

TABLE 2. FINAL NUMERICAL RESULTS

| Variable                     | Input value             | Newton-Raphson value   |
|------------------------------|-------------------------|------------------------|
| $h$                          | $25.000 \times 10^{-3}$ | $25.00 \times 10^{-3}$ |
| $k$                          | $2.2000 \times 10^{-3}$ | $2.200 \times 10^{-3}$ |
| $N_{Pe}$                     | 25.000                  | 25.00                  |
| $(N_{Pe})_{\text{particle}}$ | 2.0000                  | 2.000                  |
| $\lambda(\omega = 0.75)$     | 0.221049                | 0.2210                 |
| $\lambda(\omega = 2.00)$     | 0.582266                | 0.5823                 |
| $\lambda(\omega = 4.00)$     | 0.736841                | 0.7368                 |
| $\lambda(\omega = 5.00)$     | 0.765859                | 0.7659                 |
| $f_{1p}$                     | 0.00000                 |                        |

heat of the spherical packing  $(\rho c)_s$ , the values of  $\Pi = \ln(A_{\text{out}}/A_{\text{in}})$ , and the phase lag  $\psi$  at  $j$  values of the frequency.

#### (SIMULATED) EXAMPLE

The values of the above quantities are given in Table 1; those of  $\Pi$  and  $\psi$  were computed for a number of frequencies and the underlined values were those used in the numerical computation.

Figure 2 shows the curves  $f_{1p}(h, k) = 0$  for the various combinations of  $\omega_j$  ( $j = 1 \dots 6$ ). The true root was found by calculating  $N_{Pe}$  from Equation (8) and eliminating those that gave negative values. Table 2 lists the final values obtained; it will be seen that the unknowns  $h$ ,  $k$ , and

$N_{Pe}$  were recaptured to four significant figures by the Newton-Raphson method. (At least one graph is recommended to determine the search area and because visualization of the shape of the  $f_{1p} = 0$  lines helps in planning the computational attack.)

#### ACKNOWLEDGMENT

This work was carried out with financial support from the National Research Council of Canada; our thanks are due to this body for the operating grants and for the award of a postgraduate scholarship to M. J. Goss.

The authors are most grateful to V. Taht of the Statistics Department, University of Waterloo, Waterloo, Ontario, for writing and converting programs.

#### NOTATION

$k_e, k'_e$  = initial trial estimate of  $k$   
 $j$  = number of discrete values of the frequency  
 $N_{Pe} = UL/D$  = tube Peclet number, dimensionless  
 $p$  =  $p^{\text{th}}$  set of three frequencies  
 $r_0$  = radius of the spherical packing, cm.

#### Subscript

$i (= 1, 2 \dots) = i^{\text{th}}$  root satisfying  $(f_1)_p = 0$ ,  $p = \text{two values}$

#### LITERATURE CITED

1. Turner, G. A., *AIChE J.*, 13, 678 (1967).
2. Goss, M. J., Ph.D. thesis, Univ. Waterloo, Ontario (1969).

Manuscript received August 5, 1969; revision received April 15, 1970; paper accepted April 23, 1970.

## III. Experimental Method and Results

This part describes an experimental essay at finding simultaneously the values of the heat transfer coefficient, the dispersion coefficient, and the thermal conductivity of the packing in a packed bed of spheres. The logic of the method was presented in Part I, while the computational technique of calculating the numerical values of these quantities was presented in Part II. A sufficient number of experimental results are presented to demonstrate the potential of the method as a means of obtaining information that has previously been obtainable either with great uncertainty or not at all.

A method of finding three unknown parameters—heat transfer coefficient, thermal conductivity of the packing, and the longitudinal Peclet number—from a dispersion model of a packed bed of spheres with fluid flowing through it has been described in Part I (1) and demonstrated in Part II (2) by simulation. It was shown that the values of these parameters could be obtained to four significant figures with modest amounts of computer time, but that the allowable errors in the input quantities had to be small. This seemed likely to present an experimental challenge, and this part describes the present state of an apparatus which has produced some results. Three of these are presented: two which show that the measured values were close to reported values, while the third, although producing results of the right order of magnitude, is put in to demonstrate why care is needed in choosing ranges of the input quantities.

