

# Determination of Amount of Theoretical Zones and Feed Location in Multicomponent Distillation

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*Abstract:* - Determination of tray location in multicomponent distillation is important for the designing of a distillation columns because it determines the vapour - liquid flow rate, components purity, reflux ratio among other important parameters. This information is often calculated using theoretical methods taking into consideration initial conditions at the feed and final product concentration rather than experimental. In the present article, we analyze the case of multicomponent distillation in which a feed of 100 mol/h are added to a fractionating tower at the boiling point and 405.3 kPa pressure. The system consists of a mixture of  $C_4H_{10}$ ,  $C_5H_{12}$ ,  $C_6H_{12}$  and  $C_7H_{14}$ . We aim to determine the characterization of light and heavy phase, component distribution, tower temperature, particularly at top and bottom. Accordingly, we approach the use of approximate methods to determine the minimum reflux ratio, amount of theoretical zones and feed location.

*Key-Words:* - multicomponent distillation, process design, minimum reflux ratio, approximate method, algorithm design, Underwood method, equilibrium stages

## 1 Introduction

Distillation is one of the most common methods used in industry to achieve the separation of two or more components from homogenous mixtures. The estimated costs associated to each stage of this process are a key success in the selection of this separation method and will depend on the proper modeling design and process calculations (i.e. mass and energy balances) [1]. In this process, heat is the main source of energy required to achieve the selective separation. Other methods that can be used in the separation of homogenous mixtures are gas absorption, crystallization, ion exchange, solvent extraction, liquid membrane, affinity separation, adsorption, absorption, extraction, chromatography, separation using membranes, and electrochemistry, among other methods. Many of the distillation processes used in industry involve the separation of two or more substances. As a result, the proper understanding of such separation methods is important to achieve the desired degree of purity or components concentration [2]. There are several methods used to model multicomponent distillation and parameters determination, such as approximate and rigorous methods.

In the design of distillation columns is very useful to know the number of plates required to achieve the separation when the product is not removed from the column (total reflux) and the

minimum reflux ratio that can be used to achieve the expected separation. Even the existence of rigorous methods to determine and achieve the separation of multicomponent systems, approximate methods such as Fenske-Underwood-Gilliland, Kremser, Edmister and its variations keep being utilized for several purposes [3], such as preliminary design, parametric studies for optimum design, process simplification to determine optimum separation sequence, information about operating pressure, equilibrium stages or reflux ratio [4]. These methods can be applied using hand calculations when the physical properties are independent of composition, however, as often the calculations involve iterative procedures, it is advisable the use of computational systems [5]

Distillation requires the contact between the liquid and vapor phases, which normally flow countercurrent. This contact allows mass transfer after species distribution and is normally achieved in horizontal trays [6]. The separation of compounds by distillation occurs when the concentration of fluid phases are different from each other at equilibrium [7]. Generally, the gas phase contains the more volatile component. When the vapor flows towards the column, the concentration of the more volatile species increases in this phase, accordingly, as the liquid flows down it is enriched with components of lower volatility [8]. Mathematical

analysis of distillation and absorption process consists of material and energy balances and phase equilibrium relations. Two type of equipment are generally used, tray or packed, stagewise or continuous. Selection of the system produces stepwise changes in concentration or gradual variation concentration. A plate-type of distillation column where a fluid flows down the column is an example of stagewise system [9]. Fluid streams are mixed until thermodynamic equilibrium is reached, then separation is achieved and each phase passes countercurrently to the next stage [10]. Distillation is a separation process that is under optimization particularly in terms of energy consumption as is the most significant energy-consuming process, which can be achieved by appropriate sequencing of the columns [11]. One of the main challenges is that multicomponent systems exhibit transport characteristics completely different from classical and simple binary systems [12].

### 1.1 Importance of feed tray location

The feed of the components to the column is normally carried out in an intermediate plate. The part of the column that is above the feed is named rectification while the bottom section stripping. For binary distillation, it is normally to use the McCabe-Thiele method to determine the number of theoretical stages or trays that are necessary to achieve a desired degree of components separation. This method requires the knowledge of temperature at the feed, initial component concentrations, distillate and bottom concentration and information about reflux ratio. In the case of multicomponent distillation it is necessary to obtain information about the concentration of each and every component on each tray using methods like Lewis-Matheson or Thiele-Geddes equilibrium stage method. When the feed is located in an upper zone the reflux increases and as a result the condenser load also increases because the feed is for longer time in contact with the upcoming vapour. In the other case, if the feed tray is in a lower position the feed will be in contact with the upcoming vapour for less amount of time and reboiler activity will have to increase [13].

