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## RESEARCH NOTES

## Drawbacks of the Dissolution Method for Measurement of the Liquid–Solid Mass-Transfer Coefficients in Two-Phase Flow Packed-Bed Reactors Operated at Low and High Pressures

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The dissolution method was used predominantly at atmospheric conditions to measure the liquid–solid mass-transfer coefficient in two-phase flow packed-bed reactors. In an attempt to investigate the effect of reactor pressure and gas flow rate on the liquid–solid mass-transfer coefficient in a trickle-bed reactor using the dissolution method, difficulties were encountered in implementing this method at low- to high-pressure operation. Noticeable losses in the dissolved material were observed as a result of flashing/stripping from the liquid phase to the atmosphere and the effluent gas stream. This leads to misinterpretation of the effect of reactor pressure and to improper conclusions. In this note, we intend to alert researchers about such drawbacks, which would be encountered at atmospheric to low pressures and to provide suggestions to overcome such problems.

### Introduction

Packed-bed reactors are catalyst beds contacted by cocurrent downflow (trickle beds), cocurrent upflow (packed bubble beds), or countercurrent flow of gas and liquid. They are used widely in various processes in the petroleum, petrochemical, chemical, biochemical, and environmental industries. Packed-bed reactors, particularly trickle beds, are usually operated at elevated pressure (up to about 30 MPa). The liquid–solid mass-transfer coefficient is one of the most important parameters for design, scale-up, and performance characterization of these reactors.

A variety of methods have been used in numerous studies to measure liquid–solid mass-transfer coefficients in two-phase flow packed-bed reactors (e.g., dissolution, electrochemical, chemical reaction, dynamic adsorption, and ion exchange followed by instantaneous reaction).<sup>1,2</sup>

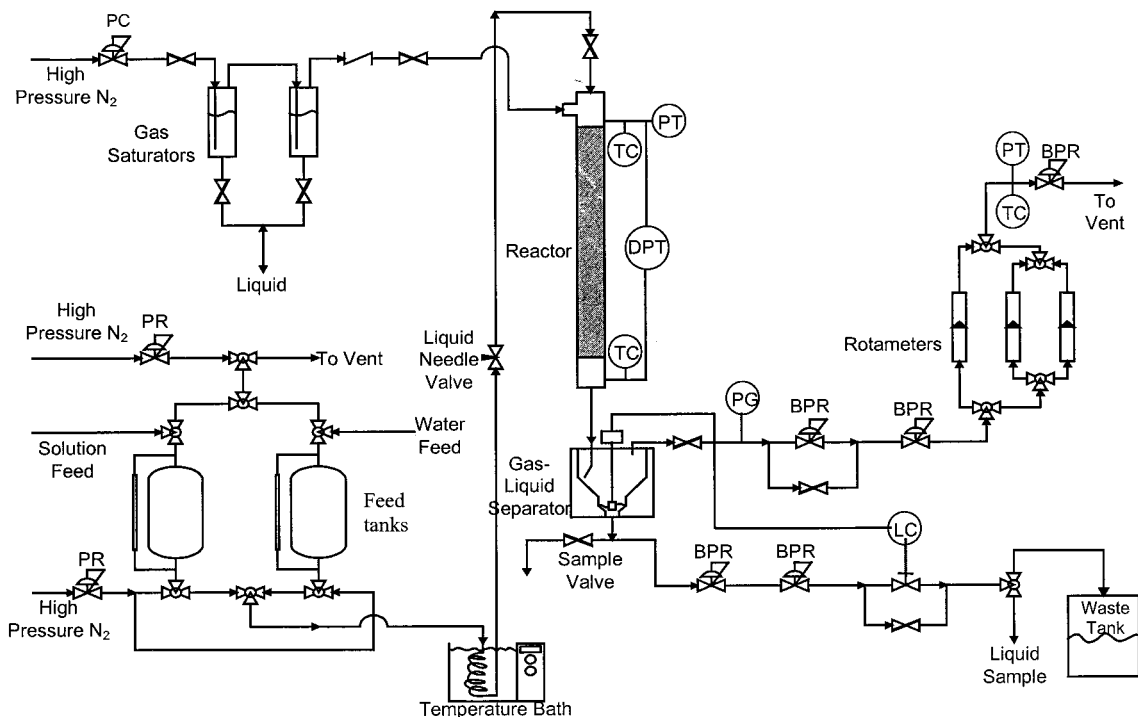
Dissolution and electrochemical methods predominantly are used to measure liquid–solid mass-transfer coefficients. The dissolution method measures an average volumetric liquid–solid mass-transfer coefficient ( $k_{ls}a$ ) for an entire bed or for a section of a bed based on the liquid saturation limit of the dissolved material (i.e., integral measurement), while the electrochemical method measures the local liquid–solid mass-transfer coefficient ( $k_{ls}$ ) for a single particle in a bed (i.e., local measurement). In addition, the dissolution method has been used to measure liquid–solid wetting efficiency in two-phase flow packed beds.<sup>3</sup>

