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RESULTS OF PERFORMANCE TESTS CONDUCTED ON THE CONSOLIDATED VACUUM CORPORATION AND THE N.R.C. CORPORATION ULTRA-HIGH-VACUUM OIL DIFFUSION PUMPING ASSEMBLIES

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ABSTRACT

Performance tests have been conducted on the new metal-gasket-sealed Consolidated Vacuum Corporation and N.R.C. Equipment Corporation ultra-high-vacuum oil diffusion pumping assemblies (PMCU and HK models respectively). These tests were performed both with and without the use of the manufacturer's zeolite trap, and with water and refrigeration coolant (-35° to -40°C range) on the baffle immediately above the pump. When the zeolite trap was used, a base pressure of 5×10^{-10} torr range was attained with the Consolidated system, and 1×10^{-9} torr was the lowest base pressure for the N.R.C. unit.

With the zeolite trap removed, the ultimate pressure was lower with the N.R.C. pump and baffle than the Consolidated unit with water cooling on the baffle. Furthermore, refrigerating the N.R.C. baffle did not improve the base pressure attainable, which was $1 - 2 \times 10^{-9}$ torr with and without refrigeration. The conclusion was reached that the addition of a zeolite trap to the N.R.C. package did not further reduce the ultimate pressure. The Consolidated pump and baffle combination was able to reach a base pressure of only 6×10^{-8} torr with water cooling, but the base pressure dropped to 4.5×10^{-9} torr when the baffle was refrigerated.

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I. INTRODUCTION

In an effort to extend the available cross section data, a facility known as the Crossed-Beam Facility is being designed and fabricated. This apparatus must be capable of operation at a very low base pressure (in the 10^{-10} torr region), but the required vacuum should be attainable through use of ultra-high-vacuum oil diffusion pumps, liquid nitrogen trapping, and titanium evaporation. This report contains a description of tests recently made at ORNL to determine the most suitable type of oil diffusion pumps for the Crossed-Beam Facility.

Recently both the Consolidated Vacuum Corporation and the N.R.C. Equipment Corporation began marketing an all-metal-gasket-sealed, ultra-high-vacuum package unit which consists of an oil diffusion pump, a water-cooled baffle, and a bakeable zeolite trap. These systems are designated as models PMCU and HK, respectively. The usual pump isolation valve has been omitted from both assemblies, so it is necessary to cool the diffusion pump fluid prior to letting the vacuum system up to atmospheric pressure.

A 6-inch size pump, baffle, and bakeable zeolite trap were purchased from each company and tested. Since the ultimate base pressure and the time required to pump down to this value were of primary concern, these features were considered the more important in the testing, and no attempt was made to measure pumping speeds either at intermediate or at base pressures. However, the poor conductance through both of the zeolite traps indicates that in each case the normal speed of approximately 600 to 800

ℓ/sec. of air for a 6-inch size pump and baffle was reduced by at least a factor of two when the zeolite traps are included in the system. The tests were conducted with and without the traps and, in the latter case, with both water and refrigeration coolant on the baffle above the pump.

II. DESCRIPTION OF EQUIPMENT

A. Consolidated Vacuum Corporation Ultra-High-Vacuum Components

1. Diffusion Pump - The pumps in this series (PMCU) are modified versions of the Consolidated PMC type with several changes made to adapt them to ultra-high-vacuum service. Heavier, ground-finish, plain-faced (ungrooved) flanges are provided on both the inlet and outlet for metal ring gasket sealing. The oil drain in the boiler has been omitted and the pump casings are type 304 SS, with nozzle assemblies made of aluminum. The 6-inch size is rated at 115 V, 1800 watts, and requires an 800 cc oil charge. The same cartridge-type insert heating elements used in the PMC model are employed in these pumps, and the boiler section is wrapped with a separate quench coil to speed oil cooling.

2. Water-Cooled Baffle - The baffles (BCRU series) consist of a single row of nickel-plated copper chevrons brazed into an outer water-cooled ring of type 304 SS. The baffle is sized to fit inside the bolt circle of the pump and does not have bolt holes. The metal gaskets are sealed by tightening a set of stud bolts which extend from the pump inlet flange through the bottom flange of the zeolite trap.

