Instruction Manual for Installing the BI-MwA

Molecular Weight Analyzer using Static Light Scattering

BI-MwA

Molecular Weight Analyzer

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Please Read

This is your instruction manual for installing your Brookhaven BI-MwA Molecular Weight Analyzer. Please read it carefully before making measurements. In Section I: Introduction, you will find a brief description of software that is used with the BI-MwA. Each software package has its own manual, except the BI-TAS used for installation and test. The part of BI-MwATASw used for installation is extremely simple. See Section VIII.

Instruments undergo periodic improvements. There are always additions and changes. As these become available, they will be added to the back of this manual as appendices. Please look at the appendices if you cannot find the answer to your questions in the main part.

Please contact the factory at info@brookhaveninstruments.com if you have questions. Please refer to the instrument model number (BI-MwA), the serial number, and date of manufacture as indi-cated on the identification sticker affixed to the instrument. Please also indicate from whom the instrument was purchased.

Remember the old saying: "When in doubt, read the instruction manual." Sometimes the solution to your problem has already been addressed. You just need to find it. Thanks for purchasing a Brookhaven.

Important Warning

Never allow any liquid to dry inside the instrument. Always flush the solute or polymer with solvent. If you are not going to work with the instrument for more than a day, **AND** you are working with a solvent in which bacteria may grow, flush the instrument with one or more compatible solvents. Flush with one or more intermediate solvents until you can replace the final liquid with ethanol or some other alcohol in which bacteria will not grow. Then cap the inlet and the outlet ports with the plastic DELRIN plugs that were included when we shipped the instrument to you.

If a blockage occurs due to dried salt or polymer, or if the instrument was left with water and bacterial growth is suspected, refer to Appendix A of this manual.

For a list of compatible solvents, see Section III of this manual.

Copyright Notice

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Catalogue Number: BI-MwA, Ver. 2.01

Software License Agreement

Warranty

Brookhaven Instruments Corporation (hereinafter known as BIC) warrants that the product is free from defective material and workmanship. Under the terms of this warranty, BIC agrees to correct by repair, or at BIC's election, by replacement any parts that prove to be defective through no fault of the user.

This warranty is limited to the original purchaser of the product.

The product shall be shipped, freight prepaid and insured in full, or delivered to a facility authorized by BIC to render the service provided hereunder, in either the original package or a similar package affording an equal degree of protection. The purchaser must contact BIC for instructions before returning the product.

The product shall not have been previously altered, repaired or serviced by anyone other than a service facility authorized by BIC. The product shall not have been subjected to accident, misuse, or abuse, or operated contrary to the instructions contained in the operating manual or manuals.

BIC shall not be liable for direct, indirect, incidental, consequential, or other types of damages, resulting from the use of this product other than the liability stated above. These warranties are in lieu of all other warranties, expressed or implied, including, but not limited to, the implied warranties of merchantability or fitness for a particular purpose.

The BIC warranty extends for a period of 90 days. This period begins from the date of receipt of the equipment, and it applies only to the original purchaser. The warranty period is automatically extended to 1 year (except as noted below) from the date of receipt of the equipment provided all invoices for said equipment, including transportation charges if applicable, are paid within 30 days after receipt of invoice.

The BIC warranty does not cover damage to any part of the cell if the wrong solvent is used. See the WARNING on Page *ii* and repeated in Section III of this manual.

Table of Contents

Section I: Introduction

The BI-MwA is a seven-angle, static light scattering instrument used to determine properties of polymers in solution. Use the following software with the BI-MwA:

- BI-MwATASW, during installation, to test instrument functionality.
- BI-ZPMwA, in batch-mode, to determine $M_{w,}$ R_g, and A_2.
- BI-ACOMP, in flow-mode, to determine M_w, and R_g.
- Compact WinGPC, in flow-mode, to determine M_w, and R_g.

The BI-MwATASW software is included with every BI-MwA. Use it during installation to confirm that the instrument is functional. See Section VIII of this manual for instructions.

Using the batch-mode (BI-ZPMwA) requires manual injection of solutions, a syringe pump (BI-SPump), or automated injection using an HPLC pump, autodilution, and a differential refractometer. [Contact the factory for more information on the automated configuration.] Weight-average molecular weights, M_w, from a few hundred to over a billion can be determined. Z-average radii of gyration, R_g, from about 10 nm to a few hundred nanometers can be determined. The second virial coefficient, A_2, can also be determined.

ACOMP (Automatic Continuous Online Monitoring of Polymerization), also known as TDSLS (Time-Dependent Static Light Scattering), is a technique for monitoring polymer solution properties *during the polymerization/depolymerization process*. These properties include M_w, R_g, $[\eta]$ intrinsic viscosity, and monomer conversion. The second virial coefficient cannot be determined using ACOMP; however, if A_2 is known, or a reasonable estimate for it is known, the value can be used to correct M_w and R_g.

Finally, Compact WinGPC, allows one to control a GPC (Gel Permeation Chromatography) system with additional detectors such as RI, UV, and viscometers for the analysis of molecular weight distribution (MWD). Also known as SEC (Size Exclusion Chromatography), GPC is the preferred method for determination of MWDs (Molecular Weight Distributions). By using the BI-MwA, M_w (and other molecular weight averages) and $R_{g,}$ independent of column standards, can be determined. This lifts the requirement for standards that interact with the column in the same way as the unknown polymer. In this way, M_w and R_g are more accurate. The second virial coefficient cannot be determined using GPC/SEC; however, if A_2 is known, or a reasonable estimate for it is known, the value can be used to correct M_w and R_g. Yet, in most cases, the concentrations used in GPC/SEC are so low that no correction is even necessary.

When using any of these software packages, except for the BI-MwATASW, please refer to the appropriate software manual included with your order.

Safety

The BI-MwA is a Class I laser product. As such, there are no laser beams available to the user. The user should not open the instrument. Only a trained technician should do so. Figure I-1 shows the position of the warning labels.

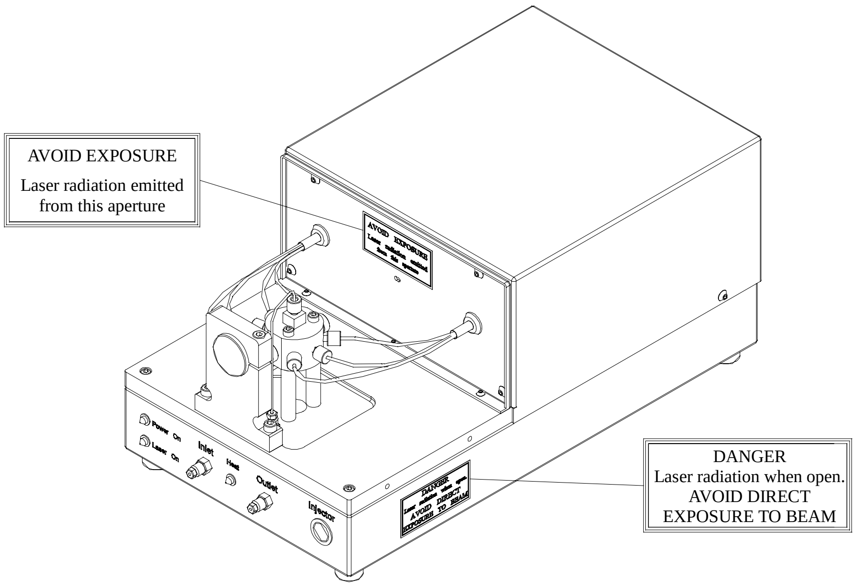

Figure I-1: Warning labels and positions.

Section II: General Specifications

The front of the instrument is shown in Figure II-1.