Despite the operating condition of interest being high pressure, all the previous investigations on the liquid–solid mass-transfer coefficient were performed under atmospheric conditions.<sup>4</sup> Few studies have been conducted to investigate and to predict the effect of reactor pressure on pressure drop, liquid holdup, wetting efficiency, gas–liquid interfacial area, and flow regime transition in trickle-bed and packed bubble-bed reactors.<sup>5</sup> Recently, HighFill and Al-Dahhan<sup>2</sup> and HighFill<sup>1</sup> studied the effect of high pressure on the liquid–solid mass-transfer coefficient ( $k_{ls}$ ) in a trickle-bed reactor using the electrochemical method. The cathode particle was placed in the center of the middle section of the bed. They found that  $k_{ls}$  increases with the increase in pressure and in the gas flow rate. The effects of the reactor pressure and gas flow rate were described on the basis of the analysis of five limited cases presented by Al-Dahhan and Dudukovic<sup>6,7</sup> and Al-Dahhan et al.<sup>5</sup> Because, as mentioned earlier, the electrochemical method provides a local measurement of  $k_{ls}$ , it is of interest to examine the effects of reactor pressure and gas flow rate on the mass transfer measured in a section of the bed or in the entire bed using the dissolution method.

Therefore, in this study the dissolution method is utilized in an attempt to investigate the effect of reactor pressure and gas flow rate on the  $k_{ls}a$  on a section of the bed and in the entire bed. Unfortunately, difficulties were encountered in implementing the dissolution method measurements in a trickle-bed reactor operated at low to high pressures, which can lead to misinterpretation of the effect of reactor pressure and to improper conclusions. Because the current trend of research on the two-phase flow packed beds is at elevated pressure (the operating conditions of interest to that number of industrial applications), the purpose

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**Figure 1.** Schematic diagram of the high-pressure trickle-bed reactor facility. PR, pressure regulator; BPR, back-pressure regulator; LC, level control; TC, thermocouple; PT, pressure transducer; DPT, differential pressure transducer.

of this note is to alert researchers about such drawbacks that would be encountered as well at atmospheric to low pressures and to provide suggestions to address such problems.

### Experimental Work

The dissolution method employs particles made from a solid material that is sparingly soluble in the liquid phase to prevent the change in the bed characteristics during the measurement. Different types of packing material have been used, such as benzoic acid,<sup>8–14</sup> benzoic acid mixed with a fluorescent dye,<sup>15</sup> naphthalene,<sup>3,16</sup> and  $\beta$ -naphthal.<sup>17</sup> During the dissolution method one must be careful not to saturate the liquid exiting the column. To avoid this, previous investigators have either used short beds consisting of only active particles (dissolved particles), or used longer beds of inert particles with a short section of active particles packed within. In addition, the active particles can be dispersed along the bed within the inert particles in an amount equivalent to a short section required to maintain the dissolved material under 60%–75% of the liquid saturation limit.<sup>1</sup>