#### APPARATUS

##### General

To use the method described in Parts I and II, the physical system had to be made to agree with the model as closely as

possible. The aim was for tolerances of meaningful quantities not to exceed one part in a thousand. Thus the temperature wave was to be of constant amplitude, of constant known frequency, and was to have as low and constant a harmonic content as possible in order to facilitate measurement.

The fluid flow was to be constant, nonrotational, and parallel to the axis of the system. Radial variations of velocity and temperature were to be small (within a few percent of the mean) at least in the central measurement region.

The apparatus is shown in Figure 1; further details are given in reference 9.

Air, supplied by the compressor 2, was cooled to its dew point in order that subsequent heating by the temperature controller raised the mean temperature to that of the laboratory. The interchanger used a glycol solution cooled in tank 11 by refrigeration unit 9. The cooled air passed downward through a vacuum jacketed glass tube shown in Figure 2 (8.6 cm. I.D. by 186 cm. long) containing the temperature control devices, the bed with its associated thermometers, and the flow straighteners. The air, after emerging from the end of this vacuum jacketed tube (which was in addition wrapped in 1 in. styrofoam insulation), passed upward, outside it, in an annular space inside tube 20. By this procedure it was possible to enjoy a combination of effective insulation and small temperature gradients. A rough calculation gave for the worst case (namely,

10°C. gradient across a silvered vacuum jacket of  $10^{-4}$  mm. Hg) gave a radial input of 0.17 C.h.u./(hr.) (sq. ft.), averaged across the inner tube. At an air flow rate of 130 cu.ft./min. this corresponds to a temperature uncertainty of 0.0005°C., which was borne out by experimental measurements on an earlier apparatus of similar type.

The pressure of the air at the entrance to the vacuum jacketed tube was controlled, to within 1 part in 500, by bubblers 23 and 24 (which could be coupled in series when the higher flow rates were used).

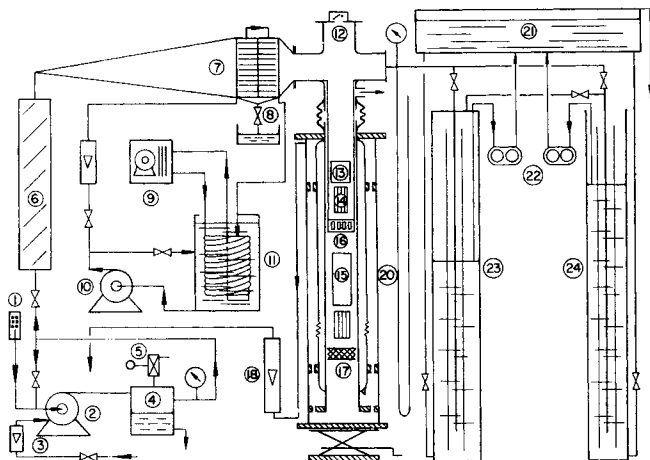


Fig. 1. General arrangement of the apparatus.

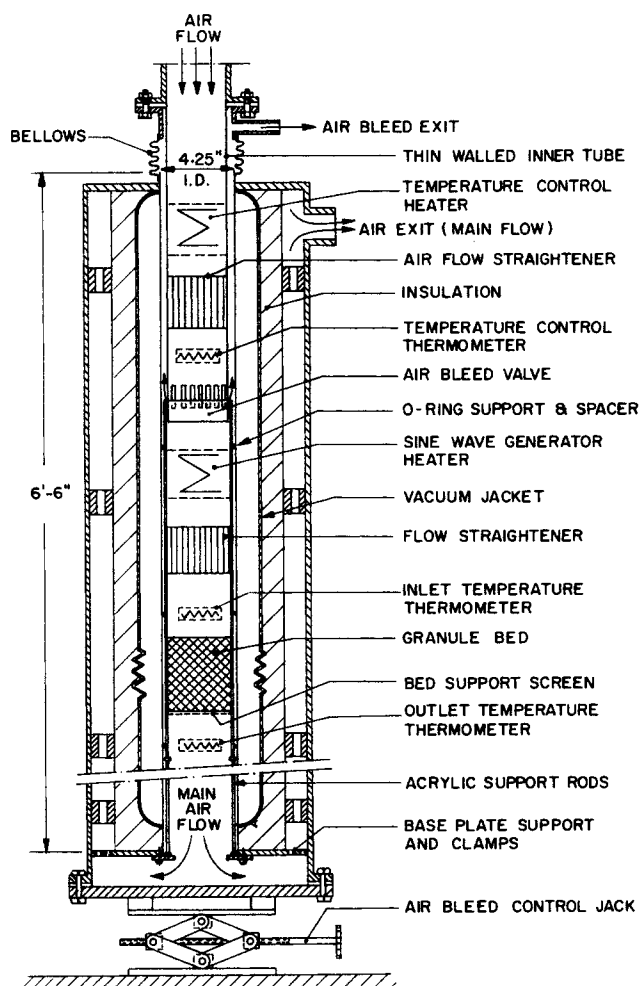


Fig. 2. Details of the vacuum jacketed tube.

