OKO-290

MASTER

DO NOT PHOTOSTAT

DEPARTMENT OF ENGINEERING RESEARCH
NORTH CAROLINA STATE COLLEGE
RALEIGH, NORTH CAROLINA

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

ORO-290

PROGRESS REPORT NO. 13
covering the specific topic of
THE DEVELOPMENT OF A PULSE-COLUMN
SAMPLING TECHNIQUE TO OVERCOME
ENTRAINMENT

Contract AT-(40-1)-1320
The Performance of Contactors for
Liquid-Liquid Extraction

Topic Personnel:

W. F. Boerner
E. E. Erickson
F. O. Mixon, Jr.
J. R. Nelli
F. P. Pike

Submitted by:

F. Philips Pike
Project Director
Professor of Chemical
Engineering

DEPARTMENT OF ENGINEERING RESEARCH
NORTH CAROLINA STATE COLLEGE
RALEIGH, NORTH CAROLINA

April 26, 1955

MAY 16 1955

6 - 101

E-3031

**THE DEVELOPMENT OF A PULSE-COLUMN SAMPLING
TECHNIQUE TO OVERCOME ENTRAINMENT**

TABLE OF CONTENTS

	Page
Table Index	
Figure Index	
Summary	1
Statement of the Problem	3
Introduction	4
Preliminary Experiments	7
Introduction	7
Apparatus	8
Filter Media	8
Procedures	13
Results	14
Conclusions	20
Preliminary Column Test	21
Column Installation of Filter Units	25
Information from Recent Paper	29
Appendix	32
A. Nomenclature	33
B. Discussion of Equation for Pressure Drop	34
C. Sample Calculations	36
D. Pressure-Drop and Flow-Rate Data	40
E. Haze Characterization and Settling Rate Studies	47
F. Cleaning Procedures for Tubular Fritted-Glass Filters	51

TABLE INDEX

Table		Page
I	Pertinent Physical Information for Various Filter Media.	13
II	Filter Constants.	16
III	Pressure-drop and Flow-rate Data for Water Flowing thru Simulated Filter-tube Neck and External Fittings.	40
IV	Data for the Filtration of Distilled Water with a 4-inch Coarse-porosity Filter.	41
V	Data for the Filtration of Distilled Water with a 4-inch Medium-porosity Filter.	42
VI	Data for the Filtration of Distilled Water with a 2-inch Coarse-porosity Filter.	43
VII	Data for the Filtration of Benzene with a 4-inch Coarse-porosity Filter.	44
VIII	Data for the Filtration of Benzene with a 4-inch Medium-porosity Filter.	45
IX	Data for the Filtration of Benzene with a 2-inch Coarse-porosity Filter.	46
X	Comparison of Various Hazes by Means of Light Absorption.	47
XI	Measurement of Settling-rates of Benzene-water Dispersions.	48

FIGURE INDEX

Figure		Page
1	Schematic Diagram of Preliminary Filter Testing Apparatus.	9
2	Preliminary Filter Testing Apparatus.	10
3	Filter Chamber.	11
4	Fritted Pyrex Tubular Filters.	12
5	Pressure-drop and Flow-rate Behavior for Water and Benzene Flowing thru Neck and External Fittings of Filter Thimbles.	15a
6	Flow Characteristics of 4-inch Coarse Filters.	17
7	Flow Characteristics of 4-inch Medium Filters.	18
8	Flow Characteristics of 2-inch Coarse Filters.	19
9	Details of Column Filtering Apparatus.	22
10	Column Filtering Unit.	23
11	Schematic Diagram of Column with Temporary Filtering Units Installed.	24
12	Filter Installation at Top of Column.	26
13	Filter Installation at Bottom of Column.	27
14	Details of Filter Fitting.	28
15	Viscosity of Water.	38
16	Viscosity of Benzene.	39
17	Settling Rates of Water-in-benzene and Benzene-in-water Dispersions.	50

THE DEVELOPMENT OF A PULSE-COLUMN SAMPLING TECHNIQUE TO OVERCOME ENTRAINMENT

Eugene E. Erickson, Forest O. Mixon, Jr.,
and F. Philips Pike

--

SUMMARY

An adequate solution has been found to the problem of obtaining a clear, homogeneous sample of the continuous phase of the unstable dispersions which are characteristic of pulse column exit streams. While for many purposes these dispersions cause little trouble, experience had indicated that even a trace of a second phase can absolutely ruin an analysis when the attempt is being made, with relatively insoluble liquids, to measure individual-film mass-transfer coefficients. Therefore, for the purposes of this project, a solution to this apparently minor problem was mandatory.

A preliminary investigation revealed that fritted-glass tubular filters adequately removed the dispersed material from either phase, even when the emulsions were more finely divided than those produced by the pulse column. Pressure-drop studies proved these filters to be practical for the low flow-rates of the sampling lines. Temporary filter units, installed on the pulse-column and tested under operating conditions, gave added assurance that the fritted glass filters would accomplish the desired purpose.

The information gained was utilized in designing effluent sampling sections, containing the fritted-glass tubular

filters, for the column discharge lines. The units were constructed, installed and tested and have provided a satisfactory solution to the entrainment problem.

The pressure-drop across the filter-tube at sampling flow-rates was compared with that pressure that will rupture the interface at the capillary entrance of a preferentially wetted membrane, as developed by Jordan* in a recent paper. Data for the separation of entrainment in the water phase indicate the pressure-drops to be well under Jordan's rupture pressure. The fact that the filter, which would be wetted preferentially by water, appears to operate satisfactorily also in the benzene phase lends support to an originally assumed analogy to the filtration of small particles suspended in a liquid.

*Jordan, G. V., Jr., Trans. ASME 77, 393-400 (1955).
"Separation of immiscible liquids by means of porous membranes."

STATEMENT OF THE PROBLEM

The purpose of this work was to develop, install and demonstrate a means for completely separating enough of the continuous phase for analysis of the various benzene-in-water and water-in-benzene dispersions which form the exit streams from the pulse column. Such a means was absolutely necessary in order to permit studies of rate of mass transfer between the phases, to obtain individual-film mass-transfer coefficients.