3. Zeolite Trap - The zeolite traps (TSMU series) are fabricated from type 304 SS with flanges at the top and bottom containing ground-finish

plain faces identical to those of the pump. The internal construction consists of an oil creep barrier at the bottom, two zeolite trays (the top one being removable), and a thin, flat, removable, sheet metal cover which sits approximately 1-1/4 inches above the top tray and forces all gas moving through the trap to pass over it. A 1/4 inch diameter stainless steel needle valve is welded into the trap body below the bottom tray to permit bringing the system to atmospheric pressure without danger of blowing zeolite pellets from the trays into the pump or the system above. A compact corset-type heating mantle, made of spun glass and resistance-heating wire, fits around the outside of the trap for use during bakeout of the zeolite. This mantle is rated at 1240 watts and is capable of producing temperatures in excess of 500°C. A small clip and set screw welded to the outside of the trap body facilitates attachment of a thermocouple to monitor temperature during the bakeout cycle.

4. Metal Gaskets - Ring gaskets of various types of metal are offered with the package. Normally, dead-soft annealed aluminum wire brazed into an "O"-ring is used on the pump outlet flange, between the pump inlet flange and baffle, and between the baffle and the bottom flange of the zeolite trap. The wire is 1100-0 alloy and has a diameter of 0.080 inch. Gaskets of this type can withstand a temperature of 350°C. The seal for the top flange of the trap, which normally becomes much hotter than the bottom seal during bakeout, is made with a fully annealed gold wire ring gasket 0.040 inch in diameter and is capable of withstanding temperatures to 500°C. Spools of indium alloy wire are also available as gasket material. Seals made from this material can be safely heated to temperatures of 125°C without damage. No fusion is required to form a gasket, since the twisted ends will cold-weld together under pressure.

B. N.R.C. Equipment Corporation Ultra-High-Vacuum Components

1. Diffusion Pump - The pumps in this series (HK) are slightly modified versions of the standard N.R.C. HS line. The only change which has been made is the substitution of heavier inlet and outlet flanges which contain both an "O"-ring groove as well as a ground, raised face for metal ring gasket sealing. The anti-backstreaming water-cooled copper cold cap located over the top jet to reduce oil backstreaming is provided as standard equipment on the HK models but is optional on the HS series. The inlet and outlet water-cooling lines for the cap are sealed where they pass through the pump body with small, 1/8 inch cross-section-diameter rubber "O"-rings. The pump body is fabricated from type 300 series SS, and the jet assembly is aluminum. Fractionating distillation is incorporated in the design through a series of raised circular rings in the boiler section of the pump. The heater is a calrod unit embedded in an aluminum casting which bolts to and completely covers the bottom of the boiler. At present the 6-inch size pump heater is rated at 1800 watts, but N.R.C. reports that their tests show better performance with a 2500 watt heater, and all future HK series pumps will be equipped with the higher power element. The boiler section of this pump is also fitted with a separate tubing quench coil to hasten cooling. The oil charge for the 6-inch size pump is only 400 cc.

2. Water-Cooled Baffle - Although N.R.C. provides a choice of either a cryogenic (Series 0315) or a water-cooled (HW model) baffle in their ultra-high-vacuum package, the tests were conducted with the latter type since it is better suited to the facility under design. The top flange of the baffle is machined with both an "O"-ring groove and a wide ground,

raised face. While the inverted dome and cooling-water tubing are non-magnetic, unfortunately N.R.C. fabricates the outside body and top flange of all their baffles from nickel-plated carbon steel. The tests to be described in this report were conducted with a nickel-plated carbon steel baffle.

The extra cross-sectional area gained by drilling and tapping holes into the bottom of the baffle body on the same bolt circle as the pump inlet flange gives the baffle better conductance and results in a higher pumping speed. However, with a stainless steel body and bolts there is danger of the bolts seizing and having to be removed by machining. Hence, the design has a pumping speed advantage but a mechanical disadvantage. The seizing problem can probably be greatly reduced if silicon-bronze bolts are used, or if the threads of stainless bolts are lubricated each time with an anti-seize compound, such as "lead-plate," before they are inserted into the baffle body.

3. Zeolite Trap - The design and dimensions of the top flange and the bottom of the outer body of the N.R.C. zeolite trap (HZ model) are identical with those of the baffle, but type 300 SS is used throughout in the trap fabrication. Thus the same bolt-seizing problem described for the baffle design is present here to an even greater extent since the bottom of the trap will normally become quite warm during the zeolite bakeout cycle. The trap contains three separate trays, none of which are removable. The 6-inch size unit is heated by six 120 V, 500 watt Chromalox strap heaters clamped in pairs around the outside of the body. A cooling tube is attached both directly beneath the upper flange and near the bottom of the trap,

supposedly to be used to prevent overheating the seals in case elastomer type "O"-rings are used. However, all test results which are reported here were obtained using metal gaskets and with no cooling in these tubes.