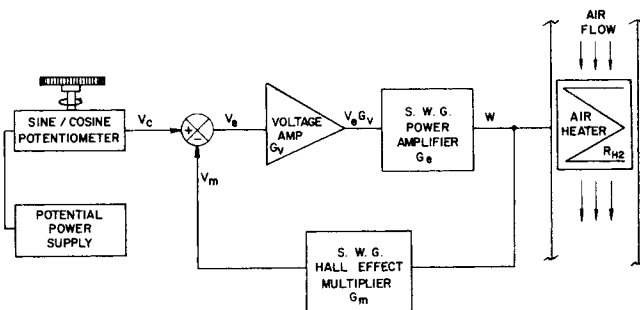


Fig. 3. Block diagram of sine wave generator.

The air finally passed through the rotameter 18 (needed only for monitoring purposes, or if  $D$ , rather than  $N_{Pe}$ , were required).

The air velocity could be varied from 210 to 1,220 cm./sec. in the empty tube, and the frequency of the sinusoidal temperature waves varied from an upper limit of about 1 cycle/sec. down to 1 cycle/day or thereabouts.

### Temperature Control

The temperature was first controlled to a steady value by an electrical proportional controller consisting of an electrical heating system (similar to that used for the sine wave generation described below) with a linearizing feedback loop (see below), and another feedback loop whose control signal was provided by a resistance thermometer situated below the electrical heater in the air stream.

### Sine Wave Generation

A sine-cosine potentiometer was driven at a constant speed by a synchronous motor with its supply frequency controlled. The sinusoidal control electromotive force from it programmed a power amplifier. The latter accepted only a unidirectional programming signal and in the present apparatus the output power of this amplifier was made proportional to the programming voltage by a Hall effect watt transducer feedback loop, shown in Figure 3.

The air heater consisted of a total of 1,945 m. of  $\sim 0.01$  mm. diameter Copel resistance wire.

The harmonic analysis of the temperature waves generated in the air stream by this method showed that the waves were indeed very pure. (The relative amplitude of the second harmonic was about 1%; that of the third harmonic was about 0.5%. Higher harmonics were negligibly small.)

### Temperature Measurement

The air temperature was measured above and below the bed by resistance thermometers of 0.01-mm. Balco wire with a total resistance of 1,000 ohms. The design and construction of these thermometers were such that it averaged the temperature across the core of about 3.7 cm. diameter.

The temperature waves above and below the bed could be either alternately or consecutively sampled, and this sampling was done by opening the gate of an electronic counter 16 times per wave for a period that was fixed at some value (for example, 0.01 sec.) by the counter. The measuring chain comprised thermometer, voltage-to-frequency converter, and counter. The device that triggered the counter was a signal from a fast photodiode, which received a light pulse through one of 16 holes regularly and precisely located in the gear wheel on the sine-cosine potentiometer shaft. In addition, grooves on this gear wheel actuated a microswitch when the input and output waves needed to be sampled alternately.

### Air Flow

The velocity profile was measured by a hot-film anemometer. The most satisfactory flow straightener was found to be a bundle of  $\frac{1}{4}$ -in. diameter paper straws about 15 cm. long, filling the tube. Two of these flow straighteners were used, one above the air bleed and one above the bed inlet thermometer.