The study of multicomponent distillation process require the solution of system of equations developed after mass balance, equilibrium, enthalpy balance, molar fraction analysis were classic methods developed after Lewis-Matheson and Thiele-Geddes reports in conventional column studies. Relaxation methods are also used, in the case that time dependent mass balance is performed

but this method requires more time to provide a steady state solution. Lewis-Matheson method requires difference equation and initial assumptions of column variables and the Thiele-Geddes method utilizes ratios of flow rates to obtain modified form of the difference equations and requires fewer amounts of initial assumptions. In many cases, the design of the multi-stage equipment requires a rigorous determination of parameters, i.e. temperature, pressure, flows and compositions of streams. Given the high no linearity of the equations it is necessary to solve the system by using iterative procedures, where the methods differ in the selection of the set of independent variables. The mathematical model of such system normally requires mass balance for each component, enthalpy balance, and phase equilibrium relations. In the present paper we aim to use approximate methods to determine the minimum reflux ratio, amount of theoretical zones and feed location in the process of distillation. Additionally, we determine liquid composition at dew point, composition of bottoms and distillate. Determination of dew point is important to ensure proper separation of the components and determine composition of each substance.

## 2 Description of the system

We start the analysis considering that  $C_5H_{12}$  will be mainly recovered in the distillate (95%) and  $C_6H_{12}$  will remain in the bottoms (95%). It is necessary to perform a mass balance for each component, ( $F = D + W$ ), where F is the feed, D is the distillate and W the bottoms fraction. For  $C_5H_{12}$  we obtain

$$x_{B_F} \cdot F = 0.28(100) = 28 \quad (1)$$

$$x_{B_F} \cdot F = y_{B_D} \cdot D + x_{B_W} \cdot W \quad (2)$$

Taking into consideration that 95% of  $C_5H_{12}$  is distilled we determine  $y_{B_D} \cdot D = 0.95(28) = 26.6$ . Therefore,  $x_{B_W} \cdot W = 0.05(28) = 1.25$ . For  $C_6H_{14}$  we would obtain the next values:

$$x_{C_F} \cdot F = 0.18(100) = 18 \quad (3)$$

$$x_{C_F} \cdot F = y_{C_D} \cdot D + x_{C_W} \cdot W \quad (4)$$

$$y_{C_D} \cdot D = 0.05(18) = 0.9 \quad (5)$$

$$x_{C_W} \cdot W = 0.95(18) = 17.1 \quad (6)$$

We assume that there is no  $C_7H_{14}$  in the distillate and no  $C_4H_{10}$  in the bottom. As a result, we can obtain the flow of  $C_4H_{10}$  and  $C_7H_{16}$  in distillate and bottoms respectively and the composition of distillate and bottoms as described next

$$y_{A,D} \cdot D = 0.4(100) = 40 \quad (7)$$

$$x_{D,W} \cdot W = 0.15(100) = 15 \quad (8)$$

**Table 1.** Characterization of distillate and bottoms.

component	Feed		Distillate, D		Bottoms, W	
	$x_F \cdot F$	$x_F$	$y_D \cdot D$	$y_D = x_D$	$x_W \cdot W$	$x_W$
	(mol/h)		(mol/h)		(mol/h)	
$C_4H_{10}$	37	0.37	37	0.5736	0	0
$C_5H_{12}$	28	0.28	26.6	0.4124	1.4	0.04
$C_6H_{14}$	18	0.18	0.9	0.014	17.1	0.481
$C_7H_{16}$	17	0.17	0	0	17	0.478
total	100	1	64.5	1	35.5	1

In order to determine the dew point of the distillate (top temperature) we use an iterative method with an initial guess of  $67^\circ\text{C}$ . Then we proceed to determine  $k_i$ ,  $\alpha_i$ , and  $K_C$  values for each component. The equilibrium  $K$  values for light hydrocarbons ( $C_4H_{10}$  to  $C_{10}H_{22}$ ) at 405.3 kPa have been determined semi-empirically and therefore can be evaluated. The parameters  $\delta$ ,  $\beta$  and  $\gamma$  correspond to constant values provided in Table 2 [14].

