Experimental Transient Response of a Pilot-Plant Distillation Column:

Part II. Response to Liquid and Vapor Rate Perturbations

M. F. BABER and J. A. GERSTER
University of Delaware, Newark, Delaware

The first paper of this series (*4*) indicated that although many mathematical models exist in the literature for describing the dynamic behavior of a distillation system, the validity of these models has not been adequately tested with experimental data (*1, 2, 5, 9, 10, 11, 12, 13, 14*). The Delaware transient response research program, initiated in 1958, was undertaken to assist in providing such information from experimental studies carried out in a 2-ft. diameter pressure column.

Part I of the Delaware research program (*4*) reported distillation column behavior when a step change in feed composition was made to the top tray of a five-tray column. The column was operated under total reflux conditions throughout the test. After introduction of a change in composition (but not the rate) of the liquid fed to the top tray, the tower pressure and the vapor rate were unchanged, while the overhead vapors were continuously removed from the system. Because of the new reflux composition a new steady state, total-reflux condition was eventually established; the time required was about 10 min., during which time the liquid on each tray was replaced about seventy to eighty times.

The second series of tests in the Delaware research program is reported in the present paper. These tests use the same column but proceed from an initial total-reflux condition to a final steady state stripping column by step changing the liquid or vapor rate to the column. In a third series of tests now well underway the dynamics of a ten-tray column fed at the center are being determined, in which case the added complications of a regular feed tray and a condenser-reflux-drum system are present.

The first paper of this series showed that in the absence of rate perturbations the experimental composition-time behavior of the tray liquids could be predicted quite well by equations developed by Lamb, Pigford, and Rippin (*8*) for characterizing the transient behavior of distillation columns. These equations employ linear perturbation techniques and are relatively easy to apply, especially when an analogue computer is available. The purpose of the present paper then is not only to determine the experimental transient response of a distillation column to changes in liquid and vapor rate, but to demonstrate the applicability of the theoretical equations for predicting the measured response.

EXPERIMENTAL

The column and its auxiliaries are the same as that employed at the University of Delaware for a five-year study of tray efficiencies sponsored by the Research Committee of the American Institute of Chemical Engineers from 1952 through 1957. During the course of the efficiency program values were determined for the tray efficiency, gas and liquid holdup on the trays, downpipe holdup, gas pressure drop, flooding point, and entrainment characteristics over a wide range of operating and design variables and for the systems acetone-benzene and pentane-xylene. The complete results of these studies were published in 1958 (*6*).

Description of the Unit

The column has an inside diameter of 2 ft. and contains five bubble-cap trays on 24-in. spacing. Segmental downpipes are employed, no inlet weir is used, and the outlet weir height is 2 in. Each tray contains seventeen round bubble caps of 3-in. diam. in an arrangement designated as "Tray 2" (*6*). The vapor for the column is supplied by a vertical, natural circulation, thermosyphon reboiler with process liquid inside the tubes. The discharge from the reboiler enters a 50-gal. separator from which unvaporized liquid is returned to the bottom of the reboiler and from which vapor is sent to the base of the column.

Scope of Experiments

Ten separate runs were made in the liquid perturbation experiments, and twelve runs are reported for the vapor perturbation tests. All tests were made with the acetone-benzene system, which has relative volatility values varying from 1.8 to 2.2; this system has enthalpy properties such that equal molar overflow prevails within the column. However in all of the tests the reflux entering the top tray was around 100°F. which was well below the top tray operating temperature. In all of the tests the reflux composition was very close to 65 mole % acetone, which gave reboiler liquid compositions of less than 5 mole % acetone. The runs were carried out at different values of gas and liquid rate which could be varied independently by varying the tower pressure. All of the step changes were in the same direction; the liquid rates were always increased and the vapor rates were always decreased so as to proceed from total reflux to stripping column operation. A summary of the operating conditions for all of the runs is given in Table 1.

Operating Procedure

During the initial steady state operation under total reflux conditions, overhead vapors from the column were totally condensed and sent to a reflux drum. The outlet from this drum flowed to Tank 6 from which reflux was pumped to the top tray. Before the step change was introduced, a check was made to insure that the compositions in the reflux drum and in Tank 6 were equal. The step change in liquid rate to the column was brought about by rapidly opening further a valve located in the reflux supply line to the column. At the same instant a valve between the reflux drum and Tank 6 was closed to insure that the reflux composition would remain constant during the transient; as the run proceeded, excess liquid from the reflux drum was emptied into an auxiliary tank. Sufficient storage capacity was available to continue the new method of operation for 10 min. or more, by which time a new steady state condition had been reached. Immediately after the step change part of the liquid flowing from the bottom tray to the reboiler was allowed to flow to storage so

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M. F. Baber is with Rohm and Haas, Philadelphia, Pennsylvania.

TABLE 1

Operating data for vapor-rate perturbation runs

Run	Reflux to column Comp. mole % acet.	Reflux to column Temp., °F.	Reflux to column Rate, lb. mole/min.	Initial steady state compositions, mole % acetone Top tray	Initial steady state compositions, mole % acetone Middle tray	Initial steady state compositions, mole % acetone Bottom tray	Top tray Temps., °F. Initial	Top tray Temps., °F. Final	Tower pressure, lb./sq. in. gauge	F factor*
M-1	65.9	98	1.874	55.0	25.0	5.3	193	186	17.3	1.84
M-3	69.7	100	2.020	59.6	29.0	7.0	205	202	23.3	1.86
M-4	58.6	106	2.319	45.5	15.9	2.7	222	218	30.0	2.13
M-5	66.1	100	2.351	54.7	24.4	5.7	249	246	49.4	1.96
M-6	65.6	102	2.114	53.9	23.0	4.7	220	218	30.1	1.91
M-7	65.6	98	1.889	54.6	23.4	4.5	176	172	7.4	2.06
M-8	66.6	99	2.518	54.6	28.3	7.1	182	190	10.4	2.64
M-9	64.9	100	1.911	53.7	22.6	4.8	261	258	57.7	1.54
M-10	65.9	97	1.662	54.4	21.9	4.0	223	221	29.4	1.52
M-11	66.2	104	2.211	55.2	26.1	5.7	171	165	5.5	2.42
M-12	66.0	99	1.914	54.8	23.6	4.7	199	194	15.3	1.95
M-13	64.5	98	2.008	55.1	26.0	6.0	276	271	58.6	1.67

