nelson and Webb 1973).

Vapor Pressure

Flammability is closely related to the vapor pressure for NPH in the region C₁₀ to C₁₄. The flash point corresponds to the temperature at which the vapor pressure equals the lower flammable limit. Calculation of flash points for mixtures requires a knowledge of the vapor pressure as a function of temperature. Perry and Chilton (1973) give the temperatures corresponding to vapor pressures of 1 to 760 mm of Hg. The data in this form are not useful since interpolation is required to obtain a vapor pressure at a given temperature. Dean (1979) gives constants for an equation relating vapor pressure to temperature, but the range of applicability does not always cover the region of interest. In the range of mutual applicability, the results usually agree within 1°C except for two values which seem to be typographical errors in Perry and Chilton's tables. The equation used by Dean is as follows:

$$\log p = A - \frac{B}{t + C}$$

where p is the pressure in torr (mm Hg) and t is temperature in $^{\circ}\text{C}$. It can be rearranged as follows to calculate temperature for a given pressure:

$$t = \frac{B}{A - \log p} - C.$$

Approximate constants for this equation were derived by using three widely separated values of vapor pressure and the corresponding temperature. These values were then refined by adjusting the value of A and using a least squares method to solve for B and C until a "best fit" was achieved.

Values for TBP were based on the limited and conflicting values presented by Moore and Walser (1980). Only three points were used to fit the three constants: boiling point of 289°C at 760 mm, steam distillation data corresponding to 0.988 mm at 100°C , and vapor pressure of 25 mm at 177°C . The other data in Moore and Walser correspond to lower vapor pressure at the same temperature.

Values of the constants for NPH components and for TBP are given in Table 2. Error values for NPH components are based on the table in Perry and Chilton (1973). Two values were not included either in deriving the constants or in evaluating errors. These are for (1) undecane at 20 mm Hg, where Perry and Chilton list 85.6°C but values of 89.1°C and 88.3°C , respectively, result from Dean (1979) and the constants in Table 2, and (2) tridecane at 5 mm Hg, where Perry and Chilton list 98.3°C but the constants in Table 2 give 89.3°C . Comparison with Dean's values over the range of applicability is generally poorer, with the largest errors at low pressures. Dean consistently indicates a lower vapor pressure at a given temperature in this pressure region.

Vapor pressure of separate phases (e.g., NPH and water) in equilibrium with air is equal to the sum of the vapor pressures of the two phases. However, when a solution exists, the vapor pressure is not the simple sum of the pure component vapor pressures but is equal to the sum of the vapor pressure of each pure component multiplied by the mol fraction of that component in the solution. Since NPH is a mixture of components, this rule (Raoult's law) must be used to compute the vapor pressure of the mixture. Also, the vapor phase composition is different from the solution phase composition.

The same effect is important in reducing the vapor pressure of water over solutions containing dissolved salts. As the salt content increases, the vapor pressure of water decreases and the boiling point of the solution increases. The effect on water vapor pressure can be approximated based on the normal boiling point of the solution since at the boiling point the product of water vapor pressure at that temperature and mol fraction of water is 760 mm of Hg. The mol fraction computed in this way can be applied at any other temperature to determine the vapor pressure of water over the solution.

When a solution saturated with NPH is evaporated, the vapor is relatively rich in water since water is more volatile than NPH. The solubility of NPH in water is quite low (Egorov 1979) and thus the mol fraction of NPH is low. As the water selectively evaporates, a second phase will form since more organic is present than is soluble in the water. As soon as the separate phase forms, it will evaporate since its vapor pressure is only limited by the vapor pressure of the pure organic instead of this vapor pressure multiplied by its mol fraction in water. Thus, the vapor phase will contain water and organic in the same ratio as was present in the saturated aqueous phase. The organic cannot condense as a separable phase until its vapor pressure has been reduced by a factor of the mol fraction of organic in a saturated solution. This factor is about 10^{-6} for NPH so that it will not form a separable phase until well below 0°C .

A layer of NPH on top of water may prevent the water from being in equilibrium with air. This would be especially true in vessels with no significant agitation, and at temperatures well below the boiling point.

