A REVERSE OSMOSIS SYSTEM FOR AN ADVANCED SEPARATION PROCESS LABORATORY*

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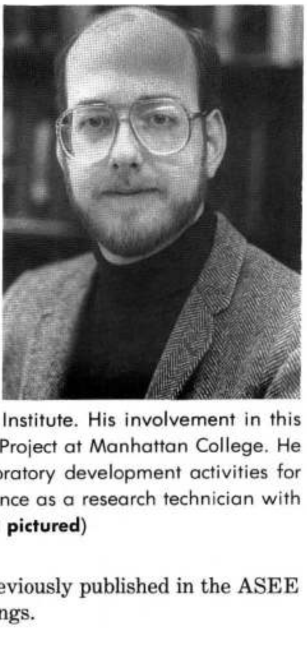
MEMBRANE PROCESSES are one of the new technologies being introduced in today's engineering curriculum. The membrane process reverse osmosis (RO) is being utilized by a broad spectrum of industries for a variety of uses. Applications are found in the agrichemical, biochemical, chemical, electrochemical, food and beverage, metal finishing, petrochemical, pharmaceutical, pulp and paper, and textile industries [1]. Reverse osmosis membrane processes are competing with the more traditional separation techniques such as distillation, evaporation, and filtration.

Reverse osmosis is considered a mass transfer unit operation and as such, theory relating to membrane transport should be presented in a mass transfer oriented course. System design and operation can be discussed briefly in a process or plant design course. Most engineering curricula present membrane technology in graduate courses, but some knowledge of the theory and operation should also be presented to undergraduates. The demonstration of reverse osmosis should occur in a senior level chemical engineering or "unit operations" laboratory. In such an experimental setting the student can understand the theory, operation and design of these systems and see the applications to industry. This could be in the form of a half-day experiment or a full semester project.



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This paper focuses on the development of a small pilot unit for use in an advanced separations process laboratory. The end goal is to develop experiments with advanced separation processes such as reverse osmosis, ultrafiltration, adsorption, chromatography, *etc.* This paper presents one step in that direction.

REVERSE OSMOSIS PRINCIPLES

In simplest terms, RO uses a thin semipermeable membrane that allows the transport of certain species while retaining others. A feed stream is introduced, and the membrane separates it into a purer stream, the permeate, and a more concentrated stream, the retentate or concentrate. The permeate is the stream that permeates the membrane barrier. Mass transfer through an RO membrane can occur by several mechanisms for which many models have been proposed [2, 3]. The solution-diffusion model describing water and solute transport through the membrane is utilized here [4]. In this model each species in solution dissolves and diffuses through the membrane at a rate corresponding to the applied transmembrane pressure, ΔP, and the concentration gradient ΔC_s, across the membrane.

The permeate flux, J_w, which in most applications is water, is directly related to the transmembrane or hydraulic pressure driving force, ΔP, minus the difference in osmotic pressure, $\Delta\pi$, on both sides of the membrane.

$$J_w = A_w (\Delta P - \Delta\pi) \tag{1}$$

The flux and pressure gradients are related by the water permeability coefficient, A_w.

The solute flux, J_s, is related to the concentration

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gradient on both sides of the membrane, ΔC_s, by a solute permeability coefficient, B_s

$$J_s = (B_s)(\Delta C_s) \tag{2}$$

Recovery is the ratio of permeate production, Q_p, to feed rate, Q_f.

$$\text{Recovery,} \quad Y = \frac{Q_p}{Q_f} \tag{3}$$

Solute rejection, R, can be measured in several ways. It can be denoted as a relationship between feed and permeate solute concentrations

$$R = 1 - \frac{C_p}{C_f} \tag{4}$$

Other ways to express rejection compare permeate to retentate concentrations or to an average of the feed and retentate concentrations. The recovery can also be incorporated into the expression [5]. It can be demonstrated from the solute and permeate flux equations that rejection is a function of pressure and concentration gradients. Water flux is dependent on pressure; therefore, an increase in pressure will increase water flux at constant solute flux, *i.e.*, decrease permeate solute concentration and increase solute rejection.

