High-pressure R&D on wheels

John P. Friedrich

A system is described for high-pressure equipment that can be adapted quickly and conveniently to changing requirements. This versatility is achieved by mobilizing the electronic controls and autoclaves, thereby permitting rapid interchange. All electronic sensing and control equipment is mounted in modules that can be rolled from cell to cell where all power supplies, receptacles, and thermocouple and transducer leads are matched both as to type and location. Autoclaves are mounted on dollies having identical dimensions. The dolly hold-down clamps and utilities and plumbing in each cell are also identical both as to type and location. Resulting mobility allows one to make maximum use of available cells.

The economical approach to the need for increased working capacity is not necessarily new construction but improved efficiency of operation. If a laboratory could be made more versatile, more adaptable, and less subject to shutdown and other delays, then the required research could be carried out in a facility of reasonable physical dimensions. While we were operating in a less than satisfactory facility, we kept a record of problems that interfered with efficient operation to act as a guide for future planning. Almost all the difficulties could be traced to two basic problems:

1. Failure of electronic sensing and control equipment
2. Immobility of autoclaves and attendant plumbing

This paper will characterize these problems and describe a solution that has proved to be satisfactory in use.

Mobile control modules

Most existing facilities employ the time-honored "control panel" in which all electronic control and sensing equipment is mounted in a stationary panel parallel to and perhaps 2 ft from the wall of the pressure cell and facing away from it. With this system even a minor electronic malfunction can become a major obstacle. Reaching the electrical connections at the back of the equipment is nearly impossible through the maze of valve handles, plumbing, and electrical conduits that traverse the area between the control panel and the cell wall. Servicing then means removing the unit from the panel. This time-consuming method usually requires special personnel, who may not be immediately available. If the problem is serious, the unit may even require factory repair which entails loss of service for an extended period. One alternative is to maintain a back-up for each piece of equipment; however, having a spare is only a partial solution. In addition to being costly, the back-up unit may require lengthy calibration to match it with other units in the system. At best, you have lost a few hours and at worst, several weeks. But in research even a few minutes delay can cause the loss of an intermediate which is more or less costly to prepare.

Since receiving his doctorate in organic chemistry at Oklahoma State in 1959, Dr. Friedrich has worked at the Northern Regional Research Lab. His studies have been concentrated on industrial utilization of fats and oils; in particular, linseed oil. Much of his chemical modification work involved high pressure which led to his present position as principal chemist in charge of high-pressure research, Peoria, Ill.

Our solution was to mount all necessary electronic equipment in *mobile* control cabinets (Figure 1). The cabinets are mounted on locking 4-in. heavy-duty soft rubber wheels, two fixed and two swivel. A folding 12-in. shelf on the front provides a convenient place to record data. A large locking steel door (Figure 2) on the back allows access to all the instruments inside. This door is louvered to provide ventilation. Two large 200-A, 120-V receptacles, one male (power in) and one female (power out), are located on top of the cabinet. The overall dimensions of this control module (66 in. high × 24 in. wide × 18 in. deep) are such that it can be rolled through standard doorways.

The control cabinet contains a strip-chart temperature recorder. Variable speed permits recording of overnight operation as well as rapid changes in time variables; a flip-out transport feature permits a continuous record of a process even if it becomes necessary to change control modules during a reaction (Figure 2). In the event that a large number of similar reactions are being run on a semiroutine basis, the flip-out chart can be used to maintain a continuous record for future reference.

Heating to the autoclave is provided by a three-function current adjusting temperature controller and a silicon-controlled rectifier power unit, which proportions ac power. The latter unit is mounted inside the control module. Power lines run from it to the receptacles on top of the cabinet.

A high-limit temperature control *with separate thermocouple* ensures against overshooting critical temperatures because of instrument failure in the primary control system. If the process reaches the preset temperature, current to the autoclave is automatically interrupted and an audible alarm is triggered.

Process pressure is recorded on a dual range recorder equipped with a front set switch which also interrupts the power to the heating load if the preset pressure is reached.

A tachometer, easily moved from one control module to another, measures the agitator speed of magnetically stirred autoclaves.