4. Metal Gaskets - Copper crush "O"-rings were furnished as the metal seals for all locations except the diffusion pump outlet flange and the water-cooled cap inlet and outlet lines. These "O"-rings have a diamond-shaped cross section and are fabricated as integral units from a length of pipe or tubing. The rings were dimensioned to fit into the "O"-ring grooves machined in each component of the system. The groove dimensions are such that standard 1/8 inch cross-section neoprene or Viton "O"-rings may also be used.

III. TEST APPARATUS AND PROCEDURES

A. Diffusion Pump, Baffle, and Zeolite Trap

For the first series of tests the zeolite trap was included in the assembled systems. While experiments of this type would normally be conducted by evacuating a bakeable vessel whose volume was at least twice that of the pumping system, the time and expense required to construct such an apparatus were not taken. Instead a one-inch-thick type 300 SS blank-off plate containing a welded Veeco RG-75 ionization gauge was sealed with a metal gasket directly to the top of the trap. This same plate, ionization gauge, and RG-75A panel were used for testing both units.

A Welch Duo-Seal (Model No. 1397) mechanical pump was employed as the forepump. The foreline of the system consisted of 1-1/2 inch diameter copper tubing, a 1-1/2 inch Veeco angle valve for isolating the forepump

from the system, a 3/4 inch Veeco valve on the diffusion pump side of the 1-1/2 inch valve for roughing down the system, a stainless steel bellows, and a Veeco thermocouple gauge.

On the company's recommendation, both the Consolidated diffusion pump and the Welch Duo-Seal forepump were charged with Convoil-20 pump oil. The N.R.C. diffusion pump used Dow 705 silicon-based fluid, which is relatively new and extremely expensive, and which has a vapor pressure quoted by N.R.C. as being an order of magnitude below that of the best previously available diffusion pump oils. During the N.R.C. test the Welch forepump still contained Convoil-20 oil.

The initial evacuation and zeolite bakeout procedures were identical. To prevent contamination of the diffusion pump oil by backstreaming while the forepump was operating under heavy gas loads, the assemblies were roughed down through a small liquid nitrogen trap and portable pump wagon to approximately 25 microns with the 1-1/2 inch system forepump isolation valve closed. Then the isolation valve was opened, the roughing valve closed, and the trap heaters were energized to rated power. Initial pressure rises in the foreline due to the release of moisture adsorbed on the surface of the zeolite pellets peaked at 150-200 microns. After approximately 1/2 hour, when the pressure had dropped to below 50 microns, the diffusion pumps were turned on at rated power, and the zeolite bakeout was continued for a 24 hour period.

The blank-off plate directly above the trap was simultaneously baked with a calrod-heated insulated box which sat down over the plate and ionization gauge. The temperatures of the trap and plate were monitored separately by iron-constantan thermocouples spot-welded to their surfaces. In both

instances the blank-off plate was baked at 450°C. Since the heating mantle on the Consolidated trap was capable of exceeding the recommended bakeout temperature of 500°C when operated continuously at rated power, it became necessary to control the power input with a variac after 500°C was reached. However, the N.R.C. trap heaters were operated at full rated power continuously, and the insulated thermocouple on the outside trap wall located midway between two of the strap heater bands reached a maximum of only 340°C.

Following the twenty-four hour bakeout the blank-off plate and trap heaters were allowed to cool down to room temperature, and pressure readings were recorded at periodic intervals. In all instances the systems were operated for a minimum of 48 hours after the cooling-down process had been completed. Efforts were made to obtain the optimum base pressures by adjusting the pump heater power over a ± 10 per cent rated range and also by carefully throttling the diffusion pump cooling water flow rates. Also, the N.R.C. diffusion pump 1500 watt heater was operated at 2500 watts during the testing of this system in an effort to gain the additional pumping efficiency. The above-outlined tests were performed several times on both units with practically identical results being observed in each instance.

B. Diffusion Pump and Water Cooling on Baffle

The pump and water-cooled baffle tests were performed by removing the zeolite trap and installing the blank-off plate and ionization gauge immediately above the baffle. The remainder of the test apparatus was identical to that already described with one exception. Since the use of the Dow 705 silicon-based fluid had already produced such satisfactory results in the N.R.C. pump and water-cooled baffle tests, the Consolidated pump

and baffle were washed with trichlorethylene and then with ethyl alcohol, and the pump was charged with 800 cc of Dow 705 for this and following tests. In both cases the baffle cooling-water outlet line was connected in series with the pump inlet line. No noticeable increase could be detected in the temperature of the cooling water flowing through either baffle.