Run	Internal vapor rate before step, or int. liq. rate, lb. moles/min.	Step decrease in int. vap. rate, lb. moles/min.	Total change in liq. composition between init. and final st. state, mole % acet. Top tray	Total change in liq. composition between init. and final st. state, mole % acet. Middle tray	Total change in liq. composition between init. and final st. state, mole % acet. Bottom tray	Holdup in gal. on Tray only	Holdup in gal. on Tray plus downpipe	Time lag on tray, sec.	Time lag in pipe from bottom tray to reboiler, sec.	Reboiler tube holdup, gal.
M-1	2.272	0.488	11.38	31.59	24.32	2.37	3.38	5.35	54.5	19.8
M-3	2.510	0.172	3.27	8.92	4.53	2.39	3.49	5.09	50.0	19.5
M-4	2.960	0.138	3.76	7.06	2.53	2.47	3.80	4.31	39.0	17.7
M-5	3.242	0.167	3.82	7.51	4.65	2.64	3.87	4.18	36.4	18.9
M-6	2.708	0.145	3.53	8.75	3.92	2.42	3.58	4.67	44.0	19.1
M-7	2.189	0.138	4.19	9.88	4.67	2.19	3.35	5.00	55.0	19.8
M-8	2.960	0.142	4.50	8.08	4.50	2.24	3.82	4.15	44.6	16.3
M-9	2.705	0.081	1.73	2.68	1.23	2.70	3.78	4.98	44.0	19.8
M-10	2.156	0.107	3.88	8.37	3.32	2.48	3.47	5.95	55.0	19.9
M-11	2.505	0.151	4.73	10.15	6.31	2.19	3.49	4.81	53.3	17.4
M-12	2.342	0.198	4.09	9.91	4.14	2.42	3.54	5.17	54.3	18.6
M-13	2.952	0.157	4.36	9.81	5.23	2.74	3.87	4.64	38.4	18.9

Operating data for liquid-rate perturbation runs

Run	Reflux to column Comp., mole % acet.	Reflux to column Temp., °F.	Reflux to column Rate, lb. mole/min. Before step	Reflux to column Rate, lb. mole/min. After step	Initial steady state compositions, mole % acetone Top tray	Initial steady state compositions, mole % acetone Middle tray	Initial steady state compositions, mole % acetone Bottom tray	Top tray temps., °F. Initial	Top tray temps., °F. Final	Tower pressure, lb./sq. in. gauge	F factor*
B-1	66.0	96	2.028	2.202	54.6	23.3	4.5	183	180	10.4	2.16
B-2	65.7	100	2.010	2.132	54.5	23.1	4.6	199	196	16.1	2.02
B-3	66.9	102	2.204	2.320	55.5	26.3	5.8	168	166	5.5	2.44
B-4	65.9	101	2.255	2.396	55.1	25.0	5.5	239	234	32.8	2.08
B-6	62.7	101	1.940	2.080	51.9	20.7	3.8	230	230	28.9	1.80
B-7	62.7	101	2.148	2.323	51.3	20.4	4.2	276	274	58.0	1.81
B-8	64.4	99	2.601	2.821	52.7	22.3	4.6	222	219	23.4	2.55
B-9	63.1	99	2.140	2.315	51.8	21.4	4.5	272	270	55.3	1.80
B-10	64.9	103	2.796	3.086	52.2	22.3	5.2	282	276	62.1	2.31
B-11	63.4	100	1.791	1.941	51.9	19.9	3.5	231	230	29.4	1.67

Run	Internal liquid rate before step, or int. vap. rate, lb. moles/min.	Step increase in int. liq. rate, lb. moles/min.	Total change in liq. composition between init. and final steady state, mole % acet. Top tray	Total change in liq. composition between init. and final steady state, mole % acet. Middle tray	Total change in liq. composition between init. and final steady state, mole % acet. Bottom tray	Holdup in gal. on Tray only	Holdup in gal. on Tray plus downpipe	Time lag on tray, sec.	Time lag in pipe from bottom tray to reboiler, sec	Reboiler tube holdup, gal.
B-1	2.401	0.180	4.69	11.45	3.98	2.21	3.48	4.78	50.6	17.6
B-2	2.453	0.148	3.38	7.78	3.02	2.28	3.52	4.81	48.8	17.8
B-3	2.528	0.132	2.95	6.92	3.84	2.17	3.63	4.56	51.2	16.5
B-4	3.050	0.173	2.51	5.90	2.87	2.48	3.77	4.00	38.9	17.8
B-6	2.522	0.176	3.82	8.56	3.72	2.47	3.61	4.78	44.2	19.6
B-7	3.131	0.243	3.36	7.16	3.09	2.67	3.88	4.06	35.1	20.0
B-8	3.320	0.265	3.09	8.05	3.62	2.43	4.09	3.49	36.6	16.6
B-9	3.068	0.184	4.73	9.72	4.37	2.81	4.02	4.43	39.4	19.4
B-10	4.106	0.384	5.71	13.20	7.10	2.93	4.54	3.38	27.1	17.0
B-11	2.341	0.201	6.10	14.78	6.81	2.38	3.42	5.06	50.2	19.5

* *F* factor is the product of the vapor rate, cu. ft./(sec.)(sq. ft. of tray bubbling area) and the square root of the vapor density, lb./cu. ft.; tray bubbling area is the total column cross section less area of two downpipes.

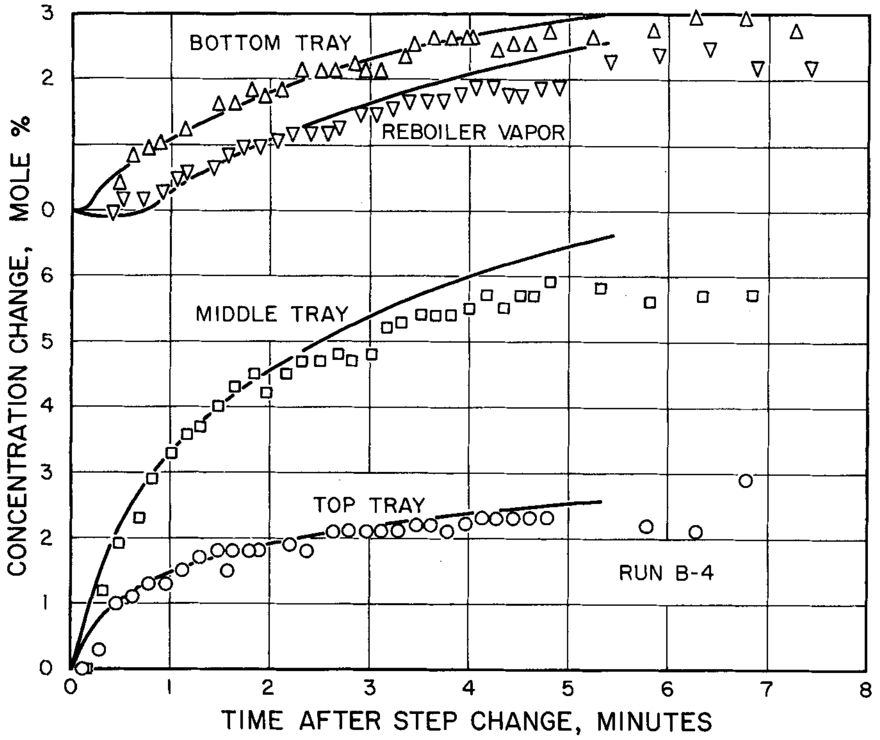

Fig. 1. Composition perturbations for tray liquids and reboiler vapor as a function of time after a step change in liquid reflux rate to the tower. Points are experimental values, solid lines are analogue predictions.

as to maintain the reboiler holdup constant during the transient.