$$k = e^{-\delta/T^2 + \beta - \gamma} \quad (9)$$

$$\alpha_i = \frac{k_i}{k_{ref}} \quad (10)$$

$$K_C = \sum \left( \frac{y_i}{\alpha_i} \right) \quad (11)$$

Then, a new value of temperature is obtained using the value of  $K_C$  determined previously, and is compared to the assumed temperature value. If both values are equal, the dew point is determined at this value, if not, the temperature is used to find new set of  $k_i$ ,  $\alpha_i$ , and  $K_C$  values and a new value of temperature is calculated with these parameters, until both values match each other. Then, the composition in the liquid is calculated using equation

$$x_i = \frac{y_i}{\alpha_i \sum (y_i/\alpha_i)} \quad (12)$$

**Table 2.** Constant values to be used in the present system [14].

component	$\delta$	$\beta$	$\gamma$
$C_4H_{10}$	1280557	7.94986	0.96455
$C_5H_{12}$	1524891	7.33129	0.89143
$C_6H_{14}$	1778901	6.96783	0.84634
$C_7H_{16}$	2013803	6.52914	0.79543

**Table 3** Composition of distillate and determination of  $k$ ,  $\alpha$  and  $y_D/\alpha$  values.

component	$y_D$	$k$	$\alpha$	$y_D/\alpha$
$C_4H_{10}$	0.5736	1.8072	6.223	0.0928
$C_5H_{12}$	0.4124	0.684	2.355	0.175
$C_6H_{14}$	0.01395	0.29	1	0.0139
$C_7H_{16}$	0	0.1232	0.424	0

**Table 4.** Example of subsequent iteration procedure.

component	T( $^\circ\text{C}$ )	$k$	$\alpha$	$y_D/\alpha$	$K_C$
$C_4H_{10}$	65.85	1.765	6.27	0.0913	0.279
$C_5H_{12}$		0.665	2.360	0.1742	
$C_6H_{14}$		0.281	1.000	0.0139	
$C_7H_{16}$		0.118	0.422	0	
$C_4H_{10}$	65.64	1.758	6.289	0.0912	0.279
$C_5H_{12}$		0.662	2.368	0.1741	
$C_6H_{14}$		0.279	1.000	0.0139	
$C_7H_{16}$		0.118	0.422	0.000	
$C_4H_{10}$	65.6	1.757	6.29	0.091	0.279
$C_5H_{12}$		0.661	2.36	0.174	
$C_6H_{14}$		0.279	1.000	0.0139	
$C_7H_{16}$		0.118	0.422	0	

**Table 5.** Liquid Composition at dew point.

T(°C)	component	$y_i / [\alpha_i \sum (y_i / \alpha_i)]$
65.6	$C_4H_{10}$	0.3265
	$C_5H_{12}$	0.6235
	$C_6H_{14}$	0.0499
	$C_7H_{16}$	0

**Table 6.** Composition of bottoms and determination of  $k$ ,  $\alpha$  and  $x_W \cdot \alpha$  values.

component	$x_W$	$k$	$\alpha$	$x_W \cdot \alpha$	$K_C$
$C_4H_{10}$	0.00000	5.01	4.18	0.00	1.2459
$C_5H_{12}$	0.0394	2.30	1.92	0.075	
$C_6H_{14}$	0.4816	1.19	1.00	0.4816	
$C_7H_{16}$	0.4788	0.61	0.51	0.245	

**Table 7.** Example of subsequent iteration procedure.

component	T(°C)	$k$	$\alpha$	$x_W \cdot \alpha$	$K_C$
$C_4H_{10}$	135.4	5.156	4.130	0.000	1.2446
$C_5H_{12}$		2.383	1.913	0.075	
$C_6H_{14}$		1.24	1.000	0.481	
$C_7H_{16}$		0.648	0.514	0.246	
$C_4H_{10}$	135.36	5.152	4.138	0.000	1.244
$C_5H_{12}$		2.381	1.913	0.075	
$C_6H_{14}$		1.246	1.000	0.481	
$C_7H_{16}$		0.641	0.514	0.246	