Purex Solvent

Specifications for NPH diluent limit the range of allowable hydrocarbons as C_{10} to C_{14} inclusive, and also limit the flash point to $>80^{\circ}\text{C}$ (EMS-G-132-00669 B-1 1983). Only normal paraffin hydrocarbons are allowed. Because of these limitations, the diluent

TABLE 2. Constants for Vapor Pressure Equation

Compound		Constants			Error, $^{\circ}\text{C}$	
Name	Formula	A	B	C	Maximum	Average
Decane	$\text{C}_{10}\text{H}_{22}$	7.61	1967.77	242.13	0.30	0.13
Undecane	$\text{C}_{11}\text{H}_{24}$	7.74	2128.96	242.34	0.46	0.23
Dodecane	$\text{C}_{12}\text{H}_{26}$	7.66	2134.98	230.90	0.38	0.18
Tridecane	$\text{C}_{13}\text{H}_{28}$	8.11	2570.11	257.50	0.64	0.19
Tetradecane	$\text{C}_{14}\text{H}_{30}$	7.50	2115.30	205.44	0.71	0.37
Tributyl Phosphate	$\text{C}_{12}\text{H}_{27}\text{PO}_4$	10.55	5299.86	402.16		

must contain mostly decane or higher hydrocarbons. Many different compositions are allowable provided the above and other specifications are met. A composition quoted by Stordeur (1982) was used as the basis for the following discussion but was normalized to a mol fraction of unity. Other compositions would vary somewhat in details but would have the same general features.

Using the data of Tables 1 and 2, the flash point of NPH is calculated to be 76.7°C, and a vapor phase organic concentration of 45 mg/L is reached at 80.4°C. Stordeur (1983) uses a different method for calculating vapor pressure from the same base data, and concludes that a vapor concentration of 45 mg/L is not reached until 86°C. Stordeur does not seem to recognize that the ratio of organic to air is important at the lower limit. As air expands thermally, a lower organic concentration in mg/L will be flammable even though the mole ratio of organic to air at the flammable limit does not change much. According to my calculations, the solvent composition cited by Stordeur would not meet specifications since it has a flash point below 80°C. The actual flash point of the solvent was not quoted by Stordeur.

Different calculations of vapor pressure and evaporation rate of NPH are compared in Table 3. The vapor pressure values of Stordeur (1983) are based on NPH with a higher content of volatile hydrocarbons than the composition adopted for this work. This increase in volatile components should cause Stordeur's values of vapor pressure to be considerably higher than this work. The vapor pressure values of Kirch (1983) were apparently calculated from an equation given in Moore and Walser (1980). The lower flammable limit (0.6 to 0.7 volume percent) would be reached at between 50 and 60°C according to the vapor pressure data of Kirch. Therefore, the vapor pressure data of Kirch appear to be high. Evaporation rate data from Kirch are based on 25% of equilibrium whereas the data from this work are based on full equilibrium. Riley (1983) assumes full water saturation of air leaving the tank. The void space above a full tank is almost 500,000 gal. so that at 150 cfm, the average residence time of air in the tank is about two days. Full saturation of the air by organics does not seem unreasonable. Also, Kirch bases his

TABLE 3. Comparison of Vapor Pressure Calculations

Temperature, °C	Vapor Pressure, mm Hg			Evaporation Rate, gal./day	
	Stordeur (1983)	Kirch (1983)	This Work	Kirch (1983)	This Work
0	0.1		0.036		0.71
30	0.25		0.34		6.1
40	0.54	2.35	0.65	11.7	11.4
50	1.11	3.79	1.2	18.9	20.4
60	1.99	5.94	2.1	29.7	35.1
70	3.45	9.07	3.6	45.3	58.4
80	5.78	13.5	5.9	67.6	94.2
85	7.41		7.6		118
90	9.43	19.7	9.5	98.5	148
100	14.92	28.2	14.9	141	225
110		39.5	23	198	336
120		54.5	34	272	491

calculations on 150 scfm of air, and this work is based on 150 cfm of vapor exhausted from the tank. Thus, there is a larger volume of vapor represented in the Kirch evaporation rate data, especially at the higher temperatures.

Because of the high boiling point of NPH, it does not evaporate rapidly. However, at elevated temperature and with large volumes of air an appreciable quantity of NPH can evaporate. Mixture with TBP reduces the rate of evaporation of NPH by reducing the mol fraction in the solution. Evaporation rates were calculated as a function of temperature for fresh NPH and for 30% TBP in fresh NPH as shown in Table 4. Evaporation removes a higher percentage of the lower hydrocarbons than is present in the liquid phase so that subsequent evaporation proceeds at a slower rate, and the flash point of the residual organic solution becomes higher.