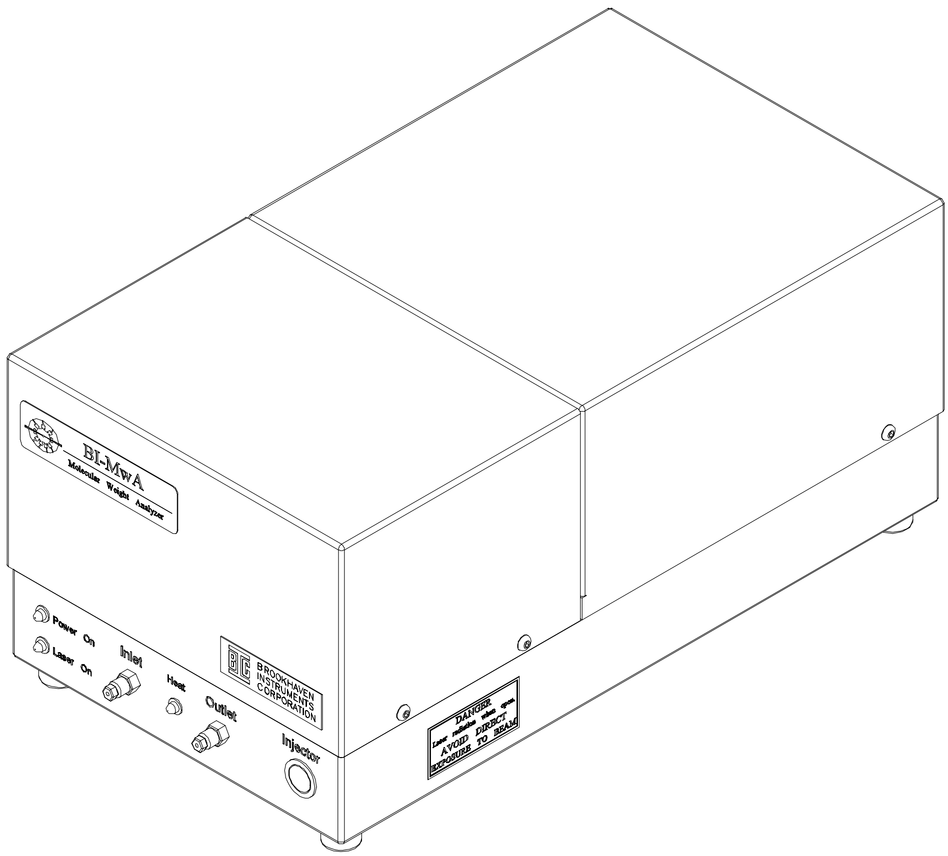

Figure II-1: Front of BI-MwA showing Power On (green), Laser On (red), Heating Status (amber) LEDS; Inlet/Outlet Ports; and Manual Injector button.

The green LED is on when the instrument is plugged into a power source. The red LED is on, indicating the laser is on, when the software is running. The amber LED indicating heater status has three states: off means not heating; continuously on means heating; blinking means the optional BI-MwATC temperature controller is not enabled.

Location of Auxiliary Inputs

Auxiliary inputs are normally analog signals coming from RI, UV/VIS, viscometry, and osmometry instruments. An injector input is also provided.

The auxiliary signal inputs are attached to a small, printed circuit board that is internal to the BI-MwA. The board is accessible via the rear of the instrument. See Figure II-2. A magnetically secured door is easily pulled down to reveal the board.

The details for these auxiliary inputs are described in Sections V and VI of this manual.

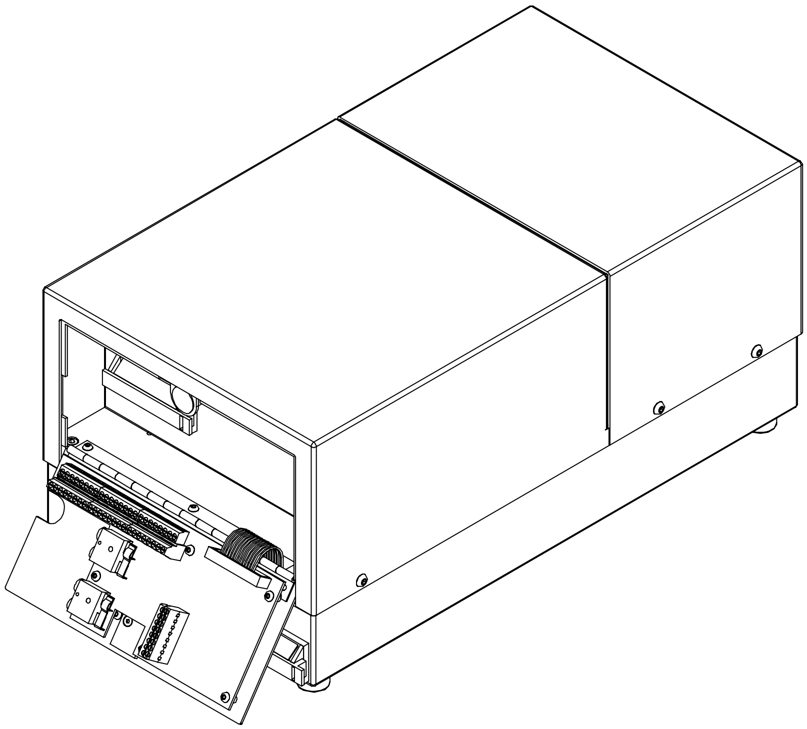

Figure II-2: Rear-view showing access to auxiliary input board.

Two Compartments of BI-MwA Defined

The BI-MwA design separates the electronics/laser part from the cell/liquid part of the instrument. In this way, should there ever be a leak, the electronics are fully protected. The electronics and laser are in the rear compartment, and the cell and tubing are in the front compartment of the instrument.

WARNING: Do not open either compartment. During operation, it is not necessary for the user to open either compartment.

Cell/Liquid Compartment

See Figure II-3. Scattered light intensity at seven angles is simultaneously measured. Flow is from bottom-to-top to facilitate the rise of bubbles up and out of the cell chamber. The chamber volume is nominally 100 μL. The scattering volume is nominally 20 nL.