**Table 8.** Liquid Composition at bottom of tower.

T(°C)	component	$x_W \cdot \alpha / [\sum (x_W \cdot \alpha)]$
135.36	$C_4H_{10}$	0
	$C_5H_{12}$	0.094
	$C_6H_{14}$	0.60
	$C_7H_{16}$	0.307

In order to determine the minimum theoretical stages ( $N_M$ ) required for total reflux we refer to Fenske equation as expressed next [15]:

$$N_M = \frac{\log [x_{Dl} \cdot x_{Wh} / x_{Dh} \cdot x_{Wl}]}{\log \left( \frac{\alpha_{Dl}}{\alpha_{Wl}} \right)} \quad (13)$$

$x_{Dl}$  refers to the mole fraction in the light phase in distillate,  $x_{Wl}$  is the mole fraction in the bottom,  $x_{Dh}$  is the mole fraction of heavy phase in distillate and  $x_{Wh}$  the fraction mol of heavy phase in the bottom.

$$N_M = \frac{\log [0.4124 \cdot 0.4816 / 0.01395 \cdot 0.03943]}{\log \left( \frac{2.369}{1.914} \right)} = 7.8 \quad (14)$$

**Table 9.** Component distribution. Determination of traces.

component	$\alpha_D$	$\alpha_W$	$\sqrt{\alpha_W \alpha_D}$
$C_4H_{10}$	6.292	4.139	5.103
$C_5H_{12}$	2.369	1.914	2.129
$C_6H_{14}$	1.000	1.000	1
$C_7H_{16}$	0.422	0.514	0.466

For  $C_4H_{10}$

$$\frac{D \cdot x_{C_4H_{10}D}}{W \cdot x_{C_4H_{10}W}} = \left( \sqrt{\alpha_W \alpha_D} \right)^{N_M} \frac{D \cdot x_{Dh}}{W \cdot x_{Wh}} \quad (15)$$

$$\frac{D \cdot x_{C_4H_{10}D}}{W \cdot x_{C_4H_{10}W}} = (5.103)^{7.79} \frac{64.75 \cdot 0.01395}{35.5 \cdot 0.481} \quad (16)$$

$$\frac{D \cdot x_{C_4H_{10}D}}{W \cdot x_{C_4H_{10}W}} = 17318.1 \quad (17)$$

The overall balance on  $C_4H_{10}$  leads to  $x_{C_4H_{10}F} \cdot F = x_{C_4H_{10}D} \cdot D + x_{C_4H_{10}W} \cdot W$ . Thus, the next equations are obtained:

$$x_{C_4H_{10}F} \cdot F = 17318 \cdot W \cdot x_{C_4H_{10}W} + x_{C_4H_{10}W} \cdot W \quad (18)$$

$$37 = 17318 \cdot W \cdot x_{C_4H_{10}W} \quad (19)$$

$$W \cdot x_{C_4H_{10}W} = 0.0021 \text{ mol} \quad (20)$$

$$x_{C_4H_{10}D} \cdot D = 17318 \cdot W \cdot x_{C_4H_{10}W} \quad (21)$$

$$x_{C_4H_{10}D} \cdot D = 36.99 \quad (22)$$

For  $C_7H_{14}$ :

$$(0.463)^{7.8} \frac{D \cdot x_{C_7H_{14}D}}{W \cdot x_{C_7H_{14}W}} = (\sqrt{\alpha_W \alpha_D})^{N_M} \frac{D \cdot x_{Dh}}{W \cdot x_{Wh}} = 0.000137 \quad (23)$$

The overall balance on  $C_7H_{14}$  leads to  $x_{C_7H_{14}F} \cdot F = x_{C_7H_{14}D} \cdot D + x_{C_7H_{14}W} \cdot W$

$$x_{C_7H_{14}F} \cdot F = D \cdot x_{C_7H_{14}D} + x_{C_7H_{14}W} \cdot W \quad (24)$$