The left side of the control module contains the electrical connections, other than primary heating, which a process may require. Figure 3 shows details of this section: The first and third receptacles are thermocouple connections for primary control and the high-limit override; the

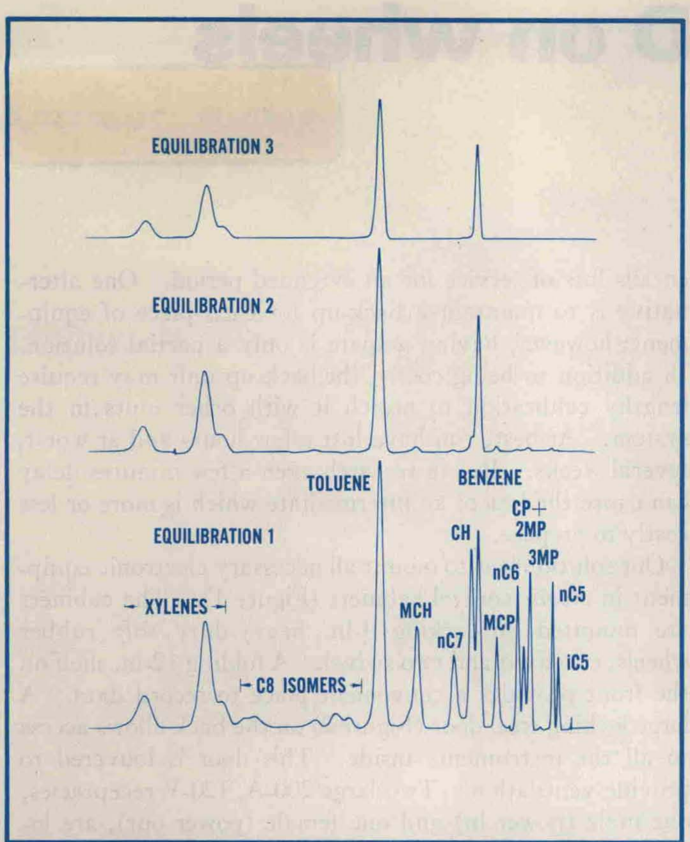

Figure 8. Hydrocarbons dissolved in tap water from a sample of crude oil. The chromatograph attenuation was constant

pound is a nonhydrocarbon. Such information even in a qualitative sense can be quite useful.

Such distribution coefficients, along with relative retention time, help identify unknown organic compounds. An actual example of the identification of organic contaminants in a tap-water sample from a city water supply is shown in Figure 7. The first equilibration shows the presence of several organic compounds which, if this were the only chromatogram, would be difficult to identify. However, distribution coefficients obtained by additional equilibrations permit positive identification of several of the contaminants.

The first peak is predominantly methane. The next prominent peak has the relative retention time for *n*-hexane, but the additional equilibrations gave a distribution coefficient that duplicates that for chloroform (as calculated from solubility and vapor pressure data). Chloroform added to water gave the same results.

Benzene and toluene were identified also by relative retention times and partitioning between gas and water phases. The peaks on either side of benzene have not been identified, but their distribution indicates that they are not hydrocarbons. Similarly, the peaks between methane and chloroform are not alkanes, which would have been completely removed after two equilibrations; they may be olefins.

The concentrations in the tap-water sample are low (less than one ppb) but readily measurable. By contrast, only methane is detectable by this method in seawater samples from Cook Inlet, Alaska (*9*).

Hydrocarbons dissolved from a crude oil sample. Although one would predict from Figure 1 that a complex mixture of hydrocarbons would partition as shown, it is always satisfying to see the actual analysis of a complex mixture. Such a mixture, a sample of crude oil from the Black Hollow oil field near Ft. Collins, Colo., was contacted with tap water (*8*). The light gases had been separated from the crude oil, so C_1's through C_4's were in low concentration.

The water containing the hydrocarbons dissolved from the crude oil was analyzed, and the results are shown in Figure 8. The first-equilibration chromatogram shows all the hydrocarbons through eight carbon atoms normally found in crude oils. As predicted, the first equilibration removed over 96% of the alkanes, leaving aromatic hydrocarbons and about 10% of the cycloalkanes. The third equilibration chromatogram shows only aromatic hydrocarbons.

Method sensitivity. When we use the described procedure and introduce a 5-ml gas sample into the chromatograph, the method is capable of detecting alkane and cycloalkane hydrocarbons in water if they are present in amounts of one to three parts in 10^{12} parts of water by weight. Aromatic hydrocarbons, because of their lower partitioning into the gas phase, can be detected if present in concentrations of 4–12 ppt. Reasonable accuracy can be obtained if the aqueous concentrations are 20 to 30 times these values. Methane is present in open ocean waters in amounts of 28–36 ppt (*10*). With the present procedure, methane can be detected at 1 ppt or less.

