# ABSORPTION OF ETHYLENE OXIDE in a PACKED COLUMN WITH VARIOUS SOLVENTS

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**Data were obtained on the solubility of ethylene oxide gas in a number of aqueous and nonaqueous solvents at various temperatures and gas concentrations. Its absorption from high concentrations in air was studied in a packed column with a cooling jacket for removal of the heat evolved. The results can be correlated with adequate accuracy in terms of the conditions at the top or dilute end of the column. The values of $(H.T.U.)_G$ and $(H.T.U.)_L$ agree approximately with known values for other systems. For the solvents tested, the liquid-film resistance is controlling at room temperatures.**

In this research the absorption of ethylene oxide has been studied under varying conditions of gas composition, temperature, and solvent; and the relationship between absorption rate and the significant variables has been investigated. Ethylene oxide is an organic gas which boils at 10.5°C. and is readily soluble in many organic and inorganic solvents. The absorption was not strictly kept under either isothermal or adiabatic conditions, but rather a cooling jacket with refrigerant flowing upward at a constant rate was employed. The immediate purpose was to investigate the practicability of recovering ethylene oxide by scrubbing the vent gases of a plant producing acetyl butyro lactone ($C_6H_8O_3$) from ethyl aceto acetate and ethylene oxide. An additional purpose was to check the suitability of present over-all methods of column design to absorption of concentrated soluble gases with heat evolution and cooling. Solvents employed included acetone, ethyl alcohol, water, 52% by volume ethyl alcohol, benzene, and ethyl aceto acetate. For each solvent the concentration of ethylene oxide in air at the inlet to the column was varied from 10 to 46 mole %. The solubility of ethylene oxide in the various solvents was also measured.

## GENERAL LITERATURE SURVEY AND ABSORPTION THEORY

Since the publication of the two-film theory (*18*) and the development of the diffusion theory (*17*), researchers have tried to correlate their absorption data on these bases. Early workers assumed that one film predominated and reported the film coefficient as a function of either the gas or liquid flow rate (*4, 8, 11*). Recent investigators have measured the resistances of both films and related them to the liquid and gas flow rates (*5, 7, 10, 14, 19*).

In terms of over-all height per transfer unit (*3*) $(H.T.U.)_{OG}$, the equation that expresses the addition of the individual gas and liquid resistances for an isothermal system following Henry's Law (so that $m$ is constant) is

$$(H.T.U.)_{OG} = (H.T.U.)_G + (H.T.U.)_L \frac{mG}{L} \frac{(1-x)_f}{(1-y)_f} \qquad (1)$$

When the experimentally determined values of $(H.T.U.)_{OG}$ vs. $\frac{mG}{L} \frac{(1-x)_f}{(1-y)_f}$ for each absorption run at some constant temperature for the same solvent are plotted, the slope of the straight line yields $(H.T.U.)_L$ and the intercept, at $\frac{mG}{L} \frac{(1-x)_f}{(1-y)_f}$ equal to zero, is $(H.T.U.)_G$. The values of the over-all and individual film H.T.U.'s are related to the over-all and individual mass-transfer coefficients by

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This article is contribution 49 from the Chemical Engineering Laboratories, Engineering Center, Columbia University, New York, and is taken in part from the M. S. thesis written by Dr. Baron at the Johns Hopkins University in 1947.

$$(\text{H.T.U.})_{OG} = \frac{G}{K_G a\,(1-y)} \qquad (2)$$

$$(\text{H.T.U.})_{G} = \frac{G}{k_G a\,(1-y)_f} \qquad (3)$$

$$(\text{H.T.U.})_{L} = \frac{L}{k_L a\,(1-x)_f} \qquad (4)$$

If the separate film coefficients are proportional to the flow rate of the corresponding streams and the concentration of solute in the streams is low, the $(\text{H.T.U.})_{OG}$ would then be a linear function of $\frac{mG}{L}\,\frac{(1-x)_f}{(1-y)_f}$. Several investigators have studied systems obeying this relationship (*1, 2, 6, 8*).

