

UHV5[™]

ULTRA-HIGH VACUUM COMPATIBLE ELECTROSPRAY DEPOSITION SOURCE

Modular electrospray deposition source capable of producing a molecular beam from an electrosprayed solution at pressures typically in the 10⁻⁷ mbar range (typically down to 10⁻⁸ mbar with an additional UHVX pumping stage). Suitable for the deposition of all molecules and nanoparticles that can be held in a stable electrospray compatible solution e.g carbon nanotubes, fullerenes, single molecule magnets, organometallic dye complexes, proteins, giant porphyrin nanorings, polymers and biomolecules. The UH5 features an inert gas environment electrospray chamber for reduced atmospheric contamination and the containment of both solvent and solute.



KEY FEATURES

- Inert gas electrospray interface
- Containment of solvent and solute
- In-situ emitter positioning
- Heatable desolvation entrance capillary
- Precision skimmers and exit aperture
- 3 differential pumping stages
- Reusable precision electrospray emitter
- Upgradable modular design

UHV-COMPATIBLE ELECTROSPRAY DEPOSITION

The interaction of atoms and molecules with pristine surface in ultra-high vacuum (UHV) has provided incredible insights into surfaces and interfaces over the years, from catalysis to bioscience. Exposure of a single crystal surface to gases or thermally evaporated molecules is a staple of the field, but when molecules become so complex that they are thermally labile or completely non-volatile, electrospray deposition can often overcome these challenges. As a nonthermal technique, electrospray forms a plume of molecules from solution. This takes place under inert gas conditions in the UHV5 by passing the solution to a precision stainless steel emitter held at a high voltage (typically 2kV) positioned in front of narrow capillary interface inside an environmental chamber. The source then extract that plume into vacuum and transport the molecules through differentially pumped skimmers and apertures to impinge on a surface held in UHV for scanning probe microscopy, electron spectroscopy and thin film deposition. The contained electrospray interface allows control over the inert gas environment around the emitter reducing atmospheric contamination at the sample, and provides containment of both the solvent vapour and the nebulised solute.

INSTALLATION & PUMPING

To mount the UHV5 electrospray deposition source all you need on your existing UHV system is a free DN35CF port facing the sample position. It is best to mount the source on a gate valve so that it can be vented when not in use without affecting your main system. The UHV5 core unit has 3 differential pumping stages. Stages 1 and 2 require roughing pumps (>6m³/hr⁻), while stage 3 requires a turbomolecular pump (>77 l/s). Stage 2 can usually be pumped by the backing pump of the stage 3 turbo so in total you will need 2 backing pumps and 1 small turbo. Depending on the pumping arrangement of your UHV chamber, this will lead to pressures in the 10⁻⁶ to 10⁻⁷ mbar, typically during the deposition. The UHV5 can be mounted in any orientation. The inert gas electrospray chamber (Stage 0) requires a gas feed and exhaust line for full operation, connected to the gas inlet and outlet ports on the front flange of the UHV5.

DEPOSITION CONDITIONS

Electrospray ionisation works best with polar solvents such as methanol and water. Molecules that require organic solvents such as toluene can still be deposited as long as the solution can tolerate the addition of some polar solvent (e.g toluene with

10% methanol). A successful combination of solvents can usually be found for most molecules.

The solution is held in a syringe, typically driven by a syringe pump at a flow rate on the order of 0.1 ml/hr) to the liquid inlet port on the front flange. The emitter voltage (connected to the emitter through a feedthrough on the front flange) is increased until the liquid at the tip is drawn into a 'Taylor-cone' by the electric field. At the tip of the cone, a jet is emitted, which breaks up into tiny, charged droplets, which undergo a cascade of Coulomb fission events to ever smaller droplets as the solvent evaporates. The result is a beam of charge nanodroplets and molecular ions. Because the molecules can be carried by the nanodroplets, even macromolecules such as polymers can be successfully deposited by electrospray onto a surface in a UHV chamber. The resulting spot-size at the sample depends on the distance from the mounting flange but is typically around 2 mm.