Sensitivity can be increased by analyzing a larger sample of the gas phase and by increasing the ratio of water to gas. Patent rights are reserved by Chevron Research Co.

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The quietest place on earth must be the area inhabited by the Mabaan tribe of the Sudan, where background noise is one-tenth as loud as the murmur of a refrigerator. Boing, the main town, hears only the moo of a cow and, occasionally, the patter of raindrops. All this has given the Mabaans perfect hearing: They can pick up a normal voice at 300 ft, and 75-year-olds can hear as well as 25-year-old city dwellers.

Forum World

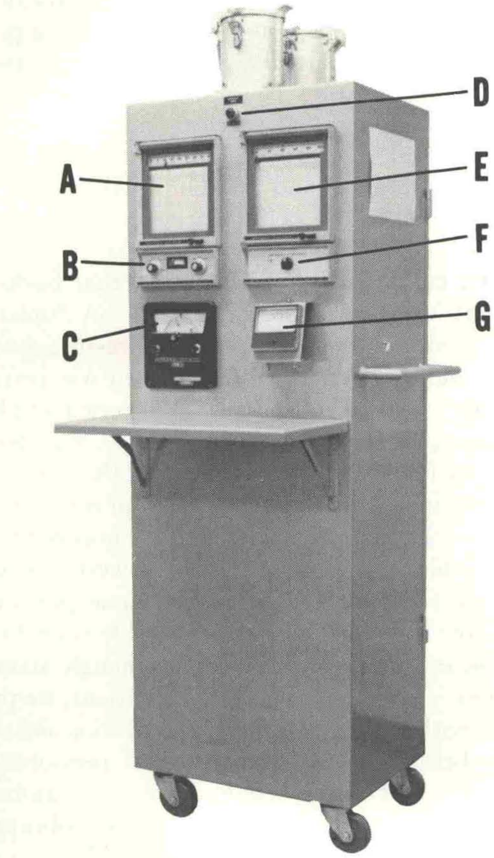

Figure 1. Front view of control module

A. Temperature recorder
B. Three-function current adjusting temperature controller
C. High-limit temperature fail-safe
D. Instrument pilot light
E. Dual-range pressure recorder
F. Pressure transducer power supply
G. Autoclave stirrer tachometer

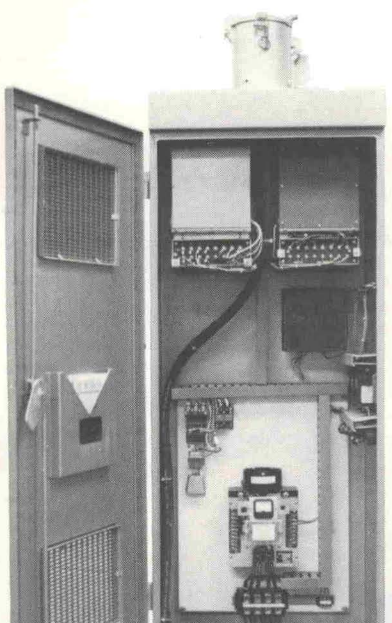

Figure 2. Rear view of control module

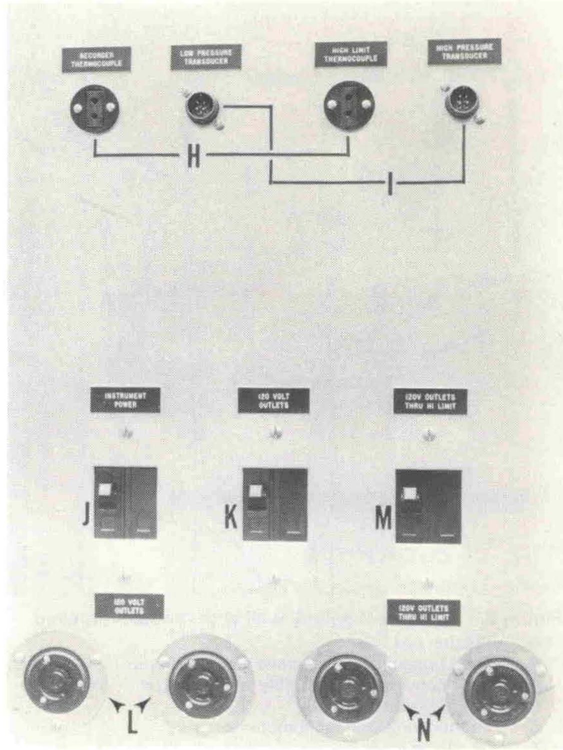

Figure 3. Left side of control module (control and sensing connections)