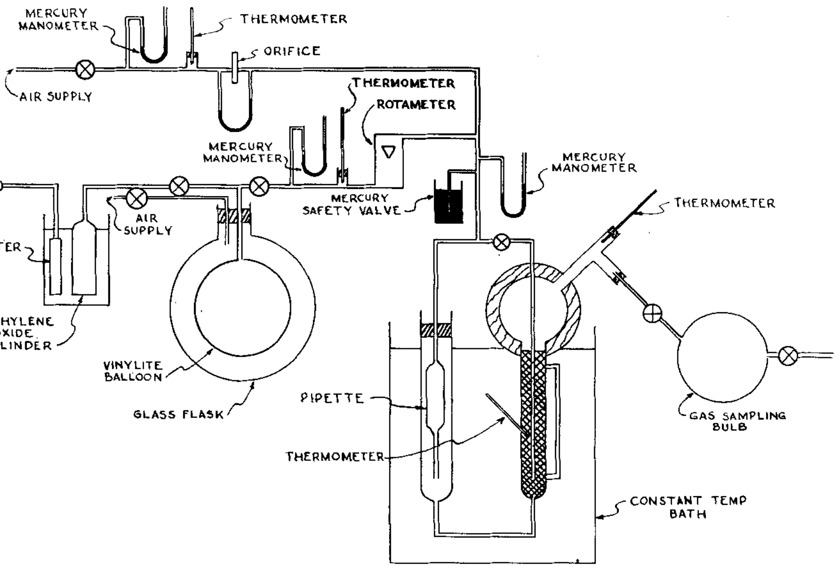

FIG. 1. SOLUBILITY APPARATUS.

Some work has been done to study the effect of temperature in isothermal absorption. The results have shown that the liquid-film coefficient is very sensitive to temperature whereas the gas-film coefficient is only slightly affected by temperature (*5, 8, 15*). However, no over-all analyses of concentrated systems involving a soluble gas have been located.

## METHOD OF CALCULATION

The solubility data as tabulated in Table 1* show that at the determined temperatures Henry's Law holds, namely $y = mx$. When there is no solute in the entering solvent, Colburn (*3*) has shown that where $(mG/L)$ is constant, the number of transfer units $N_{OG}$ is determined by the equation:

$$N_{OG} = \frac{2.3}{\left(1-\frac{mG}{L}\right)} \log\left[\left(1-\frac{mG}{L}\right)\frac{y_B}{y_T} + \frac{mG}{L}\right] \qquad (5)$$

The dilute, and thus substantially isothermal, runs, in which the outlet solvent temperature was within ± 2°C. of its inlet temperature, were first analyzed.

For this case Equation (1) substantially equals

$$(\text{H.T.U.})_{OG} = (\text{H.T.U.})_{G} + (\text{H.T.U.})_{L}\,\frac{mG}{L}\,\frac{1}{(1-y_T)} \qquad (6)$$

By a plot of $(\text{H.T.U.})_{OG}$ vs. $\frac{mG}{L}\,\frac{1}{(1-y_T)}$ for any given solvent at the same temperature, the values of $\text{H.T.U.}_G$ and $\text{H.T.U.}_L$ were determined.

When the solvent temperature changed by more than two degrees in each set of runs with a given solvent inlet and jacket temperatures, values of $m$ corresponding to the range of solvent temperatures were used in calculating the over-all H.T.U. That value of $m$ which gave best agreement in $(\text{H.T.U.})_{OG}$ between the isothermal and nonisothermal runs at the same average temperature was considered as representing the mean value for the column. As the calculated results in Table 3 indicate, the temperature corresponding to the mean $m$ is in general approximately the same as the solvent temperature at the top of the column. This fact substantiates the approach of correlating the data with the conditions at the top or the dilute end of the column, as is recommended (*12*) by Perry for quick approximate solution in nonisothermal cases.

## ANALYTICAL PROCEDURE AND SOLUBILITY APPARATUS

Ethylene oxide when absorbed in aqueous hydrochloric acid in the presence of calcium chloride reacts quantitatively with the hydrochloric acid to form ethyl chlorhydrin. The excess hydrochloric acid is back-titrated with sodium hydroxide; bromphenol blue is the indicator. Experiments run previously at the laboratory of U. S. Industrial Chemicals, Inc., showed immediate absorption of ethylene oxide by this method if the water is approximately saturated with calcium chloride and the normality of the hydrochloric acid is between 0.7 and 0.8. The calcium chloride is first dissolved in the water and the solution filtered to remove insoluble impurities, apparently from the calcium chloride. Concentrated hydrochloric acid is added to bring the solution to the correct normality. A stock solution containing 3.2 lb. of calcium chloride (anhydrous), 2,600 ml. of water, and 200 ml. of concentrated hydrochloric acid was used for all ethylene oxide determinations.

The solubility data were obtained by passing a mixture of ethylene oxide and air in a constant ratio through the solvent at a specific temperature until the liquid was saturated. The gas and liquid phases were then analyzed for ethylene oxide. The equipment is shown in Figure 1.

