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Retractable capillary doser

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A retractable capillary doser is described which can place a reproducible coverage of molecules on a substrate in an ultrahigh-vacuum environment. An integral valve assembly controls the flow of molecules from the doser volume to the substrate. Automatic operation is achieved by controlling the valve and the position of the doser orifice from a commercial IEEE interface.

In surface physics experiments it is often desirable to place a reproducible quantity of a gas phase species onto a solid substrate while maintaining high-vacuum conditions in its vicinity. This can be accomplished by expanding a fixed volume of the gas (at a fixed pressure) into a vacuum chamber through a capillary tube directed at the substrate. If the orifice of the capillary tube is placed a fixed distance from the substrate, and if the substrate remains in the path of the emerging gas for a precise length of time, a highly reproducible surface coverage can be obtained. This principle forms the basis of an automated capillary doser which we designed in order to condense a precisely controlled quantity of benzene on to a cryogenically cooled field-emitter tip in the field-ion tomographic microscope. Image reproducibility and isothermal field-desorption spectra² suggest that successive depositions can be controlled to within the thickness of a few condensed monolayers.

The capillary doser is shown schematically in Fig. 1. A cryoshield completely surrounds our specimen except for a small aperture through which the dosing gas and imaging beam must pass. The cryoshield is kept at 50 K, well below the condensation temperature of benzene so that the field-emitter tip will be completely shielded until the capillary tube is fully extended and its orifice positioned directly in front of the aperture.

A valve, placed at the base of the capillary tube, shuts off the supply of benzene when the capillary tube is fully retracted. The valve seals by pressing a stainless-steel knife edge into an annealed gold seat. A sealing pressure of ~ 70 lbs/ in² is provided by two small, pneumatic air cylinders whose stroke determines the total distance traveled by the capillary tube. The two air cylinders are connected to a common manifold and pressurized. They are controlled by a so-

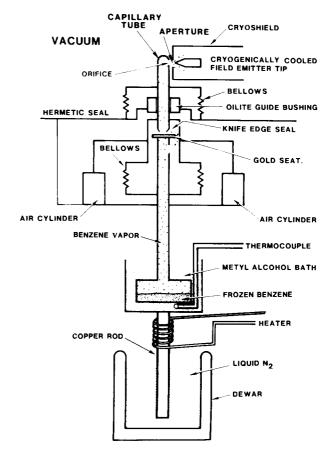


FIG. 1. A schematic view of a typical, capillary doser assembly.

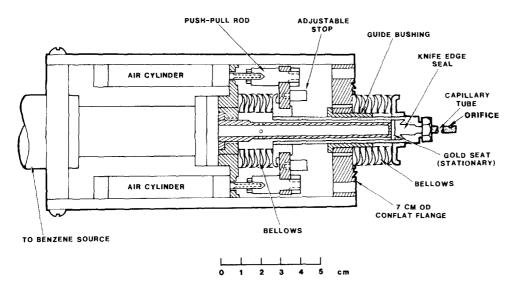


FIG. 2. A cross-sectional view of the automated capillary doser described in the text.

lenoid valve that is energized by an ac current which flows through a relay remotely controlled via an IEEE-488 bus.³

As the pneumatic cylinders are energized, the valve opens and the orifice in the capillary tube moves toward the aperture in the cryoshield. During transit, gas begins to emerge from the orifice. By the time the orifice is positioned opposite the aperture, the flow of emerging gas has reached its equilibrium value. It then condenses directly onto the emitter tip until the air pressure to the pneumatic cylinders is

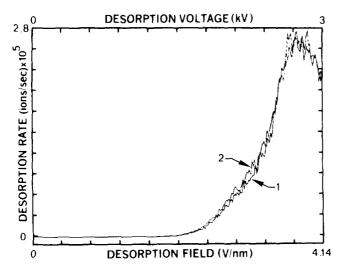


FIG. 3. Ramp desorption spectra of benzene removed from a field-emitter tip coated with ferritin molecules (see Ref. 1). Spectrum 1 and 2 were recorded on different days and demonstrate the reproducibility of the capillary dosing system described in the text.

removed. The capillary tube retracts, the valve closes and seals, and any residual gas emerging from the capillary orifice condenses onto the cryoshield or is pumped away.

The doser volume is large enough to ensure that its pressure will remain essentially constant during a maximum 10 s deposition cycle. The pressure of gas within the doser volume is determined by the temperature of a benzene reservoir connected to it. The reservoir temperature is controlled to within one degree kelvin by a liquid-nitrogen bath and the heat from a 70-W electrical filament whose actual power is determined by a proportional temperature controller which monitors the bath temperature. Figure 2 shows the capillary doser in greater detail.

Figure 3 shows two isothermal field-desorption spectra of benzene condensed onto our substrate using the doser shown in Fig. 2.² The area under each curve is proportional to the total amount of benzene condensed onto the specimen. The reproducibility of the spectra suggests that successive deposition conditions are reproduced to within 1% or approximately the thickness of a single benzene layer.

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