H. Recorder and high-limit thermocouple connections
I. High- and low-range pressure transducer connections
J. Instrument power supply and circuit breaker
K,L. 120-V circuit breaker and outlets
M,N. 120-V circuit breaker and outlets with high-limit temperature and pressure override

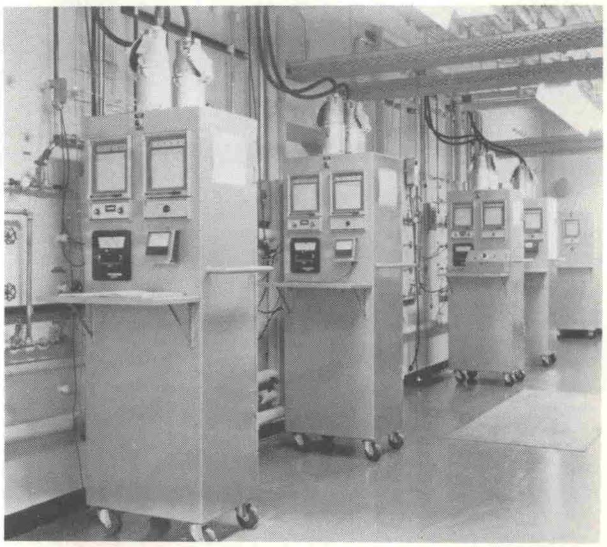

Figure 4. Control gallery with interchangeable modules in place

other two are for the high- and low-range pressure transducers. A 15-A circuit breaker serves the instrumentation. While a 20-A breaker controls each pair of 110-V ac outlets, one of which is wired through the high-limit control. Since one pair of 110-V circuits is used to power agitation, the operator has a choice of maintaining or stopping agitation whenever preset pressure or temperature is exceeded.

In the event of any malfunction, it is a 3-min job to break all connections and move another cabinet into place. A back-up module is desirable; however, one module is usually available because all cells are seldom in operation simultaneously.

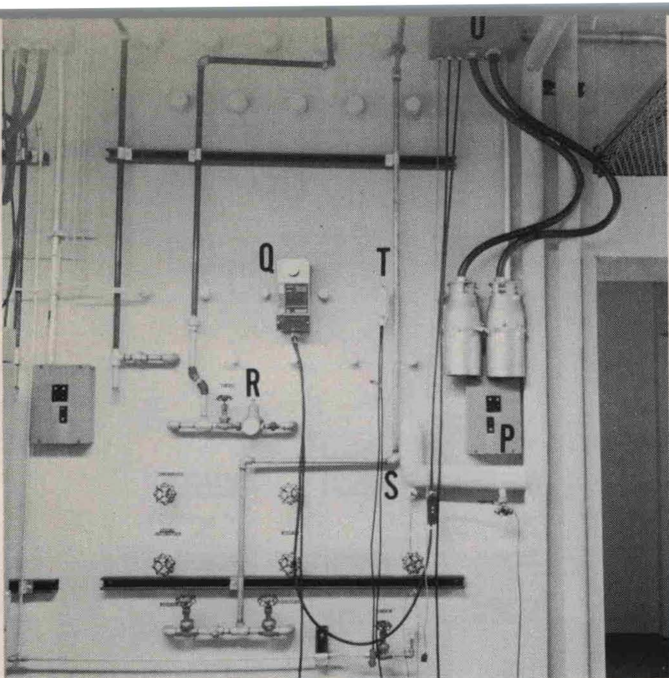

Figure 5. Section of gallery wall with module removed
O. Junction box
P. Circuit breakers for autoclave heating circuits
Q. Ac-dc converter and variable speed control
R. High-pressure air supply
S. Thermocouple and tachometer leads
T. Transducer leads