$$17 = 1.00018 \cdot W \cdot x_{C_7H_{14}W} \quad (25)$$

$$W \cdot x_{C_7H_{14}W} = 16.997 \text{ mol} \quad (26)$$

$$x_{C_7H_{14}D} \cdot D = 0.000036 \cdot W \cdot x_{C_7H_{14}W} \quad (27)$$

$$x_{C_7H_{14}D} \cdot D = 0.0023 \quad (28)$$

Therefore, the new composition is as described in Table 10

**Table 10.** Characterization of distillate and bottoms.

compound	Distillate, D		Bottoms, W	
	$y_D = x_D$	$x_D \cdot D$ (mol/h)	$x_W$	$x_W \cdot W$ (mol/h)
$C_4H_{10}$	0.5736	36.9979	0.0001	0.0021
$C_5H_{12}$	0.4124	26.6	0.0394	1.400
$C_6H_{14}$	0.014	0.9	0.4817	17.100
$C_7H_{16}$	0.000036	0.0023	0.478	16.997
Total	1.0000	64.50	1.000	35.500

This shows that the traces are  $x_{C_7H_{14}D} = 3.6 \times 10^{-5}$  and  $x_{C_7H_{14}W} = 1 \times 10^{-4}$

The Underwood method is used to determine the minimum reflux ratio. In this method it is required to consider all the components under the conditions of minimum reflux [16]. However, it is important to consider also different alternatives for the determination of minimum reflux ratio, such as exact methods developed previously by Tavana and Hanson [17]. The equations used to calculate the minimum reflux ratio are described in (29) and (30) where  $q=1$  when feed is at boiling point

$$1 - q = \sum \frac{\alpha_i \cdot x_{iF}}{\alpha_i - \theta} \quad (29)$$

$$R_m + 1 = \sum \frac{\alpha_i \cdot x_{iD}}{\alpha_i - \theta} \quad (30)$$

$$T = (T_{top} + T_{bottom})/2 = \frac{65.6+135.36}{2} = 100.48 \quad (31)$$

At this temperature, values of  $k$  and  $\alpha$  are determined, as shown in Table 11.

**Table 11.** Determination of  $\alpha$  and  $x_D$  values required for Underwood method.

component	$x_F$	$k(98.8^\circ\text{C})$	$\alpha$	$x_D$
$C_4H_{10}$	0.370	3.243	4.956	0.574
$C_5H_{12}$	0.280	1.373	2.098	0.412
$C_6H_{14}$	0.180	0.654	1.000	0.014
$C_7H_{16}$	0.170	0.309	0.472	0.000036

$$1 - 1 = \frac{4.956 \cdot 0.37}{4.956 - \theta} + \frac{2.098 \cdot 0.28}{2.098 - \theta} + \frac{1 \cdot 0.18}{1 - \theta} + \frac{0.472 \cdot 0.17}{0.472 - \theta} \quad (32)$$

$$0 = \frac{1.8337}{4.956 - \theta} + \frac{0.58744}{2.098 - \theta} + \frac{0.18}{1 - \theta} + \frac{0.08024}{0.472 - \theta} \quad (33)$$

Using an algorithm developed to solve the previous equation using implicit method, the values of  $\theta$  that satisfy the equation are 0.5398 and 1.1789. However, the real value has to be between  $\alpha_{C_5H_{12}}$  and  $\alpha_{C_6H_{14}}$ , therefore, the value corresponds to  $\theta = 1.1789$ . As a result, the minimum reflux ratio is given by

$$R_m = \frac{4.956 \cdot 0.574}{4.956 - 1.1789} + \frac{2.098 \cdot 0.412}{2.098 - 1.1789} + \frac{1 \cdot 0.014}{1 - 1.1789} + \frac{0.472 \cdot 0.000036}{0.472 - 1.1789} - 1 \quad (34)$$

$$R_m = 0.6153 \quad (35)$$

We then proceed to determine the number of theoretical stages at different operational reflux ratio, when  $R = 1.5R_m = 1.5 \cdot 0.6153 = 0.923$ . Using the Erbar-Maddox correlation, we have

$$\frac{R}{R+1} = \frac{0.923}{1+0.923} = 0.4799 \quad (36)$$

$$\frac{R_m}{R_m+1} = \frac{0.6153}{1+0.6153} = 0.381 \quad (37)$$

Following, it is possible to estimate the requirements in terms of number of ideal stages required using empirical correlation methods like Erbar and Maddox, which describes the ratio of stages required to the total reflux, as a function of the reflux ratio, considering the minimum reflux ratio as a parameter. Hence,  $N_M/N = 0.6$ . Following,  $N = N_M/0.6 = 7.8/0.6 = 13$  theoretical

stages. Feed point location can be estimated considering Kirkbride method.