EXPERIMENTAL SYSTEM DESIGN

Since the membrane is the critical element in the RO system, proper understanding of structure and configuration is necessary before system design can commence. Reverse osmosis membranes are characterized by a high degree of semipermeability, high water flux, mechanical strength, chemical stability and economically acceptable cost. The early RO membranes were made out of cellulose acetate, but restrictions on process stream pH and temperature, including low rejection of some organics, spurred the development of non-cellulosic and composite materials. Polysulfones, polyamides, among others, and composite structures are popular alternatives because they do not have the draw-back of cellulose acetate [6]. The conventional composite membranes, normally called thin film composite membranes, do have a drawback in their ability to tolerate chlorine.

The membranes are configured into certain geometries for system operation. The four basic configurations are plate and frame, tubular, spiral wound, and hollow fiber. Since the studies employed in this paper use the spiral wound module, emphasis will be given to its characteristics with the reader referred to Leeper *et al* [7] for more design details. The spiral wound configuration consists of two membrane sheets forming an envelope for a permeate channel with feed channels on the outside of the envelope wrapped in a "jelly-roll" pattern around a perforated tube (Figure 1). The feed flows axially through the unit, exiting as retentate. The permeate enters the membrane envelope and travels spirally to the perforated center tube where it exits the membrane at the retentate end. The plate and frame and tubular membrane modules employ far more simple geometries, as their names imply. The hollow fiber membrane configuration utilizes thousands of "hair-like" hollow fibers in a fiber bundle.

This paper focuses on the development of a small pilot unit for use in an advanced separations process laboratory. The end goal is to develop experiments with advanced separation processes such as reverse osmosis, ultrafiltration, adsorption, chromatography, etc.

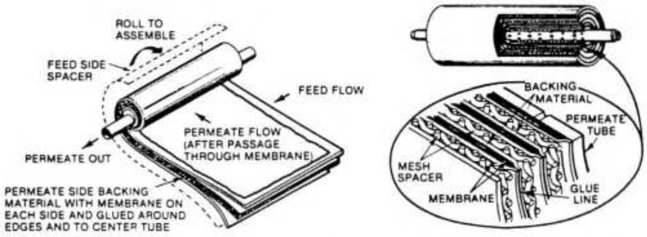

FIGURE 1. Spiral wound membrane configuration (from McNulty, K. J. and P. R. Hoover, EPA-600/2-80-084, U.S. Environmental Protection Agency, Cincinnati, OH, 1980, page 5).

The aforementioned membrane modules can be arranged in series and parallel to design a commercial scale unit. In most large-scale commercial systems, this is needed to accommodate the higher feed rates and produce a high recovery. The functioning of one module is the basis for commercial system design and scale-up. Therefore, experiments on one membrane element can be utilized by a student to develop a system for a process or plant design course.

A simple laboratory RO system was designed to be as versatile as possible. It can be operated in such a way that different process parameters can be evaluated. A single (or small multiple) membrane system was chosen. It has the capability to independently vary the following process parameters: membrane feed rate, operating pressure and temperature. It is designed so that modifications can be easily done and membrane replacement can be quickly accomplished. Use of other membrane configurations is also a design consideration. Since spiral wound RO membrane systems are the most prevalent in the industry the system was designed on this basis. The system can be easily retrofitted for hollow fiber and tubular configurations.

System design rational and a detailed discussion of the system's components are presented in Slater and Paccione [8]. A list of manufacturers of spiral wound membrane modules can be acquired by writing the author. Table 1 and the accompanying Figure 2 present the components and layout of the system. Some key features of the system are

- **Stainless steel construction to allow for durability and for the processing of a broad range of fluids**
- **Sizing to accommodate all 2.5 inch diameter spiral wound membranes and most of the 4 inch diameter modules**
- **Independent setting of feed rate (0-10 gpm) and pressure (0-1000 psi) to obtain desired recoveries**
- **Construction with tubing and standard compression fittings to allow for easy alterations**
- **Layout which permits easy control of system and direct observation of flow patterns**

System cost is always a consideration in a laboratory development project. It is encouraging to note that many membrane vendors and equipment suppliers give educational institutions discounts ranging up to 20% and some will even supply membranes. The system described in this paper, including a variety of membrane types, costs approximately \$12,000. Obviously this figure does not include the labor involved in design and fabrication. It is important to note that this cost could be reduced by not using stainless steel construction, by using a modest pump and drive, and by using house water for temperature regulation. A simple system on the order of \$5000 can be constructed for basic experiments if the future use and utility of the system is not important. Regardless of the complexity of the system involved, it is always cheaper to build your own system, and the involvement of students in the design, fabrication, and startup activities broadens the scope of the project.

