

High-pressure R&D on wheels

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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 shut-down 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



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.

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.

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 \times 24 in. wide \times 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 semi-routine 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

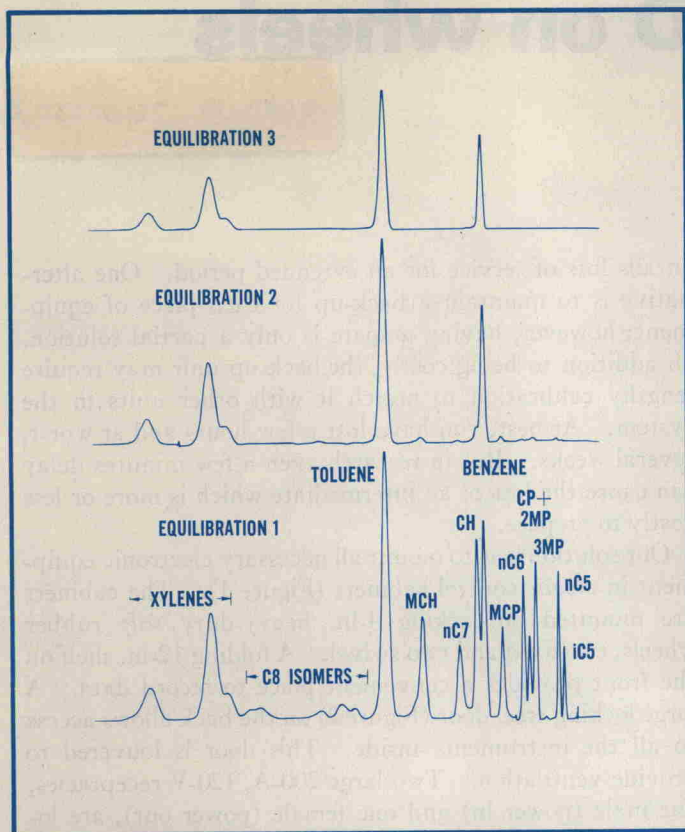


Figure 8. Hydrocarbons dissolved in tap water from a sample of crude oil. The chromatograph attenuation was constant

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.

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