TABLE 1. SUMMARIZED RESULTS

| Air flow,<br>kg./hr.<br>(number of runs)                                                                | $(N_{Re})_p$ | $h^{\circ} \times 10^3$ | $k^{\circ} \times 10^3$ | $N^{\circ}_{St}$  | $(N_{Pe})^{\circ}_p$ | Remarks                                  |
|---------------------------------------------------------------------------------------------------------|--------------|-------------------------|-------------------------|-------------------|----------------------|------------------------------------------|
| Soda lime glass. $d_p = 0.399 \pm 0.0013$ cm. Reported $k = 2 \times 10^{-3}$ (7) (English Glass)       |              |                         |                         |                   |                      |                                          |
| 219.5 (12)                                                                                              | 2416         | 22.3 (2.1)              | 1.96 (0.16)             | 0.088 (0.003)     | 1.05 (0.13)          | 0.092                                    |
| 142.9 (6)                                                                                               | 1570         | 17.9 (1.7)              | 1.89 (0.21)             | 0.109 (0.002)     | 1.05 (0.11)          | 0.108                                    |
|                                                                                                         |              |                         |                         |                   |                      | Above are $N_{St}$ from<br>reference (8) |
| Borosilicate glass. $d_p = 0.5$ cm. Reported $k = 2.7 \times 10^{-3}$ (7) (Corning)                     |              |                         |                         |                   |                      |                                          |
| 219.5 (3)                                                                                               | 3020         | $23.1 \pm 0.5$          | $2.71 \pm 0.9$          | $0.09 \pm 0.002$  | $1.0 \pm 0.1$        | Null method                              |
| 219.5 (2)                                                                                               | 3020         | $23.9 \pm 1.0$          | $2.45 \pm 0.02$         | $0.095 \pm 0.004$ | $1.0 \pm 0.1$        |                                          |
| 142.9 (1)                                                                                               | 1963         | 18.9                    | 2.87                    | 0.115             | 0.8                  |                                          |
| Methyl methacrylate. $d_p = 0.48$ cm. Reported $k = 0.4$ to $0.6 \times 10^{-3}$ (7) (Johnson Plastics) |              |                         |                         |                   |                      |                                          |
| 219.5 (1)                                                                                               | 2899         | 30.2                    | 0.3                     | 0.119             | 0.6                  |                                          |

\* Reported as either Mean (standard deviation) or midrange value  $\pm \frac{1}{2}$  range.

## PROCEDURE

A run was made at a particular air flow rate (set by adjusting the bleed valve and the bubbler depth of immersion) with beds of different depths. The input and output waves were sampled, as described above, for a minimum of three cycles; in this way the presence of subharmonics could be detected. The values of the sampled temperature were analyzed to give the relative amplitude and phase lag of input and output waves, as well as information on the purity of the waves. Reference 9 contains full details. An alternative potentiometric (null) method was also tried, using the signal from other sine-cosine potentiometers (at fundamental and harmonic frequencies). The amplitude and phase angle of this signal could be adjusted.

## RESULTS

The final mean values and scatter of the thermal conductivity  $k$ , the heat transfer coefficient  $h$ , the Peclet number  $N_{Pe}$ , and the Stanton number  $N_{St}$  at two air velocities are given in Table 1, which also contains approximate values of the particle Peclet number  $(N_{Pe})_p$ . These values are however considerably less accurate than the previous parameters since their calculation requires estimates of the bed porosity and the bed depth  $L$ , and these in turn were not measured as accurately as the frequency response data. The reproducibility is indicated in Table 2.

Sample graphs of the experimental  $f_{1p} = 0$  curves in the  $h, k$  plane is illustrated in Figure 4. The root that gave the solutions has been circled; the other roots were eliminated by the fact that Peclet numbers corresponding to them were negative. (See Part II.) The movement of the intersections away from the theoretical single inter-

section is due to experimental errors in the frequency response measurements when more than four frequencies are used. Computation of all the lines shown in Figure 4 from four frequencies required about 2 min. of IBM 360-75 computer time. A Newton-Raphson determination required less than 10 sec.

## DISCUSSION

### Measured Values: Conductivity

Most work was done with soda lime glass spheres with the results summarized in Table 1. The measured conductivity was close to the reported value (7).

### Heat Transfer Coefficient

To compare the values of the heat transfer coefficients, the Stanton numbers at the two air flow rates are also listed in Table 1, along with values calculated from Meek's correlations (8), namely,  $N_{St} = 1.77 N_{Re}^{-0.38}$ . Agreement is very good. It should be noted that Meek's correlation was obtained after elimination of wall effects, while other correlations, listed in reference 8, and summarized as  $N_{St} = 0.72 N_{Re}^{-0.30}$ , agreed with the values Meek obtained when wall effects were not eliminated.