The same procedure was followed for the runs where a step change in vapor rate was introduced, except that instead of increasing the reflux rate to the tower the steam rate to the reboiler was lowered. The valve between the reflux drum and Tank 6 was closed at the same instant, and bottoms product was withdrawn. The liquid rate step change was executed in less than a few seconds, but the reboiler response to a change in steam rate was such that the decrease in vapor rate took place over about a 20-sec. period.

During the transient, composition-time measurements were taken of the liquid leaving the top, middle, and bottom trays and the reboiler vapor. A portion of each tray liquid or reboiler vapor was continuously removed from the column through fine-bore tubing which passed through coolers before discharging to the atmosphere. The sample streams flowed at a rate of 1 cc./sec.; portions of each sample stream were collected for a period of 2 sec. every 10 sec. after the step change. The contents of each sample bottle were analyzed later for composition with a five-place refractometer. As acetone and benzene have considerably different indexes of refraction, the precision of the analytical method was better than 0.1 mole %.

The holdups of the sample system were carefully determined; plug flow within the sample lines was assumed in computing sample lags.

The flow rate of liquid within the column was calculated from knowledge of the metered flow of cold reflux to the column and enthalpy balances on the top tray. The flow rate of vapor within the column was calculated from measurements taken on the rate of steam condensate from the reboiler and on the rate and temperature rise of the water flowing through the condenser.

Values of liquid holdup on the trays and in the downpipes were measured experimentally in previous steady state runs made with this system (*7*). The operating liquid holdups in the reboiler system were also determined experimentally.

EXPERIMENTAL RESULTS

Figure 1 shows the composition-time behavior obtained experimentally for Run B-4 in which the cold reflux rate to the top tray was increased by 6.3%. The time shown is the actual time the sample discharged from the tray rather than the time the sample arrived at the sample bottle. The shape of the curves obtained is typical of the response to a step change which is normally encountered, a rapidly changing initial response followed by an asymptotic approach to a final steady state value. A typical set of experimental response data for a vapor perturbation run is shown in Figure 2. Response data for all runs are summarized in Table 2* and in the thesis of Baber (*3*).

Tray efficiencies were evaluated from McCabe-Thiele diagrams at the initial and final steady state conditions. At the initial condition of total reflux the tray efficiencies ranged from 70 to 85% for all trays except the top tray depending upon the vapor and liquid flow rates. The top tray efficiency was 10 to 20 efficiency % lower than the other trays because the reflux entering that tray was cold; from 15 to 30% of the vapor entering the top tray remained permanently condensed in the top tray liquid to supply the enthalpy required to raise the cold reflux to the boiling point. Thus from 15 to 30% of the tray was inoperable. It is likely that the lowered length of liquid path available for normal mass transfer on the top tray is responsible for the lowering of the top tray efficiency. The predicted difference between the point efficiency and the Murphree efficiency for a normal tray in the present instance averages about 15 efficiency %, and much of this efficiency improvement is probably lost on the top tray.

Tray compositions at the final steady state were obtained by extending the response data typified by Figure 1 to reasonable final values. Tray efficiencies were computed from these final tray compositions by McCabe-Thiele diagrams in which the operating line slope corresponded to the L/V existing in the column after the step change (L/V at the final condition was always greater than unity). These tray efficiencies were very close in value to those found for the initial steady state condition (Figure 3).

MATHEMATICAL PREDICTIONS

Perturbation Equations

The Lamb, Pigford, and Rippin equations (*8*) assume that changes in pressure, composition, and temperature within the distillation system can be expressed as small, linear perturbations from a steady state value. Other assumptions include lack of appreciable vapor holdup, constant tray efficiency, and a completely-mixed tray liquid. The vapor-liquid equilibrium relationship is most conveniently taken to be linear for each tray but is allowed to have a different slope for each tray. For the case of a 100% tray efficiency the perturbation equations for a typical tray n reduce to the following:

$$\frac{dx_n}{d\theta} = (\bar{x}_{n+1} - \bar{x}_n)(L_{n+1}/\bar{L}_n) - \bar{y}_n (V_n/\bar{L}_n) + \bar{y}_{n-1} (V_{n-1}/\bar{L}_n) + x_{n+1} (\bar{L}_{n+1}/\bar{L}_n) + m_{n-1} x_{n-1} (\bar{V}_{n-1}/\bar{L}_n) - x_n \left(1 + \frac{\bar{V}_n}{\bar{L}_n} m_n\right) \quad (1)$$

A bar over a variable indicates that it is a steady state value, while variables lacking the bar are the deviations or perturbations from the steady state value; for example the liquid composi-

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* Tabular material has been deposited as document 7157 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for $1.25 for photoprints or for 35-mm. microfilm.

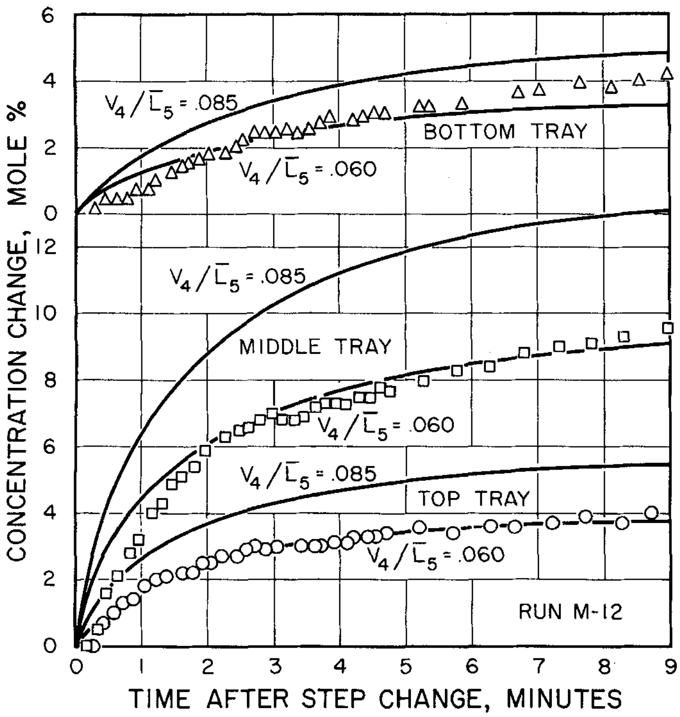

Fig. 2. Composition perturbations for tray liquids as a function of time after a step change in vapor rate to the tower. Points are experimental values. Two prediction cases are shown as solid lines, each with a different value of ratio of perturbation rate to steady state liquid rate ($V_4/\bar L_5$).

tion on tray n at any instant is $\bar x_n + x_n$. The variable θ is the ratio of elapsed time to the time required for the liquid to traverse a tray. Equation (1) does not assume equal molal overflow, but because it assumes lack of appreciable vapor holdup in the column, $V_{n+1} = V_n = V_{n-1}$; that is any perturbation in vapor rate is felt immediately throughout the column.