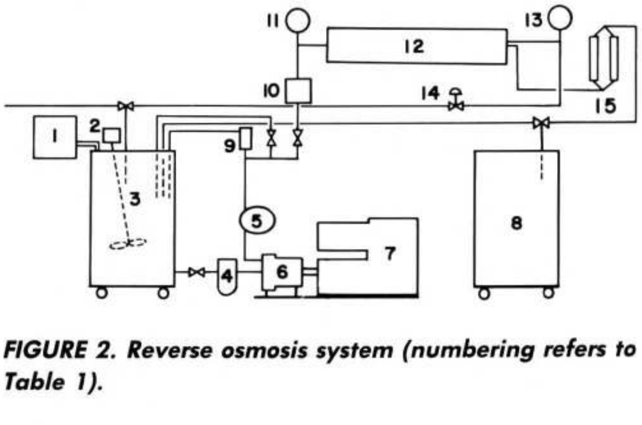

FIGURE 2. Reverse osmosis system (numbering refers to Table 1).

Several modes of operation are possible with the RO system and are shown in Figure 3. The steady-state recycle pattern of operation was used in the detailed experiments that follow. The critical process stream characteristics are flow rate and solute concentration. Permeate flux is obtained by dividing the permeate flow rate by the respective membrane surface area. Typical units are m^3/m^2d, cm^3/cm^2s and gal/

TABLE 1
Reverse Osmosis System Component Listing

Component	Model	Manufacturer-Distributor
1. Temperature Control Unit	**2095**	**Forma Scientific Marietta, OH**
2. Agitator	**T-Line Lab Model 104**	**Talboys Engineering Corp. Emerson, NJ**
3. Feed Tank	**Model SSD-55**	**Utensco, Inc. Port Washington, NY**
4. Prefilter	**Big Blue Model T-1508-70 w/1508-78**	**Cole-Parmer Instrument Co., Chicago, IL**
5. Pulsation Dampener	**850990**	**Greer Products, Los Angels, CA**
6. Pump	**Hydra-Cell D 10S**	**Wanner Engineering, Mahwah, NJ**
7. Drive	**Reeves Vari-Speed 5 Hp—Size 331**	**Reliance Electric Co., Columbus, IN**
8. Permeate Tank	**Model CC-55**	**Utensco, Inc. Port Washington, NY**
9. Pressure Relief Valve	**R3A**	**Nupro Co., Willingboro, OH**
10. Feed Rotameter	**10A 2227A**	**Fischer & Porter Warminster, PA**
11. Feed Pressure Gauge	**Master Series E 9672B**	**Marsh Instrument Co., Skokie, IL**
12a. Membrane Pressure Vessel*	**2S-1-2521**	**Advanced Structures, San Marcos, CA**
12b. Membrane Module*	**SW 30-2521**	**FilmTec Corp. Minneapolis, MN**
13. Retentate Pressure Gauge	**see component 11**	
14. Back Pressure Regulator	**26-1723-28-035**	**Tescom Corp. Elk River, MN**
15. Permeate Rotameters	**3202,3,4-20**	**Gilmont Instruments, Great Neck, NY**

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*Used in the specific experiments described in the paper. Other suppliers and specifications listed in ref. [8]

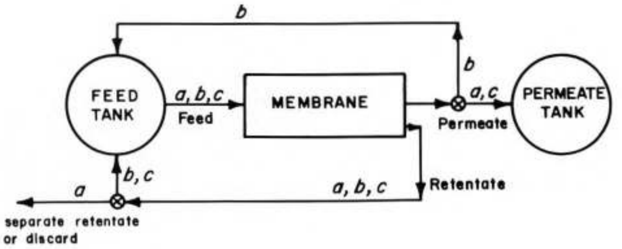

FIGURE 3. System operational schematic. Flow options: a) single pass, b) recycle steady state, c) recycle unsteady state.

dayft^2, also written as gfd. Solute concentration for a simple solute system is determined by measuring process stream conductivity and correlating the value to solute concentration.