INTRODUCTION

Pulse column operation, because of its forceful back and forth surges thru the plate perforations, is prone to develop unstable emulsions of the two liquid streams present. The column end-sections, almost invariably larger in diameter than the column proper, serve to some degree to settle out the coarser components of these dispersions in the exit streams. Yet each of the exit phases tends to contain a fine dispersion of the other phase.

Inspection of column operation reveals that at times this emulsion formation is much worse than at others. Some conditions of pulse frequency, pulse amplitude and flow-rates permit almost clear exit streams; some, on the other hand, cause dispersions of droplets so fine and so numerous as to be milky in appearance. It might be presumed that this trouble would affect only the continuous phase, since the pulse column action is designed specifically to be always dispersing the discontinuous phase. However, both exit streams exhibit this phenomenon.

These emulsions of the exit streams are very troublesome in many cases. In the present work, where the measurement of individual-film mass-transfer coefficients is a goal, the presence of even a trace of dispersion makes it impossible to carry out the necessary analytical measurements. This is so because the two phases being employed, benzene and water, are practically insoluble in each other. For instance, at saturation around room temperature, benzene dissolves only

about 0.060 weight percent water. Since the exit stream analysis normally is over 90 percent of saturation, it is obvious that even a trace of droplet contamination, say 0.006 weight percent, would cause serious error. The result is that the samples taken for analysis of the exit streams must be de-entrained completely and without question.

The problem as stated above may seem strange, since it is common practice in pulse column work to ignore the emulsion situation (except in regard to decontamination). The samples are merely set aside long enough for complete separation to take place, then analyzed. However, in all of those cases the interest lies in a third component, whose distribution is rarely changed appreciably by the haze present. Only when one is interested in the mutual degree of saturation of the two liquid phases, the present case, does the problem become severe.

For a number of reasons it appeared best to plan not to de-entrain the entire exit stream, but rather to clarify only that portion sampled for analysis. Every device or means considered added to the time of contact provided by the pulse column, and therefore caused added mass-transfer to take place. This disadvantage had to be accepted. The additional mass-transfer effect would then be merely added to that already known to exist for the column ends, as distinct from the pulse column proper. However, to minimize this additional end-effect, the separating means should be small and installed as close as possible to the column.

The unit operation of filtration appeared to be the simplest available means of carrying out the desired separation. This preliminary selection raised questions of the separating ability of various filtration media, their pressure drop, wettability, size, shape, and ease of installation and replacement. Glass members were favored, although finely woven wire screen seemed a possibility.

PRELIMINARY EXPERIMENTS

Introduction

The preliminary experiments were made to test filtration as a means of clarification, and to provide quantitative information on the performance of various filter media. These experiments fell into three classes, as follows.

1. Experiments concerning the ability of various media to clarify emulsions of water and benzene. This was done by filtering fine dispersions of water in benzene and benzene in water thru various filter media, and observing the nature of the filtrate.

2. Experiments concerning pressure drop and flow rate behavior. For this phase, pure benzene and pure water were forced thru the filters at measured rates under measured pressures. At the time these measurements were made, one idea was to install the filtering unit inside the column end-section and to filter the entire exit stream. Such a procedure made pressure drop measurements necessary. An advantage to this method would have presumably been a reduction of the mass transfer end-effect. Later, it was decided to filter only the portion sampled. In that case, the pressure drop data became desirable but not necessary.

3. Miscellaneous experiments to test clogging and plugging tendencies, and the cleaning procedures required for tubular fritted-glass filter units.

Apparatus

The equipment used for the preliminary testing of fritted-glass filter units consisted mainly of standard laboratory glassware. The laboratory air supply was first filtered, to remove solid or liquid particles that might tend to clog the filter units being tested. This filtered air then pressurized a solution chamber containing the liquid phases to be tested, forcing one of them through ^{the filter} unit which was installed inside a glass tee. By suitably raising or lowering the outlet tube from the solution chamber, either of the two phases could be forced out. Auxiliary openings on the glass tee permitted measurements of temperature and pressure.

The arrangement of apparatus is shown schematically in Figure 1. A photograph of the entire assembly, an enlarged view of the filter-chamber and a photograph of two of the filter-tubes may be seen in Figure 2, 3 and 4 respectively.

When wire screening was tested, a piece of 2-inch Pyrex pipe, 5 inches long, was attached to the discharge end of the tee (see Figure 1) with a flange assembly employing two Ameripole rubber gaskets. The sample of metal screen was sandwiched between the two gaskets.

Filter Media

The filter media evaluated are listed in Table 1. They consisted of Pyrex fritted-glass tubular units and ~~stainless metal~~ ~~steel~~ screening of close weaves.

FIGURE 1

SCHEMATIC DIAGRAM OF PRELIMINARY FILTER
TESTING APPARATUS

FIGURE 2

PRELIMINARY FILTER TESTING APPARATUS

FIGURE 3
FILTER CHAMBER

202 016

FRITTED PYREX TUBULAR FILTERS
FIGURE 4

TABLE I

Pertinent Physical Information for Various Filter Media

Filter	Material of Con- struction	Mesh	Approximate Pore Size Microns	Out- side Diam. in.	Length in.	Filter- ing Area in. ²
Screen	Phosphor- bronze	200x200				
Screen	Brass wire	100x100				
Screen	Stainless steel	100x100				
Screen	Stainless steel	700x60				
Tubular* filters	Fritted glass	Coarse	40-60	0.615	3.969	7.620
"	"		40-60	0.606	3.969	7.556
"	"	"	40-60	0.630	1.844	3.650
"	"	"	40-60	0.632	1.969	3.910
"	"	Medium	10-15	0.598	3.906	7.341
"	"	"	10-15	0.605	3.938	7.485

*Corning tubular fritted-glass filters, Catalog No. 35,000.

Procedures

It was not practical to obtain the liquid-liquid dispersions for testing from the pulse column unit. Furthermore, these dispersions were not stable enough for testing. Much more stable emulsions were manufactured by heating mixtures of benzene and water to about 65°C, agitating vigorously for a short time to approach saturation in both phases, then cooling to room temperature. This procedure yielded very fine

dispersions, or mists, that appeared to be finer than those obtained under even the worst conditions of pulse column operation. A description of the trials that led to standardization of the procedure for manufacturing the test haze is given in the Appendix.