The saturator was a 1-liter flask used in an inverted position; to the neck of the flask was blown a 1 3/8-in. I.D., 1 1/2-ft.-long glass extension tube packed with 1/4-in. Raschig rings through which the gases were admitted into the absorber. A 1/8-in. glass return tube to the bottom of the packing gave good liquid recirculation. Sealed at the bottom of the packed tube was a 1/2-in. glass tube

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*Tabular data are available as document 4474 from A. D. I. Auxiliary Publications Project, Photoduplication Service, Library of Congress, Washington 25, D.C., for $2.50 for photoprints or $1.75 for 35-mm. microfilm.

that held a rubber stopper and a pipette for sampling the liquid. The 1-liter flask was thermally insulated with asbestos cement 1/4 in. thick provided with peep holes. The exit-gas sampling bulb was a 2-liter flask of measured volume with rubber connections and screw clamps. When a sample was taken, the inlet connection was closed first and then the outlet connection.

The air was obtained from the regular laboratory compressed-air–supply system. The ethylene oxide was stored in liquid form in a 50-lb. cylinder which fed to a 5-lb. gas cylinder in a heated water bath to provide the necessary heat to vaporize the ethylene oxide. Owing to irregular boiling of the ethylene oxide in the cylinder, large fluctuations in the pressure occurred. A constant ethylene oxide gas pressure slightly above atmospheric pressure was accomplished by employing as a gas holder a Vinylite balloon, of the type used by the Army Weather Bureau, in a round 50-liter flask. An accurately controlled air pressure was connected to the space between the flask and the balloon. To inflate the balloon, its outlet connection was shut off, the space between the flask and balloon vented to the atmosphere, and the connection from the 5-lb. cylinder to the balloon opened. When the balloon completely filled the flask, the ethylene oxide was shut off and the air supply to the flask turned on. A constant air pressure on the balloon caused a steady stream of ethylene oxide gas to pass into the absorber.

The ethylene oxide flow rate was measured by means of a rotameter calibrated with air against two wet-test meters which agreed with each other. Correction to the specific gravity of ethylene oxide of 1.52 was made by the nomograph supplied by the manufacturer. The air-flow rate was measured by an orifice calibrated against the wet-test meters. The ratio of ethylene oxide to gas-flow indications was also directly checked by the analysis for ethylene oxide in the exit gas after saturation. Thermometers and manometers were always used, as required, in the air lines and during calibration tests, so that the flow rates might be corrected to standard conditions. All gas-line connections were sealed with cellulose nitrate solutions.

The saturator was thermostatted in a 20-gal. barrel with a coolant at the desired temperature. Below 0°C., ethanol cooled in a deep-freeze unit was circulated through the barrel by a centrifugal pump. The temperature was controlled by settings on the freezer and by the rate of flow of the ethanol. Above 0°C. the ethanol was also employed as the coolant.

The saturator was filled with the solvent to be tested to about 1 in. below the top of the packing and then immersed in the barrel to the top of the packing.