C. Diffusion Pump and Refrigeration on Baffle

To perform this series of tests the baffle cooling supply and return lines were disconnected from the cooling water system, evacuated, and connected to a 1 H.P. Copelamatic refrigeration unit. The refrigeration gas was Freon 502. Otherwise the test setup was the same as that outlined above with water cooling on the baffles. In both instances the diffusion pumps were heated up and pumped for approximately ten minutes to reduce the system pressure to the 10^{-5} torr range before the baffle refrigeration was started. The inlet and outlet temperature of the refrigerant circulating through each baffle was monitored by insulated thermocouples attached to the lines as close to the baffle as possible. The refrigeration lines were insulated but none was placed on either baffle body. However, since the temperatures were in the -35° to -40°C range, a layer of frozen condensation approximately 1-1/2 inches in thickness soon formed on the outside of each of the baffle bodies, and this layer acted as a form of natural insulation.

IV. TEST RESULTS AND OBSERVATIONS

The ultimate base pressures attained by the Consolidated and N.R.C.

pumping assemblies under the various test conditions were as follows:

	CONSOLIDATED CORP.		N.R.C. CORP.	
Test Conditions	Diffusion Pump Fluid	Ultimate Base Press. (Torr)	Diffusion Pump Fluid	Ultimate Base Press. (Torr)
Zeolite Traps Incl. in System	Convoil-20	5×10^{-10}	Dow 705	1×10^{-9}
Traps Removed - H_2O Cooling on Baffle	Dow 705	5.8×10^{-8}	Dow 705	1.3×10^{-9}
Traps Removed - Baffle Refrigerated (-35° to -40°C)	Dow 705	4.5×10^{-9}	Dow 705	1.2×10^{-9}

The base pressures listed above when using the traps in the systems were reached approximately eight hours after cooling to room temperature following the twenty-four hour zeolite bakeout cycle. The pressure of the Consolidated assembly increased to 1×10^{-9} torr after operating overnight, and the N.R.C. unit gradually climbed to 4×10^{-9} during a twenty-eight hour period. Since these pressure increases occurred with no known external gas load being introduced into either system, they probably indicate the rates at which the adsorbing surfaces of the zeolite pellets were gradually becoming saturated. To re-establish the original base pressure, an additional shorter (normally eight hours) reactivation bakeout cycle was necessary. A completely new zeolite charge is recommended following the third heating cycle.

With water cooling on the baffles, both units reached base pressure after approximately twenty-four hours of pump operation. The optimum N.R.C. pump heater power was about 1650 watts, and additional power caused immediate noticeable system pressure increases. Varying the Consolidated pump

heater power by ± 10 per cent from the rated value had very little effect on the base pressure. However, a slight reduction from the 1800 watt value to 1740 watts produced the lowest pressure. Cooling water flow rate adjustments over a wide range had no noticeable effect on either unit.

When the baffles were refrigerated to a temperature between -35° and -40°C, approximately thirty-six to forty-eight hours elapsed before the minimum base pressures were attained. While the Consolidated assembly base pressure did not seem to be affected by pump power adjustments, the N.R.C. unit operated most steadily at 2100 watt heater power, and raising it to the recommended value of 2500 watts caused rapid, very sharp pressure fluctuations extended from 9×10^{-10} to 1.4×10^{-9} torr.

In all cases where the Dow 705 pump fluid was used some form of pressure fluctuations was observed. Their frequency and magnitude could be altered by adjustments in pump power and cooling water flow rates. Although the fluctuations were often reduced to a frequency of only 1 - 2 per minute and to very small magnitudes in the 10^{-9} torr pressure range, they could never be completely eliminated. In many instances the magnitudes of the fluctuations were so slight that they probably would not have been observed had the pumping system been connected to a larger volume instead of being blanked off immediately above the baffle or trap.

The metal gaskets performed very satisfactorily in both assemblies, and after each initial seal was made no leaks were detected prior to or following the zeolite bakeout cycle or when the baffles were refrigerated. However, the bolts compressing the aluminum wire "O"-rings used on the Consolidated assembly were torqued (at the company's recommendation) to much higher values than the N.R.C. copper crush rings. For example, the

7-3/4 inch I. D. aluminum "O"-ring for the 6 inch size pump was compressed by applying 125 ft.-lb of torque to eight 3/4 inch bolts on a 9-1/2 inch diameter bolt circle. This resulted in the aluminum ring being reduced from 0.080 inch in diameter to approximately 0.033 inch in thickness and 0.115 inch in width.