### Longitudinal Peclet Number

The mean particle Peclet numbers are also given in Table 1, while the mean for all runs was 1.03, with a

TABLE 2. STATISTICAL PARAMETERS OF SAMPLES OF OBSERVATIONS

| Samples | Means     |           | Standard deviations |         |
|---------|-----------|-----------|---------------------|---------|
|         | $\bar{h}$ | $\bar{k}$ | $s_h$               | $s_k$   |
| A       | 0.0258    | 0.00181   | 0.0008              | 0.00013 |
| B       | 0.0249    | 0.00209   | 0.0014              | 0.00015 |
| C       | 0.0223    | 0.00196   | 0.0021              | 0.00016 |

A: one run; 16 estimates of roots. B: three runs, responses averaged; 17 estimates of roots. C: overall results; all sources of error included.

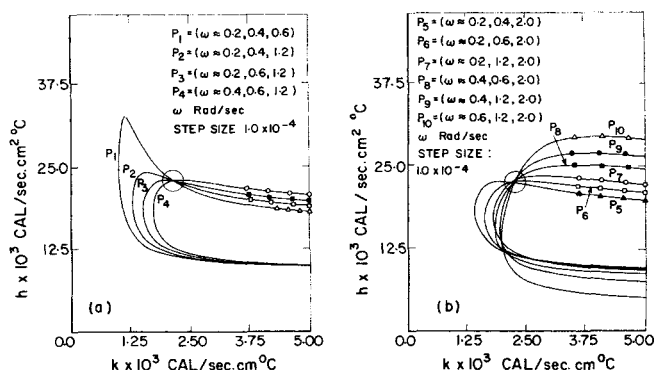


Fig. 4. Typical  $f_{1p} = 0$  curves (glass beads). (a) From four frequencies; (a) and (b) together from five frequencies.

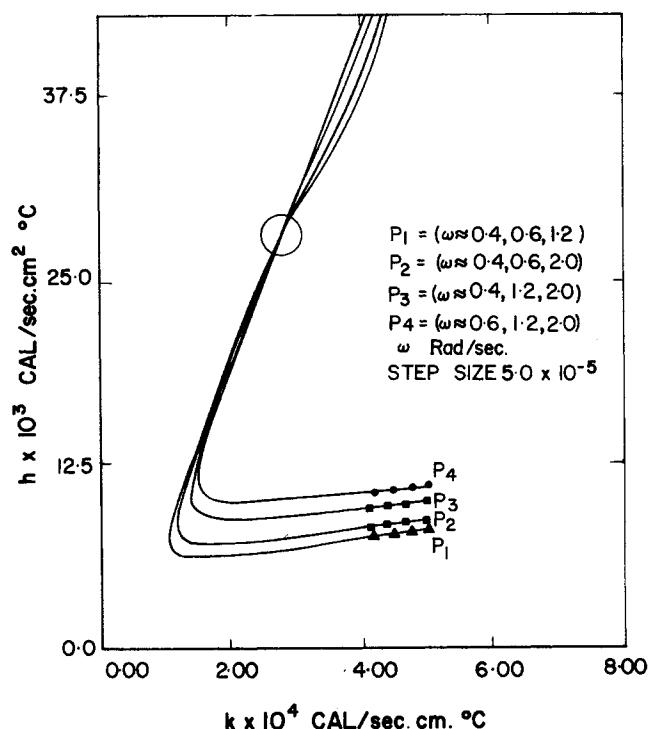


Fig. 5. The  $f_{1p} = 0$  curves illustrating the results of a restricted range of frequencies (for acrylic spheres).

standard deviation of 0.21. The value of  $\sim 2$  usually accepted has been measured at values of the particle Reynolds number much lower than those used here. There is evidence (12, 13) that at higher values of the Reynolds number the values of  $(N_{Pe})_p$  are markedly lower.

Other measurements are also reported in Table 1; however, fewer runs have been made than for soda lime glass.

The results with borosilicate glass are good, those obtained by using the null method being less close to the reported values.

Values of  $k$  for methyl methacrylate spheres were less satisfactory; the reason is probably that this lower value of the conductivity required a value of the frequency higher than the apparatus could generate; in consequence the value of the parameter  $\lambda$  lay in a restricted range of 0.15 (imposed by the theory) to 0.29 (imposed by the limitations of the present apparatus). The result was that the  $f_{1p} = 0$  curves are nearly parallel in the region of their intersection, which is therefore sensitive to small errors. Figure 5 is included to show this and thus to demonstrate the need for a sufficient span of frequencies.