Assuming plug flow and performing steady-state mass balance, yield the following equation to evaluate  $k_{1s}a$ ,

$$k_{1s}a = \frac{Q}{AZ} \ln \left( \frac{C_{A,\text{sat}}^L - C_{A,\text{in}}^L}{C_{A,\text{sat}}^L - C_{A,\text{out}}^L} \right)$$

where  $Q$  is the volumetric liquid flow rate ( $\text{m}^3/\text{s}$ ),  $A$  is the bed cross-sectional area ( $\text{m}^2$ ),  $Z$  is the packed-bed length (m),  $C_{A,\text{sat}}^L$  is the saturation concentration of the dissolved material,  $C_{A,\text{in}}^L$  is the concentration of the dissolved material in the inlet flow, and  $C_{A,\text{out}}^L$  is the concentration of the dissolved material in the outlet flow. Usually,  $C_{A,\text{in}}^L$  is equal to zero. Hence, when  $Q$  and  $C_{A,\text{out}}^L$  are measured,  $k_{1s}a$  can be evaluated.

In this work, naphthalene as the packing material and a system of water–nitrogen were used to measure  $k_{1s}a$ . A stainless steel mold was designed and constructed to manufacture  $0.32 \times 0.32$  cm ( $1/8 \times 1/8$  in.) cylindrical naphthalene particles using a heat press.<sup>1</sup>

Figure 1 shows the high-pressure trickle-bed reactor facility<sup>2,6,7</sup> that was used to measure  $k_{1s}a$  by a dissolution method. The facility can be operated up to 7 MPa (1000 psi). It consists of the reactor and gas–liquid distributor, the liquid delivery system, the gas delivery system, the separator, the gas and liquid exit systems, and the data acquisition system. The reactor used was 2.2 cm in diameter and 50 cm in height. The height of the bed packed with naphthalene cylindrical particles ( $0.32 \times 0.32$  cm) was  $\sim 3.2$  cm (1.25 in.), which resulted in an outlet concentration of 25%–75% of the saturation level for the range of liquid and gas flow rates used. This active bed was either placed in the middle height of the reactor or dispersed along the bed. For the former case, the bed below and above the naphthalene particles was packed with  $0.32 \times 0.32$  cm ( $1/8 \times 1/8$  in.) cylindrical glass particles.

Samples from the reactor outlet stream were drawn from the separator via a 0.16 cm ( $1/16$  in.) needle valve mounted on a 0.16 ( $1/16$  in.) tubing as shown in Figure 1. A UV spectrophotometer (Bausch & Lomb Spectronic 21 model U.V.-D) was used to measure the concentration of naphthalene in water. For the range of light necessary for naphthalene (ultraviolet region), quartz cuvettes were utilized. It was found that a wavelength of 274 nm gives the maximum absorbency<sup>1,3</sup> and, hence, it was used for the measurement of naphthalene concentration in water.

Table 1 shows the experimental conditions used to measure  $k_{1s}a$  by dissolution of naphthalene particles that represent the trickle flow regime.<sup>7,18,19</sup>

**Table 1. Operating Conditions and Bed Characteristics**

system	water–nitrogen
liquid velocity	0.036–0.28 cm/s
gas velocity	1.0–7.9 cm/s
pressure	0.31–3.55 MPa (30–500 psig)
bed diameter	2.2 cm
cross-sectional area of the bed	3.8 cm <sup>2</sup>
bed length	49.7 cm
equivalent active bed, height of naphthalene	3.2 cm
bed porosity	0.33
average naphthalene particle size	0.33 cm (diameter) × 0.43 cm
average glass particle size	0.25 cm (diameter) × 0.31 cm

## Results and Discussion

At all pressures, the measured  $k_{\text{LS}}a$  values increase with the increase in liquid flow rate as shown in Figure 2 a,b. All of the previous studies showed such a trend. At constant pressure and liquid flow rate,  $k_{\text{LS}}a$  increases with the gas flow rate for the pressures investigated. This trend of  $k_{\text{LS}}a$  with gas velocity coincides with the findings reported in the literature.<sup>2,3,9,11,13–15,20–22</sup> However, in this work,  $k_{\text{LS}}a$  values obtained at 30 psig (0.31 MPa) are larger than those obtained at 500 psig (3.55 MPa) where the gas density is larger (see Figure 2a,b). This is not consistent with the findings of HighFill and Al-Dahhan<sup>2</sup> where the  $k_{\text{LS}}a$  measured by the electrochemical method increases with the reactor pressure at constant liquid and gas flow rates and with the trend of the effect of reactor pressure on particle wetting efficiency, liquid holdup and pressure drop. In addition, Bliss,<sup>14</sup> in experiments that covered trickle and pulse flow regimes, found that  $k_{\text{LS}}a$  measured by dissolution of benzoic acid particles increased with gas velocity and gas density using helium, nitrogen, and argon gases.