Liquid holdup on the trays of a column does change with liquid rate, and for this reason a liquid perturbation to the top tray is not felt immediately on the other trays. Liquid perturbations on adjacent trays are related to each other by the relationship

$$\frac{dL_n}{d\theta} = (1/\beta_n)(L_{n+1} - L_n) \qquad (2)$$

where $\beta_n = (\bar L_n/\bar H_n)\ (d\bar H_n/d\bar L_n)$, and $\bar H_n$ is the steady state holdup on tray n.

Assumption of 100% tray efficiency does not impose an undue limitation upon the use of Equation (1). For cases where the efficiency is not 100%, a pseudoequilibrium curve is employed: this curve is drawn with vapor compositions taken as the true equilibrium vapor compositions multiplied by the Murphree vapor tray efficiency E_{MV}; the dashed line in Figure 3 illustrates such a pseudocurve for Run B-9. Thus Equation (1) is valid for all efficiencies provided the slopes of the equilibrium curve (m-values) are taken from the pseudoequilibrium curve. This concept reduces the complexity of the required equations.

The equation analogous to Equation (1) for a well-mixed, constant holdup, 100% efficient reboiler is

$$\frac{dx_R}{d\theta}\bar h_R = \bar x_1\ (L_1/\bar L_1) - \bar x_1\ (L_B/\bar L_1) - y_R\ (V_R/\bar L_1) + x_1\ (V_R/\bar L_1) - x_R\ (\bar V_R/\bar L_1)\ m_R \qquad (3)$$

This equation applies, as in the present instance, to a column wherein the bottoms product is part of the liquid leaving the bottom tray; $\bar L_B + L_B$ is the flow rate of this bottoms product. In the first series of tests reported in this paper a step increase in liquid reflux rate is applied to the total reflux column without changing the vapor rate, and the reboiler holdup is maintained constant by withdrawing bottoms product; in this case $L_1 = L_B$ and $V_R = 0$ so that Equation (3) reduces to

$$\frac{dx_R}{d\theta} = (x_1/\bar h_R)\ (\bar V_R/\bar L_1) - x_R\ (\bar V_R/\bar L_1)\ (m_R/\bar h_R) \qquad (4)$$

In the second series of tests reported in this paper a step decrease in vapor rate from the reboiler is applied to the total reflux column without changing the reflux rate at the top, and the reboiler holdup is maintained constant by withdrawing bottoms product; in this case — $V_R = L_B$ and $L_1 = 0$ so that Equation (3) once more reduces to Equation (4).

Perturbation Equation for Top Tray

As mentioned before the nature of the system employed insured equal molal overflow on all internal trays of the column. However the reflux to the top tray was well below the boiling point so that the liquid rate to and from the top tray differed by from 13

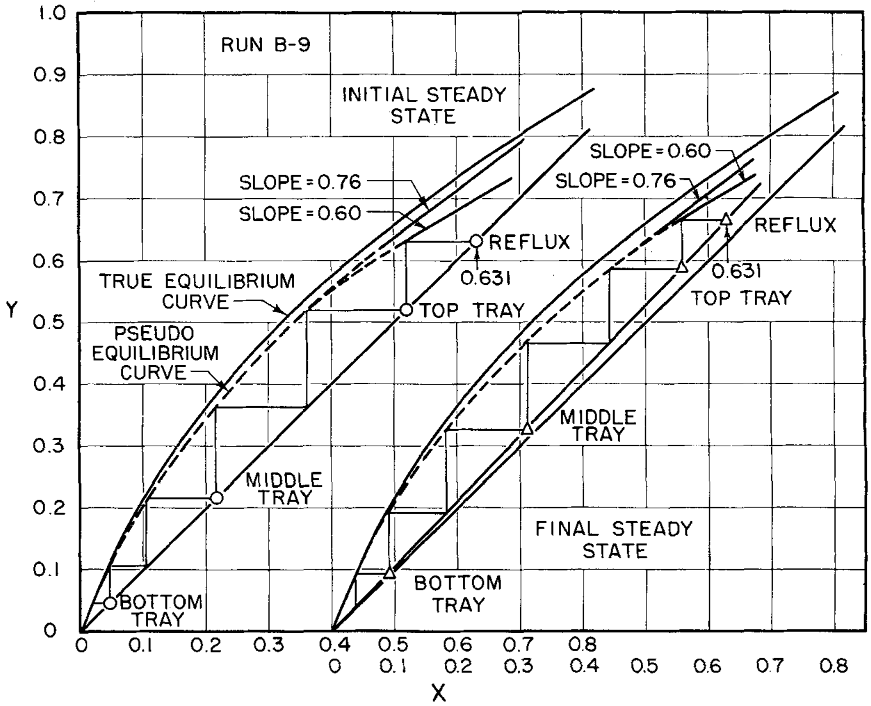

Fig. 3. McCabe-Thiele diagram showing initial and final steady state conditions within tower. Points show experimental tray compositions.

to 32%. As liquid and vapor rates to and from the top tray could be computed by enthalpy balances, it was first believed that Equation (1) would be suitable for predicting top-tray behavior providing proper values were used. However predicted results differed considerably from the experimental values (as shown later), so a different approach was employed.

TABLE 3. KEY TO ANALOGUE CIRCUIT GIVEN ON FIGURE 4

Potentiometer number	Designation for Liquid-rate runs	Vapor-rate runs	Numerical value for typical liquid-rate run, B-4
1	$L_C/\bar{L}_5$	$V_4/\bar{L}_5$	0.0450
2	$(\bar{x}_C - \bar{x}_{5a})/F$	$\bar{y}_4$	0.096
3	$m_4(\bar{V}_4/\bar{L}_5)$	same	0.814
4	$1/F$	same	0.385*
5	$1/(1-F)$	same	0.135*
6	$\frac{1 + (\bar{V}_5/\bar{L}_5)m_5}{(1-F)}$	same	0.239*
7	$m_4(\bar{V}_4/\bar{L}_5)$	same	0.814
8	$1 + (\bar{V}_4/\bar{L}_4)m_4$	same	0.1814*
9	$m_3(\bar{V}_3/\bar{L}_4)$	same	0.1040*
10	$1 + (\bar{V}_3/\bar{L}_3)m_3$	same	0.2040*
11	$m_2(\bar{V}_2/\bar{L}_3)$	same	0.1345*
12	$1 + (\bar{V}_2/\bar{L}_2)m_2$	same	0.2345*
13	$m_1(\bar{V}_1/\bar{L}_2)$	same	0.1894*
14	$1 + (\bar{V}_1/\bar{L}_1)m_1$	same	0.2894*
15	$m_R(\bar{V}_R/\bar{L}_1)$	same	0.2450*
16	$(m_R/\bar{h}_R)(\bar{V}_R/\bar{L}_1)$	same	0.3410
17	$(1/\bar{h}_R)(\bar{V}_R/\bar{L}_1)$	same	0.1352
18	$(\bar{x}_{5a} - \bar{x}_5)/(1-F)$	$(\bar{y}_5 - \bar{y}_4)$	0.096
19	$(\bar{x}_5 - \bar{x}_4)$	$(\bar{y}_4 - \bar{y}_3)$	0.140
20	$(\bar{x}_4 - \bar{x}_3)$	$(\bar{y}_3 - \bar{y}_2)$	0.145
21	$(\bar{x}_3 - \bar{x}_2)$	$(\bar{y}_2 - \bar{y}_1)$	0.130
22	$(\bar{x}_2 - \bar{x}_1)$	$(\bar{y}_1 - \bar{y}_R)$	0.080
23-30	$1/\beta$	not used	0.508†

Dead time

D.T.-1

Blocks on Figure 4 with this designation were used to simulate the dead time in the downpipes between trays; the actual circuit used is given as a detail on the figure. The magnitude of the downpipe dead time, in units of θ, was 0.522 for Run B-4 and 0.462 for Run M-3.