EXPERIMENTAL STUDIES

Several simple experimental studies can be conducted to examine process parameters and membrane mass transfer characteristics. Data can be analyzed quickly to determine if the experiment was carried out correctly. In most of these experiments certain process parameters are kept constant and others varied. Process feed rate, concentration, temperature, pH, recovery, and pressure can be adjusted within certain operating limits.

The two major characteristics to be observed in a reverse osmosis experiment are separation efficiency and permeate production. The effect of process variables on these, and the verification of membrane mass transfer models, can be accomplished with simple experiments. A summary of some typical experiments is presented below. Specific details are omitted so that instructors can construct the proper experiment for the appropriate student audience and time frame.

Pressure Study

The effect of pressure on permeate production and solute rejection can be easily demonstrated. A particular membrane module should be chosen for this study—particularly one that will allow for a wide range of operating pressures. A simple solute, *e.g.*, NaCl, can be utilized as the feed at a certain concentration, *e.g.*, 5000 mg/L. Process parameters such as temperature and pH are maintained at certain values. The recovery for each run can be held constant by regulating the feed flow rate. It is important to remember to operate within the appropriate feed flow and/or recovery limits established by the manufacturer to lessen the problems associated with concentration polarization. Runs are conducted at various pressures, at 25 or 50 psi increments, depending on the amount of data to be collected. Membrane manufacturers give a maximum operating pressure for each membrane along with a normal or recommended operating range. This range is usually 10 to 25% less than the maximum. The system's safety relief valve can be set at the upper end of the pressure range to protect the membrane from excessive pressures resulting from operator error or mechanical malfunction.

In the experiment the student first observes the minimum pressure needed to produce permeate. As the pressure increases, the permeate flow will increase and the permeate concentration will decrease. The effect of increased pressure on permeate production can be explained by the permeate flux model (Eq. 1). By plotting permeate flux *vs* pressure the relationship is observed. At each of the pressures a sample of the permeate is analyzed using the conductivity meter, and its salt concentration is determined. The permeate concentration will decrease exponentially with increasing pressure. This phenomena is a result of solute flux being independent of applied pressure (Eq. 2). The membrane's solute rejection can be calculated by one of the expressions given earlier (Eq. 4). The rejection will increase and appear to approach a maximum as pressure is increased.

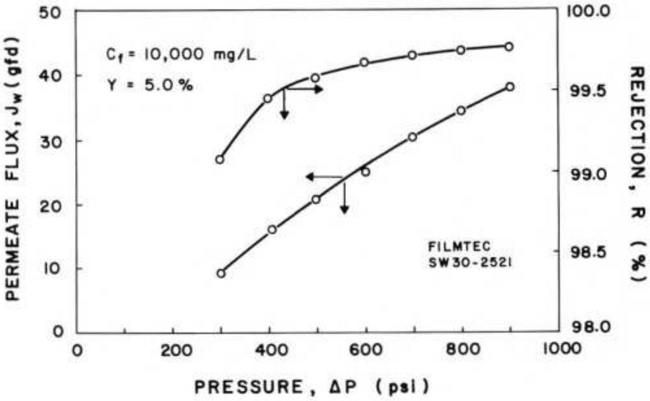

FIGURE 4. Results of a typical experimental study examining the effect of pressure on permeate flux and solute rejection.

An example of typical experimental output is shown in Figure 4. This study utilized a FilmTec SW30-2521 (2.5 in. diameter × 21 in. length) thin film composite spiral wound membrane. A 10,000 mg/L solution of NaCl was used as the feed at a temperature of 20°C and pH of 6. The study was run at a recovery of 5.0% at pressures ranging from 300 to 900 psi.

Feed Concentration Study

The effect of feed solute concentration on permeate

The two major characteristics to be observed . . . are separation efficiency and permeate production. The effect of process variables on these, and the verification of membrane mass transfer models, can be accomplished . . .

flux and concentration can be readily observed. In this experiment the only parameter varied is the solute concentration. The feed and standard processing conditions utilized in the previous study can be employed here. A particular operating pressure is selected for all runs. The recovery can also be maintained.