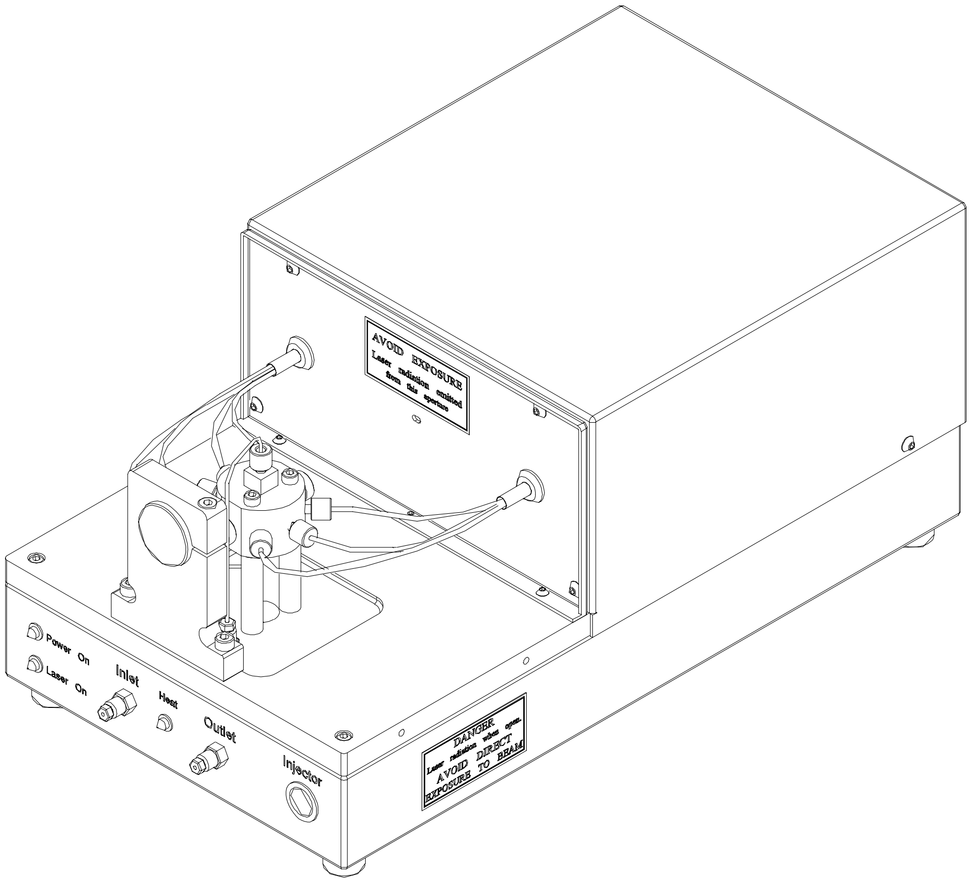

Figure II-3: Cell/liquid compartment, F-configuration.

The stainless steel tubing between the inlet port and the bottom of the cell, and between the top of the cell and the outlet port is standard 0.010” I.D. (0.25 mm) and 1/16” O.D. (1.59 mm). Each of these SS tubes is 25 cm long. The volume for tubing of this I.D. is 0.5 μL/cm. Thus, the volume for both the inlet and outlet is 12.5 μL each.

For BI-MwA instruments dedicated to batch-mode operation, 0.020” I.D. (0.50 mm) tubing can be provided. This makes it easier to manually inject viscous polymer solutions. Please contact the factory if you would like this option installed. In this case, the tubing volumes are increased by a factor of four.

If you are working with samples that should not come in contact with stainless steel, the tubing can be changed to PEEK poly(etheretherketone) or to some other type. Please contact the factory.

Electronics/Laser Compartment

The 635 nm wavelength, solid-state laser is a highly stabilized laser. Internally, the laser power is monitored and the output is used as a feedback to stabilize the laser. The feedback signal is used to normalize the signals from the seven scattering angles. In this way, residual, minor drifts in laser power are compensated for.

The fibers from the two sides of the sample cell are routed through the wall separating the two compartments. They are fed through a holder to the CCD. The CCD detector is mounted below the holder and is part of the electronic board that controls the entire instrument.

Figure II-4 shows the arrangement of the scattering angles. This is a cross-sectional view looking down on the scattering cell. Flow is from the bottom of the cell to the top, perpendicular to this cross-sectional view. The small circle in the middle of the drawing is the liquid-containing part of the cell assembly. This is the scattering chamber.

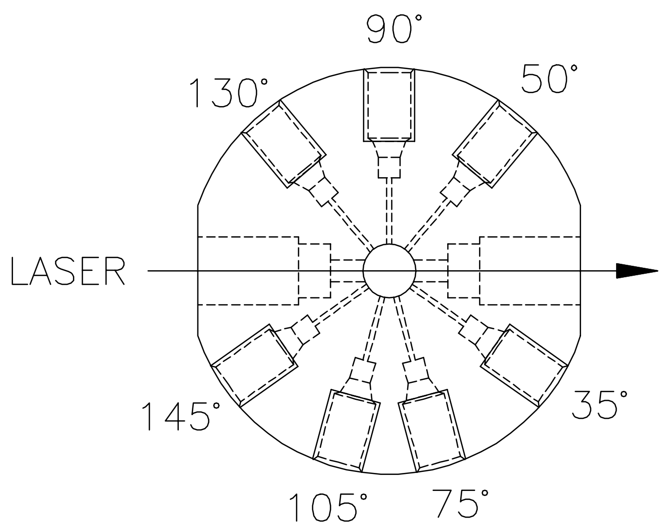

Figure II-4: Scattering angles in standard BI-MwA.

Figure II-5 shows the underside of both compartments. The right side shows the underside of the electronics/laser compartment; the left side shows the underside of the cell/liquid compartment.

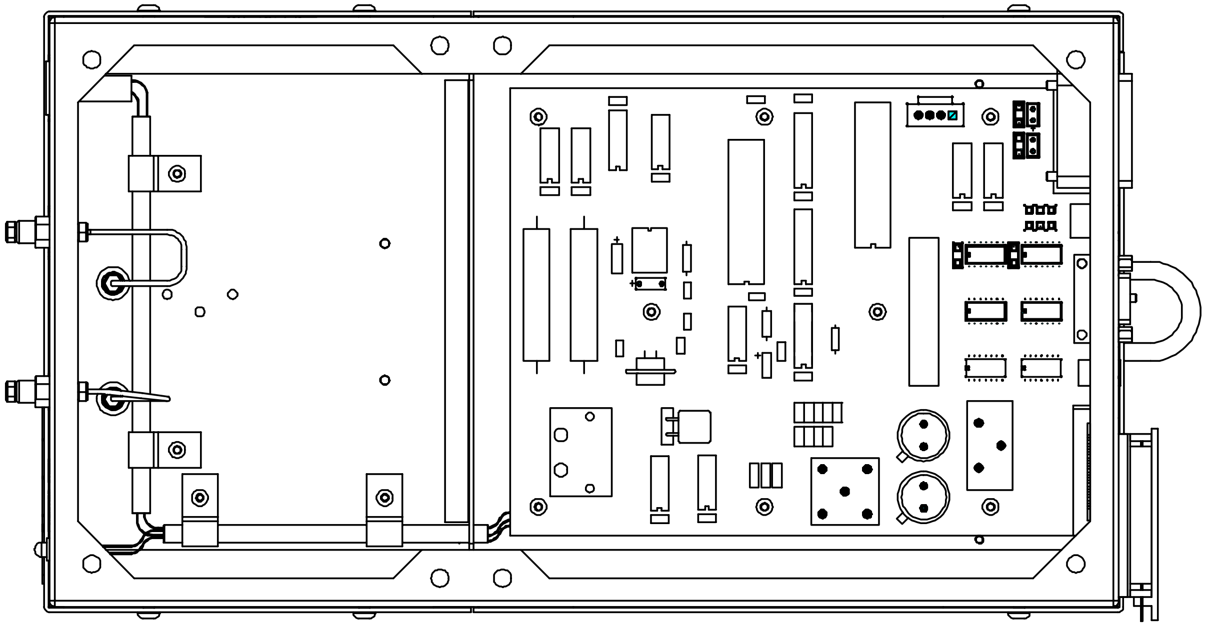

Figure II-5: Electronics/laser compartment (right) and cell/tubing compartment (left).

General Specifications

Molecular Weight Range: <10^3 to >10^9 Daltons, depending on dn/dc and concentration. [High end for dendrimers and other compact structures.]

Cell/Optics

Angles: 7, nominally 35, 50, 75, 90, 105, 130, and 145.
Fiber: Low numerical aperture, integral to cell.
Reference: Internal laser intensity monitor.
Back Pressure: 3.5 MPa (500 psi) maximum.
Fittings: Standard HPLC Inlet/Outlet on front panel.
Volume: Cell, 100 μL nominal; Scattering, 20 nL nominal.
Laser: Nominal 35 mW, 635 nm, vertically polarized, precision power control and active temperature stabilization.
Cell: PEEK, poly(etheretherketone), options on request.
Tubing: Inlet to cell, 25 cm, 0.010" I.D. 316 stainless steel, Outlet to cell, 25 cm, 0.010" I.D. 316 stainless steel. Options available such as 0.020" I.D. for viscous polymer solutions in batch mode and PEEK tubing for materials that should not contact stainless steel.