D.T.-2

This block on Figure 4 was used to simulate the dead time in the transfer pipe between the bottom tray and the reboiler. The same circuit used for downpipe dead time was used in this instance also. The value of θ was 9.73 for Run B-4 and 9.84 for Run M-3.

Sign of input voltage

It will be noted that the 100-v. input shown on Figure 4 is negative for the liquid-rate runs and positive for the vapor-rate runs. The reason for the difference in sign is that in the liquid-rate runs the perturbations were positive, that is the liquid rate was increased, while in the vapor-rate case the perturbations were negative (vapor rate was decreased).

* Actual values have been divided by 10, as a gain of 10 is used in the following amplifier.
† Actual values have been divided by 5, as a gain of 5 is used in the following amplifier.

The procedure used for the top tray is to assume that it consists of two parts: a portion which totally condenses vapor to raise the entering cold liquid to the boiling and a portion which receives and discharges saturated liquid and vapor streams as a normal tray. Application of Equation (1) to the first portion of the tray gives

$$\frac{dx_{na}}{d\theta}\bar{h}_a = \bar{x}_{n+1}(L_{n+1}/\bar{L}_{na}) - \bar{x}_{na}\left[\frac{L_{n+1} + F\,V_{n-1}}{\bar{L}_{na}}\right] + \bar{y}_{n-1}\,(FV_{n-1}/\bar{L}_{na}) + x_{n+1}\,(\bar{L}_{n+1}/\bar{L}_{na}) + m_{n-1}\,x_{n-1}\,(F\bar{V}_{n-1}/\bar{L}_{na}) - x_{na} \quad (5)$$

The second liquid portion on the top tray acts as a normal tray except that its holdup and vapor loading are less than that of a normal tray. Thus its equation becomes

$$\frac{dx_n}{d\theta}\bar{h}_b = (\bar{x}_{na} - \bar{x}_n)(L_{na}/\bar{L}_n) - \bar{y}_n\,(V_n/\bar{L}_n) + \bar{y}_{n-1}\,(1-F)(V_{n-1}/\bar{L}_n) + x_{na}\,(\bar{L}_{na}/\bar{L}_n) + m_{n-1}\,x_{n-1}\,(1-F)(\bar{V}_{n-1}/\bar{L}_n) - x_n\left(1 + \frac{\bar{V}_n}{\bar{L}_n}m_n\right) \quad (6)$$

(It is well to note that $\bar{h}_b = 1 - F$, and $\bar{h}_a = F$, and that in the general case, but not the present case, F is a variable changing with the magnitude of L_{n+1} and the temperature of $\bar{L}_{n+1}$.)

Solution of Equations by Analog Computer

The composition-time behavior of the five trays and reboiler used in the present experiments were predicted by solving simultaneously the differential equations applying to each tray and to the reboiler. This was accomplished using the university's Pace analogue computer with the analogue circuit shown in Figure 4. Designations for each potentiometer setting and values employed for a typical run are shown in Table 3.

For the runs involving a step change in reflux rate to the column there were no vapor perturbations and no change in reflux composition. Thus in Equations (5) and (6) used for the top tray V_{n-1}, V_n, and x_{n+1} were each zero. The value of $\bar{L}_n$ was taken as the average of the initial and final steady state values. For the reboiler vapor-rate perturbation runs values of L_{n+1}, L_{na}, and x_{n+1} were each zero in Equations (5) and (6). In both sets of runs, when Equation (1) was applied to each of the lower four trays and Equation (4) was applied to the reboiler, averages of the initial and final steady state values were employed for steady state compositions, flow rates, and slopes of the equilibrium curve (m-values).

Liquid holdup in the downpipes averaged about 50% of the tray liquid holdup. To account for this comparatively large holdup, plug flow of liquid through each downpipe (dead time) was assumed in the analogue program.

The liquid transfer line from the bottom tray to the reboiler also contained considerable holdup, and no mixing in this line was assumed to occur in setting up the analogue circuit. The main reboiler system was taken to be a single well-mixed pool of liquid.

RESULTS

The experimental changes in composition of the tray liquids and the reboiler vapor during the transient period were closely predicted by the perturbation equations. Comparison of the experimental and predicted composition-time behavior for four typical runs is shown in Figures 1, 2, 5, and 6; the

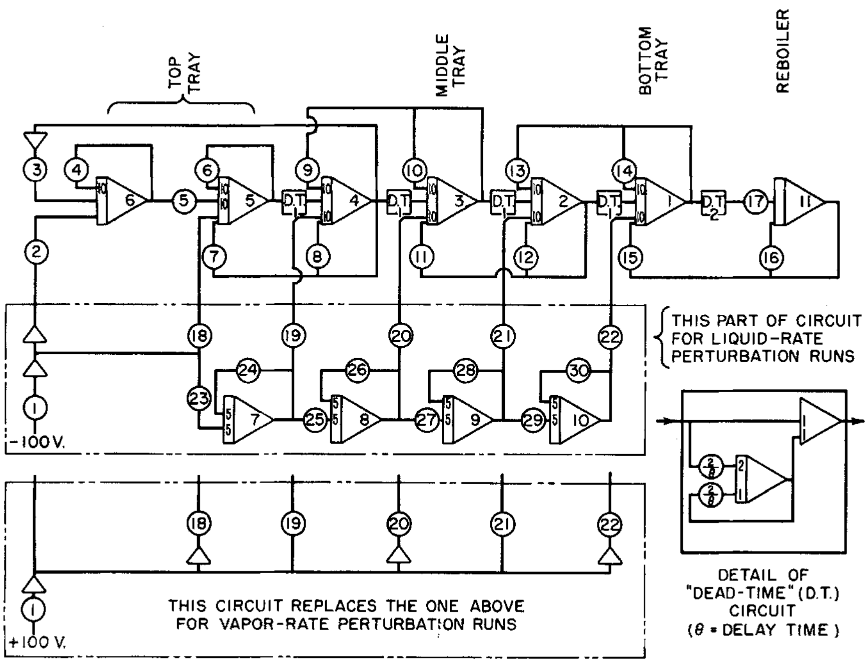

Fig. 4. Analogue circuit used to solve Equations (1) to (6) for five-tray column.

points are the experimentally-determined values, while the solid lines are the analogue-computer predictions. A comparison of experimental and predicted values for all of the runs is shown in Table 2*, while a summary of deviations of predicted values from experimental values is shown in Table 4. Study of this latter table shows that the predicted changes in composition fall both above and below the experimental values with an absolute average deviation of 0.37 mole %. This figure may be compared with the average total composition change on each of the trays, which was 3.5 mole %. The deviations are about the same for any of the tray liquids or the reboiler vapor when considered individually; they were not appreciably different at various values of θ, that is at various points during the transient.

