

STEAM

DISTILLATION

Aim: To study the characteristics of steam distillation.

To determine the values of vaporization efficiency (η_v) and thermal efficiency (η_t) for steam distillation of aniline

Apparatus: Distillation flask, steam generator, water cooled condenser, thermometer, separating funnel, specific gravity bottle, measuring jar.

Theory: Steam distillation is the term applied to a batch of continuous distillation process with open steam. The liquid is distilled by feeding open steam directly into the distillation still, so that the steam carries with it the vapors of volatile liquid component and is then condensed to separate the liquid from water. Steam distillation is possible only when,

1. The substance does not react with steam at the given conditions of temperature and pressure.
2. The substance is insoluble in water.

Steam distillation method is used for the separation of high-boiling substances from non-volatile impurities or for the removal of very high-boiling volatile impurities from still higher-boiling substances. The process has special value where it is desired to separate substances at temperature lower than their normal boiling points because of heat sensitivity or other reasons. The condensed organic liquid and water do not miscible in each other. Therefore partial pressure of each component is equal to vapor by each liquid. If P is total pressure, P_A and P_w are vapor pressure of organic liquid and water respectively. Then $P = P_A + P_w$. For ambient distillation, $P = 1 \text{ atm} = 101.3 \text{ kpa}$. Therefore P_A v/s T and $(101.3 - P_w)$ v/s T plots intersects and this point corresponds to distillation temperature.

Steam Requirements: Let y_A and y_w represent mole fraction of organic liquid and H_2O respectively in vapor phase. Then by Dalton's law $P^\circ_A = P y_A = P [N_A / (N_A + N_w)]$ and $P^\circ_w = P y_w = P [N_w / (N_A + N_w)]$ where N_A and N_w are moles of A and W and P°_A and P°_w are partial pressure of A and W.

Therefore $P_A^o/P_W^o = N_A/N_W = (W_A/M_A)$. We have $P_A^o = P_A$ and $P_W^o = P_W$ for steam distillation.

$$\text{Therefore } W_w/W_A = (P_W/P_A) \cdot (M_W/M_A) \quad (1)$$

Obtain P_W and P_A at distillation temperature from literature. The term (W_w/W_A) is the amount of open steam condensed per unit weight of liquid condensed however the actual requirement is higher. In equation (1) vapor pressure of liquid is assumed that be equal to its partial pressure.

FORMULAE TO BE USED:

Vaporization efficiency, η_v

$$\eta_v = \frac{\text{(wt. of aniline distilled/unit wt. of steam) actual}}{\text{(wt. of aniline distilled / unit wt. of steam) ideal}} = \frac{[W_s/W_w]_{\text{actual}}}{[W_s/W_w]_{\text{Ideal}}}$$

$$[W_s / W_w]_{\text{actual}} = \frac{\text{[Vol. of aniline in distillate x density. of aniline in distillate]}}{\text{[Vol. of water in distillate x density. of water in distillate]}}$$

$$[W_s / W_w]_{\text{ideal}} = [P_s M_s] / [P_w M_w]$$

Where

P_s = Vapor pressure of aniline at distillation temperature

P_w = Vapor pressure of water at distillation temperature

M_s and M_w = Molecular weights of aniline (93.12) and water (18)

Thermal efficiency, η_t

$$\eta_t = \frac{\text{[Distillation requirement of steam/unit wt. of sample distilled]}}{\text{[Actual requirement of steam / unit wt. of sample distilled]}}$$

i.e. $\eta_t = S_t / S_a$

where,

$$S_t = \frac{(1+R) C_{p,s} (T_d - T_r) + \lambda_s}{\lambda_w + (T_s - T_d) C_{p,w}} + \frac{[P_w M_w]}{[P_s M_s]}$$

$$S_a = \frac{[\text{Volume of H}_2\text{O in residue} \times \text{density of H}_2\text{O in residue}] + [\text{volume of H}_2\text{O in distillate} \times \text{density of H}_2\text{O in distillate}]}{[\text{volume of ANILINE in distillate} \times \text{density of ANILINE in distillate}]}$$

$$R = \frac{\text{Wt. of aniline in residue}}{\text{Wt. of aniline in distillate}}$$