It was found that the measured outlet naphthalene concentration decreases as the pressure increases at fixed flow rates of gas and liquid. Figure 3 shows that the measured molar flow of the naphthalene,  $Q_{\text{L}}C_{\text{L}}^{\text{exp}}$  (mol/s), decreases with pressure. Such a decrease is larger at higher liquid flow rates where more naphthalene is dissolved. It is obvious from eq 1 that if  $C_{\text{A,out}}^{\text{L}}$  decreases at constant liquid and gas flow rates, then the evaluated value of  $k_{\text{LS}}a$  decreases.

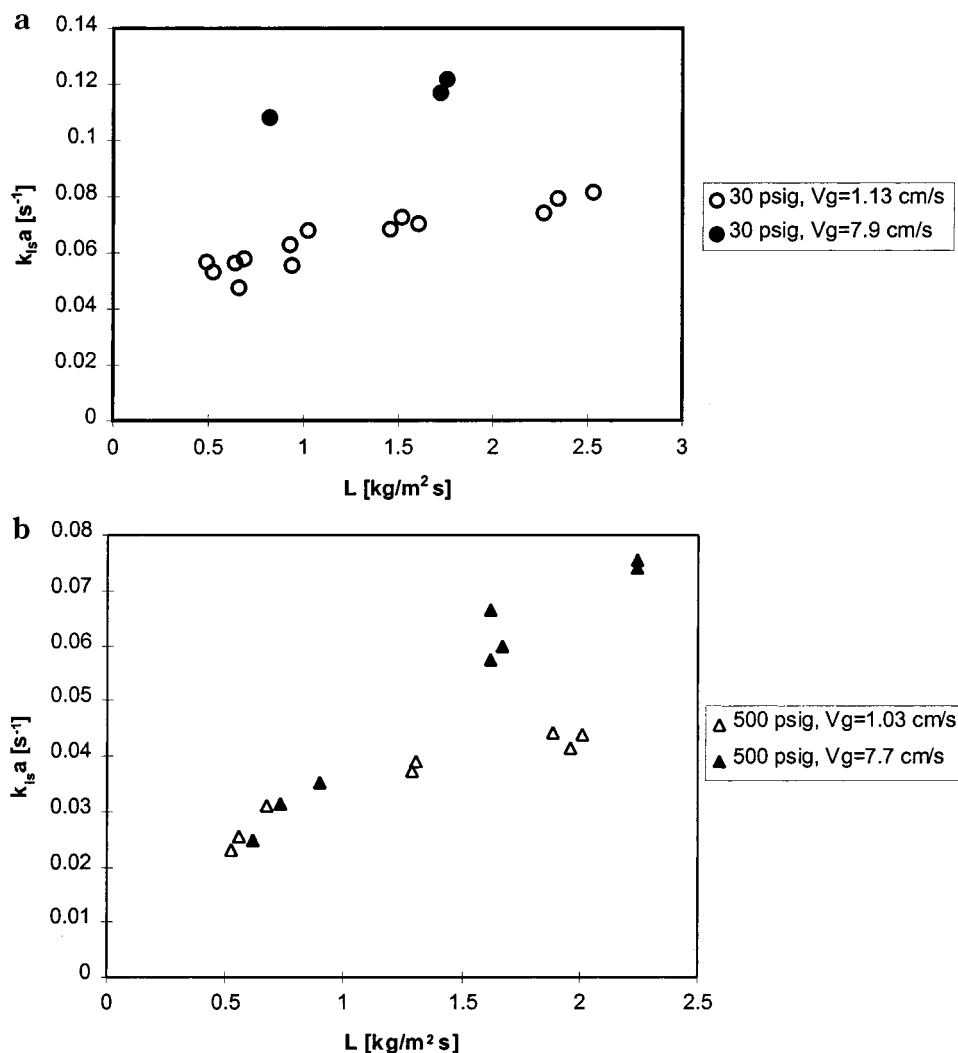
It is believed and confirmed experimentally in the following section that the reason for the decrease in the naphthalene concentrations with pressure is that the naphthalene is being flashed/stripped from the liquid phase during the sampling process. As shown in Figure 1, the samples were drawn from the high-pressure gas–liquid separator (which is under the same pressure of the reactor) via a  $1/16$  in. needle valve that is opened to the atmospheric conditions (room environment). Therefore, the drop in the pressure across the sampling valve caused flashing, thereby inducing naphthalene to transfer from the liquid to the atmosphere. As the reactor pressure was increased, the pressure drop across the sampling valve was increased, causing more flashing and hence more naphthalene transfers from the liquid to the atmosphere. Therefore, at higher reactor pressures, a lower liquid effluent concentration was found at constant liquid and gas flow rate, which yields a lower  $k_{\text{LS}}a$  value according to eq 1. Because the molar flow rates of naphthalene in the saturated air ( $Q_{\text{g}}C_{\text{g}}^*$ ) and liquid ( $Q_{\text{L}}C_{\text{L}}^*$ ) at the operating conditions used are comparable and of the same order of magnitude as shown in Table 2,<sup>23</sup> there would be a possibility of the