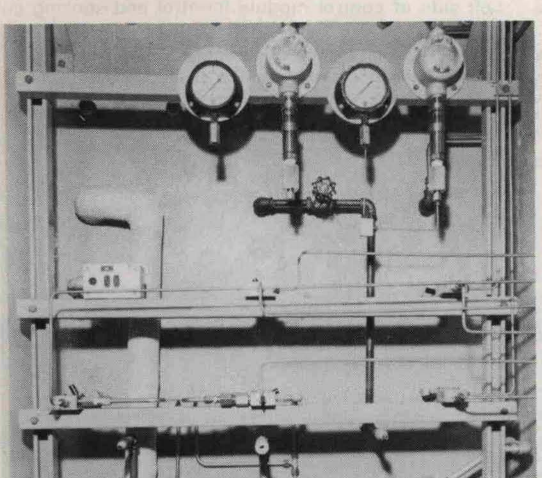

Figure 6. Interior cell wall backing control gallery

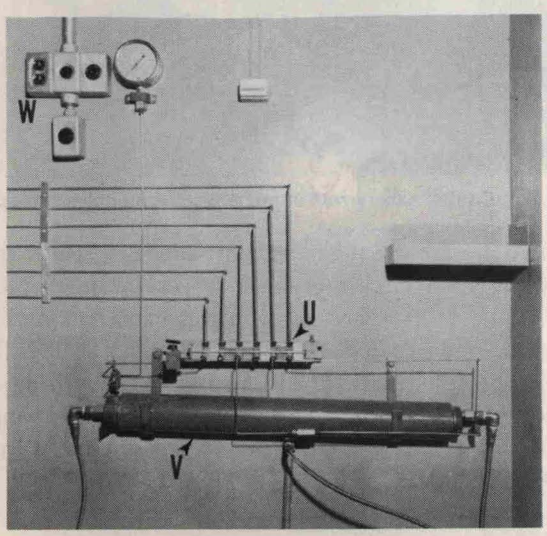

Figure 7. Interior cell wall facing entrance and perpendicular to control gallery wall
U. Terminal block for permanent pressure plumbing
V. Pressure intensifier
W. Electrical connections

Outside the cells. Our control gallery with cabinets in place is shown in Figure 4, and Figure 5 shows a typical exterior cell wall. Our autoclaves are wired with two or three circuits to facilitate control, particularly at low temperatures. Nonelectrical services common to each cell are 125 psi steam, cold water, soft water for cooling coils, high-pressure air, and vacuum. All valves and utilities are located in the same relative positions in each cell.

Inside the cells. The inside cell wall that backs the control gallery wall is shown in Figure 6. A "unistrut" frame was used for mounting valves, pressure gauges, transducers, and the like. The frame itself was installed 10 in. from the wall to avoid utilities. All permanent high-pressure valves, fittings, and tubing are $^1/_4$-in. nominal diameter rated for 60,000 psi. Utilities, other than electrical, are terminated in either "quick connects" or flare fittings and are brought out flush with the unistrut frame. These are visible below the third and lowest horizontal bar. Because they are located in the same position in each cell, short standard lengths of flexible hose can be quickly attached to the autoclave. Although standard hydraulic hose is suitable for many applications, we prefer $^3/_8$-in. Teflon with braided stainless steel covering and stainless fittings because it withstands higher pressures and temperatures and, of course, is quite inert. The autoclave vent, accumulator vent, and rupture disk assemblies are all

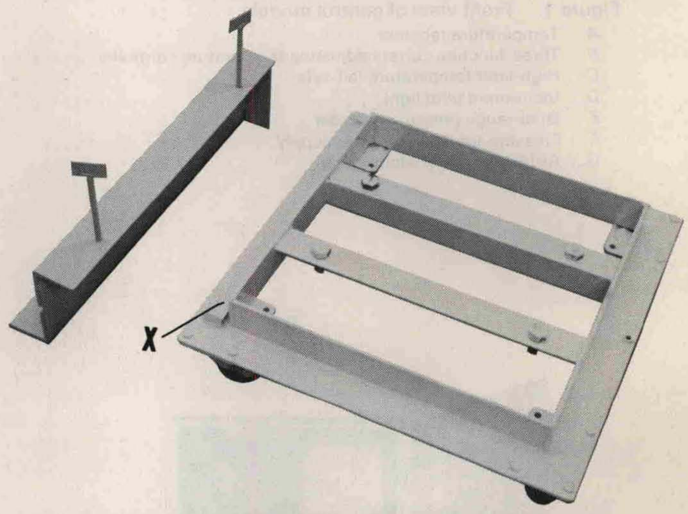

Figure 8. Equipment dolly and hold-down clamp
X. Alignment guide for hold-down clamp

