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The main components of this closed-system recirculator-purifier are a circulating pump, a pressure-regulating system, and a purification bed. Gas flow rates of up to 35 ft³/h may be obtained while the spark chamber is held at a preset pressure plus or minus 0.02 in. of water. In normal routine operation the impurity level of the gas can be held to less than 50 parts per million.

Increasing use of spark chambers has necessitated development of an apparatus to recirculate the gas used. The demand for spark chamber neon gas (nominally 90% neon, 10% helium) has reduced its cost severalfold in the last few years. However, the present price of this gas is still somewhat more than a dollar per ft³, and this precludes its usage in a "once-through-and-vented" fashion for all except the very smallest of spark chambers. Any device to recirculate the gas should necessarily purify it, not only to remove gaseous compounds formed or liberated (from solid surfaces) by the sparks, but also to give the experimenter a wider choice in his selection of materials for the construction of the spark chamber. The ideal apparatus would then allow the experimenter to maximize the flow rate in order to get good sparks rather than minimize it because of economic necessity, and this ideal apparatus would also remove all gases except those the experimenter wanted.

The apparatus described here is a reasonable approximation of the ideal (see Fig. 1). Molecular sieve is used to purify the gas; two identical purification units are connected in parallel so that one can be regenerated while the other is in use. In each of these units the gas to be purified flows first through a bed of 13X molecular sieve 1 at room temperature, then through a heat exchanger to a bed of 5A molecular sieve 1 in a liquid nitrogen bath. The 13X sieve removes water, alcohol, and other vapors; the 5A sieve removes nitrogen, oxygen, and all other gases whose critical temperature is above 77° K. The purpose of the 13X sieve is to reduce the water load on the 5A sieve and also to avoid the possible consequences of sequestering alcohol and liquid oxygen in the same bed of sieve. The only gases that are not trapped or sequestered

by this system are neon, hydrogen, and helium. Some of the neon-helium mixture is adsorbed on the surface of the sieve; the loss of about 4 ft³ of gas per change of purifier unit is more than one would expect from the PVT relation.

The gas flows to and from the cold sieve through heat-exchanger tubing of the Collins type; we made this tubing only because we could not buy it. Each sieve bed contains 2 pounds of sieve in 4-in. nominal copper tubing about a foot long, with copper tube caps hard-soldered on as closures. The container for the cold sieve has copper fins hard-soldered to the inside of the tubing. Metallic-sheathed 750-watt electric heaters are hard-soldered to the outside of each copper container so the sieve bed can be heated while it is regenerated by vacuum pumping. Thermostats attached to the outside of the copper containers limit the maximum temperature to 250°F. There is a solenoid valve in the vacuum pumping line; this valve is electrically in parallel with the vacuum pump motor to prevent backflow of pump oil if there is loss of electric power.

The circulating pump is a Model G-3 Dia-Pump²); the nylon-reinforced neoprene diaphragm in this pump has a lifetime to failure of several thousand hours. Other diaphragm pumps that we have tried have had much shorter lifetimes; vane-type pumps do not work in this application because the gas is too dry. Typical operating parameters for this pump are: inlet pressure, 11 in. Hg vacuum; outlet pressure, 3 psig for a flow of 35 ft³/h.

The pressure-regulating system consists of two valves in series with the output of the circulating pump and two valves in series with the

input. These four valves are all modifications of the Matheson Model 70B fore pressure regulator³). All four valves are modified with new larger seats, O-ring-sealed plugs, and corrugated Mylar⁴) diaphragms. The fore pressure regulator after the pump discharge has an original-size diaphragm, but the second fore pressure regulator (right after, and in series with, the first) has a diaphragm twice the original diameter, for greater sensitivity.

From this high-sensitivity fore pressure regulator the gas flows to the spark chamber, next back from the spark chamber to a high-sensitivity back pressure regulator, then to a standard back pressure regulator, and finally to the input of the circulating pump. These back pressure regulators are identical to the fore pressure regulators except that their internal mechanical linkages have been inverted. The high-sensitivity regulators are mounted with their diaphragms in the vertical plane to reduce the effect of the weight of the parts on the sensitivity. The purification beds are located between the pump outlet and the first fore pressure regulator so that the positive pressure there can make the sequestering process more efficient. Typical pressures around the system loop for a flow of 35 ft 3 /h are:

| Between pump outlet and purifier | 3 psig |
|---|---------------------------|
| Between purifier and first fore pressure regulator | 2.75 psig |
| Between first and second fore pressure regulators | 0.5 psig |
| Between second regulator and spark chamber | 0.23 in. H ₂ O |
| Between spark chamber outlet and first pressure regulator | 0.11 in. H ₂ O |
| | |

Between first and second back pressure regulators 1 in. Hg vacuum

Between second regulator and pump inlet 11 in. Hg vacuum

For this flow rate the high-sensitivity regulators shut off the flow at ±0.4 in. H₂O. If one of the hoses carrying the gas to or from the spark chamber should be blocked, the pressure in the chamber would rise to +0.4 in. H₂O or fall to -0.4 in. H₂O, depending on which regulator is still connected to the chamber. For those cases in which these excursions from atmospheric pressure are too great, pressure and vacuum reliefs, respectively, are provided for the lines to and from the spark chamber. These reliefs are tubes that are immersed to the necessary depth in glass jars of mineral oil. For those experimenters who wish to dope the gas with alcohol there is plumbing between the first and second fore pressure regulators to tap off a part of the gas flow, saturate it with alcohol, and return it to the main gas flow.