Standerfer (1979) gives an annual emission rate of 12 metric tons of volatilized hydrocarbons per year based on continuous PUREX operation. This corresponds to 32 kg per day which at a density of 760 g/L would be 43 L or 11.4 gal per day. The rate of addition of fresh NPH is 50 gal/day (Reberger 1983) so that about 23% is volatilized. This value is surprisingly low in view of the evaporation rates shown in Table 4 for 150 cfm since the total 291-A-1 stack flow is 6.32×10^{10} ft³/year (US EROA 1975 p. II. 1-C-70) or 120,000 cfm. If this flow of air contained 100 gal. per day of NPH, the NPH would not condense until well below 0°C. Even with only 11.4 gal/day evaporation loss, there will be an increase of flash point of PUREX solvent during operations as the volatile components are selectively removed by evaporation. With the arbitrary assumption of 10,000 gal. total solvent inventory, the flash point would increase by slightly over 3°C as a result of this continued loss.

TABLE 4. Evaporation Rates of PUREX Diluent and Solvent

Temperature, °C	Evaporation Rate, Gal/Day*		
	Pure NPH	30% TBP in NPH	
		NPH	TBP
30	6.1	4.5	0.16
40	11.4	8.4	0.22
50	20.4	14.8	0.40
60	35.1	25.7	0.70
70	58.4	42.7	1.18
80	94.2	68.8	1.96
90	148	108	3.19
100	225	165	5.09

*Based on air flow rate of 150 cfm and full equilibrium.

Another effect of the NPH being a mixture is that the flash point of condensed vapor will be lower than the flash point of the solution which evaporated to form the vapor. If the solvent is evaporated at 60°C and condensed at 30°C, about 2/3 of the vapor will condense and the flash point of the condensate will be about 10°C lower than the flash point of the original solution.

Applications

The waste tanks of concern may be divided into four categories:

1. Aging waste tanks and associated off-gas treatment
2. Double-shell slurry tanks and associated off-gas treatment
3. Feed tank for the evaporator/crystallizer
4. Evaporator/crystallizer and associated off-gas treatment.

Safety can be assured if one or both of the following conditions are met:

1. Vapor phase concentration of organic material below the lower flammable limit
2. Vapor phase concentration of water at least 33 volume percent (but dilution with air may produce a flammable mixture).

Safe conditions exist if the organic concentration in air is below the lower flammable limit. At atmospheric pressure and in the absence of mist or aerosol of organic, this condition is obtained if the surface temperature is below the flash point. At other pressures or with a mist or aerosol, the limiting condition is usually expressed in volume percent, but by the gas law this is equivalent to mole percent.

The second condition can be met if both separable organic and aqueous phases are in equilibrium with the vapor phase, and the partial pressure of water over the aqueous phase is sufficiently high. The partial pressure of water is dependent on the salt content of the aqueous phase. However, a mixture that is safe because of water content may become unsafe when mixed with air. Furthermore, in a quiescent system a layer of organic on top of the water may prevent the vapor phase from being in equilibrium with the aqueous phase.

At 80°C, the vapor pressure of water is 355 mm which corresponds to 46.7% by volume at atmospheric pressure. The vapor pressure of salt solutions can be estimated from the normal boiling point of the solution. At boiling, the vapor pressure is 760 mm, which is equal to the vapor pressure of water at the boiling temperature times the mole fraction of water in the solution. The

mole fraction determined in this way can be used to determine the vapor pressure of water over a salt solution at a given temperature. A solution with a boiling point of 110°C will have 33.0 volume percent water in the vapor phase at 80°C. Solutions with a somewhat higher boiling point may also be safe since the effect of water vapor at below 33 volume percent is to increase the flash point (Zabetakis 1965).

Aging waste tanks receive material only from PUREX Tank F16 which cannot contain separable organic (Reberger 1983). Organic present in dilute waste is removed by decanting prior to concentration, and further heating cycles assure complete removal of separable organics before material is added to TK-F-16 (Moore and Walser 1980). Organic may be present as a saturated aqueous solution. As the waste is concentrated, the quantity of organic which can remain in solution is reduced, and a separable organic phase is formed. This separable phase will immediately evaporate with a maximum concentration well below the limit of flammability. Thus, the aging waste tanks are always safe with respect to organic flammability.

Double-shell slurry tanks receive waste which has been processed through the evaporator/concentrator. This process step removes separable organics but leaves an aqueous phase saturated with organics. Waste from UNC (Schofield 1981) potentially contains a separable organic phase, but this organic phase is removed before the waste is shipped to RHO. Thus, the double-shell slurry tanks are always safe with respect to flammability.