naphthalene to be stripped from the liquid phase inside the reactor and separator and from the collected samples to the atmosphere. A similar case would happen to the benzoic acid as shown in Table 2 where the estimated  $Q_{\text{g}}C_{\text{g}}^*$  is of an order of magnitude larger than the estimated  $Q_{\text{L}}C_{\text{L}}^*$ . To confirm experimentally the loss of the dissolved material to the atmosphere and/or to the gas effluent stream by flashing/stripping, the following test was performed.

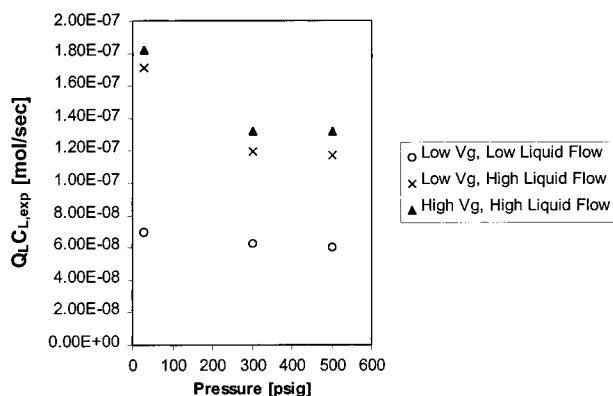
**Experimental Confirmation for the Loss of the Dissolved Material.** The same facility described earlier (Figure 1) has been used to quantitatively characterize the loss of the dissolved naphthalene and benzoic acid by flashing/stripping. In this experimental test, no active particles (i.e., naphthalene or benzoic acid particles) were used. Instead, the reactor was packed only with  $0.32 \times 0.32$  cm ( $1/8 \times 1/8$  in.) cylindrical glass beads. Solutions with known concentrations of the naphthalene and another one with benzoic acid were prepared and charged into the feed tanks. The setup was operated using the same operating procedure for measurement of the liquid–solid mass-transfer coefficient. The concentrations of naphthalene (when naphthalene solution was used) and benzoic acid (when benzoic acid solution was used) in the feed tank were measured for each operating condition used. As mentioned earlier, the samples were analyzed using a Bausch & Lomb UV spectrophotometer at wavelengths of 274 nm for naphthalene and 268 nm for benzoic acid concentrations.

At steady-state operating conditions (i.e., maintaining for a few minutes for constant system pressure, gas and liquid flow rates, and liquid level in the separator), samples were drawn from the sampling valve connected to the separator (Figure 1) where the concentrations were measured by the UV spectrophotometer. The differences between the measured concentrations at the exit of the reactor (withdrawn from the gas/liquid separator) and in the feed tank represent the loss that occurred for the dissolved material by flashing/stripping in the reactor and separator and to the atmosphere via the sampling procedure. The reproducibility of the measurements has been evaluated and found to be within  $\pm 3\%$ .