T_r = room temperature

T_d = distillation temperature

T_s = Steam temperature

$C_{p,s}$ = specific heat of aniline at T_d

$C_{p,w}$ = specific heat of water at T_d

λ_s = latent heat of vaporization of aniline at T_d

λ_w = latent heat of vaporization of water at T_d

Note that $[\lambda_s / \lambda_w] = [M_w / M_s] [d(\ln P_w) / d(\ln P_s)]$

Where, M_w and M_s are molecular weights of water and aniline respectively.

$$\lambda_s = [M_w / M_s] \times [d(\ln P_w) / d(\ln P_s)] [\lambda_w]$$

PROCEDURE:

1. Take 100 ml aniline in the distillation flask and set up the apparatus.
2. Pass the steam at a pressure of 0.25 kg/cm²
3. Note down the distillation temperature.
4. Pass the steam till about 60-70 % of the liquid distills.
5. Stop the steam and allow the residue and distillate to get cooled.
6. Using the separating funnel, separate aniline and water in residue and distillate.
7. Measure the volume of aniline and water in residue and in distillate and find out specific gravities.

8. Draw a graph of $\ln P_w$ vs $\ln P_s$ and calculate the slope. Then calculate λ_s .
9. Draw Hans-brandt chart [plot of $(760 - P_s)$ vs. temperature and P_w vs. temperature) to find out the theoretical distillation temperature and compare it with the actual value.

OBSERVATIONS AND CALCULATIONS:

Volume of aniline taken = 100 ml

Distillation temperature $T_d = \text{ }^\circ\text{C}$

Room temperature $T_r = \text{ }^\circ\text{C}$

Steam temperature $T_s = \text{ }^\circ\text{C}$ (from steam tables)

Steam pressure, $P = 0.25 \text{ kg/cm}^2 - 0.3 \text{ kg/cm}^2$

$C_{p, s} = \dots\dots\dots$ (from perry's H.B)

$C_{p, w} = \dots\dots\dots$ (from Perry's H.B)

$\lambda_w = \dots\dots\dots$ (from Perry's H.B)

To get λ_s , obtain Vapor pressure data for aniline and water (from Perry's H.B)

Then

$$\lambda_s = [M_w / M_s] [d(\ln P_w) / d(\ln P_s)] \times [\lambda_w]$$

$[d(\ln P_w) / d(\ln P_s)]$ is given by slope of $\ln P_w$ Vs. $\ln P_s$ plot

Wt. of empty sp. gr. bottle = $W_1 = \dots\dots\dots$ g

Wt. of empty sp. gr. bottle + water = $W_2 = \dots\dots\dots$ g

Description		Volume (ml)	Wt. of sp. gr Bottle + soln. (g)	Specific gravity
Distilate	Water			
	Aniline			
Resiude	Water			
	Aniline			

To plot $\ln P_w$ Vs. P_s graph and Hans-brandt graph, the data extracted from Perry's Hand book is given below.

Sl. No.	Temperature (°C)	P _s (mm Hg)	P _w (mm Hg)	(P-P _s) P=760 mm Hg	ln P _s	ln P _w
1	34.8	1	41.71	759		
2	57.9	5	136.08	755		
3	69.4	10	223.73	750		
4	82.0	20	384.90	740		
5	96.7	40	674.60	720		

For calculation of η_v and η_t

$$\eta_v = \frac{[W_s / W_w]_{\text{actual}}}{[W_s / W_w]_{\text{ideal}}} \times 100 = \dots\dots\dots\%$$

$$\eta_t = [S_t / S_a] \times 100 = \dots\dots\dots\%$$

(Show one specimen calculation to find out specific gravity)

Results:

For steam distillation of aniline

Vaporization efficiency, $\eta_v = \dots\dots\dots\%$

Thermal efficiency, $\eta_t = \dots\dots\dots\%$