Electronics/Detector

Control: Integrated, dedicated, powerful microcontroller.
Detector: CCD, ultra-high sensitivity and spatial uniformity.
Monitor: Processor core temperature readout. Tubing temperature readout.
Inputs: 4 standard, with 16 or 24 bit resolution, 15 additional analog channels (optional) with 24 bit resolution. Computer selectable gain adjustment. Suitable for use with most common RI, UV, viscometer, thermocouple, thermistor, injector, pressure gauge and pump outputs.

Power Requirements

100/115/220/240 VAC, 50/60 Hz, 25 Watts

Dimensions

Size: 175(H) x 210(W) x 380(D) mm
Weight: 5.5 kg

Options

Software

BI-ZPMwA: Zimm, Berry, Debye Plot software for use in batch-mode measurements.
BI-ACOMP: Software for automatic continuous on-line monitoring of polymerization reactions.
Compact WinGPC: Software for GPC/SEC measurements of MWD.

Hardware

BI-MwATC: Temperature controller. Room temperature to 80 ˚C, stability ± 0.030 ˚C.
BI-MwAXB: Expansion of 15 additional, 24-bit, analog signals
BI-DNDC: Differential Refractometer (RI detector).
BI-ViSC: Viscometer for monitoring polymer solutions in ACOMP.
BI-SPump: Syringe pump for flushing and cleaning BI-MwA.

The BI-MwA is protected by US Patent #6,052,184.

Section III: Solvent Compatibility

Cells

The BI-MwA is supplied with a PEEK cell. PEEK stands for poly(etheretherketone). Depending on the solvent, this polymer is stable to approximately 200 ˚C and to most, common solvents. See the Table III-1 for specific compatibilities.

Earlier versions of the BI-MwA were manufactured with a different cell material. The manufacturing I.D. label, after the model name, indicates which type of cell you have. BI-MwA/PEEK is most common. The I.D. label is located on the right side of the instrument near the rear.

The only materials in contact with the solvent are PEEK, stainless steel 316 tubing*, the Teflon gasket material (top and bottom of cell) and glass from the entrance/exit windows and the fibers.

Table III-1: CHEMICAL RESISTANCE OF PEEK

SEE KEY AT END OF LIST FOR DESCRIPTION OF LETTERS A, B, C

CHEMICAL	73°F (23°C)	212°F (100°C)	392°F (200°C)
ACIDS			
Acetic Acid, 10% Conc.	A	A	
Acetic Acid, Conc.	A	A	A
Acetic Acid, Glacial	A	A	
Acrylic Acid	A	A	
Aqua Regia	C	C	C
Benzene Sulfonic Acid	C		
Benzoic Acid	A	A	
Boric Acid	A	A	
Carbolic Acid	A		
Carbonic Acid	A	A	
Chloroacetic Acid	A	A	
Chlorosulfonic Acid	C	C	C
Chromic Acid, 40% Conc.	A		
Chromic Acid, Conc.	C	C	C
Citric Acid	A	A	
Formic Acid	B	B	
Hydrobromic Acid	C	C	C
Hydrochloric Acid, 10% Conc.	A	A	
Hydrochloric Acid, Conc.	A	B	
Hydrocyanic Acid	A	A	
Hydrofluoric Acid, 40% Conc.	C	C	C
Lactic Acid	A	A	
Maleic Acid	A	A	
Nitric Acid, 10% Conc.	A	A	
Nitric Acid, 30% Conc.	B		
Nitric Acid, 50% Conc.	C	C	C

* If stainless steel is inappropriate for your materials, PEEK tubing is an option. PEEK tubing is often used in LC/HPLC for biomaterials. Please contact the factory.

CHEMICAL	73°F (23°C)	212°F (100°C)	392°F (200°C)
Nitric Acid, Conc.	C	C	C
Nitrous Acid, 10%	A		
Oleic Acid	A		
Oleum	C	C	C
Oxalic Acid	A	A	
Perchloric Acid	A	A	
Phosphoric Acid, 10% Conc.	A	A	A
Phosphoric Acid, 50% Conc.	A	A	A
Phosphoric Acid, 80% Conc.	A	A	
Phthalic Acid	A	A	
Picric Acid	A	A	
Silicic Acid	A	A	
Sulfuric Acid, <40% Conc.	B	B	B
Sulfuric Acid, >40% Conc.	C	C	C
Sulfurous Acid	A	A	
Tannic Acid, 10% Conc.	A	A	
Tartaric Acid	A	A	
Trifluoromethyl Sulfonic Acid	C	C	C
ALCOHOLS			
Benzyl Alcohol	A		
Butanol	A		
Cyclohexanol	A		
Ethanol	A	A	
Ethylene Glycol	A	A	B
Ethylene Glycol, 50% Conc.	A	A	A
Glycerol	A		
Gylcols	A	A	
Isopropanol	A		
Methanol	A	A	
Propanol	A		
ALDEHYDES/KETONES			
Acetaldehyde	A	A	
Acetone	A	A	
Benzaldehyde	A		
Cyclohexanone	A		
Formaldehyde	A	A	
Formalin	A		
Ketones	A		
Methylethyl Ketone (MEK)	A	B	C
N-Methyl-2-Pyrrolidone (NMP)	A		
BASES			
Ammonia 880	A		
Ammonia Anhydrous	A	A	A
Ammonia Aqueous	A	A	A
Ammonium Hydroxide, 10% Conc.	A		
Ammonium Hydroxide, Conc.	A		
Calcium Hydroxide	A		
Hydrazine	A	A	
Hydroxides	A		
Magnesium Hydroxide	A		
Potassium Hydroxide, 10% Conc.	A		
Potassium Hydroxide, 70% Conc.	A		

CHEMICAL	73°F (23°C)	212°F (100°C)	392°F (200°C)
Sodium Hydroxide, 10% Conc.	A	A	A
Sodium Hydroxide, 50% Conc.	A	A	A
Sodium Hydroxide, Conc.	A		
ESTERS			
Aliphatic Esters	A	A	
Amyl Acetate	A	A	
Butyl Acetate	A		
Dibutyl Phthalate	A		
Dimethyl Phthalate	A		
Dioctyl Phthalate	A		
Ethyl Acetate	A		
Oils (Di-Ester and Phosphate Ester Based)	A	A	
ETHERS			
Diethylether	A	A	
Dioxane	A		
Ether	A	A	
Ethylene Oxide (EtO)	A		
Tetrahydrofuran (THF)	A		
HALOGENATED ORGANICS			
1,1,1 Trichloroethane (Genklene*)	A		
1,2 Dichloroethane	A		
Carbon Tetrachloride	A	A	
Chorobenzene	A	A	
Chloroform	A	A	
Dibromoethane	A		
Dichlorobenzene	A		
Dichloroethane	A		
Ethylene Dichloride	A		
Freon* 11 Trichlorofluoromethane	A		
Freon 113 Trichlorotrifluoroethane	A		
Freon 114 1,1 Dichloro 1,2,2,2 Tetrafluoroethane	A		
Freon 12 Dichlorodifluoromethane	A		
Freon 22 Chlorodifluoromethane	A	A	
Freon 134a	A		
Freon 502	A	A	
Methylene Chloride	A		
Perchloroethylene	A	A	
Trichloroethylene	A	A	
HYDROCARBONS			
Acetylene	A	A	
Aromatic Solvents	A	A	
Aviation Hydraulic Fluid	A		
Benzene	A	A	
Brake Fluid (Mineral)	A	A	A
Brake Fluid (Polyglycol)	A	A	A
Butane	A		
Crude Oil	A		
Cyclohexane	A	A	
Diesel Oil	A		
Ethane	A		