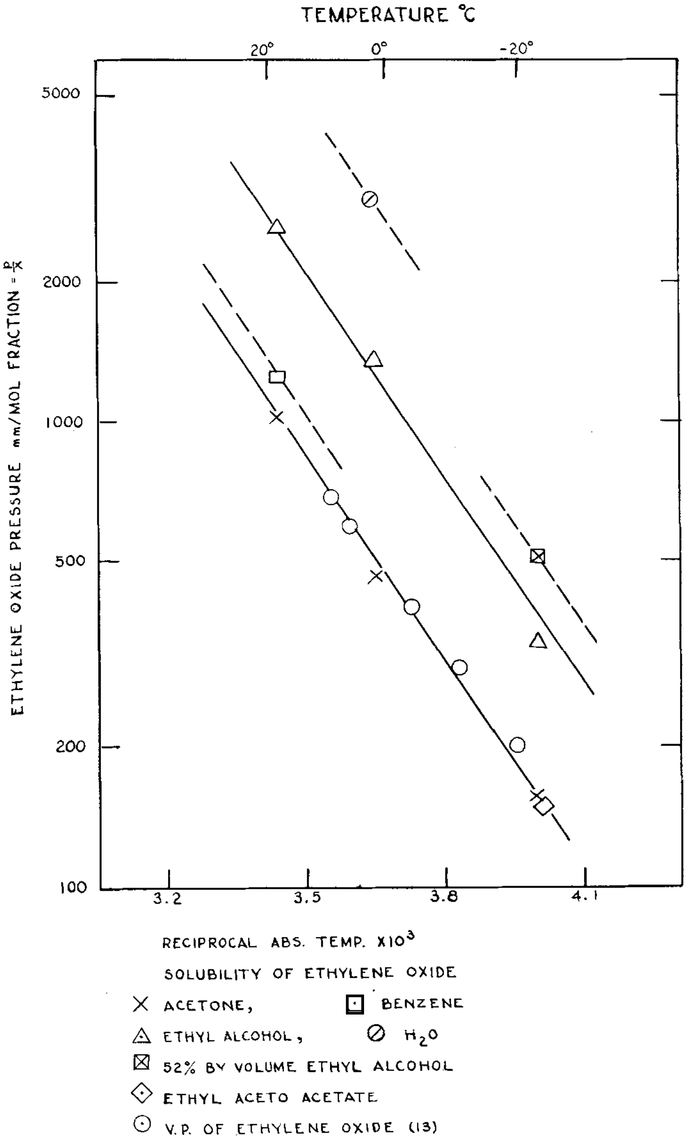


FIG. 2.

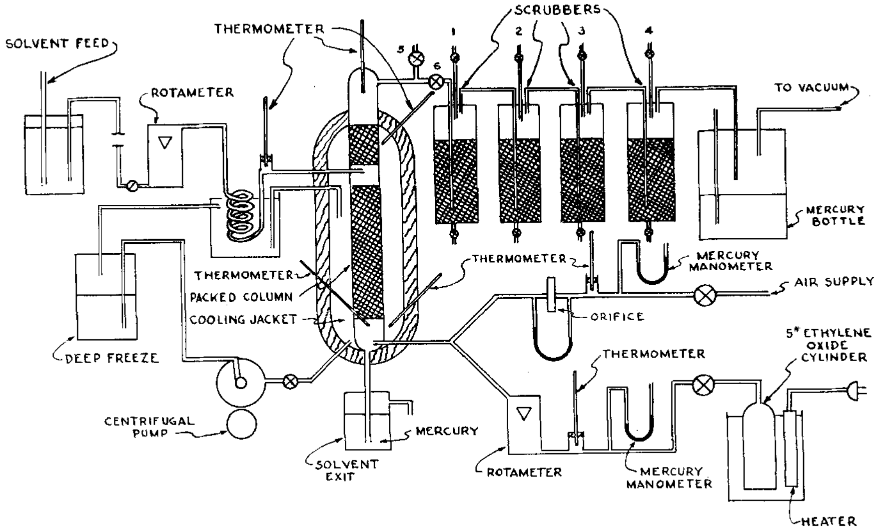
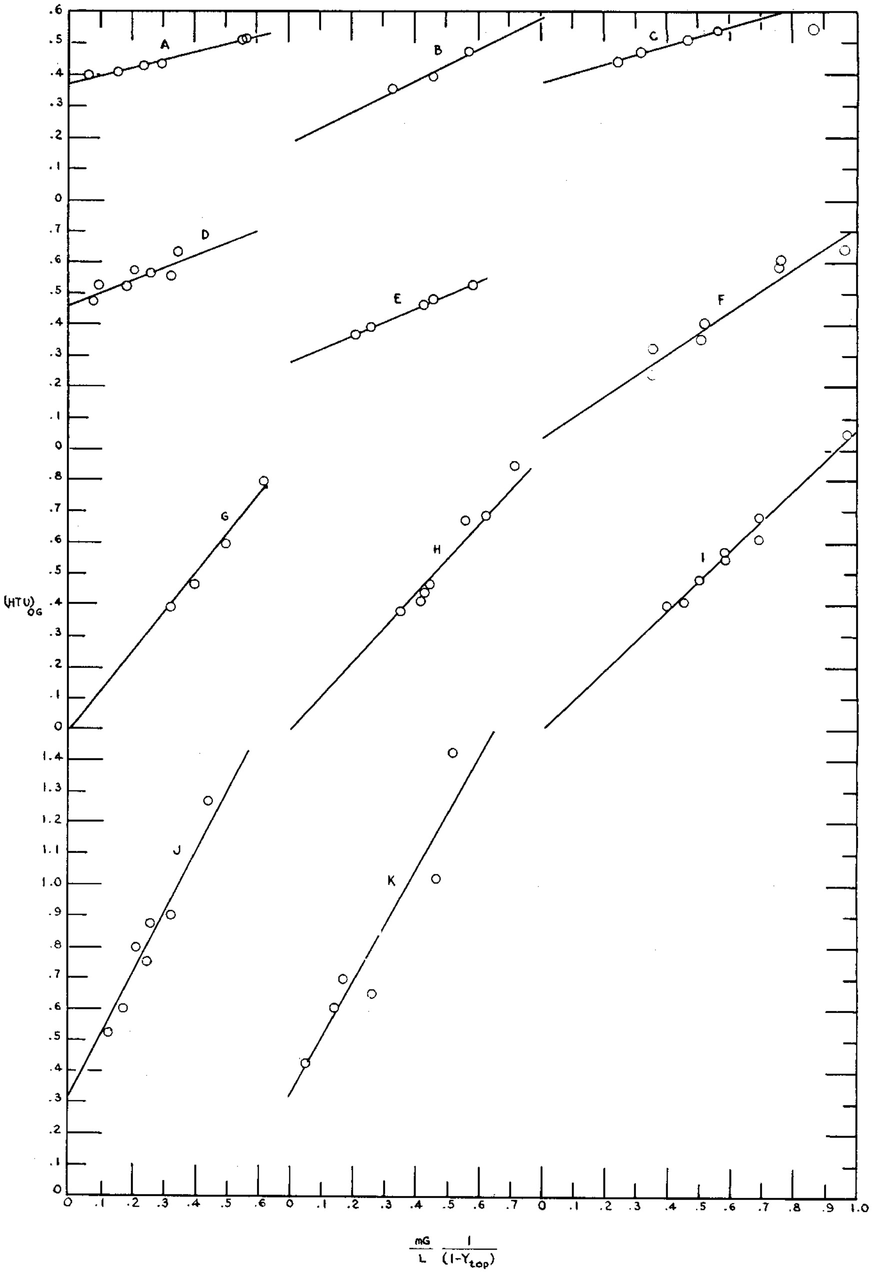