The various filter media were tested for their ability to produce a clear filtrate from the hazy test solutions. The criterion was the visible appearance of the filtrate, and the relative lack of droplets.

The procedure for obtaining flow-rate and pressure-drop data was quite similar. The (pressure) flask was charged with the liquid to be tested, and the pressurizing air was adjusted to give a predetermined pressure drop across the filtering device; after a brief period to assure steady-state operation, the rate of discharge from the filtering chamber was measured by timing the flow into a volumetric flask of appropriate size. A separate set of measurements were made to determine the pressure drop due to external connections to the filter unit.

Results

The results are summarized here. Most of the supporting information is placed in the Appendix.

1. Evaluation of Various Filter Media. All of the woven-wire filter clothes were found to be unsatisfactory for the filtration of both the benzene phase and the water phase dispersions. These woven clothes included the tightest weaves available on the market.

It is significant that the 700x60 mesh dutch twill cloth had previously been found to be fairly effective on actual pulse column dispersions. The fact that it was completely ineffective on the test dispersions indicated that the adopted test procedure was a relatively severe test. In other words, the haze emulsion must have been much finer than the pulse column emulsions, as indeed they so appeared by eye.

It was found that the fritted-glass filter units separated the dispersions rather effectively. For the benzene-in-water dispersions, both the coarse and medium grade filters were perfectly satisfactory. For the water-in-benzene dispersions, the coarse grade units were not always completely satisfactory. They did effect a marked reduction in the number of droplets but still let some material thru.

2. Pressure Drop and Flow Information. It was found that the pressure drop across a filter unit can be considered to be made up of two component parts, the pressure drop across the fritted section and the pressure drop due to the glass neck and outlet tube. In order to separate these two components, a small glass tee was made up to approximately the dimensions of the neck of the tubular filter. The pressure drop was then measured across this unit as a function of flow rate. The results are plotted on Figure 5, where the actual data points were obtained using water as the fluid. The dashed line for benzene was obtained by calculation, assuming that $\Delta P_{\text{Benzene}} = \Delta P_{\text{H}_2\text{O}} \left(\frac{\rho_{\text{B}}}{\rho_{\text{W}}} \right)$, which is approximately correct. These ΔP values were used to correct the observed ΔP to provide only the ΔP across the filtering section.

FIGURE 5

PRESSURE DROP AND FLOW RATE BEHAVIOR FOR
WATER AND BENZENE FLOWING THROUGH NECK AND
EXTERNAL FITTINGS OF FILTER THIMBLES

For the flow thru the fritted-glass section, it was assumed that the ordinary filtration equation, based upon Poiseuille's law, was applicable. The form used was as follows.

$$\frac{\mu V}{A\theta} = K \frac{\pi D^4 g}{128 L} \Delta P$$

$$\left(\frac{\mu}{A}\right)q = K' \Delta P, \text{ where } q = \left(\frac{V}{\theta}\right)$$

See the Appendix for the nomenclature.

The value of K' is characteristic of the grade of fritted glass. It was determined as the slope of a plot of $\frac{\mu V}{A\theta}$ vs. ΔP , where the ΔP has been corrected for the pressure drop in the extraneous connections. Plots of this type are shown in Figures 6, 7 and 8. A dashed line is used to indicate the total ΔP as measured, where the above corrections are appreciable. The properly corrected values were used to determine the values of the constant K' for various filters, which are tabulated in Table II. As K' characterizes a given filter, once it has been determined, the pressure-drop through the porous media at any given flow-rate can be found quite readily. The ΔP for the external fittings can be estimated from Figure 5.

TABLE II

Filter Designation	Length of Fretted Section	Stated Porosity	Value of K'
			$\frac{(C_p)(\text{sec.})}{(\text{in.}^2)(\text{min.})(\text{in. Hg})}$
4-C-1	4 in.	Coarse	0.0400
4-C-2	4 in.	Coarse	0.0500
4-M-1	4 in.	Medium	0.0048
4-M-2	4 in.	Medium	0.0014
2-C-1	2 in.	Coarse	0.0156
2-C-2	2 in.	Coarse	0.0360

FIGURE 6

FLOW CHARACTERISTICS OF FOUR-INCH,
COARSE FILTERS

FIGURE 7

FLOW CHARACTERISTICS OF FOUR-INCH MEDIUM FILTERS

CORRECTIONS FOR PRESSURE DROP THROUGH
NECK AND FITTINGS ARE NEGLIGIBLE

○ □ DATA ON FLOW OF WATER

● ■ DATA ON FLOW OF BENZENE

FIGURE 8

FLOW CHARACTERISTICS OF TWO-INCH.
COARSE FILTERS

If the entire exit stream flow is to be filtered, both of the pressure drop components are significant. However, if only the sample portion is to be filtered, the pressure drop for the neck section can be ignored.

3. General Observations. It was noted that under some conditions the filter units could become clogged. When an attempt was made to filter tap water, rather than distilled water, a coarse filter unit became badly plugged after the passage of only about 500 ml. of liquid. However, when distilled water was used, there was no noticeable decrease in flow rate, at constant pressure, after filtering as much as 5 liters of liquid.

It was found that the filter units could be cleaned and unclogged by following the cleaning procedure recommended by the manufacturer. Procedures suitable for cleaning are presented in the Appendix.

Conclusions

It was concluded that fritted-glass tubular filter units would probably be satisfactory in filtering ability, flow capacity, size, shape and ease of cleaning.

The various wire clothes, even the most tightly woven ones obtainable, did not possess the ability to clarify properly the test hazes.

The procedure for evaluating the filter media employed dispersions more difficult to clarify than those encountered in pulse column operations.

PRELIMINARY COLUMN TEST

While material for a permanent installation was being acquired, a preliminary column test was made to check the conclusion that fritted-glass filter units were suitable means of de-entraining the samples from the pulse column. Temporary units were constructed simply of brass, Ameripole rubber and stainless steel. A drawing of the temporary filtering unit is shown in Figure 9, and a photograph in Figure 10. A schematic diagram of the installed positions is shown in Figure 11.