Tables 3 and 4 summarize the experimental results obtained for the naphthalene and benzoic acid experiments, respectively. It is obvious that the outlet concentrations of the dissolved materials decreases as the reactor pressure increases. It is noteworthy that the percentage differences for benzoic acid concentrations are larger than those of naphthalene despite the percentage saturation of the benzoic acid solution being much lower than that of the naphthalene solution in the feed tank. This is due to the differences in solubility, vapor pressure, and Henry's law constant as indicated in Table 2. Moreover, a noticeable decrease in the concentrations was observed, even at low pressures (30 psig), particularly for benzoic acid solution. This indicates that some losses of the dissolved benzoic acid to the effluent stream would occur, even at atmospheric pressure and to room atmosphere where most of the previous studies were performed (as mentioned earlier) using benzoic acid particles to measure the liquid–solid mass-transfer coefficient at atmospheric pressure. In all these studies, no attempt was performed to a priori characterize whether there is a noticeable loss of the dissolved material to account for. On the basis of the results of this work, such an evaluation should be



**Figure 2.** (a) Volumetric liquid–solid mass-transfer coefficients ( $k_{L,a}$ ) vs superficial liquid mass velocity ( $L$ ), measured at 30 psig (0.31 MPa) operating pressure. (For high superficial gas velocity ( $V_g$ ) data:  $V_g = 7.9$  cm/s, bed voidage ( $\epsilon_B$ ) = 0.33, bed height ( $Z$ ) = 49.7 cm, equivalent active section length = 3.2 cm. For low  $V_g$  data:  $V_g = 1.13$  cm/s,  $\epsilon_B = 0.33$ ,  $Z = 49.7$  cm, equivalent active section length = 3.2 cm). (b) Volumetric liquid–solid mass-transfer coefficients ( $k_{L,a}$ ) vs superficial liquid mass velocity ( $L$ ), measured at 500 psig (2.17 MPa) operating pressure. (For high  $V_g$  data:  $V_g = 7.7$  cm/s,  $\epsilon_B = 0.33$ ,  $Z = 49.7$  cm, equivalent active section length = 3.2 cm. For low  $V_g$  data:  $V_g = 1.03$  cm/s,  $\epsilon_B = 0.33$ ,  $Z = 49.7$  cm, equivalent active section length = 3.2 cm).



**Figure 3.** Molar flow rate of naphthalene in the liquid-phase found during the dissolution experiments.

performed for each operating condition used whether the operation is at elevated or atmospheric pressure.

### Concluding Remarks

According to these findings, careful consideration needs to be given to the problems discussed above in implementing the dissolution method for measuring the

liquid–solid mass-transfer coefficient and catalyst wetting efficiency in two-phase flow packed beds under high and atmospheric pressures. One way to remedy such problems is by using high-pressure on-line analytical equipment to analyze the liquid effluent stream under similar reactor pressures. This is not straightforward and high-pressure analytical equipment is commonly not available. In addition, the gas effluent stream needs to be analyzed to close the mass balance and to check whether really naphthalene or benzoic acid is stripped in the reactor and gas–liquid separator to the gas stream. It is also found that the equipment and the method to analyze the gas phase for very low concentrations of naphthalene and benzoic acid are not handy and/or straightforward.<sup>1</sup>

In the absence of the above-mentioned analytical techniques, it is suggested that a priori calibration of the system at the operating conditions of interest should be performed to identify a correction factor for the loss of the dissolved material from the withdrawn samples. This can be done by performing the following steps:

(1) Fill the reactor with glass particles of similar shape and dimensions as those of the active bed.

**Table 2. Molar Flow Rate of Naphthalene and Benzoic Acid in the Air That Is in Equilibrium with Water Saturated with These Materials for the Conditions Shown in Table 1<sup>23</sup>**