All this equipment is carried in a four-wheeled cart. One side of the cart is a plywood panel on which the plumbing schematic has been painted. Flowmeters appear in their proper places and as many valves as possible have been brought to the panel for operating convenience. See Fig. 2.

The hoses should be connected so that the input to the spark chamber is at the top of the chamber and the outflow is at the bottom, although in actual practice experimenters seem to use the reverse configuration about half of the time.

About 20 volume changes are required before a chamber, initially full of air, contains gas of sufficient purity for operation. For example, a 16-ft³ chamber "cleaned up" in 12 hours at a flow rate of 25 ft³/h; the purifiers were changed four times during this period. In normal operation experimenters fall into the habit of changing purifiers

once a day, particularly if they are using alcohol. A set of very large (and very clean) chambers operated 15 days without a purifier change; at the end of this time the impurity level measured with a gas chromatograph was still very low. Usually impurity levels are lower than the following "least count" of the mass spectrometer on which gas samples were analyzed: O₂, 25 ppm; N₂, 25 ppm; Ar, 20 ppm, CO₂, 15 ppm.

This recirculator-purifier has been in use at Lawrence Radiation Laboratory since 1964; we now have 18 of these devices, and at least 12 of them are in operation at any given time. They perform well and faithfully.

Victor Perez-Mendez was the first to tell us of the need for such a device; we express our appreciation for the numerous stimulating conversations that have followed.

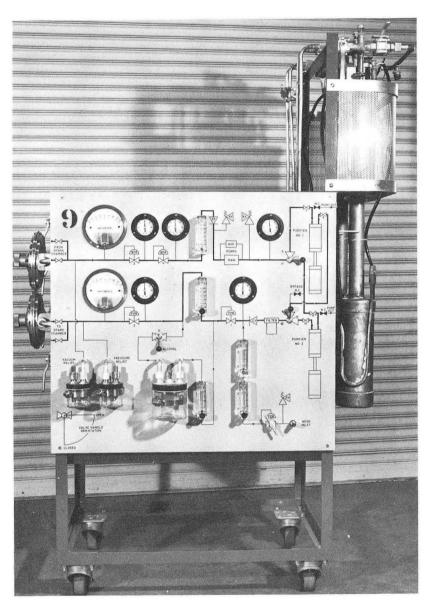
FOOTNOTES AND REFERENCES

 st Work done under the auspices of the U. S. Atomic Energy Commission.

- 1. Linde Division of Union Carbide Corporation is one of the manufacturers of 13X and 5A molecular sieve. We use 1/16-in.-diameter pellets, but other sizes would probably work just as well.
- 2. Air Control, Inc., 450 Narberth Avenue, Narberth, Pennsylvania.
- 3. The Matheson Co., Inc., East Rutherford, New Jersey. Valves that are similar are sold by other vendors as different model numbers.
- 4. E. I. Du Pont de Nemours and Co., Inc.

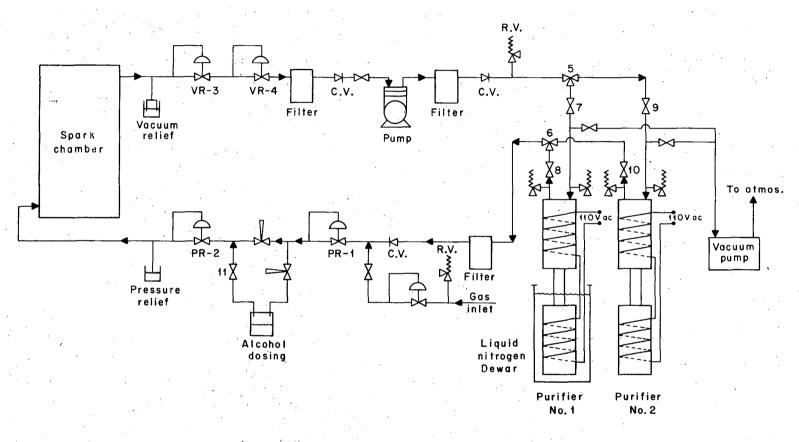
FIGURE CAPTIONS

- Fig. 1. Schematic flow diagram.
- Fig. 2. Apparatus mounted on its cart, with schematic diagram on panel, and valves and indicators in appropriate positions.



GPR 3058

Fig. 1



XBL691-1680

Fig. 2

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