CHEMICAL	73°F (23°C)	212°F (100°C)	392°F (200°C)
Fuel Oil	A		
Gas (Manufactured)	A		
Gas (Natural)	A		
Gasoline	A	A	
Heptane	A		
Hexane	A		
Hydraulic Fluid	A		
Iso-Octane	A		
Kerosene	A		
Lubricating Oil	A		
Methane (Gas)	A	A	A
Motor Oil	A	A	A
Naphtha	A	A	
Naphthalene	A	A	
Oils (Petroleum)	A	A	
Oils (Vegetable)	A	A	
Pentane	A		
Petroleum Ether	A		
Propane	A		
Styrene (Liquid)	A		
Toluene	A		
Transformer Oil	A	A	
Vaseline	A		
Xylene	A		
INORGANICS			
Aluminum Chloride	A	A	
Aluminum Sulfate	A	A	
Alum, Saturated	A	A	
Ammonium Chloride, 10% Conc.		A	A
Ammonium Nitrate	A	A	
Antimony Trichloride	A	A	
Barium Salts (Chloride, Sulfide)	A		
Bleach	A	A	
Brine	A	A	
Bromine	C	C	C
Bromine (Dry)	C	C	C
Bromine (Wet)	C	C	C
Bromine Water, Saturated	A	A	
Calcium Bisulfide	A	A	
Calcium Carbonate	A		
Calcium Chloride	A	A	
Calcium Hypochlorite	A	A	
Calcium Nitrate	A		
Calcium Sulfate	A	A	
Carbon Dioxide (Dry)	A		
Carbon Monoxide (Gas)	A	A	A
Chlorine	C	C	C
Copper Acetate	A	A	
Copper Carbonate	A	A	
Copper Chloride	A	A	
Copper Cyanide	A	A	
Copper Fluoride	A	A	
Copper Nitrate	A	A	
Copper Sulfate	A	A	
Cupric Fluoride	A	A	
Cupric Sulfate	A	A	

CHEMICAL	73°F (23°C)	212°F (100°C)	392°F (200°C)
Cuprous Chloride	A	A	
Ethylene Nitrate	A		
Ferric Chloride	B	B	
Ferric Nitrate	A		
Ferric Oxide	A	A	
Ferric Sulfate	A		
Ferrous Chloride	A		
Ferrous Nitrate	A		
Ferrous Sulfate	A	A	
Fluorine	C	C	C
Hydrogen Peroxide	A	A	
Hydrogen Sulfide (Gas)	A	A	A
Iodine	B		
Lead Acetate	A	A	
Lime	A	A	
Magnesium Chloride	A	A	
Magnesium Sulfate	A	A	
Mercuric Chloride	A	A	
Mercurous Chloride	A		
Mercury	A	A	
Nickel Acetate	A	A	
Nickel Chloride	A	A	
Nickel Nitrate	A	A	
Nickel Salts	A		
Nickel Sulfate	A	A	
Nitrogen	A		
Nitrous Oxide	A		
Oxygen	A		
Ozone	A	B	
Phosphorous Chlorides	A	A	
Phosphorous Pentoxide	A	A	
Potassium Aluminum Sulfate	A	A	
Potassium Bicarbonate	A		
Potassium Bromide	A	A	
Potassium Carbonate	A		
Potassium Chlorate	A	A	
Potassium Chloride	A	A	
Potassium Dichromate	A		
Potassium Ferricyanide	A		
Potassium Ferrocyanide	A		
Potassium Hydroxide	A	A	
Potassium Nitrate	A	A	
Potassium Permanganate	A		
Potassium Sulfate	A	A	
Potassium Sulfide	A		
Silicone Fluids	A	A	
Silver Nitrate	A	A	
Sodium Acetate	A		
Sodium Bicarbonate	A		
Sodium Carbonate	A	A	
Sodium Chlorate	A	A	
Sodium Chloride	A	A	
Soldium Hypochlorite	A	A	
Sodium Nitrate	A	A	
Sodium Nitrite	A		
Sodium Peroxide	A	A	
Sodium Salts	A		
Sodium Silicate	A	A	

CHEMICAL	73°F (23°C)	212°F (100°C)	392°F (200°C)
Sodium Sulfate	A	A	
Sodium Sulfide	A	A	
Sodium Sulfite	A	A	
Sodium (Hot)	C	C	C
Stannic Chloride	A	A	
Stannous Chloride	A	A	
Steam	A	A	A
Sulfites	A	A	
Sulfur	A	A	
Sulfur Chloride	A	A	
Sulfur Dichloride	A	A	
Sulfur Dioxide	A	A	A
Sulfur Hexafluoride (Gas)	A		
Sulfur Trioxide	A	A	
Tar	A		
Tetraethyl Lead	A		
Water, Distilled	A	A	
Water	A	A	A
Water, Sea/Salt	A	A	
Zinc Chloride	A	A	
Zinc Sulfate	A	A	
MISCELLANEOUS			
Adhesives (not cyanoacrylates)	A		
Apple Juice	A		
Aviation Spirit	A		
Beer	A	A	
Cooking Oil	A		
Creosote	A		
Detergent Solutions (non-phenolic)	A	A	
Edible Fats & Oils	A		
Fatty Acids	A	A	
Fruit Juice	A	A	
Gelatin	A	A	
Ketchup	A		
Linseed Oil	A		
Milk	A	A	
Mineral Oil	A		
Molasses	A	A	
Olive Oil	A	A	
Peanut Oil	A	A	
Paraffin	A	A	
Sewage	A	A	
Soap Solution	A		
Starch	A	A	
Tallow	A	A	
Turpentine	A		
Urea	A	A	
Varnish	A		
Vinegar	A	A	
Wax	A		
White Spirit	A		
Wines and Spirits	A		
Yeast	A	A	

CHEMICAL	73°F (23°C)	212°F (100°C)	392°F (200°C)
ORGANO-NITROGENS			
Acetonitrile	A		
Aniline	A	B	
Dimethyl Formamide (DMF)	A		
Diethylamine	A		
Nitrobenzene	A	C	
Pyridine	A	A	
PHENOLS			
Phenol (Conc.)	C	C	C
Phenol (Dilute)	A		
SULFUR COMPOUNDS			
Carbon Disulfide	A	A	
Dimethylsulfoxide (DMSO)	B	B	
Diphenylsulfone (DPS)	B	C	C
Ethylene Sulfate	A		

KEY

A — No attack. Little or no absorption of solvent.

B — Slight attack. Satisfactory use of PEEK will depend on the application.

C — Severe attack. PEEK should not be used for any application where these chemicals are present.

Tubing

You can use stainless steel tubing to connect the BI-MwA to any other device. Use 1/16” O.D. (1.59 mm) and 0.010” I.D. (0.25 mm) tubing. For continuous use with a variety of solvents or for higher temperature, stainless steel is preferred.

However, for use with water or in batch-mode operation with less aggressive solvents, consider using PEEK tubing. PEEK, poly(etheretherketone), can withstand temperatures up to 200 °C (depending on the solvent) when used continuously under pressure. It is an excellent choice for most chromatography applications except when used with these solvents:

Do NOT use these solvents with PEEK tubing:

DMSO (dimethyl sulfoxide)
Concentrated HNO_3 (nitric acid)
Concentrated H_2SO_4 (sulfuric acid)

When using PEEK tubing, use 1/16” O.D. (1.59 mm) and 0.010” I.D. (0.25 mm).