Although N.R.C. furnishes the diamond-shaped cross section crush rings for their ultra-high-vacuum units, they were unable to define the standard bolt torque values which are normally required to form a vacuum-tight seal. Therefore an arbitrary value of 60 ft.-lbs. per bolt was applied to eight bolts of the same size and on the same bolt circle diameter as listed for the Consolidated pump. This created a vacuum-tight seal and flattened the top and bottom sharp edges of the diamond-shaped cross-sectional rings to a width of approximately 1/64 inch. It is quite possible that a leak-tight seal could have been made with a lower bolt torque value, but this point was not investigated further. Thus the aluminum wire formed a sealing surface with a relatively large width, while the seals made using the copper crush rings were of a sharp edge, low-contact area type.

Following each of the various tests the assemblies were dismantled for examination of the component parts. The N.R.C. water-cooled optical baffle was extremely effective in stopping backstreaming from the diffusion pump. While the underside of the inverted baffle dome had a thin coating of pump fluid, none was detectable by visual observations anywhere in the system above this point, neither on the inside of the baffle body, the top of the dome, nor on the blank-off plate.

Both sides of chevrons in the Consolidated baffle, the baffle wall, and the blank-off plate were heavily coated with oil. Also, when the

zeolite trap was in the system, some traces were found on the bottom of the trays. Refrigeration cooling on the baffle did not seem to improve visibly its backstreaming efficiency, although lower system base pressures were obtained than when cooling water was used. The wall of the 3-inch diameter forearm of the Consolidated pump was saturated with oil following each test, while the 1-7/8 inch diameter, baffled N.R.C. pump foreline remained fairly clean at all times.

V. CONCLUSIONS AND RECOMMENDATIONS

With zeolite traps included in the pumping systems the test results show the Consolidated package using Convoil-20 diffusion pump oil is capable of attaining a lower minimum base pressure following the bakeout cycle than the N.R.C. assembly with Dow-705 silicon-based fluid. This is probably due to the inefficiency of the trap heaters of the latter assembly. While their combined power rating is 3000 watts compared to 1240 watts for the equivalent Consolidated heating element, the maximum N.R.C. trap body temperature attained is only 340°C with the units energized at full power rating continuously. The Consolidated heating mantle raises the temperature of the trap body to 500°C within a few hours, and thereafter the power input has to be reduced with a variac to approximately one-half the 1240 watt value to prevent exceeding 500°C.

In addition, the inside of the N.R.C. zeolite trap for convenience should be redesigned to permit removal of the top two trays. The inaccessibility of the lowest one of the three trays makes unloading and charging almost impossible without unbolting the trap from the baffle. Failure to

do this invariably results in zeolite pellets being dropped through the baffle into the diffusion pump, regardless of the degree of care exercised.

With the traps removed from the systems, the N.R.C. pump and baffle performance is preferred to that of the Consolidated unit with both water cooling and refrigeration (-35° to -40°C) on the baffle. The test procedure and equipment in each case was identical, including the use of Dow 705 fluid in both diffusion pumps. While refrigerating the N.R.C. baffle did not alter the base pressure which has been attained with water cooling, the Consolidated base pressure was decreased by better than a factor of ten. This difference is due to the ineffectiveness of the Consolidated baffle in preventing severe pump fluid backstreaming, as is evidenced when the equipment is dismantled for inspection following each test. The Consolidated package construction is such that the bottoms of the zeolite trap trays act as backstreaming barriers. Also, it is suspected that the immersion style heater elements of the diffusion pump cause localized superheating and flashing of the fluid in direct contact with them, thereby causing entrainment of large droplets of oil upward through the jet assembly and into the vacuum system above.

The assumption was made during these tests that the Dow Corning silicon-based 705 fluid was superior in vapor pressure and performance to any other pump fluid commercially available at present, but the test procedures were such that this point was not verified conclusively. While the fluid performed well in the N.R.C. system, it is possible that the Consolidated pump with its immersion type heaters may have attained lower base pressures in the water-cooled and refrigerated baffle tests if it had been operated

with Convoil-20. Also, because of the excessive cost of the 705 fluid at present, more complete comparative testing should be conducted to determine if the base pressures and pumping speeds attainable with it justify its additional expense over more economically priced fluids, especially for use in larger pumps.

Zeolite becomes saturated readily, particularly when exposed to gas at high pressures, and a substantial amount of time is required to complete the bakeout cycle. Therefore, the usefulness of zeolite is primarily restricted to systems which are normally let up to atmospheric pressure infrequently, and also those which have relatively small external gas loads being introduced during experimentation.

Since the N.R.C. pump using 705 fluid and the water-cooled baffle are capable of readily attaining a base pressure of 1×10^{-9} torr, the decision was made to use this type of system on the Crossed-Beam Facility. These pumps along with cryogenic-Ti gettering pumping should result in the desirable operating pressure.

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