The feed tank for the evaporator/crystallizer may contain separable organics (Reddick 1983). Feed is accumulated in this tank until enough has been accumulated for an evaporation cycle. Bottoms from the concentrator are returned to the feed tank until adequate concentration is achieved (Fineman 1981). When feed begins, the temperature of the tank is below 80°C, and the boiling point of the tank contents are below 110°C so that the tank is safe.

During evaporation, the tank may be heated to 125°F (52°C) at a rate of up to 10°F (5.6°C) per hour, but above 125°F the average heating rate may not exceed 3°F (1.7°C) per day (TF&EPC 1983). During concentration, any separable organic phase initially present would evaporate before the feed tank temperature could exceed the flash point. A sensitive period may exist whenever the tank temperature is above 80°C and the boiling point of the tank is above 110°C. During this period, only waste streams with no possible separable organics can be added to the tank. Administrative controls will be required to assure safety of this tank. Maintaining the evaporator feed tank below 80°C is recommended.

The principal opportunity for flammable organic vapors is in the vacuum evaporator when the normal boiling point of the contents is over 110°C. Under normal equilibrium operation there should be no problem because of inadequate air in the vapor phase, but problems could exist during startup or shutdown,

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or by air inleakage to dilute the vapor phase. The principal safety measures for this vessel are the provisions to exclude separable organic and the absence of ignition sources. However, a more detailed analysis of the operating cycle might demonstrate complete safety even if both separable organics and an ignition source were present. Based on limited information, the evaporator seems to be safe by water volatility during startup (boiling point below 110°C), absence of air during normal operation, and absence of organic during shutdown. However, the variability of potential operating conditions does make this analysis questionable. It is safe if organics are excluded.

Mist formation (Stordeur 1982, Stordeur 1983) was not considered since no agitators are present in the waste tanks of interest.

Some revisions to safety documentation seem to be desirable. The evaporator/crystallizer SAR (Christensen 1977) needs to be revised on pages 30 to 32 to add additional basis for safety in the description, to revise the detection scenario to allow for possible disabling of alarms as a result of an incident, to make minor changes to the consequences, and to rewrite the precautions section to state the minimum necessary and sufficient precautions.

The roadmap OSR (Schreiber and Vanselow 1981) needs to be revised on p. 23 to use appropriate references under Hazards, state actual safety limits, change limiting conditions to permit organic in the feed tank, add significant surveillance items, and update references.

The 241-AW, AN SAR (Safety Analysis Reports Staff 1980) needs to be revised on p. 30 to clarify the event sequence, modify the consequences, and add to the preventative measures.

The principal revisions are to the "Roadmap OSR" (Schreiber and Vanselow 1981). Some of the revised requirements are based on SAR documentation and probably do not yet exist in SOPs or operating specifications. I have indicated this by the notation "Specification to be written". I included a reference to an OSAR under "Limiting Conditions for Operation". It may be better for this to reference an operating specification if one exists.

My major changes are a complete rewrite of the material under "Hazard", allowance for separable organic under "Safety Limits" and "Limiting Conditions for Operation", and inclusion of "Surveillance Requirements" for temperature in the evaporator feed tank and liquid level in the same tank. References to RHO-MA-151 were changed to OSD-T-151. Other references in the old version were not checked or changed.

The principal change is the temperature in the evaporator feed tank which is limited as a "Safety Limit" to the flash point of PUREX solvent (TBP + NPH), and as a "Limiting Condition for Operation" to the more conservative flash

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point of pure PUREX diluent. I haven't calculated the margin of safety between the safety limit and operating limit, but it is over 5°C.

Surveillance is also required at PUREX in transferring material to the evaporator feed tank, perhaps to include a maximum temperature of solutions to be transferred as well as a limit on separable organics. Surveillance should also be done on catch tanks C-100 and A-417, but these do not fit with a tank farm OSR.

Consequences of various events were analyzed by Quinn et al., (1980), but an organic vapor fire was not analyzed per se. The closest event was a hydrogen explosion in the dome void. They state, "gases would cause a tank breach at an excess pressure of 60 in. of water." However, a recent analysis (DeFigh-Price 1983) shows that the tank will not yield until 40 psi, and even at 80 psig the secondary liner and concrete tank structure should not be damaged.

Gale (1983) extrapolates to 92,500 gal. of organic in solvent treatment waste from processing 10,000 MTU of fuel. At a rate of 10 MT per day and 100 gal. of organic per day (Reberger 1983), the total organic added is only 100,000 gal. in this period. This leaves little additional organic for discharge by the other routes identified by Reberger: (1) evaporation, (2) discharge to crib, and (3) soluble organic and organic degradation products. The organic balance across the PUREX plant is not well known.

Yours truly,

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