Important Warning

Never allow any liquid to dry inside the instrument. Always flush the solute or polymer with solvent. If you are not going to work with the instrument for more than a day, **AND** you are working with a solvent in which bacteria may grow, flush the instrument with one or more compatible solvents (see end of this section). Flush with one or more intermediate solvents until you can replace the final liquid with ethanol or some other alcohol in which bacteria will not grow. Then cap the inlet and the outlet ports with the plastic DELRIN plugs that were included when we shipped the instrument to you.

If a blockage occurs due to dried salt or polymer, refer to Appendix A of this manual.

Compatible Solvents Used For Flushing

Methanol and ethanol are intermediate solvents that are miscible with water and with less polar solvents like THF, ethyl acetate, toluene, and benzene.

When changing from water to a nonpolar solvent, first flush the salt or polymer solution with water until the signal is stable (if you have software running). This should take about 20 mL. Then slowly flush (about 1 mL/minute) with an intermediate solvent like ethanol. Check for signal stability again. This should take about 20 mL.

When changing from a nonpolar solvent to water, first flush the polymer solution with the solvent until the signal is stable. This should take about 20 mL. Then slowly flush (about 1 mL/minute) with an intermediate solvent like ethanol. Check for signal stability again. This should take about 20 mL, depending on the miscibility of the two liquids.

We recommend using a syringe pump like the BI-SPump for slowly flushing the system. The BI-SPump is a fixed-speed, syringe pump. With a 20 mL syringe, the speed is about 1 mL/minute.

By flushing the salt or polymer solution first with the solvent, you prevent salt or polymer from precipitating, and then clogging the small-diameter tubing. For example, if you flush a salt solution with an intermediate solvent, the salt may precipitate. Therefore, in this case, flush first with water and then isopropanol, the intermediate solvent.

Section IV: Plumbing Connections

Figure IV-1 shows a close up of the Inlet/Outlet ports. Both are standard, stainless steel, bulkhead, zero-dead volume (ZDV) union assemblies with 0.020” (0.50 mm) thru-holes. They require 10-32 male nuts with ferrules.

When using stainless steel tubing, we recommend Upchurch Scientific U-400 male nuts and the corresponding U-401 ferrules for use with 1/16” O.D. (1.59 mm) tubing. However, any supplier of standard LC/HPLC fittings should have the equivalent hardware.

When attaching stainless steel tubing to the bulkhead union using the Upchurch U-400 male nuts and U-401 ferrules, follow these procedures recommended by Upchurch. Slide the male nut onto the tubing. Slide the ferrule onto the tubing leaving a 3/16” of the tubing protruding past the ferrule. Push the assembly into the bulkhead union, making sure the tubing is touching the bottom of the union. Tighten the male nut by hand while pushing on the tubing. When you can no longer tighten by hand further, use the two wrenches supplied with the BI-MwA.

Brookhaven supplies a 1/4” and a 3/8” wrench. Use the 3/8” wrench to hold the bulkhead union while tightening the nut with the 1/4” wrench. Upchurch recommends turning the male nut 3/4ths of one full turn to *swage* the ferrule onto the tubing. Other manufacturers have their own recommendations that should be followed if you use their fittings.

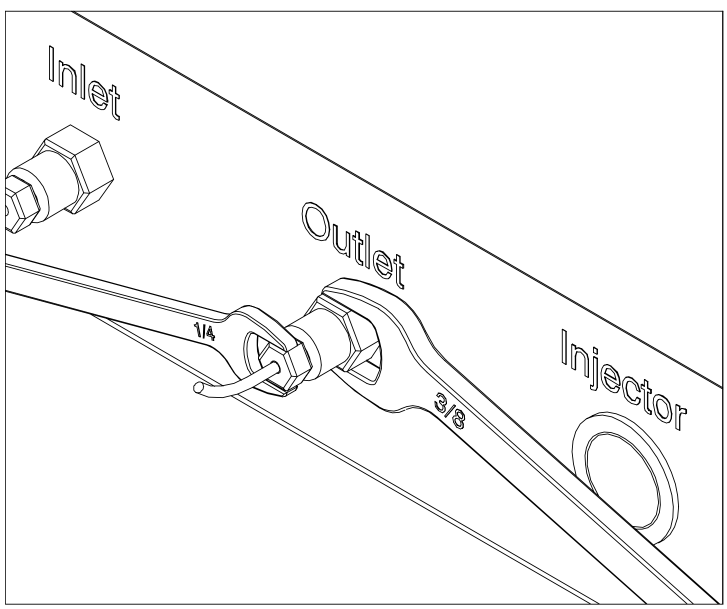

Figure IV-1: Inlet/Outlet Ports showing close-up of bulkhead ZDV unions.

When using PEEK tubing, we recommend Upchurch Scientific F-120 “Fingertight” fittings. These are made of PEEK. They are one piece and do not require a ferrule. If you change the fittings often, you may prefer to use a two-piece fitting with a ferrule.

Important Warning

Never allow any liquid to dry inside the instrument. Always flush the solute or polymer with solvent. If you are not going to work with the instrument for more than a day, **AND** you are working with a solvent in which bacteria may grow, flush the instrument with one or more compatible solvents. Flush with one or more intermediate solvents until you can replace the final liquid with ethanol or some other alcohol in which bacteria will not grow. Then cap the inlet and the outlet ports with the plastic DELRIN plugs that were included when we shipped the instrument to you.

For a list of compatible solvents and a list of intermediate solvents used for flushing, refer to Section III of this manual.

If a blockage occurs due to dried salt or polymer, refer to Appendix A of this manual.

Where to Purchase More Tubing and Fittings

We recommend Upchurch Scientific Inc., currently owned by IDEX Health & Science. They make a variety of plastic and metal tubing as well as fittings, unions, and ferrules.

Section V: Electrical Connections: Auxiliary Analog Inputs

Note: If you want to add the optional, additional 15 analog input channels, please contact the factory. The input specifications for the 1st, four, standard channels as well as the optional 15 additional ones are listed in Table V-1.

Table V-1: Analog Input Specifications.

Input	Voltage Specs	Recommended Detector
Injector	Contact Closure to Ground	Injector
Ch. #1, Std.	± 10 volts, fixed, grounded	Viscometer
Ch. #2, Std.	± 2.5 volts, fixed, grounded	UV, 1st wavelength
Ch. #3, Std.	± 2.5 volts, fixed, grounded	UV, 2nd wavelength
Ch. #4, Std.	± 2.5 volts, in 8 steps to ± 20 mV**, floating	RI, BI-DNDC
Ch. #5-#19, Opt.	± 2.5 volts, in 8 steps to ± 20 mV**, floating	

**The actual voltage steps, software selectable, are as follows:

0.020 Volts (20 mV)
0.040,
0.080,
0.160,
0.320,
0.640,
1.280,
2.560 volts.

Digital Resolution

Channels #1 to #3 use a 16-bit analog-to-digital conversion. Channels #4 to #19 use a 24-bit analog-to-digital conversion.

Figure V-1 shows the PC board with all 19 analog inputs, the injector input, and two thermocouple inputs. This board is standard, though only the 4, standard analog inputs and the injector input are enabled as standard. Contact the factory to enable the 15 optional analog inputs and the thermocouple inputs once you have ordered this option.