Runs are conducted at various concentrations from a low value to a high value , *e.g.*, 1000 mg/L to 35,000 mg/L. Increments are chosen depending on time; five to ten different concentrations are usually sufficient. At each different concentration the permeate production and its concentration are determined. As concentration increases, permeate flux decreases because of the increased osmotic pressure of the feed (Eq. 1). The solute flux is dependent on the concentration (Eq. 2) so it will increase as the feed concentration is increased. Therefore an increase in the permeate concentration and a decrease in solute rejection with increased feed concentration will be demonstrated.

Experimental output from a series of runs with a FilmTec SW30-2521 membrane at increasing NaCl feed concentrations can be seen in Figure 5. The plot illustrates the decrease in permeate flux and the increase in permeate concentration as the concentration of the NaCl feed solution is increased. The runs were performed at an applied pressure of 600 psi at 20°C with a feed rate of 3.0 gpm.

An extension of this and the initial study would be to examine different membrane models from each manufacturer. This comparison would show the different membrane characteristics within a particular membrane product line.

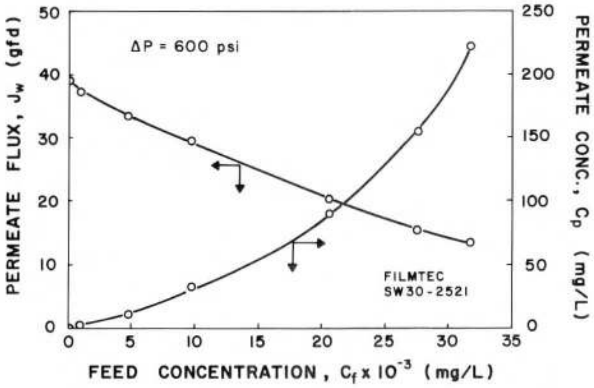

FIGURE 5. Results of a typical experimental study examining the effect of feed concentration on permeate flux and concentration.

Additional Studies

Many other studies can be performed utilizing the RO system. Publication of a thorough description of each study is planned for some later date and will include

- **The effect of temperature on permeate production and separation efficiency**
- **Determination of mass transfer coefficients**
- **Analysis of concentration polarization and related models**
- **The effects of fouling on permeate production and separation efficiency**
- **Operational characteristics of other membrane configurations, *e.g.*, tubular and hollow fiber**
- **The effect of different membrane materials on organic solute rejection**
- **The effect of recovery and feed rate on permeate production and separation efficiency**
- **The effect of pH on separation efficiency and membrane life**

CONCLUSIONS

Reverse osmosis theory and system operation can be demonstrated with a series of experiments on a small pilot-scale unit. The system design is simple and yet quite versatile. The system is made with an "open-end" design and can be easily changed to incorporate future design modifications and be used with different types of membranes. The system that was developed for an advanced separations laboratory was based on using small spiral wound membranes and can operate at feed rates to 10 gpm and pressures to 1000 psi. Feed flow, solute concentration, temperature, pressure, and recovery can all be independently varied. The system can operate in various flow schemes. Experiments investigate simple operational parameters and mass transfer characteristics. Permeate production and solute rejection are studied. More detailed experiments involving organic separation, concentration polarization and fouling and other membrane configurations can be performed.

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McGovern of FilmTec for their assistance with this project.

NOMENCLATURE

A_w	Water permeability coefficient $[L^{-1}t]$
B_s	Solute permeability coefficient $[Lt^{-1}]$
C_f	Feed solute concentration $[ML^{-3}]$
C_p	Permeate solute concentration $[ML^{-3}]$
C_r	Retentate solute concentration $[ML^{-3}]$
C_s	Solute concentration $[ML^{-3}]$
J_s	Solute flux $[L^3L^{-2}t^{-1}]$, $[ML^{-2}t^{-1}]$
J_w	Water or permeate flux $[L^3L^{-2}t^{-1}]$, $[ML^{-2}t^{-1}]$
ΔP	Applied pressure gradient $[ML^{-1}t^{-2}]$
Q_f	Volumetric flow rate of feed $[L^3t^{-1}]$
Q_p	Volumetric flow rate of permeate $[L^3t^{-1}]$
Y	Recovery, single-pass operation [unitless]
$\Delta\pi$	Osmotic pressure gradient $[ML^{-1}t^{-2}]$