FIG. 3. ABSORPTION APPARATUS.

The ethylene oxide and air flow were started at the same time. A screw clamp at the saturation inlet was set so that the gas flow was divided about equally between the absorber and the pipette. Every 5 min. during a run the exit-gas sampling bulb was analyzed for ethylene oxide. When the ratio of ethylene oxide to air was the same in the bulb as on entering the absorber, then the liquid was considered saturated, and a sample of the liquid was withdrawn with the 5-ml. pipette and analyzed for ethylene oxide. It was found that the dilute gas mixtures would saturate the solvent in 10 min. and the more concentrated mixtures in 15 min. The saturation pressure was taken to be that at the gas inlet to the packing, which would be substantially correct from the location of the pipette.

Solubility runs were started with dilute gas mixtures, and the concentration increased after each run. At the end of a run the screw clamp before the gas bulb was closed, the gas releasing through the mercury valve. The gas flows were then shut off, and a liquid sample was immediately taken. Since the pipette was calibrated and the liquid density known, it was possible to analyze the liquid for ethylene oxide without weighing it. This method gave reproducible results and avoided the necessity of weighing the sample and the possibility of losing ethylene oxide in the process.

The gas sampling bulb, completely pinched off, was allowed to warm up to room temperature and the excess gas was released. A measured amount of the hydrochloric acid solution between 50 and 100 cc. was placed in a separatory funnel and about three fourths allowed to flow into the bulb. The bulb was shut off and the hydrochloric acid swirled for about 5 min. The remaining hydrochloric acid was admitted and again swirled and then drained out and the bulb washed twice with equal volumes of water. The washes were added to the hydrochloric acid and the sample was titrated, the results being plotted in Figure 2. It is evident that solutions in acetone and ethyl aceto acetate obey Raoult's Law, with increasing positive deviations both for less polar (benzene) and more polar (water) liquids. The equations of the five lines plotted are given in Table 2.