After installation the pulse column was set into operation at the following conditions, which were known to and did cause quite distinct emulsification of the exit streams.

Pulse Amplitude = 2.0 inches

Pulse Frequency = 50 cycles per minute

$V_D = 35 \text{ ft/hr}$

$V_C = 105 \text{ ft/hr}$

$V_D/V_C = 0.33$

$V_D+V_C = 140 \text{ ft/hr}$

Over a period of time various samples were withdrawn from the column and examined visually and carefully. They gave every appearance of being free of suspended droplets. This preliminary column test was considered successful.

FIGURE 9
DETAILS OF COLUMN FILTERING APPARATUS

FIGURE 10
COLUMN FILTERING UNIT

FIGURE 11

SCHEMATIC DIAGRAM OF COLUMN WITH TEMPORARY
FILTERING UNITS INSTALLED

COLUMN INSTALLATION OF FILTER UNITS

Based upon the preliminary column test, permanent units were planned, constructed and installed. In part, the end connections of the pulse column were changed to permit the units to be placed in the exit lines in a manner to meet the following specifications.

1. Installation as close as practical to the point of exit from the column.
2. Installation in a vertical position, discharging upward, to avoid trapping air or vapor.
3. Location so that the hydrostatic head on the column would be adequate for the sample flow thru the filter unit.
4. Special consideration to avoid contamination.
5. Use of a medium porosity filter unit in the benzene phase, a coarse porosity unit in the water phase.
6. The sizing of the flow path of the exit streams thru the sampling zone to provide low resistance.

The sampling zones were built of standard Pyrex pipe sections and couplings, fabricated brass end plates, and "Swagelok" tubing connectors. The details of the installation at the top of the column is shown in Figure 12, that at the bottom, in Figure 13. The manner of connection of the filter-tube to the sample line is shown in Figure 14.

Operation of these units has been satisfactory. The samples taken have at all times been clear with no trace of suspended particles. In addition, the analyses of the water phase for benzene have provided values less than saturation, as required.

FILTER INSTALLATION AT TOP OF COLUMN

FIGURE 13

FILTER INSTALLATION AT BOTTOM OF COLUMN

FIGURE 14
DETAILS OF FILTER FITTING

INFORMATION FROM A RECENT PAPER

A paper published recently that is pertinent to this report came to our attention after the study was completed. Jordan* has discussed the theory of separation of entrainment and emulsions by porous membranes, and has described the application of the theory to industrial problems. The separation of entrainment in each phase is carried out by means of a membrane that is preferentially wet by the phase to be filtered.

The paper presents a simple method of determining the allowable pressure-drop across a capillary bed before the dispersed phase will be carried through. The force necessary to cause a rupture of the interface at the capillary entrance is PA' , where P is the rupture pressure and A' , the capillary cross section area. The force resisting the rupture is σc , where σ is interfacial tension (at the interface of the dispersed phase and the film wetting the capillary wall) and c is the circumference of the capillary opening. Then

$$\sigma c = PA'.$$

If the capillary opening is circular with a radius r ,

$$2\pi r\sigma = \pi r^2 P, \text{ or}$$

$$P = 2\sigma/r.$$

Jordan has plotted the relationship between the maximum capillary radius of the interfacial support and the

*Jordan, G. V., Jr., Trans. ASME 77, 393-400 (1955).
 "Separation of immiscible liquids by means of porous membranes."

rupture pressure for a considerable range of interfacial tensions.

From Jordan's graph, the filter-tube capillary size, and the assumption of circular capillary paths, the rupture pressure for our system can be obtained.

1. Water phase, 4" coarse porosity filter. The pore diameter from Table I is 40-60 microns. Using a maximum $r = 30$ microns, and assuming the thickness of the film of wetting liquid to be negligible,

$$P = 0.34 \text{ psi or } 0.69 \text{ in. Hg.}$$

It can be seen from the pressure-drop data tabulated in the Appendix that the pressure-drop across the filter is well below this value, for a maximum sampling rate (about 0.05 psi for 200 ml/min).

2. Benzene phase, 4" medium porosity filter. If the tube were preferentially wet with benzene, the value of the rupture pressure for a pore radius of 7.00 microns (Table I), obtained as above, would be about 1.2 psi or about 2.44 in. Hg. This is also well above the observed pressure drop, which was about 0.3 psi for flow rates of interest.

Since the glass is preferentially wet by water, however, perhaps the second comparison is unwarranted. This problem was recognized during the earlier investigation, but it was felt that the filter unit, in view of the small amount of dispersed phase and its apparently significant droplet size, might treat the matter as an ordinary filtration operation and exclude the unwanted water particles. The pulsing flow past the filter would tend to prevent the build-up of water

droplets at the filter surface. In any case, the unit appears to operate in a satisfactory manner in both streams.

APPENDIX

	Page
A. Nomenclature	33
B. Discussion of Equations for Pressure Drop	34
C. Sample Calculations	36
D. Pressure-Drop and Flow Rate Data	40
E. Haze Characterization and Settling Rate Studies	47
F. Cleaning Procedures for Tubular Fritted-glass Filters	51

A. NOMENCLATURE

- A = Filtering area (outside) of tubular filters.
 A' = Cross-section area of capillary.
 c = Circumference of interface support (capillary opening).
 D = Diameter of opening.
 f = Fanning friction factor.
 g = Gravitational conversion factor.
 H = Hydrostatic-head correction for pressure-drop calculations.
 K = Constant, capillaries per unit area, in Poiseuille's equation.
 K' = Filter constant, $= \frac{K\pi D^4 g}{128L}$.
 L = Length of path for fluid-flow.
 P = Rupture pressure at capillary interface.
 q = Volumetric liquid discharge rate, ml/min.
 q' = Volumetric liquid discharge rate, gpm.
 r = Radius of capillary opening.
 t = Temperature--°C.
 V = Volume of liquid discharged.
 ΔP = Pressure drop.
 ΔP_c = Correction term applied to pressure-drop calculations for filters tested, and equal to the pressure-drop through the neck and external filter fittings.
 ΔP_f = Pressure drop after application of the correction terms.
 θ = Time--minutes.
 μ = Absolute viscosity--centipoises.
 σ = Interfacial tension.