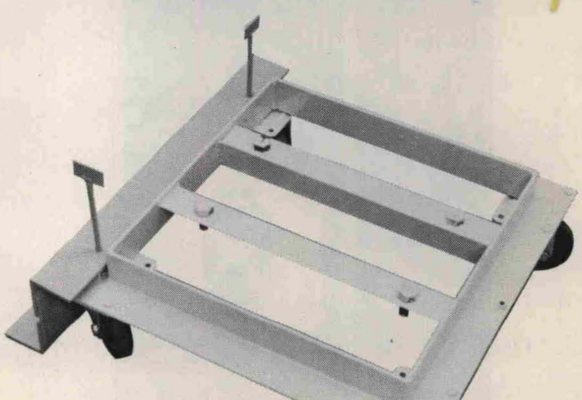

Figure 9. Equipment dolly in clamped position

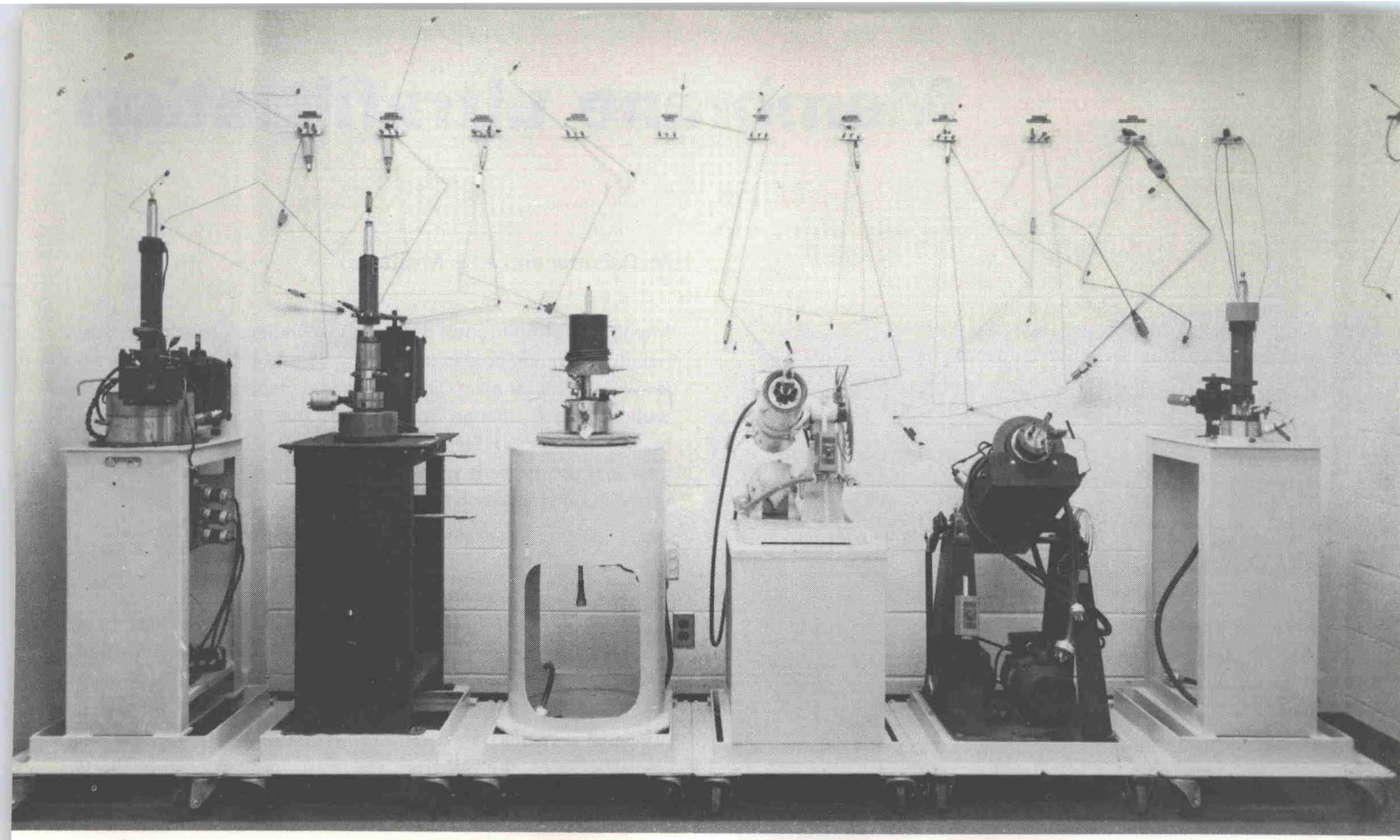

Figure 10. Stored autoclaves and attendant standardized tubing hookups

connected to a $^1/_2$-in. line, which extends through the *outside* wall of the cell and terminates under the lower exhaust stack. Steam (125 psi) is connected to the other end of the vent line and is used to purge it. By removing the dust cap from the exhaust stack, it is possible to purge the line directly into the sand pit outside the cell. We find this steup more efficient and cleaner than overhead vent lines.