The close agreement between predicted and experimental results was unaffected by the magnitude of the step change in liquid rate, which increased by as much as 8.6% of the entering reflux rate. The vapor-rate step changes were 8.5% or less of the entering vapor rate for all runs except M-1, which was 21.5% of the original vapor rate; the magnitude of the vapor step change also had no effect upon the accuracy of the predictions except for Run M-1. In this latter instance such large composition changes were involved that the assumption of linearity for the equilibrium curve over any one tray was considerably in error.

The results were equally consistent regardless of the operating pressure, temperature, vapor flow rate, or liquid flow rate which was employed. An exception to this is Run B-8, which was at a vapor rate corresponding to 91% of flooding. In this instance predicted compositions at any θ were as much as 1.6 mole % higher than the experimental values. It is possible that high entrainment caused this run to have greater deviations between predicted and experimental values than the other runs.

Perturbations in liquid or vapor rate gave equally good agreement with theory. It may be concluded that the perturbation equations used are applicable to a semicommercial distillation unit over a wide range of conditions.

The prediction equations assume a well-mixed tray liquid (except for the top tray which was specially treated) and a constant tray efficiency. The predicted and experimental results indicate that the assumptions are valid.

It is of interest to note that transient oscillatory responses were not found in the present series of tests, either experimentally, or in the analogue predictions.

Agreement over Entire Transient Period

In proceeding from the initial to the final steady state the composition of the tray liquids changed in several runs by as much as 10 mole % and on the average changed about 5 mole %. These composition changes cause some change in the values for m, the slope of the pseudoequilibrium curve, during the transient period. No attempt was made in the analogue program to consider these variations. Instead average values of m were employed. Thus perfect agreement between experimental and predicted composition perturbations over the entire transient period cannot be expected; these differences are most pronounced in Figure 1, as in most of the runs, near the final steady

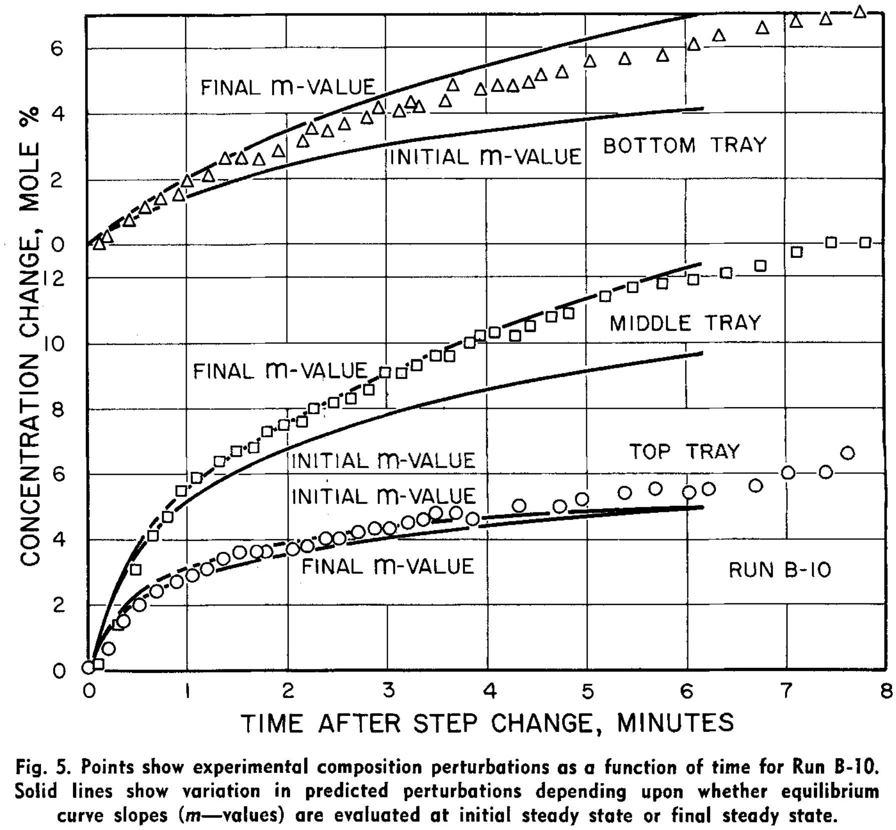

Fig. 5. Points show experimental composition perturbations as a function of time for Run B-10. Solid lines show variation in predicted perturbations depending upon whether equilibrium curve slopes (*m*—values) are evaluated at initial steady state or final steady state.

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* See footnote on page 409.

state condition. If m values corresponding to final steady state compositions had been used in the predictions, the final steady state would be predicted exactly, but the predicted relationship during the early part of the transient would deviate more from the true values.

Some idea of the changes in the predicted result which occur when m values other than the average are used may be obtained from study of Figure 5. In this figure two predicted curves are shown: one with all m values evaluated at the initial steady state condition and one with all m values evaluated at the final steady state condition. The prediction for the case where average m values are employed (not shown on the figure) lies intermediate between the two extreme values shown. Figure 5 shows that variations in the values employed for m are not too important during the early part of the transient, which is the period of importance insofar as automatic control is concerned.

Evaluation of Top Tray

As discussed previously, special equations were required to characterize closely the top tray behavior because of the cold reflux stream entering the tray. It was originally hoped that Equation (1) would be suitable in this regard, but predicted values for the top tray were considerably in error (Figure 7). And because the top tray perturbations were predicted high corresponding values for the other trays were also high, although the effect became less in proceeding away from the top tray. This trend is clearly shown in Figure 7. Dividing the top tray into two portions or pools for the purpose of making the transient predictions solved the problem.

Another problem presented itself in evaluation of the top tray, and this was proper choice of an m value for this tray. The markedly lower efficiency for the top tray caused a shift in the slope of the pseudoequilibrium curve at the top tray. Also, because no tray existed above the top tray, location of the pseudoequilibrium curve at the top tray was more difficult than for the other trays. Figure 3 shows how, in absence of firm efficiency values for the top tray, the pseudoequilibrium curve slope might easily be taken as 0.76 for Run B-9. The marked change in all of the predictions when the average slope for this one tray is not properly evaluated is shown in Figure 6.