B. DISCUSSION OF EQUATIONS FOR PRESSURE DROP

According to theory, the pressure-drop behavior thru the porous path of the filter should follow the Poisseuille equation, which is normally expressed for a filtration process as

$$\frac{dV}{d\theta} = K \frac{\pi D^4 \Delta P g A}{128 \mu L}$$

A constant temperature integration of the above differential equation gives

$$\left(\frac{V}{\theta}\right) = K \frac{\pi D^4 \Delta P g A}{128 \mu L} + \frac{C}{\theta}$$

where c is an integration constant and vanishes when V and θ are taken as zero simultaneously. Substituting this boundary condition gives

$$g = \left(\frac{V}{\theta}\right) = K \frac{\pi D^4 \Delta P g A}{128 \mu L}$$

Rearranging

$$\frac{\mu V}{A \theta} = \frac{\mu}{A} g = K \frac{\pi D^4 g}{128 L} \Delta P = K' \Delta P$$

where

$$K' = K \frac{\pi D^4 g}{128 L}$$

is a characteristic constant for a given filter and is represented by K' .

Thus the characteristic constant is equal to the slope of the line obtained by plotting $\frac{\mu V}{A \theta}$ as a function of pressure drop. These constants may be computed and tabulated for the various filters. This straight-line relationship, of course, assumes no plugging of the filter pores due to what might be considered a "filter-cake."

The pressure drop thru the neck of the tubular filter should follow the Fanning equation, which may be written

$$\Delta P = \frac{32 f L' g^2}{\pi^2 g D^5}$$

feet of fluid flowing.

Assuming that the empirical friction factor f is constant, this equation predicts that the volumetric flow rate of a specified fluid through a given circular channel is proportional to the square root of the pressure drop. Furthermore, a logarithmic plot of volumetric flow rate as a function of pressure drop through a given circular channel should, again assuming constancy of the friction factor, give a straight line having a slope of $1/2$.

A close examination of Figure 5, which is a logarithmic plot of the flow rate vs the pressure drop measured for a glass tube of approximately the same dimension as the neck of a filter tube, reveals a slight curvature in the line obtained. Also, the slope is a little less than the expected value of $1/2$. This apparent discrepancy with the theory can be attributed to the erroneous assumption of constancy in the friction factor. Actually, this factor is not constant, but for a given pipe is a function of the Reynolds number

By correcting the total pressure-drop measured for the pressure-drop thru the neck of the tube (obtained from Figure 5), the pressure-drop line for the filter section can be obtained. The filter constants discussed above are measured from this "corrected" curve.

A sample calculation and the tabulated data and calculation results are given in the following sections.

C. SAMPLE CALCULATIONS

A sample calculation is shown for the last data point in Table IV, corresponding to water flowing through a four-inch, coarse porosity filter under an apparent pressure-drop of three inches of mercury. The raw data indicate that 1000 ml of distilled water is 28.8°C under a pressure differential of three inches of Hg will flow through the filter and neck in 0.285 minutes. This corresponds to a volumetric flow rate of

$$\frac{V}{\theta} = q = \frac{1000 \text{ ml}}{0.285 \text{ min}} = 3508 \frac{\text{ml}}{\text{min}}, \text{ or } q' = 3508 \frac{\text{ml}}{\text{min}} \times .0002642 \frac{\text{gal}}{\text{ml}} \\ = 0.927 \text{ gpm.}$$

Reference to Figure 15 indicates that the viscosity of water at 28.8°C is 0.822 centipoises. Thus

$$\mu q' = (0.822 \text{ cps})(0.927 \text{ gpm}) = 0.762 \frac{\text{cps-gal}}{\text{min}}$$

Since the filtering area for this particular filter is 7.556 in.²,

$$\frac{\mu q'}{A} = (0.762 \frac{\text{cp-gal}}{\text{min}}) (\frac{1}{7.556 \text{ in.}^2}) = 0.1008 \frac{\text{cp-gal}}{\text{in.}^2\text{-min.}}$$

The hydrostatic head on the filter was approximately 2.0 inches of water. This corresponds to about 0.15 inches of mercury. The total pressure drop across the filter and external fittings, then, is 3 in. Hg plus 0.15 in. Hg, or 3.15 in. Hg. Reference to Figure 5 indicates that the pressure drop through the external fittings at a flow rate of 0.927 gpm is about 1.10 in. Hg. Therefore, the pressure drop across the porous medium itself is 3.15 in. Hg minus 1.10 in. Hg, or 2.05 in. Hg.

The calculations for benzene were made in a similar manner, using the appropriate information. The viscosity of benzene was obtained from Figure 16.

FIGURE 15

VISCOSITY OF WATER*

FIGURE 16

VISCOSITY OF BENZENE*

VISCOSITY - CENTIPOISES

*TIMMERMANS, J., "PHYSICO-CHEMICAL CONSTANTS
OF PURE ORGANIC COMPOUNDS", P. 146, ELSEVIER
PUBLISHING COMPANY, INC., NEW YORK, 1950

D. PRESSURE-DROP AND FLOW-RATE DATA

TABLE III

Pressure-Drop and Flow-Rate Data for Water Flowing
thru Simulated Filter-Tube Neck and External Fittings

ΔP in. H ₂ O	V ml.	θ min.	t °C.	ΔP in. Hg	q=V/ θ ml/min	q' gpm
1	1000	0.78	30.7	0.0735	1282	0.339
1	1000	.76	30.7	.0735	1316	.348
2	1000	.64	30.7	.147	1562	.413
2	1000	.63	30.7	.147	1587	.419
3	1000	.55	30.5	.221	1818	.480
3	1000	.56	30.5	.221	1785	.472
4	1000	.49	30.6	.294	2040	.539
4	1000	.50	30.7	.294	2000	.528
5	1000	.46	30.7	.368	2174	.574
5	1000	.46	30.6	.368	2174	.574
6	1000	.42	30.6	.441	2381	.629
6	1000	.42	30.7	.441	2381	.629
8	1000	.38	30.7	.588	2632	.695
10	1000	.34	30.7	.735	2941	.777
12	1000	.31	30.7	.882	3226	.852