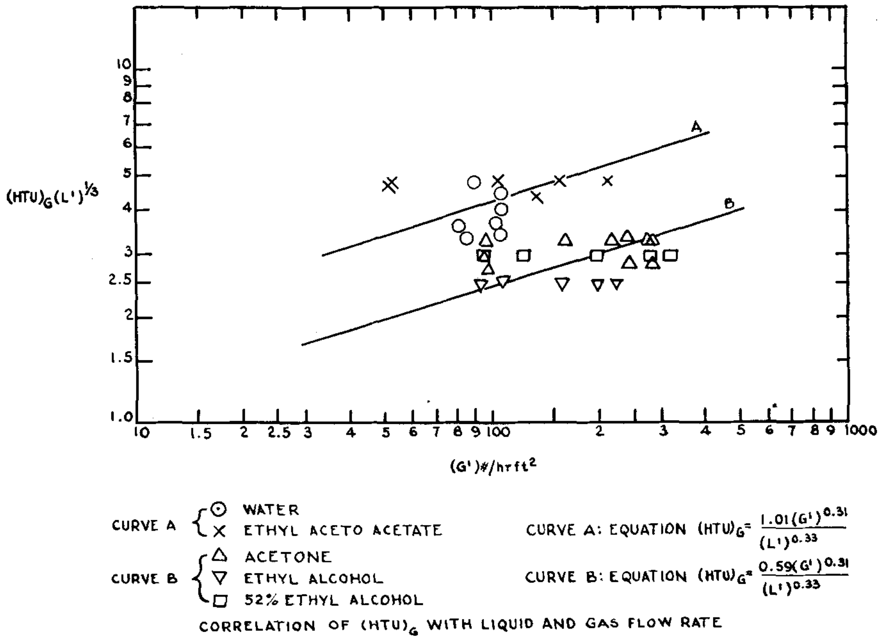


| CURVE | SOLVENT | TEMPERATURE RANGE | $(HTU)_G$ | $(HTU)_L$ |
|---|---|---|---|---|
| A | ACETONE | +2 TO -12.5 °C | 0.37 | 0.25 |
| B | ACETONE | 17 TO 19 °C | 0.18 | 0.50 |
| C | ACETONE | 4.5 TO 8 °C | 0.37 | 0.31 |
| D | ETHYL ACETO ACETATE | -5 TO -10 °C | 0.46 | 0.43 |
| E | ETHYL ALCOHOL | -5 TO -10 °C | 0.28 | 0.43 |
| F | ETHYL ALCOHOL | 13 TO 18 °C | 0.04 | 0.69 |
| G | ACETONE | 22 TO 25 °C | 0.00 | 1.26 |
| H | BENZENE | 22 TO 26.5 °C | 0.00 | 1.10 |
| I | ETHYL ALCOHOL | 24 TO 27 °C | 0.00 | 0.97 |
| J | WATER | 6 TO 14 °C | 0.33 | 1.80 |
| K | 52% ETHYL ALCOHOL | -3 TO -10 °C | 0.32 | 1.82 |

FIG. 4. COLBURN PLOT FOR ETHYLENE OXIDE ABSORPTION BASED ON TOP (PINCH) CONDITIONS.

## ABSORPTION APPARATUS

The apparatus used for the absorption-rate data is shown in Figure 3. An air-ethylene oxide mixture of known composition was passed up the absorption column, and a measured liquid flow rate passed down. Scrubbers on the gas-outlet line measured the amount of ethylene oxide leaving the column. The difference was the amount of ethylene oxide absorbed.



CORRELATION OF $(HTU)_G$ WITH LIQUID AND GAS FLOW RATE

FIG. 5.

The absorption column measured 1.1 in. I.D. and 45 in. high. The liquid level in the bottom of the column was maintained constant and gas leakage was prevented by a mercury seal on the outlet tube. The gas inlet and bottom of the packing were just above the liquid level.

The column was packed randomly with a thorough mixture of three sizes of glass Raschig rings to a height of 30.5 in. One-third of the total number was 21/64 in. O.D., 13/64 in. I.D., and 21/64 in. long; 1/3 was 9/32 in. O.D., 5/32 in. I.D., and 21/64 in. long; and 1/3 was 8/32 in. O.D., 4/32 in. I.D., and 9/32 in. long. The calculated surface is 215 sq. ft./cu. ft. Above the liquid inlet there was a 3-in. entrainment-removing section. A cooling jacket surrounded the packed column, which contained thermometers in the liquid at the bottom, in the exit gas stream at the top, and in the inlet and outlet streams of the coolant. The column was completely insulated with asbestos 0.85 in. thick.

The outlet gases bubbled through four glass-Raschig-ring-packed scrubbers 1 ft. high in series. The first and third scrubbers contained the hydrochloric acid– and calcium chloride–absorbing solution and the others contained water to catch any entrained or vaporized hydrochloric acid. The gas line from the last absorber, as well as a line through which the gas was allowed to by-pass the absorbers at the start of the run, led to a mercury bottle and air tube connected to a vacuum pump. By adjusting the submergence of the air tube it was possible to maintain enough vacuum in the air bottle just to counteract the liquid head in the four scrubbers and maintain atmospheric pressure in the absorption column.