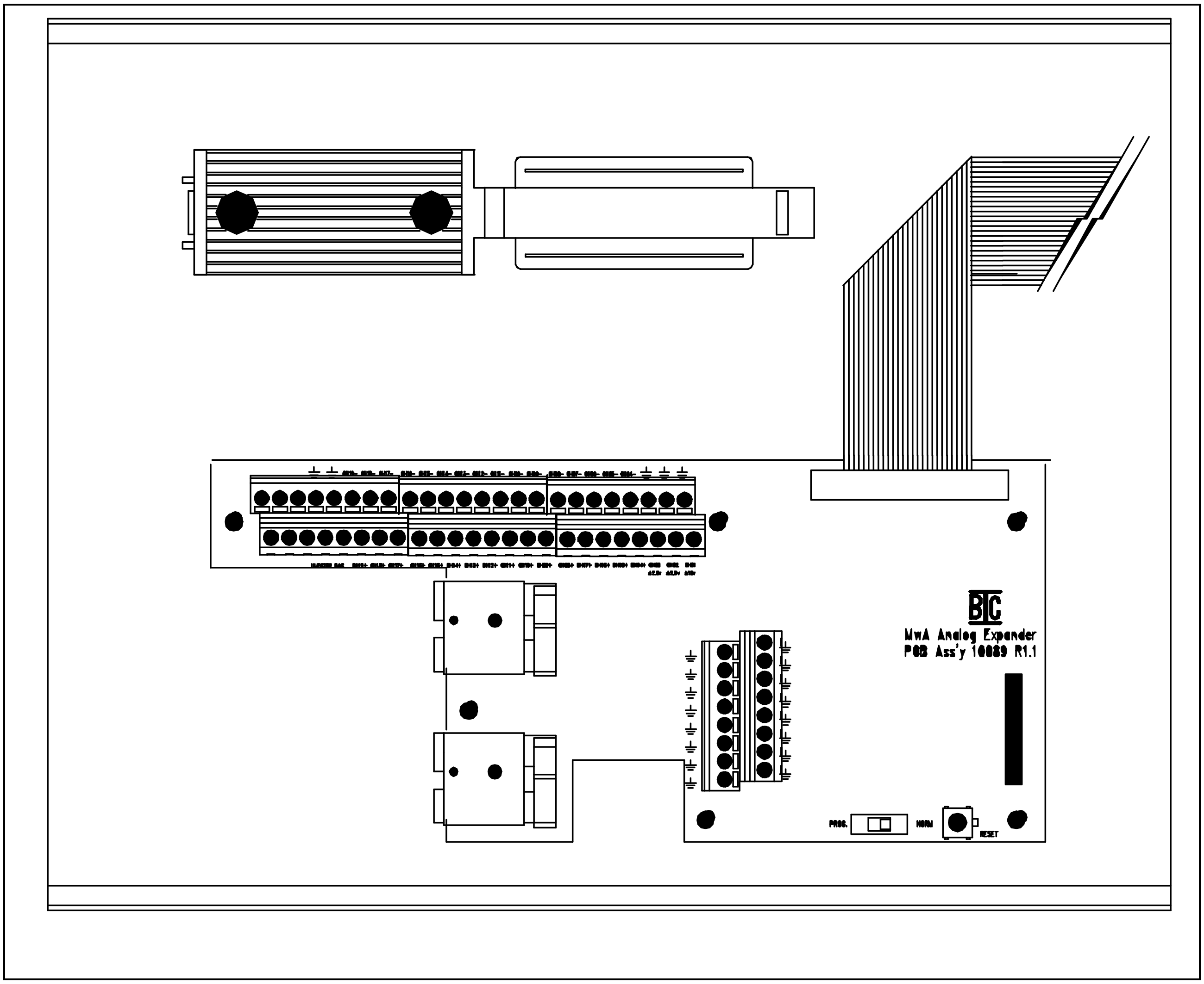

Figure V-1: PC board showing all analog inputs.

The switch in the lower right is left in the NORM position during normal operation. When changing the firmware (See Appendix B.) the switch is moved to the PROG position.

Figure V-2 shows a close-up of extended analog input board showing connectors and channel numbers. Notice that channels 4 through 19 are differential (floating). Channels 1, 2, 3, and the injector are not floating.

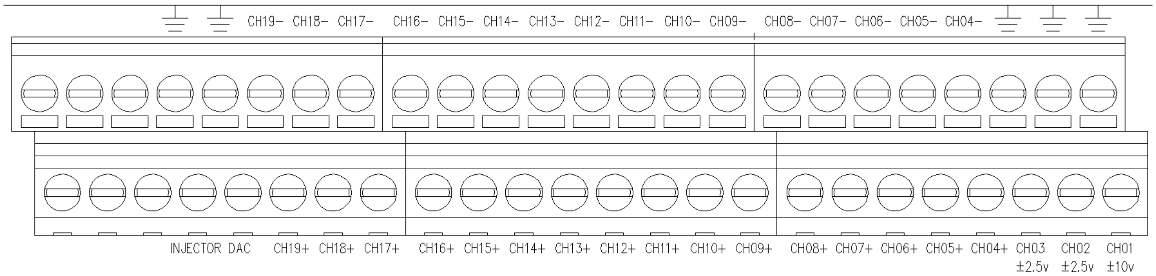

Figure V-2: Analog Input Connectors, All 19 plus Injector.

Section VI: Electrical Connections: Autosampler and Manual Injectors

When using the BI-MwA as a GPC/SEC detector, you will have to signal when an injection occurs.

If you have an autosampler or a manual injector, you must connect the two wires to the injector terminals as indicated in Section V.

If your injector does not provide a position-sensing switch (Load/Inject), use the one on the BI-MwA. It is located next to the Inlet/Outlet ports. See Figure VI-1. After injecting the sample, press the injector button on the BI-MwA. As acknowledgement, a yellow light will remain on for a few seconds.

Figure VI-1 shows a close-up of the front panel. Notice the manual injector button. The light is built into the switch.

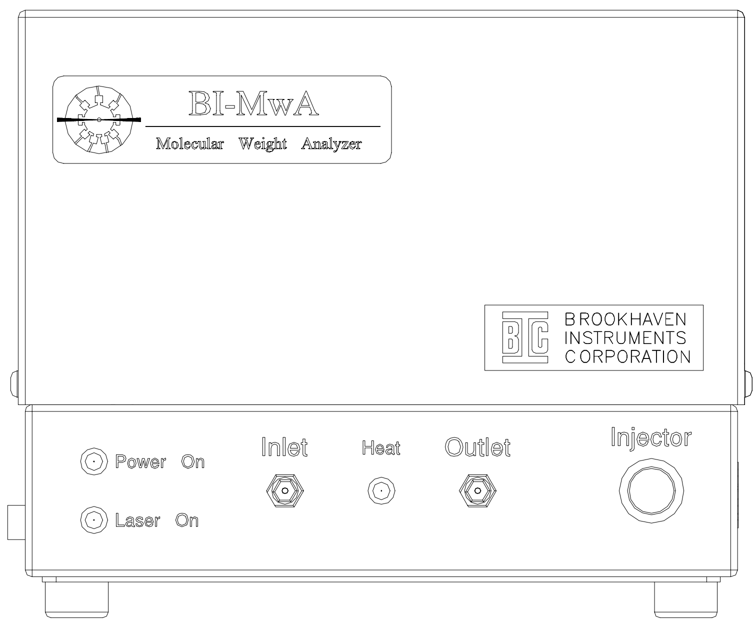

Figure VI-1: Front panel of showing injector button.

Using an appropriate software command, the time since the last injection signal can be read with a resolution of $1/128^{th}$ second (7.8 ms). The command is available to anyone writing software for the BI-MwA. Software provided by Brookhaven and its authorized software partners includes the command.

Section VII: Electrical Connections: Power, USB, and COM

The various connectors are located on the rear of the instrument opposite the inlet/outlet ports. See Figure VII-1.

The USB port is used for communication between the BI-MwA and a PC. See Appendix B for the communication protocol.

The COM port, designated the RS-232C port, is only used to re-flash the firmware inside the BI-MwA. See Appendix C for instructions on re-flashing.