UGRADES & ACCESSORIES

Molecularspray offers a range of additional components and accessories, in no particular order:

- UHVX additional differential pumping stage
- Electrospray emitters
- MV5 portable high-voltage electrospray power supply
- HIU heated inlet
- SP1 syringe pump
- EV3 visualiser
- CP1 coupling tee and bellows



UHVX Additional differential pumping stage

By providing an additional turbo-pumped aperture, the UHVX reduces the pressure at the surface during the deposition. The final aperture is matched to the beam size to reduce losses in flux. The standard UHVX is a DN35CF tee (also available as a cross on request for customers that want to install a pressure gauge on this stage or beam current monitor).

Electrospray emitters

Our electrospray sources come with one NewObjective® stainless steel TaperTip® which can be reused for a lifetime unless it suffers accidental damage such as bending, blunting or becoming irreversibly blocked. Molecularspray holds replacements of these critical components in stock in case you need to purchase a replacement.



MV5 portable high voltage electrospray power supply

Electrospray requires a bias to be applied to the emitter. Our MV5 power supply is a variable, portable DC high voltage power supply from 0 to 5kV with positive polarity as standard (negative polarity and different maximum voltages available on request). The MV5 comes with a high voltage cable with a connector matched to the electrospray source.



HIU Heated inlet upgrade

In some cases it can be beneficial to heat the entrance capillary of the electrospray source to aid desolvation and to reduce condensation of solvent at the entrance. We offer a heated inlet upgrade at the time of manufacture incorporating a PID-controlled heater chip factory set to maintain the entrance capillary at a constant temperate of 100 °C.

SP1 Syringe pump

Maintaining a constant flow rate of the solution to the emitter is critical to maintaining a stable electrospray. The SP1 syringe pump accepts the standard 10 ml gas-tight syringe provided with your electrospray in addition to a wide variety of syringe size both larger and smaller, offering controllable flow rates as low as 0.01 ml/hr to upwards of 10 ml/hr (typical optimal flow rate for standard configurations is 0.1-0.3 ml/hr).

CP1 Coupling tee and bellows

For sharing one roughing pump between Stage 2 and the backing line of the Stage 3 turbo pump.



EV3 Visualiser

The mode of electrospray is determined by balancing the voltage and flow rate. The optimal condition is a cone-jet mode in which the liquid is drawn into a Taylor cone and a jet is emitted from the tip of the cone which expands into a plume. Below this threshold the electrospray will be characterised by large droplets and incomplete ionisation, while above the optimum voltage multijet mode will result in several jets being emitted offaxis. To help maintain the condition of the spray it is useful to visually monitor it during operation. The EV3 visualiser comprises a 25X microscope that can be operated with or without the included HD video camera and mounts directly along with its light source to the inert gas electrospray chamber.

APPLICATION NOTES

A wide range of molecules and nanoparticles have been deposited using our UHV-compatible electrospray deposition sources including but not limited to: triple stranded porphyrin nanobelts, porphyrins, graphene nanoribbons, nanorings, fullerenes, single molecule magnets, organometallic dye complexes, carbon nanotubes, polymers, and functionally active proteins (see cited papers at www.molecularspray.co.uk)

Electrospray compatible solvents include Acetonitrile (CH_3CN); dichloromethane (CH_2Cl_2) if mixed with methanol; dichloroethane (CH_2ClCH_2Cl); tetrahydrofuran; ethanol (CH_3CH_2OH); propanol ($CH_3CH_2CH_2OH$); methanol (CH_3OH); nitromethane (CH_3NO_2); toluene ($C_6H_5CH_3$) if mixed with methanol or acetonitrile; water and several other combinations.

ORDERING & ENQUIRIES

For order enquiries or to obtain a quotation contact sales@molecularspray.co.uk