The cooling system consisted of the deep-freeze unit filled with 30 gal. of alcohol, which was circulated at 1.3 liters/min. by a small centrifugal pump through the column cooling jacket and next through a Dewar flask that precooled the liquid feed stream and then was returned to the deep freeze.

The solvent for the absorption was fed from a 2-liter constant-head bottle stationed about 10 ft. above the column. The liquid flowed through a rotameter calibrated with the same solvent through three turns of 1/8-in. copper tubing 3 in. in diam., in the Dewar bottle, and then into the column.

The air came from the previously mentioned air system and orifice meter. At the higher flow rates used in the absorption tests the boiling of the ethylene oxide was much steadier and the plastic bag was unnecessary; otherwise the system was as before. The gases mixed in a 1-ft.-long tube before entering the column.

In starting a run the coolant alcohol was cooled to the desired temperature and circulated through the column for 1/2 hr. Then the solvent and gases were started through the column for 15 min., the gases bypassing the scrubbers. Stopcock 5 was closed and 6 opened, and timing started with a stopwatch. Absorption was continued from 10 to 20 min., but not to exceed the capacity of the first scrubber. All temperatures, pressures, and flow rates were recorded every 5 min. At the end of the run the streams were stopped, and the hydrochloric acid scrubbers drained, washed, and analyzed. The first scrubber was found always to have caught all the ethylene oxide.

## RESULTS

For each run first the number of transfer units, $N_{OG}$, was computed by Equation (5) by use of $(mG/L)$ at the top of the column, then $(H.T.U.)_{OG}$ was computed by $(H/N_{OG})$. On analysis of the data it was found that $(H.T.U.)_{OG}$ correlated better with the function $\frac{mG}{L}\,\frac{1}{(1-y_T)}$ than with any fractional powers of gas or liquid flow rates, where $(mG/L)$ again was for the top of the column (Figure 4).

When the temperature is approximately constant, this method should be reliable since the operating and equilibrium lines diverge in going from the top to the bottom of the column, most of the transfer units being at the dilute end. When the temperature rises in passing down the column, the equilibrium line approaches the operating line, and the concentrated end, or bottom, of the column has an increasing number of transfer units. The effective mean temperature for the column would then be intermediate between those at the top and bottom of the column.

Four of the runs (*AL, AM, AN,* and *AO*), however (Table 4), determine a mean temperature as described above which is higher than both the inlet and outlet solvent temperatures. No explanation is evident, for it does not seem reasonable that absorption was faster in the middle or the top of the column than in the bottom and so overheated the liquor in the middle.

At mean temperatures above 20°C. with the solvents ethyl alcohol, acetone, and benzene, the liquid film showed as the only resistance to mass transfer. With water in the temperature range from 6° to 14°C., $(H.T.U.)_L$ is five times greater than $(H.T.U.)_G$. The 52% ethyl alcohol at —3° to —10°C. yielded $(H.T.U.)_L$ six times greater than $(H.T.U.)_G$. At temperatures below 0°C., ethyl alcohol, acetone, and ethyl aceto acetate have $(H.T.U.)_L$ and $(H.T.U.)_G$ of the same order of magnitude.

Within the range of accuracy of the experimental results, ethyl alcohol, acetone, benzene, and ethyl aceto acetate have approximately the same value of $(H.T.U.)_L$ at the same solvent temperature. With water and 52% ethyl alcohol as solvents, the value of $(H.T.U.)_L$ is about four times as great as for the other above-mentioned solvents.

Figure 5 is a comparison of the experimental results of $(H.T.U.)_G$ with the equation (curve $A$) proposed by Sherwood and Pigford (*16*) based on absorption data for ammonia, sulfur dioxide, and chlorine with water. The solvents water and ethyl aceto acetate seem to fall reasonably close to the proposed curve. The solvents acetone, ethyl alcohol, and 52% ethyl alcohol average some 40% lower, clustering around the curve $B$ represented by the equation

$$(H.T\,U.)_G = \frac{0.59\,(G')^{0.31}}{(L')^{0.33}} \qquad (7)$$

Considering only the results herein reported, however, the first two solvents fall closer to the line $A'$, or $(H.T.U.)_G(L')^{1/3} = 4.5$, and the other three to line $B'$, or $(H.T.U.)_G(L')^{1/3} = 2.9$. The relative variation in $(H.T.U.)_G(L')^{1/3}$ does not match the variation in solubility shown in Figure 2, but there is a partial correlation with surface tension of the solvent, since water and ethyl aceto acetate have higher surface tensions than have the other solvents.