TABLE IV

Data for the Filtration of Distilled Water with a 4-inch Coarse-porosity Filter

ΔP in. Hg	V ml.	θ min.	t °C	μ cps	q=V/ θ ml/min	q' gpm	$\frac{\mu}{A} q'$	H in. Hg	ΔP_c in. Hg	ΔP_f in. Hg
<u>Filter No. 1</u>										
0	200	0.58	30.4	0.795	345	0.0911	0.0095	0.15	0	0.15
1/2	1000	.83	30.4	.795	1205	.318	.0332	.15	0.05	.60
1	1000	.59	30.1	.800	1695	.448	.0470	.15	0.19	.96
1	1000	.64	32.4	.763	1562	.413	.0413	.15	0.14	1.01
1	1000	.61	32.4	.763	1639	.433	.0433	.15	0.18	0.97
2	1000	.39	32.2	.766	2564	.677	.0681	.15	0.65	1.50
2	1000	.39	32.0	.769	2564	.677	.0684	.15	0.56	1.59
3	1000	.30	31.6	.776	3333	.881	.0897	.15	0.88	2.27
3	1000	.30	31.5	.777	3333	.881	.0899	.15	0.88	2.27
4	1000	.26	31.4	.779	3846	1.016	.1038	.15	1.26	2.89
4	1000	.255	31.2	.782	3921	1.036	.1063	.15	1.30	2.85
<u>Filter No. 2</u>										
0	200	0.83	28.7	0.824	241	0.0637	0.00694	0.15	0	0.15
0	200	.68	28.7	.824	294	.0777	.00847	.15	0	0.15
0	200	.87	28.7	.824	230	.0608	.00663	.15	0	0.15
1/2	1000	.83	28.7	.824	1205	.318	.0347	.15	0.02	0.63
1/2	1000	.89	28.7	.824	1124	.297	.0324	.15	0.02	0.63
1	1000	.60	28.7	.824	1667	.440	.0480	.15	0.20	0.95
1	1000	.58	28.8	.822	1724	.455	.0495	.15	0.21	0.94
2	1000	.37	28.8	.822	2703	.714	.0777	.15	0.60	1.55
2	1000	.37	28.8	.822	2703	.714	.0777	.15	0.60	1.55
3	1000	.285	28.8	.822	3508	.927	.1008	.15	1.10	2.05
3	1000	.285	28.8	.822	3508	.927	.1008	.15	1.10	2.05

TABLE V

Data for the Filtration of Distilled Water with a 4-inch Medium-porosity Filter

ΔP in. Hg	V ml.	θ min.	t °C	μ cp	q=V/ θ ml/min	q' gpm	$\frac{\mu}{A} q'$	H in. Hg	ΔP_c in. Hg	ΔP_f in. Hg
<u>Filter No. 1</u>										
1	200	0.975	28.8	0.822	205	0.0542	0.00607	0.15	0	1.15
1	200	.980	28.8	.822	204	.0539	.00603	.15	0	1.15
2	200	.54	28.8	.822	370	.0978	.0109	.15	0	2.15
2	200	.555	28.8	.822	360	.0951	.0106	.15	0	2.15
3	200	.395	28.8	.822	506	.134	.0150	.15	0	3.15
3	200	.395	28.8	.822	506	.134	.0150	.15	0	3.15
4	200	.31	28.8	.822	645	.170	.0191	.15	0	4.15
4	200	.31	28.8	.822	645	.170	.0191	.15	0	4.15
5	200	.245	28.8	.822	816	.216	.0242	.15	0	5.15
5	200	.250	28.8	.822	800	.211	.0236	.15	0	5.15
6	200	.210	28.8	.822	952	.252	.0282	.15	0	6.15
6	200	.220	28.8	.822	909	.240	.0268	.15	0	6.15
<u>Filter No. 2</u>										
1	200	3.29	29.0	0.818	60.8	0.0161	0.00176	0.15	0	1.15
1	200	3.01	29.0	.818	66.4	.0175	.00191	.15	0	1.15
2	200	1.72	29.0	.818	116	.0306	.0033	.15	0	2.15
2	200	1.73	29.0	.818	116	.0306	.0033	.15	0	2.15
3	200	1.22	30.4	.795	164	.0433	.00459	.15	0	3.15
3	200	1.25	30.4	.795	160	.0420	.00446	.15	0	3.15
4	200	.98	30.0	.802	204	.0539	.00577	.15	0	4.15
4	200	.98	30.0	.802	204	.0539	.00577	.15	0	4.15
5	200	.82	29.8	.822	244	.0644	.00707	.15	0	5.15
5	200	.81	29.8	.822	247	.0653	.00717	.15	0	5.15
6	200	.68	29.8	.822	294	.0377	.00854	.15	0	6.15
6	200	.685	29.8	.822	292	.0771	.00847	.15	0	6.15