The wall of the cell opposite the entrance is shown in Figure 7. The $^1/_4$-in. high-pressure tubing terminates in "L" fittings which are mounted on a length of unistrut and attached to the wall. This terminal block is located in the same position in each cell. A gas intensifier or accumulator is hung directly below this terminal block. The hook-shaped intensifier hangers are also located in the same position in each cell so that the intensifier and attendant tubing can be moved from cell to cell. The tubing and fittings between the terminal block and the accumulator, as well as between the block and the autoclave, are $^1/_8$-in., 15,000 psi.

Different types of electrical receptacles were used to prevent inadvertent mismatching of supply and load. These connections are not explosion-proof since most of our autoclaves are not equipped with explosion-proof motors or nitrogen-shrouded heaters.

Mobile autoclaves

The changing requirements of research demand a diversity of high-pressure reactors. To avoid having a cell for each piece of equipment, the equipment is arranged to move readily in and out of the cells. Autoclaves, which range in size from 50 cc to 3 gal, are mounted on 2 × 2-ft dollies (Figure 8 and 9) which are constructed of $2^1/_2$ × $^1/_4$-in. angle iron with appropriate braces for bolting to the fixed stand in each cell. A length of 1-in. angle iron welded along one side of the dolly acts as an alignment guide for setscrews in the hold-down clamp. The angle iron frame is mounted on 4-in. soft-rubber heavy-duty wheels. All wheels swivel to permit maneuvering equipment in tight places. The hold-down clamp is constructed of two 26-in. lengths of 3 × $^1/_4$-in. angle iron welded as shown in Figure 8. A plate is welded to one end of the clamp to ensure proper alignment of the dolly. The clamp is fastened to the floor similarly in each cell so that the dolly with autoclave can be clamped into the same relative position. Figure 9 shows a rocking clave in place within a cell.

Short lengths of tubing, which are peculiar to each autoclave, are held in labeled clamps on the wall of the autoclave storage room with unused autoclaves (Figure 10).

With this system an autoclave can be disconnected, removed from a cell, and replaced with another in a matter of minutes. Furthermore, having each autoclave mounted on a dolly has another less obvious advantage. When opening or closing an autoclave containing noxious or poisonous materials, it is possible to roll the unit into a full-length hood, secure it to the floor with the clamp previously described, and carry out the entire operation quickly and safely.

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Membrane ultrafiltration

M. C. Porter and A. S. Michaels

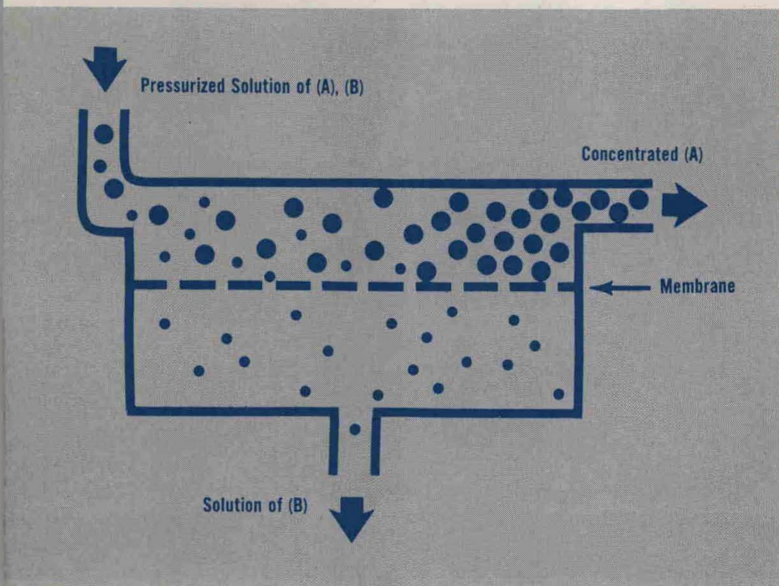

Figure 1. Schematic diagram of membrane ultrafiltration process

Membrane ultrafiltration removes low-molecular-weight materials from liquids containing polymeric and other high-molecular-weight materials. This molecular separation is effected without phase change which gives rise to unique technical results and costs. This first of five articles describes the process and compares it with competitive ones from an operability and economic viewpoint.