REFERENCES

1. Slater, C. S., R. C. Ahlert, and C. G. Uchrin, *Desalination*, 48, 171 (1983).
2. Sourirajan, S., and T. Matsuura, *Reverse Osmosis/UltraFiltration Process Principles*, National Research Council of Canada, Ottawa, Canada, 1985.
3. Belfort, G. (ed.), *Synthetic Membrane Processes: Fundamentals and Water Applications*, Academic Press, New York, NY, 1984.
4. Weber, W. J., Jr., "Membrane Processes" in *Physiochemical Processes for Water Quality Control*, Wiley, New York, NY, 1972.
5. Burns and Roe Industrial Services Corp., *Reverse Osmosis Technical Manual*, Office of Water Research Technology, Department of the Interior, Washington, DC, 1979.
6. Turbak, A. F. (ed)., *Synthetic Membranes: Vol I.*, American Chemical Society, New York, NY, 1981.
7. Leeper, S. A., D. H. Stevenson, P. Y. Chiu, S. J. Priebe, H. F. Sanchez, and P. M. Wikoff, *Membrane Technoloqy and Applications: An Assessment*, EG & G Idaho, Inc., Idaho Falls, ID, 1984.
8. Slater, C. S., and J. D. Paccione, "Design of a Pilot-Scale Reverse Osmosis System for an Advanced Separations Laboratory," 1986 Annual Conference Proceedings of the American Society for Engineering Education, Washington, DC, pp. 812-820 (1986). ☐

ChE book reviews

FUNDAMENTALS AND APPLICATIONS OF ION EXCHANGE

Edited by L. Liberti and J. R. Millar
Martinus Nijhoff Publishers, Dordrecht,
The Netherlands, 1985. 484 pgs, $65.50

Reviewed by
Friedrich G. Helfferich
Pennsylvania State University

Fundamentals and Applications of Ion Exchange is a collection of thirty contributions to a NATO Advanced Study Institute held in 1982 in Maratea, Italy. A wide range of topics is covered, from a crystal-ball perspective "Ion Exchange Towards the Twenty-First Century" by the unforgettable Calvin Calmon, to highly specialized industrial problems such as reduction of regenerant acid use in water desalination (Hendry), start-up of a RIM-NUT plant for recovery of nutrients from eutrophic aqueous discharges (Liberti *et al*), and copper and nickel recovery from plating plant effluents (Stortini), and to complex theoretical studies *e.g.* of the microscopic basis and limits of the Nernst-Planck-Poisson system (Buck) and use of the Stefan-Maxwell flux equations in multicomponent ion exchange (Graham).

The attractively produced volume constitutes the third crop harvested from the Maratea NATO Advanced Study Institute. The thirteen main lectures were published in a previous volume of the NATO ASI Series (*Mass Transfer and Kinetics of Ion Exchange*, No. 71, Nijhoff, The Hague, 1983) and a selection of fifteen other contributions of general interest appeared in *Reactive Polymers* (Vol. 2, Nos. 1, 2, January 1984). The residue collected here is of mostly high, if uneven, quality. Perhaps the harshest criticism that can be voiced on this score is that the volume has no cohesion, no common denominator other than a loose relation to ion exchange. The seemingly unorganized side-by-side of specialized pragmatic, abstract complex theoretical, and review-style papers is a little disconcerting.

Perhaps more disturbing is that at least five of the thirty papers of the book have been published previously [Hogfeldt *et al* on a method of summarizing equilibria data, and Meagher *et al* on Mossbauer and electron microprobe studies of precipitation in Nafion membranes, in *Reactive Polymers*, 2 (1984) 19 and 51, respectively; Bolto *et al* on recycling of waste water constituents, in *Effluent Water Treat. J.*, (1983) 23; Buck on the Nernst-Planck-Poisson system, in *J. Membrane Sci.*, 17 (1984) 1; Drummond *et al* on kinetics in Zeolite A in *J. Phys. Chem.*, 87 (1983) 1967]. No references to such prior publications are given in the book.

The ion exchange expert will welcome this volume as a reasonably priced collection of specialized information. No other reader is likely to be interested, and the didactic value for course work is nil. ☐