TABLE VI

Data for the Filtration of Distilled Water with a 2-inch Coarse-porosity Filter

ΔP in. Hg	V ml.	θ min.	t °C	μ cp	q=V/ θ ml/min	q' gpm	$\frac{\mu}{A}$ q'	H in. Hg	ΔP_c in. Hg	ΔP_f in. Hg
<u>Filter No. 1</u>										
1/2	200	1.19	29.8	0.822	168	0.0444	0.0100	0.15	0	0.65
1/2	200	1.11	29.8	.822	180	.0476	.0107	0.15	0	0.65
1	200	.67	29.8	.822	299	.0790	.0178	0.15	0	1.15
1	200	.65	29.8	.822	308	.0814	.0183	0.15	0	1.15
2	200	.355	29.8	.822	563	.149	.0336	0.15	0	2.15
2	200	.355	29.8	.822	563	.149	.0336	0.15	0	2.15
3	200	.250	29.8	.822	800	.211	.0475	0.15	0	3.15
3	200	.245	29.8	.822	816	.216	.0486	0.15	0	3.15
4	500	.450	29.8	.822	1111	.294	.0662	0.15	0	4.15
4	500	.450	29.8	.822	1111	.294	.0662	0.15	0	4.15
5	500	.37	29.8	.822	1351	.357	.0805	0.15	0.09	5.06
5	500	.36	29.8	.822	1389	.367	.0827	0.15	0.10	5.05
6	500	.32	29.8	.822	1563	.413	.0929	0.15	0.15	6.00
6	500	.32	29.8	.822	1563	.413	.0929	0.15	0.15	6.00
<u>Filter No. 2</u>										
1/2	200	0.40	30.0	0.802	500	0.1321	0.0271	0.15	0	0.65
1/2	200	.39	30.0	.802	513	.1355	.0279	0.15	0	0.65
1	200	.235	30.0	.802	851	.2248	.0460	0.15	0	1.15
1	200	.235	30.0	.802	851	.2248	.0460	0.15	0	1.15
2	200	.145	30.0	.802	1379	.3643	.0747	0.15	0.09	2.06
2	200	.145	30.0	.802	1379	.3643	.0747	0.15	0.09	2.06
3	500	.26	30.0	.802	1923	.508	.104	0.15	0.26	2.89
3	500	.25	30.0	.802	2000	.528	.108	0.15	0.29	2.86
4	500	.195	30.0	.802	2564	.677	.139	0.15	0.52	3.63
4	500	.19	30.0	.802	2631	.695	.142	0.15	0.58	3.57
5	500	.16	30.0	.802	3125	.826	.169	0.15	0.84	4.31
5	500	.16	30.0	.802	3125	.826	.169	0.15	0.84	4.31

TABLE VII

Data for the Filtration of Benzene with a 4-inch Coarse-porosity Filter

ΔP in. Hg	V ml.	θ min.	t °C	μ cp	$q=V/\theta$ ml/min	q' gpm	$\frac{\mu}{A} q'$	H in. Hg	ΔP_c in. Hg	ΔP_f in. Hg
<u>Filter No. 1</u>										
0	250	0.685	33.5	0.535	365	0.0964	0.0068	0.13	--	0.13
0	250	.765	33.5	.535	327	.0863	.0061	.13	--	.13
1/2	500	.325	33.4	.536	1538	.406	.0286	.13	0.125	.505
1/2	500	.325	33.4	.536	1538	.406	.0286	.13	.125	.505
1	500	.240	33.3	.538	2083	.550	.0388	.13	.285	.845
1	500	.240	33.3	.538	2083	.550	.0388	.13	.285	.845
1 1/2	500	.180	33.1	.539	2777	.734	.0519	.13	.569	1.061
1 1/2	500	.190	33.1	.539	2632	.695	.0492	.13	.498	1.132
2	500	.160	33.0	.540	3125	.826	.0585	.13	.700	1.43
2	500	.160	33.0	.540	3125	.826	.0585	.13	.700	1.43
<u>Filter No. 2</u>										
0	250	0.685	33.0	0.540	364	0.0961	0.0069	0.13	--	0.13
0	250	.580	33.0	.540	431	.114	.0081	.13	--	.13
1/2	500	.320	33.0	.540	1562	.413	.0295	.13	0.135	.495
1/2	500	.300	33.0	.540	1666	.440	.0315	.13	.160	.470
1	500	.225	32.9	.541	2222	.587	.0421	.13	.340	.790
1	500	.225	32.9	.541	2222	.587	.0421	.13	.340	.790
1 1/2	500	.180	32.8	.542	2778	.734	.0527	.13	.550	1.080
1 1/2	500	.180	32.8	.542	2778	.734	.0527	.13	.550	1.080
2	500	.150	32.7	.543	3333	.881	.0632	.13	.780	1.350
2	500	.150	32.7	.543	3333	.881	.0632	.13	.780	1.350

TABLE VIII

Data for the Filtration of Benzene with a 4-inch Medium-porosity Filter

ΔP in. Hg	V ml.	θ min.	t °C	μ cp	q=V/ θ ml/min	q' gpm	$\frac{\mu}{A}$ q'	H in. Hg	ΔP_c in. Hg	ΔP_f in. Hg
<u>Filter No. 1</u>										
1/2	250	1.24	33.3	0.538	201	0.0531	0.0039	0.13	--	0.63
1/2	250	1.22	33.3	.538	205	.0542	.0040	.13	--	0.63
1	250	.770	33.1	.539	325	.0859	.0063	.13	--	1.13
1	250	.770	33.1	.539	325	.0859	.0063	.13	--	1.13
1 1/2	250	.560	32.9	.541	446	.118	.0087	.13	--	1.63
1 1/2	250	.550	32.9	.541	455	.120	.0088	.13	--	1.63
2	250	.445	32.9	.541	562	.148	.0109	.13	--	2.13
2	250	.435	32.9	.541	575	.152	.0112	.13	--	2.13
2 1/2	250	.365	32.9	.541	685	.181	.0113	.13	--	2.63
2 1/2	250	.370	32.9	.541	676	.179	.0132	.13	--	2.63
3	250	.325	32.9	.541	769	.203	.0150	.13	--	3.13
3	250	.325	32.9	.541	769	.203	.0150	.13	--	3.13
4	250	.250	32.9	.541	1000	.264	.0195	.13	--	4.13
4	250	.250	32.9	.541	1000	.264	.0195	.13	--	4.13
5	250	.205	32.9	.541	1219	.322	.0237	.13	--	5.13
5	250	.205	32.9	.541	1219	.322	.0237	.13	--	5.13
<u>Filter No. 2</u>										
1	250	2.69	33.4	0.536	92.9	0.0246	0.0018	0.13	--	1.13
1	250	2.42	33.4	.536	103.3	.0272	.0020	.13	--	1.13
2	250	1.35	33.3	.538	185	.0489	.0035	.13	--	2.13
2	250	1.36	33.3	.538	184	.0486	.0035	.13	--	2.13
3	250	.96	33.3	.538	260	.0687	.0050	.13	--	3.13
3	250	.96	33.3	.538	260	.0687	.0050	.13	--	3.13
4	250	.755	33.1	.539	331	.0875	.0063	.13	--	4.13
4	250	.755	33.1	.539	331	.0875	.0063	.13	--	4.13
5	250	.605	33.1	.539	413	.1090	.0079	.13	--	5.13
5	250	.605	33.1	.539	413	.1090	.0079	.13	--	5.13