Treatment of Downpipe Holdup

Values of liquid holdup in the downpipes were measured experimentally with a manometer attached to the floor of the downpipe; the result was checked by visual observation through a window placed in one of the downpipes. These holdup values are given in Table 1, where it may be seen that they averaged about one half as much as the average liquid holdup on a tray. The analogue predictions were made with

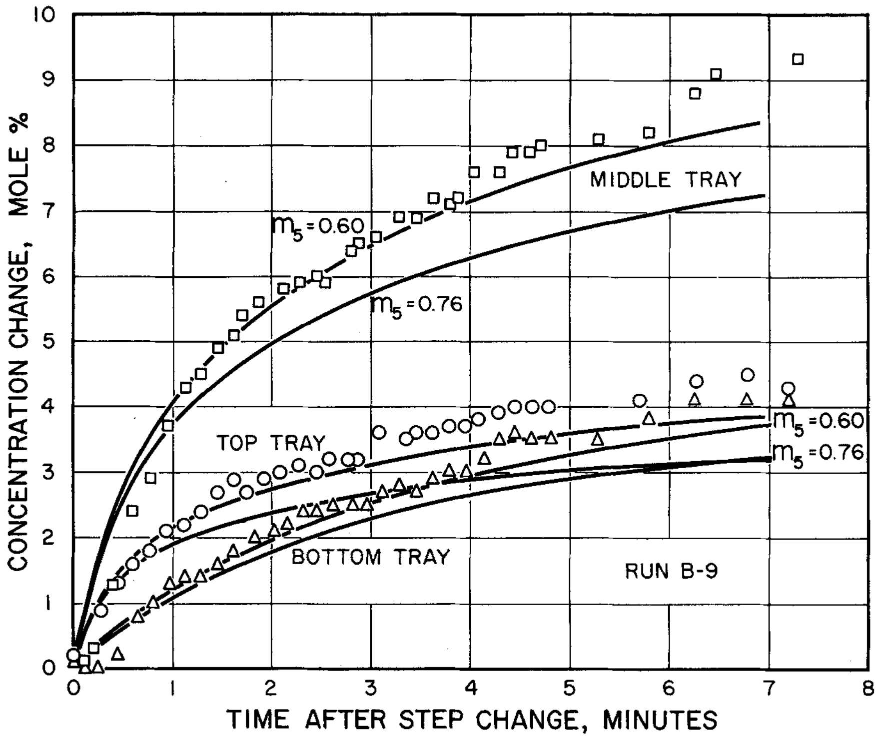

Fig. 6. Points show experimental composition perturbations as a function of time for Run B-9. Solid lines show variation in predicted perturbations with average value employed for equilibrium curve slope of top tray (m_5).

Table 4. Summary of Deviations of Predicted Values of Tray Liquid and Reboiler Vapor Compositions from Experimental Values
(Taken from Table 2)

	Average of deviations, mole %	Absolute average of deviations, mole %
Liquid-rate perturbation runs		
Top-tray liquid compositions	+0.25	0.39
Middle-tray liquid compositions	+0.25	0.58
Bottom-tray liquid compositions	—0.07	0.31
Reboiler-vapor compositions	—0.03	0.38
Tray liquid and reboiler vapor compositions at		
$\theta = 10$	+0.14	0.32
$\theta = 30$	—0.01	0.35
$\theta = 50$	+0.11	0.42
$\theta = 80$	+0.17	0.57
Average for all liquid-rate runs	+0.10	0.41
Vapor-rate perturbation runs*		
Top-tray liquid compositions	—0.14	0.28
Middle-tray liquid compositions	+0.41	0.51
Bottom-tray liquid compositions	—0.02	0.28
Reboiler-vapor compositions	+0.01	0.25
Tray liquid and reboiler vapor compositions at		
$\theta = 10$	+0.15	0.32
$\theta = 30$	+0.02	0.29
$\theta = 50$	+0.02	0.31
$\theta = 80$	+0.11	0.41
Average for all vapor-rate runs	+0.07	0.33
Average for all runs	+0.09	0.37

(The average total composition change on each of the trays was 3.5 mole %.)

* Does not include Run M-1.

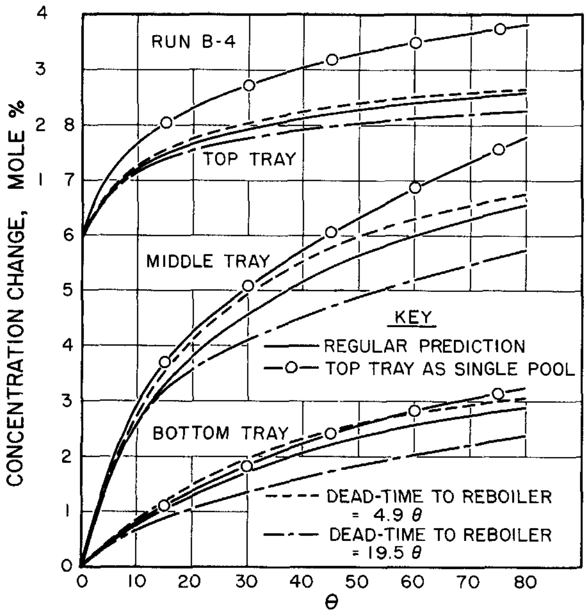

Fig. 7. Effect of treating top tray (where cold reflux enters) as single well-mixed pool upon predicted result. Also shown is effect upon predicted result of varying magnitude of dead time in pipe between bottom tray and reboiler.

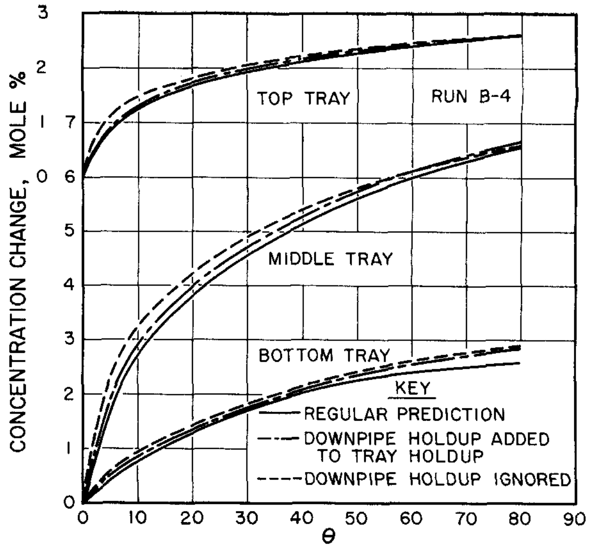

Fig. 8. Effect upon predicted result of not including downpipe holdup in analogue program. Also shown is effect of adding downpipe holdup to tray holdup.

liquid flow through each downpipe treated as plug flow or dead time (see Figure 4). Thus the composition perturbation in the liquid flowing from an upper to a lower tray was delayed by the downpipe by about 0.5 θ. Inspection of any of the response curves (Figures 1, 2, 5, or 6) shows that a 0.5 θ delay is quite small compared with the overall time for the column to arrive at a new steady state.

To demonstrate the relative importance of including downpipe holdup in the analogue predictions a predicted result for Run B-4 is shown in Figure 8 for the case where the downpipe holdup is taken as zero. This result, shown as a dashed line, indicates a slightly faster response for the case where downpipe holdup is ignored compared with the regular prediction result (solid line). A prediction was also made with downpipe holdup treated as a first-order lag, and in this instance the predicted result was coincident with the dead-time case.