$$\log \frac{N_e}{N_s} = 0.206 \log \left[ \left( \frac{x_{HF}}{x_{LF}} \right) \frac{W}{D} \left( \frac{x_{LW}}{x_{HD}} \right)^2 \right] \quad (38)$$

$$\log \frac{N_e}{N_s} = 0.206 \log \left[ \left( \frac{0.18}{0.28} \right) \frac{35.5}{64.5} \left( \frac{0.0394}{0.014} \right)^2 \right] \quad (39)$$

$$\log \frac{N_e}{N_s} = 0.092189 \quad (40)$$

$$\frac{N_e}{N_s} = 1.23 \quad (41)$$

$N_e = 1.23 \cdot N_s$ ;  $N = N_e + N_s = (13)1.23 = 2.23N_e$ ;  $N_e = 7.17$  and  $N_s = 5.82$ . Therefore, the feed tray is located at 7.17 stages from top.

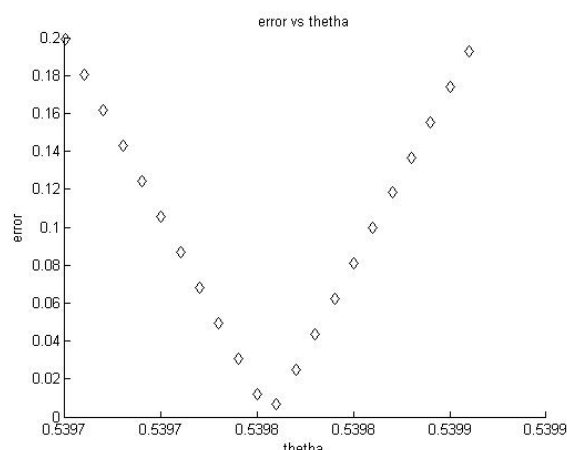
### 3 Algorithm design

An example of the algorithm developed in Matlab software that was used to solve the equation (33) using implicit method to find the values of  $\theta$ :

```

alfaA=4.956;
xA=0.37;
alfaB=2.098;
xB=0.28;
alfaC=1;
xC=0.18;
alfaD=0.472;
xD=0.17;
ymax=0.7;
ystep=0.00001;
ypoints=ymax/ystep;
for j = 1:ypoints;
    y(j) =(j-1)*ystep;
    IZQ(j)=1-(alfaA*xA/(alfaA-y(j)))+(alfaB*xB/(alfaB-
y(j)))+(alfaC*xC/(alfaC-y(j)))+(alfaD*xD/(alfaD-
y(j)));
    DER=1;
    error(j)= abs(((IZQ(j)-DER)/DER)*100);
    if error(j)<0.2;
        err=error(j)
        yfound=(y(j))
        figure(1)
        hold on
        plot (yfound,err,'kd')
        title('error vs thetha')
        xlabel('thetha')
        ylabel('error')
    end
end
end

```



**Fig. 1.** Example of the output obtained from the algorithm used to solve the equation (33) using implicit method to find one possible values of  $\theta$

### 4 Conclusion

As described previously, dew point can be evaluated after selecting a component to be the reference component, which in the present case was hexane and assuming an initial value of temperature and the values for relative volatility are determined at the corresponding  $K$  values for the selected temperature. Then, the value of  $K$  for the reference compound is determined after evaluating  $K = 1/\sum \alpha_i x_i$ . The calculated value of temperature is compared to the assumed temperature value. Multicomponent distillation problems can be solved manually by means of tray-by-tray calculations of heat and material balances and vapor-liquid equilibrium. Alternative methods have been described widely in literature and are showed in the present paper. Data obtained using these methods provide information that can be used in iterative methods for numerical analysis. It is important to consider that the design of equipment to be used in the industry is complemented with formulations that include theory of binary mass transfer and applied to multicomponent systems in matrix forms. This has also been included in computational algorithms taking into account simultaneous heat transfer effects. These advanced models have been incorporated to design software for mass and heat transfer process like distillation, absorption, extraction, and condensation. After the characterization of light and heavy phases, determination of component distribution, tower temperature at top and bottom, we used approximate methods to determine parameters such as minimum

reflux ratio, amount of theoretical zones and feed location.

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