TABLE IX

Data for the Filtration of Benzene with a 2-inch Coarse-porosity Filter

ΔP in. Hg	V ml.	θ min.	t °C	μ cp	q=V/ θ ml/min	q' gpm	$\frac{\mu}{A} q'$	H in. Hg	ΔP_c in. Hg	ΔP_f in. Hg
<u>Filter No. 1</u>										
1/2	250	0.685	33.0	0.540	365	0.0964	0.0143	0.13	--	0.63
1/2	250	.685	33.0	.540	365	.0964	.0143	.13	--	0.63
1	250	.435	33.0	.540	575	.152	.0225	.13	--	1.13
1	250	.450	33.0	.540	556	.147	.0217	.13	--	1.13
2	250	.270	33.0	.540	926	.245	.0362	.13	--	2.13
2	250	.280	33.0	.540	892	.236	.0349	.13	--	2.13
3	250	.200	32.9	.541	1250	.330	.0490	.13	--	3.13
3	250	.200	32.9	.541	1250	.330	.0490	.13	--	3.13
4	500	.32	32.8	.542	1563	.413	.0611	.13	0.133	3.997
4	500	.33	32.8	.542	1515	.400	.0592	.13	.116	4.014
5	500	.27	32.7	.543	1852	.489	.0723	.13	.210	4.92
5	500	.27	32.7	.543	1852	.489	.0723	.13	.210	4.92
<u>Filter No. 2</u>										
1/2	250	0.27	29.8	0.562	926	0.245	0.0353	0.13	--	0.63
1/2	250	.25	29.8	.562	1000	.264	.0378	.13	--	.63
1	500	.35	29.7	.563	1429	.378	.0542	.13	0.094	1.036
1	500	.37	29.7	.563	1351	.356	.0511	.13	.074	1.056
2	500	.235	29.5	.566	2128	.562	.0808	.13	.310	1.820
2	500	.235	29.5	.566	2128	.562	.0808	.13	.310	1.820
3	500	.186	29.4	.568	2688	.710	.1020	.13	.520	2.610
3	500	.185	29.4	.568	2703	.714	.1025	.13	.520	2.610
4	500	.145	29.3	.569	3448	.911	.1309	.13	.820	3.31
4	500	.150	29.3	.569	3333	.881	.1266	.13	.780	3.35

E. HAZE CHARACTERIZATION AND SETTLING RATE STUDIES

Prior to the start of filtration experiments, a little work was done on the subject of haze characterization and settling rates. This work consisted of nothing more than the manufacture of benzene-in-water and water-in-benzene dispersions by several procedures in an attempt to find the most satisfactory method of making hazes for test purposes. In order to obtain qualitative measurements on the dispersions which were made, the optical density was measured with a Lumetron Colorimeter, Model No. 400, using no light filter. The manufacturer is the Photovolt Corporation, New York City. Two methods of making dispersions were tested. These were thermal methods and mechanical methods.

- 1.a A mixture of 1 liter of benzene and 1 liter of water was heated to about 65°C, agitated, and then cooled rapidly to about 20°C.
 - b Same as above, but cooling was slower and was only to about 30°C.
 - c Mixture was agitated at room temperature and cooled rapidly to about 4°C.
 - d Mixture was agitated at room temperature and cooled rapidly to about 20°C.
2. Dispersion was made merely by vigorous mechanical agitation.

Table X shows a summary of the observations which were made on dispersions described above.

TABLE X

Comparison of Various Hazes by Means of Light Absorption

Method of Preparation	Rate of cooling	Initial Temp. °C	Final Temp. °C	Optical Density	
				Water-in-Benzene	Benzene-in-Water
(1a) Thermal	Rapid	64.3	20.0	1.00	1.55
(1b) Thermal	Slow	63.1	32.3	0.85	0.65
(1c) Thermal	Rapid	33.2	2.8	0.30	0.30
(1d) Thermal	Rapid	33.2	20.0	0.68	0.20
(2) Mechanical	--	33.2	33.2	0.05	0.10

On the basis that a high optical density is indicative of a large number of finely divided droplets, method (1a) appeared to produce the finest dispersion in both phases. Consequently, another batch of hazes was prepared by this method, and observations were made relative to the rate of settling of the dispersed phase. Data are listed in Table XI, and are shown graphically in Figure 17.

TABLE XI

Measurement of Settling-Rates of Benzene-Water Dispersions

Time after Preparation min.	Optical Density of Dispersion	
	Water in Benzene	Benzene in Water
0	4.1	4.5
5	3.8	4.0
10	3.7	3.7
15	3.3	3.6
20	2.9	3.3
25	2.8	3.2
30	2.6	3.2
190	1.55	0.90
195	1.40	0.80
200	1.35	0.55
205	1.25	0.95
210	1.25	0.85
215	1.00	0.75

These data for thermal methods indicated that, although the dispersions did settle, the rate was sufficiently slow to allow them to be used easily in the filtration tests.

FIGURE 17

SETTLING RATES OF WATER-IN-BENZENE AND
BENZENE-IN-WATER DISPERSIONS

F. CLEANING PROCEDURE FOR TUBULAR FRITTED-GLASS FILTERS

The suggested procedure for cleaning fritted ware is to wash with hot hydrochloric acid and rinse with distilled water before use to remove any foreign matter such as dust. According to information from the manufacturer, many precipitates can be removed from the filters by washing thoroughly from the reverse side with distilled water. However, several chemical means are suggested. In the course of the work reported here, the filters were originally cleaned with hydrochloric acid, then subsequently cleaned when necessary in hot sulfuric acid plus a few crystals of potassium nitrite. This latter procedure was found to be quite satisfactory for the purposes at hand.