An alternate method for treatment of downpipe holdup is to assume that the tray holdup consists of the actual tray holdup plus the downpipe holdup. The analogue circuit is then the same as that for the case of zero downpipe holdup. The dimensionless time θ is now the ratio of elapsed time to the time required for the liquid to traverse a tray plus its downpipe, and $\bar{h}_R$ is the ratio of reboiler holdup to the holdup on a tray plus its downpipe. Dead time in the pipeline to the reboiler must be expressed in terms of the new θ. Predicted results for this situation, shown in Figure 8, indicate a closer agreement with the regular prediction results than if the downpipe holdup is ignored completely. And, because this treatment reduces markedly the number of amplifiers required in the analogue program without reducing too greatly the accuracy of the predicted result, it may be preferred for practical reasons in situations where the number of trays is large.

Treatment of Reboiler System

The thermosyphon reboiler system used with the experimental column is more complex than the simple pot employed in some test systems. To make certain that the response of the reboiler system could be predicted accurately, the experimental composition-time behavior was obtained in all of the runs for the liquid leaving the bottom tray (to the reboiler system) and the vapor entering the bottom tray (from the reboiler system). A typical experimental result is shown in Figure 1, where it may be seen that the reboiler vapor composition lags the bottom tray liquid composition by a nearly constant value of time. These two compositions are exactly equal at the initial steady state and are very nearly equal at the final steady state. Inspection of the lag times showed them to be very nearly equal to the time required for the liquid to travel in plug flow through the 6-in. pipe connecting the bottom tray and the entrance to the reboiler. With this in mind the reboiler system was simulated in the analogue program as a combination of dead time plus a completely-mixed, 100%-efficient reboiler. The holdup in the separator and in the pipe from the separator to the reboiler entrance were both ignored; the reboiler holdup was taken as the liquid holdup within the tubes of the thermosyphon reboiler under operating conditions. This latter quantity was measured experimentally.

Figure 7 shows for Run B-4 the importance of properly simulating the reboiler response in the analogue prediction. The regular program shown as a solid line utilizes 9.73 θ, the experimentally-measured value, as the dead time in the pipeline to the reboiler. If this dead time is taken as either one half or twice this value, the tray responses as shown on Figure 7 are altered considerably. Changes in the tray responses were also found if the reboiler holdup values were markedly increased. Doubling of the reboiler holdup did not produce as great a change in the tray responses however as changing the dead time in the pipe to the reboiler by a factor of 2.

The analogue circuitry used to simulate pure dead time is shown on Figure 4. This circuitry did not give an exact time delay, but, as shown by the reboiler-vapor predicted line on Figure 1, an extremely good representation was possible.

Some analogue explorations were carried out with a more complex representation for the reboiler system. The components considered were pure dead time in pipe to reboiler, zero time lag within reboiler itself, contents of separator as well-mixed liquid, pure dead time in pipe from separator to reboiler. The program was not too useful because no knowledge was available as to the internal liquid circulation rate between the separator and reboiler. Separate experimental studies are underway to evaluate the response of the reboiler system more carefully, although it may be safely concluded that the model used in the regular analogue prediction program of this paper gave an adequate representation of the reboiler system.

Propagation of Liquid Rate Perturbations Down the Column

For the series of runs where a step change in reflux rate to the top tray was employed the analogue predictions were made with Equation (2) to predict the rate at which the liquid perturbation worked its way down the column. An experimental value was used for β_n, the rate of change of tray liquid holdup with respect to a change in liquid rate; a flow change of 41.6 gal./min. is required to effect a change in tray holdup of 1 gal. In the runs of this study, where the step change in reflux rate averaged 1.95 gal./min., the corresponding change in tray holdup was only 0.047 gal. which was 2% of the average tray holdup of 2.6 gal. When an analogue prediction for Run B-4 was made with β_n taken as zero, the tray responses were identical to those found previously where the experimental value of β_n was employed; thus in the present case it would have been satisfactory to assume that the liquid rate perturbation to the top tray was felt immediately by all other trays of the column.

Importance of Perturbation Rate

In the analogue predictions small changes in the magnitude of the vapor or liquid-rate perturbations cause comparatively large differences in the predicted results. In the liquid-rate perturbation runs the magnitude of the step change was measured with extremely good precision by calibrated rotameters in the reflux supply line. However in the vapor-rate perturbation runs, where the quantity of steam condensate from the reboiler was used as a means to determine the vapor rate within the column, the magnitude of the vapor perturbation was not known as accurately. In four of the vapor perturbation runs the predicted results were improved markedly when small changes were made in the values used for the vapor perturbations; the final vapor rates were changed by only −1.7, +0.8, +2.1, and −2.2% of the measured values, but these changes caused the magnitude of the perturbation to differ by −27%, +18%, +46%, and −29%, respectively. Figure 2 shows the effect of an uncertainty in the magnitude of the vapor perturbation upon the predicted result for Run M-12, where it may be seen that a −29% change in this quantity effects a comparatively large change in the predicted result.

CONCLUSION

Linear perturbation types of equations have been shown to predict satisfactorily the composition-time behavior of the tray liquids within a distillation column when a step change is made in either the liquid rate or the vapor rate to the column. It is permissible to assume that the tray liquids are completely mixed, that downpipe holdups can be ignored, and that average values for the pseudoequilibrium curve slope for each tray can be employed. Special procedures are suggested for including the effects of tray efficiency and cold reflux or cold feed to a tray. It may be concluded that the relatively simple perturbation equations of Lamb, Pigford, and Rippin (8) may be used with confidence to characterize the transient behavior of distillation columns.

ACKNOWLEDGMENT

Funds for this study were kindly made possible by a grant from the National Science Foundation.

NOTATION

F = fraction of total vapor entering a tray which remains permanently condensed because of cold reflux to tray
$\bar{H}$ = steady state liquid holdup on tray
$\bar{h}_a$ = fraction of $\bar{H}$ which discharges no vapor because of cold reflux to tray
$\bar{h}_b$ = $1 - \bar{h}_a$
$\bar{h}_R$ = ratio of reboiler holdup to tray holdup at steady state
$\bar{L}$ = steady state liquid rate, lb. moles/hr.
L = perturbation in liquid rate, lb. moles/hr.
m = slope of vapor-liquid equilibrium curve or of pseudoequilibrium curve
$\bar{V}$ = steady state vapor rate, lb. moles/hr.
V = perturbation in vapor rate, lb. moles/hr.
$\bar{x}$ = steady state liquid composition, mole fraction
x = perturbation in liquid composition, mole fraction
$\bar{y}$ = steady state vapor composition, mole fraction
y = perturbation in vapor composition, mole fraction
β = $(\bar{L}/\bar{H})(d\bar{H}/d\bar{L})$
θ = ratio of elapsed time to time required for liquid to traverse a tray

Subscripts

B = bottoms product, withdrawn from liquid entering reboiler
C = cold reflux stream to top tray
n = any tray in distillation column
$n+1$ = tray above tray n
$n-1$ = tray below tray n
na = position on tray between liquid portion which discharges no vapor (due to cold reflux) and liquid portion which does discharge vapor
R = reboiler
1,2, etc. = tray number, counting up from reboiler

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Manuscript received January 8, 1962; revision received March 26, 1962; paper accepted April 4, 1962. Paper presented at A.I.Ch.E. New York meeting.