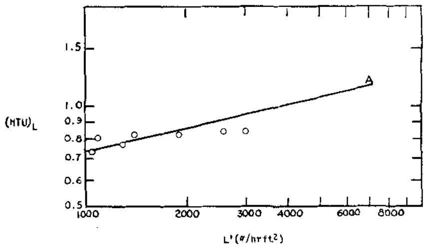


FIG. 6. CORRELATION OF $(H.T.U.)_L$ WITH LIQUID-FLOW RATE. CURVE A = 1-IN. CERAMIC RINGS, DESORPTION OF $O_2$ FROM $H_2O$ AT 25° C. (*15*); ○ = ETHYLENE OXIDE — WATER SYSTEM CORRECTED TO OXYGEN-WATER SYSTEM AT 25°C.

Figure 6 compares the experimental results of $(H.T.U.)_L$ for the ethylene oxide–water system with the data obtained by Sherwood and Holloway (*15*) for the desorption of oxygen from water. The test results were first corrected to 25°C. by use of Sherwood and Holloway's recommended corrections for temperature variation. Then, based on the diffusivity of the ethylene oxide–water system the test values of $(H.T.U.)_L$ were converted to the oxygen-water basis, as at the same solvent flow rate $(H.T.U.)_L$ is inversely proportional to the one-half power of diffusivity. The diffusivity of the ethylene oxide–water system was estimated to be equal to the known value of the glycerine–water system ($0.72 \times 10^{-5}$ sq. cm./sec. at 20° C.) since ethylene oxide converts to a glycol upon hydrolysis. The test results lie reasonably close to the data of Sherwood and Holloway for 1-in. rings of curve $A$. The other solvent systems were not compared as their diffusivities are not known.

## CONCLUSIONS

1. It is feasible to recover ethylene oxide from process waste gases by countercurrent absorption in a packed column. Of the solvents tested, ethyl alcohol, acetone, benzene, and ethyl aceto acetate require the lowest column height for a particular temperature and change in gas and liquor concentrations.
2. At room temperature the liquid-film resistance is controlling under practical conditions. At solvent temperatures below 0°C. the gas- and liquid-film resistances become comparable in magnitude.
3. The absorption of ethylene oxide gas in the high concentration range (10 to 46 mole %) can be correlated with adequate accuracy for most design purposes in terms of the conditions at the dilute end of the column.
4. The values of $(H.T.U.)_G$ and $(H.T.U.)_L$ for the ethylene oxide–water system agree generally with the known values for other systems. The $(H.T.U.)_L$ of the other solvents were not compared because of inadequate diffusivity data and insufficient published results on nonaqueous systems.

## NOTATION

$G$ = gas exit flow rate, lb. moles $(hr.^{-1})\ (ft.^{-2})$
$G'$ = gas exit flow rate, lb./$(hr.^{-1})\ (ft.^{-2})$
$H$ = absorption (packed) height of column, ft.
$(H.T.U.)_{OG}$ = height of over-all transfer unit, ft.
$(H.T.U.)_G$ = height of gas-film transfer unit, ft.
$(H.T.U.)_L$ = height of liquid-film transfer unit, ft.
$K_g a$ = over-all mass transfer coefficient, lb. moles/$(hr.^{-1})\ (ft.^{-3})\ (atm.^{-1})$
$L$ = solvent inlet flow rate, lb. moles/$(hr.^{-1})\ (ft.^{-2})$
$L'$ = solvent inlet flow rate, lb./$(hr.^{-1})\ (ft.^{-2})$
$N_{OG}$ = number of transfer units
$a$ = interfacial area, $ft.^2/ft.^{-3}$
$k_g a$ = gas film coefficient, lb. moles/$(hr.^{-1})\ (ft.^{-3})\ (atm.^{-1})$
$k_L a$ = liquid film coefficient, lb. moles/$(hr.^{-1})\ (ft.^{-3})$ lb. moles $/ft.^{-3})$
$m$ = slope of the equilibrium line on $x$-$y$ coordinates
$p$ = partial pressure of ethylene oxide in air, mm.
$t$ = temperature, °C.
$t_m$ = mean temperature, °C.
$x$ = mole fraction of solute in body of liquid phase
$y$ = mole fraction of solute in body of gas phase
$(1-x)_f$ = logarithmic mean of $1-x$ and $(1-x)$ at equil.
$(1-y)_f$ = logarithmic mean of $1-x$ and $(1-x)$ at equil.

### Subscripts

$B$ = bottom of the column
$T$ = top of the column

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