#### Reproducibility

The standard deviations in Table 2 get larger from samples A to C, as is to be expected. Furthermore it is encouraging that the standard deviation of  $h$  increases much more markedly than for  $k$ . The latter should be independent of the bed, but  $h$  is a function of both the air velocity and the mode of packing, and so  $h$  might be expected to show more variability, even without random errors.

#### The Veracity of the Apparatus

The results reported above seem to indicate that there was a sufficient closeness between experimental and mathematical models in the present set of experiments. The greater discrepancies are likely to occur at the radial and longitudinal boundaries.

**Radial Variations.** Traverses of the duct with thermometers and anemometers showed that temperature and velocity profiles away from the walls were flat to within a few

percent of the mean.

The thermometers of the apparatus were constructed to average the temperatures over the central 30% of the cross-sectional area. The indication (given earlier) is that the heat leak is very small while radial dispersion in the bed and the tube are small. There is no basis yet for deciding what the radial tolerances of temperature or velocity should be.

**Entrance and Exit Effects.** The theory (Part I) has been developed on the assumption of an infinite bed. The effects of a finite bed can be removed by making measurements on beds of two different lengths, as has been reported (3 to 5). The conditions under which the procedure is valid have been examined by Turner (11). The results obtained here, involving the differential response of beds of different lengths, seemed self-consistent except when the bed was very short; there are probably two explanations for this: the dispersion model probably does not hold very well, and the reflected wave from the exit boundary may extend too far upstream, as explained in reference 11.

#### ACKNOWLEDGMENT

This work was carried out with financial support from the National Research Council of Canada; our thanks are due to this body for the operating grants and for the award of a postgraduate scholarship to M. J. Goss.

The authors are most grateful to Dr. D. S. Scott, Chairman, Department of Chemical Engineering, University of Waterloo, for making the space and facilities of his Department available for this work.

The apparatus, developed over the period 1965-1969, evolved from an earlier apparatus of Turner and Dosser (10) built at Fisons Fertilizers Ltd., Bramford Development Station, Suffolk, England, over the period 1961-1964.

#### NOTATION

- $(N_{Pe})_p$  = particle Peclet number,  $2Ur_0/D$ , dimensionless  
 $N_{Re}$  = particle Reynolds number,  $2U_0r_0\rho/\mu$ , dimensionless  
 $N_{St}$  = Stanton number,  $h/(\rho c)_f U_0$ , dimensionless  
 $U$  =  $U_0/\epsilon$  = interstitial velocity, cm./sec.  
 $U_0$  = superficial (empty tube) velocity, cm./sec.  
 $\mu$  = dynamic viscosity, poise

#### Subscript

- $f$  = fluid

#### LITERATURE CITED

- Turner, G. A., *AIChE J.*, 13 (4), 678 (1967).
- Goss, M. J., and G. A. Turner, *ibid.*,
- McHenry K. W., Jr., and R. H. Wilhelm, *ibid.*, 3, 83 (1957).
- Liles, A. W., and C. J. Geankoplis, *ibid.*, 6, 591 (1960).
- Turner, G. A., *Chem. Eng. Sci.*, 7, 156 (1958).
- Ebach, E. A., and R. R. White, *AIChE J.*, 4 (2), 161 (1958).
- Weast, R. C., Ed., "Handbook of Chemistry and Physics," 49th edit., Chemical Rubber Co., Cleveland, Ohio (1968).
- Meek, R. M. B., *Rept. No. 54*, Natl. Eng. Lab., East Kilbride, Scotland (1962).
- Goss, M. J., Ph.D. thesis, Univ. Waterloo, Ontario (1969).
- Turner, G. A., and G. C. Dosser, *Rept. No. D1*, Fisons Fertilizers Ltd., Levington, Ipswich, Great Britain (1964).
- Turner, G. A., paper presented at AIChE/Puerto Rican Inst. Chem. Eng. Meeting, San Juan, Puerto Rico (May 1970).
- Scott, D. S., private communication.
- Urban, J. C., and A. Gomezplata, *Can. J. Chem. Eng.*, 47, 353 (1969).

Manuscript received August 5, 1969; revision received April 15, 1970; paper accepted April 22, 1970.