NOTES

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Partial rejuvenation of Bayard–Alpert ionization gauge tubes

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We describe a simple procedure for the partial restoration of Bayard–Alpert ionization gauge tubes that have been contaminated by a combination of mechanical and silicone diffusion pump oil vapors. The method consists in filling the tube with 1 M NaOH, immersing it in a hot water bath (>80 °C, ca. 1 h), thorough rinsing with deionized water and reagent acetone, and air drying. This procedure was tested on nine inoperable gauges; after restoration seven of the gauges read within a factor of 2 of a new gauge. © *1997 American Institute of Physics.* [S0034-6748(97)00104-4]

Difficulty with the operation of Bayard–Alpert ionization gauges[1] in diffusion-pumped vacuum systems employing silicone oil pump fluid has been known for many years.[2] The main modes of gauge tube failure are anomalously low pressure readings (providing a false sense of security to students operating the vacuum system) and, with extensive contamination, failure to obtain the required emission current. With the feedback regulated circuits employed in modern controllers, the tube then fails to light, and must be replaced (providing a true sense of frustration at the extra down time). The former failure is due to the buildup of insulating deposits on the collector wire, and the latter to deposits on the grid. Contaminated tubes also generally display a heavy brown, semi-opaque coating on the glass envelope. In the days of generous funding for scientific research, replacing these tubes was merely a nuisance, but now the expense must also be considered.

Over the years our research group has amassed a large collection of spent tubes, on which several cleaning strategies have been tried and found wanting. The procedure to be described here has been carried out on ca. 20 tubes (Granville–Phillips model 274002, Nonex glass envelope, thoriated iridium filament, tungsten collector, unspecified refractory metal grid, and Pyrex tubulation, operated by Granville–Phillips 260 series controllers) using equipment and chemicals common to most small laboratories. All of the tubes had gone through both stages of failure in vacuum systems pumped by Varian diffusion pumps charged with Dow–Corning DC-704 and DC-705 silicone oils, both in trapped and untrapped chambers.

Tubes containing liquid oil (common for a horizontal vacuum connection) are first drained and rinsed with acetone. The tube is clamped by its neck, open end up, the envelope immersed completely in a deionized water bath (preheating is permissible but not necessary), and filled (ca. 250 mL) with 1 M NaOH aqueous solution (40 g NaOH pellets dissolved in deionized water and diluted to 1 L). 2 M NaOH was not found to be sensibly more effective. The bath is heated to >80 °C on a hot plate, and the tube allowed to remain immersed for 1 h or more, depending on the extent of contamination. In observing several tubes as they were heated through the 80 °C mark, we noted the coating on the glass began to lift away at the temperature threshold, gradually accumulating at the top. Convection within the tube provides adequate stirring; ultrasonic agitation is not recommended, as it breaks the filament spot-welds. The base solution expands considerably in going from room temperature to 80 °C; a disposable pipet is used to withdraw solution from the tube if necessary, preventing contamination of the bath. Heating is essential to the cleaning operation; soaking tubes overnight with room temperature NaOH is ineffective. We used a 170×90 mm crystallizing dish and a Corning model PC-320 heater-stirrer for the water bath.

When it appears that deposits have been removed or loosened, the tube is removed from the bath, allowed to cool for ca. 10 min, and the contaminated base solution is then poured out and discarded. The tube is rinsed with 4 ca. 50 mL portions of deionized water (stoppering and shaking thoroughly each time) followed by 2 ca. 25 mL rinses with reagent grade acetone, and thorough draining. The cleaned tubes are allowed to air dry for ca. 24 h, then capped and stored.

In every tube, deposits on the glass walls and the collector wire were nearly completely removed to the eye, while some deposit tended to remain on the grid in most cases. Cleaner grids were obtained in tubes of earliest manufacturing date. A few tubes showed slight etching of the Nonex envelope. Breaking open a tube to expose a contaminated grid revealed that the remaining grid deposit was loosely adhering and was easily rubbed off.

In order to assess the viability of the cleaned tubes, a three-port tube adapter was installed on a nozzle chamber in our crossed beams apparatus. This chamber has a volume of 30 L, and is pumped by an untrapped Varian VHS-6 diffusion pump equipped with a ''Mexican hat'' cold cap and charged with DC-704 oil. The backing pump is a copper-

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TABLE I. Test results for partially rejuvenated gauges.[a]

Gauge No.	P_1 Torr ($\times 10^{-6}$)	P_2 Torr ($\times 10^{-4}$)	Gauge	P_1 Torr ($\times 10^{-6}$)	P_2 Torr ($\times 10^{-4}$)
New	1.2	1.5	5	4.2	⋯
1	1.2	1.5	6	1.1	1.4
2[b]	1.7	2.7	7[b]	2.2	⋯
3[c]	(0.3)	⋯	8[b]	1.7	⋯
4[b]	1.2	2.0	9	1.8	⋯

[a]P_1 is no-load pressure, P_2 with helium nozzle on, uncorrected.
[b]Grid still visibly contaminated after cleaning.
[c]Operated at 0.2 mA emission current; tube would not light at 1 mA.

gauze-trapped Sargent-Welch 1397B rotary pump with Duo-Seal oil. After one day of pumping from atmosphere, this chamber reaches 1.2×10^{-6} Torr, with an eventual base pressure of 3×10^{-7}. For each test run, a ''new'' gauge—one well burned in, but still operating reliably—was compared with one or two cleaned gauges, using the same controller unit, with one day's pumping. Each cleaned gauge was allowed to degas for 15–30 min, with a total warmup time of 1–2 h. Slight run-to-run variations in ''true'' pressure were renormalized to 1.2×10^{-6} Torr using ratios. In a few cases helium was admitted to a nozzle beam source housed in the chamber, testing response to a gas load. Table I presents results for nine cleaned gauges. All but one started and degassed readily, and yielded pressure readings that were generally high, but again with one exception well within a factor of 2 of that from the new gauge. Operation of one of these tubes for a more extended period gradually improved its accuracy. Several renewed tubes have now been called back into service in our vacuum systems. One of these tubes has shown signs of depressed pressure readings after six months of nearly continuous operation in an untrapped chamber, a shorter time to failure than for a newly installed tube, but the others, with comparable length of service, are still functional.

It is clear that these tubes are only partially rejuvenated, and need to be tested for accuracy. They are generally operable, and certainly suffice for many of our purposes, for which reproducibility is more important than absolute accuracy.

Presuming that some of the Si–O–Si siloxane bonds of the polymeric silicone oils remain when deposits are formed, breakdown and cleanup is likely to occur by nucleophilic attack of OH^- on Si, ultimately forming water soluble hydroxyl compounds and alkyl silicate salts.[3] Lifted brown deposits that accidentally reached the water bath were observed eventually to dissolve in it.

Finally, we mention briefly a few cleaning methods that failed: ultrasonic cleaning with commercial cleaning solution; soaking in various detergents; chromic-sulfuric cleaning solution at 90 °C; 0.1 M HF; jarring and scratching with various rods and tools.

[1]R. T. Bayard and D. A. Alpert, Rev. Sci. Instrum. **21**, 571 (1950).
[2]F. Rosebury, *Handbook of Electron Tube and Vacuum Techniques* (Addison-Wesley, Reading, MA, 1965), p. 401.
[3]R. R. McGregor, *Silicones and Their Uses* (McGraw-Hill, New York, 1954), p. 260.