naphthalene			
		$Q_g C_g^*$	$Q_L C_L^*$
low gas velocity 1.1 cm/s (4.2 cm <sup>3</sup> /s)		$2.6 \times 10^{-8}$	low liquid velocity 0.09 cm/s (0.33 cm <sup>3</sup> /s)
high gas velocity 7.8 cm/s (29.6 cm <sup>3</sup> /s)		$1.8 \times 10^{-7}$	high liquid velocity 0.25 cm/s (0.96 cm <sup>3</sup> /s)
solubility = 0.03 g/L Henry's constant = $4.9 \times 10^{-4}$ ((bar m <sup>3</sup> )/mol)			
benzoic acid			
low gas velocity 1.1 cm/s (4.2 cm <sup>3</sup> /s)		$3.1 \times 10^{-5}$	low liquid velocity 0.09 cm/s (0.33 cm <sup>3</sup> /s)
high gas velocity 7.8 cm/s (29.6 cm <sup>3</sup> /s)		$2.14 \times 10^{-4}$	high liquid velocity 0.25 cm/s (0.96 cm <sup>3</sup> /s)
solubility = 2.7 g/L Henry's constant = $7.1 \times 10^{-8}$ ((bar m <sup>3</sup> )/mol)			

**Table 3. Naphthalene Concentrations Measured at the Inlet and Outlet of the Trickle-Bed Reactor System**

inlet concn (ppm)	% saturation	$V_g$ (cm/s)	$V_L$ (cm/s)	outlet concn (ppm)	% difference between inlet and outlet concentration <sup>a</sup>
Pressure = 30 psig (0.31 MPa)					
9.34	28.02	2.37	0.32	8.06	13.69
9.36	28.08	2.37	0.05	8.60	8.15
10.14	30.42	7.95	0.27	9.04	10.80
10.09	30.27	7.95	0.05	8.20	18.74
Pressure = 300 psig (2.17 MPa)					
9.00	27.00	1.05	0.07	4.74	47.33
9.17	27.51	1.05	0.27	4.40	52.06
9.11	27.33	4.72	0.33	3.31	63.69
9.40	28.20	4.72	0.06	4.45	52.62

<sup>a</sup> % difference between inlet and outlet concentration =  $\{[\text{inlet concn.} - \text{outlet concn.}]/\text{inlet concn.}\} \times 100\%$ .  $V_g$ : superficial gas velocity;  $V_L$ : superficial liquid velocity.

**Table 4. Benzoic Acid Concentrations Measured at the Inlet and Outlet of the Trickle-Bed Reactor System**

inlet concn (ppm)	% saturation	$V_g$ (cm/s)	$V_L$ (cm/s)	outlet concn (ppm)	% difference between inlet and outlet concentration <sup>a</sup>
Pressure = 30 psig (0.31 MPa)					
9.94	0.28	1.05	0.24	3.16	68.15
9.84	0.27	1.05	0.06	2.07	79.01
10.03	0.28	4.55	0.07	3.16	68.45
9.66	0.27	4.55	0.24	4.17	56.83
Pressure = 300 psig (2.17 MPa)					
6.09	0.17	1.01	0.05	1.52	75.10
9.48	0.26	1.01	0.23	0.97	89.79
8.47	0.24	6.77	0.04	4.81	43.21
10.94	0.30	6.77	0.24	4.90	55.20

<sup>a</sup> % difference between inlet and outlet concentration =  $\{[\text{inlet concn.} - \text{outlet concn.}]/\text{inlet concn.}\} \times 100\%$ .  $V_g$ : superficial gas velocity;  $V_L$ : superficial liquid velocity.

(2) Use feed solution of known concentrations of naphthalene or benzoic acid.

(3) Operate the system at the same operating conditions that need to be used for  $k_{ls}a$  measurements (i.e., similar pressure, gas and liquid flow rates, and temperature).

(4) Withdraw samples and measure the naphthalene concentrations by following the same procedure used for  $k_{ls}a$  measurements.

(5) Repeat steps (2)–(4) using a suitable range of naphthalene or benzoic acid concentrations that cover

the range of concentrations obtained during the  $k_{ls}a$  measurements.

(6) Develop a calibration curve and/or correction factors for the operating conditions of interest that represent the measured concentrations versus the inlet feed concentrations. The differences between them represent the correction factors that need to be added to the measured naphthalene or benzoic acid concentrations during  $k_{ls}a$  measurement to account for the loss of naphthalene or benzoic acid during the sampling procedure.

Moreover, it is recommended, even at atmospheric conditions, to conduct a similar procedure to check if there is a noticeable loss of the dissolved material by flashing/stripping that may occur.

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