The emergence of low-pressure membrane ultrafiltration as an economic unit operation has generated considerable interest in several areas, among which is the food processing industry where it may be used for the concentration and purification of liquid foodstuffs, and for the recovery of valuable by-products from food-processing plant waste effluents. Virtually any aqueous food product whose nutritive values exist as macromolecular or colloidal substances can be inexpensively dewatered and/or demineralized by this versatile separations technique.

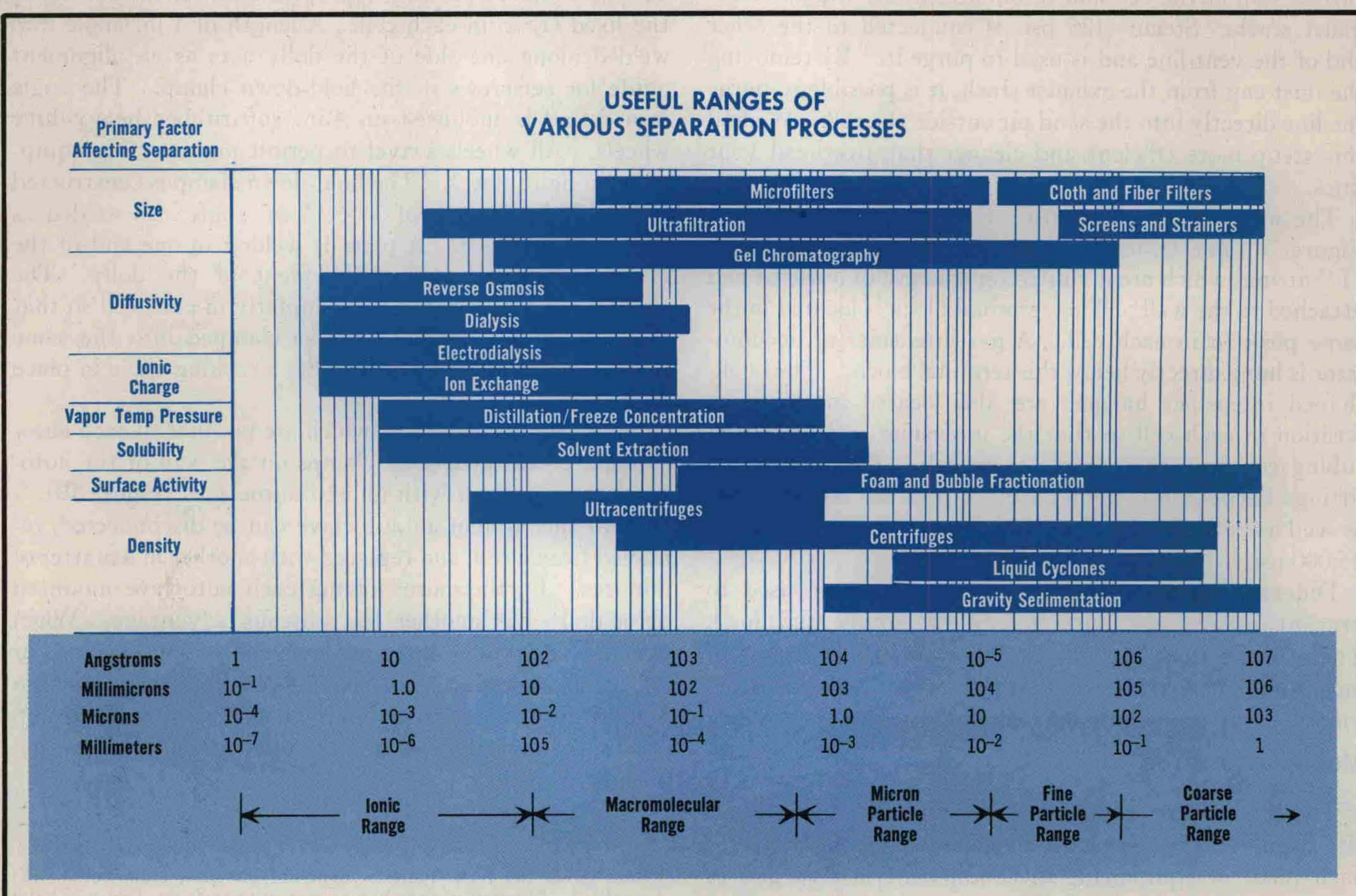

Figure 2. Useful ranges of various separation processes