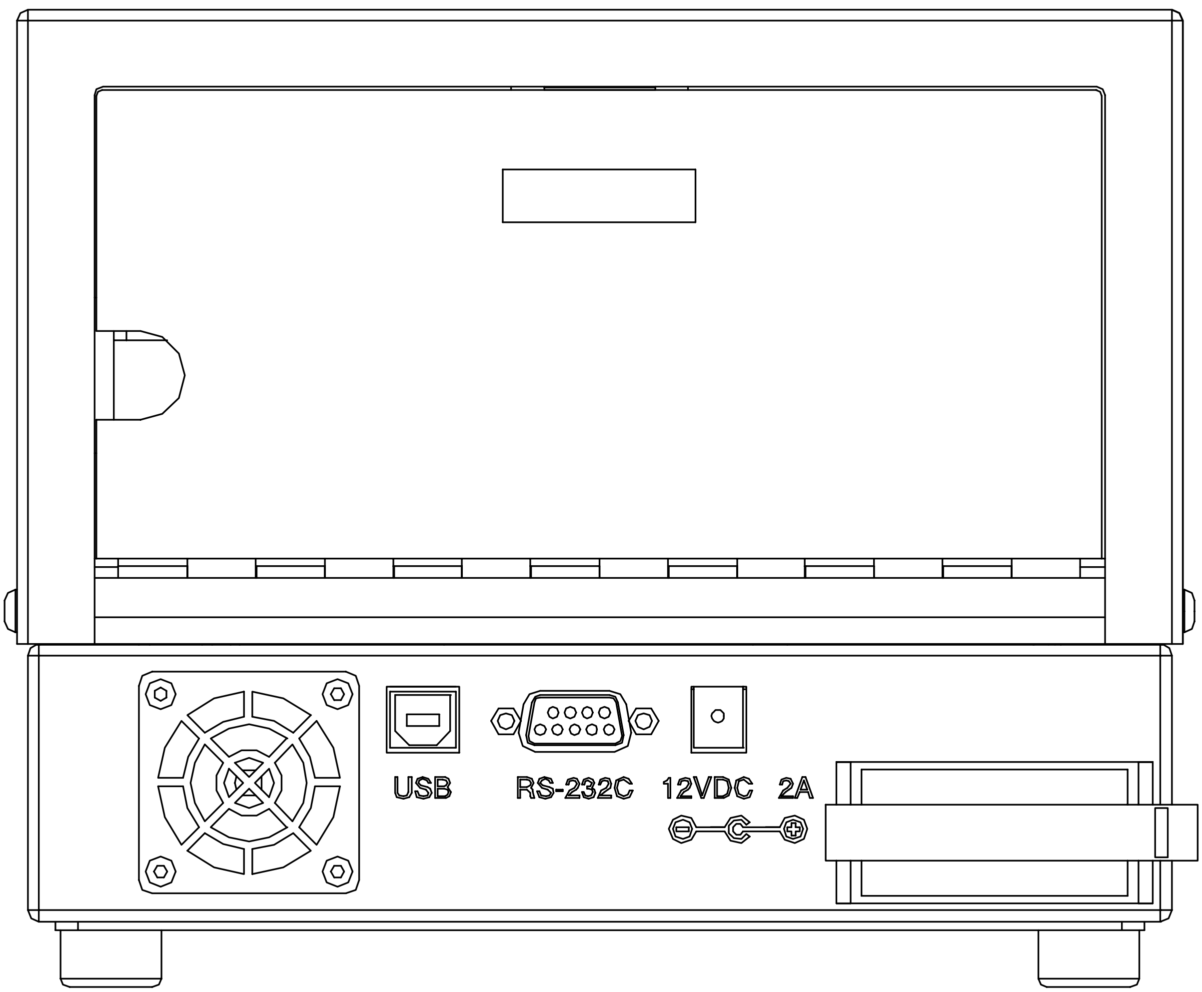

Figure VII-2: Electrical connector locations in rear of BI-MwA.

Section VIII: Installation

Electrical

Plug the power brick provided into a suitable source of mains supply. Then plug the low voltage connector into the 12 VDC mating connector on the rear of the BI-MwA. The green power light on the front panel will glow.

Make sure the switch marked “Prog” and “Norm” is in the “Norm” position on the auxiliary input board. See Figures VIII-1.

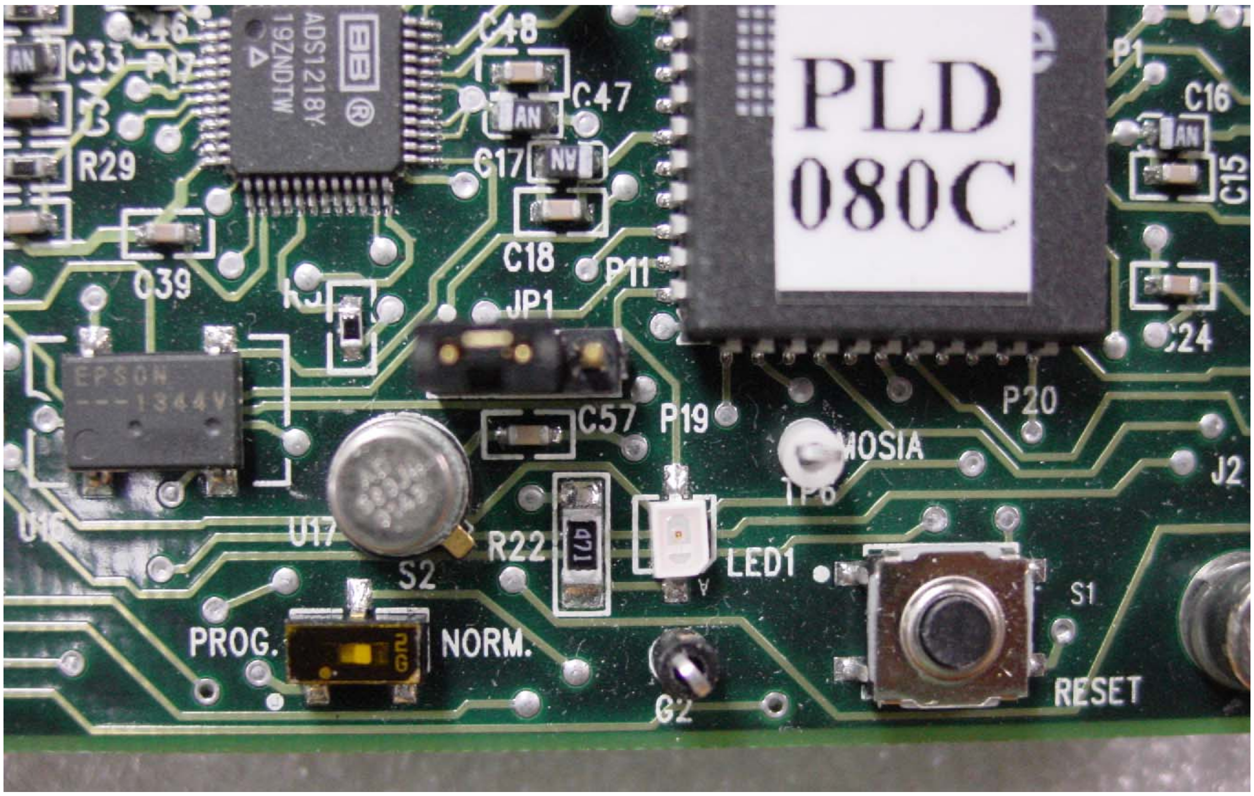

Figure VIII-1: Lower right-hand section of Auxiliary Input Board showing Prog/Norm switch.

Follow the table and figures in Section V for connecting the analog inputs.

Follow the table and figures in Section V for connecting the autosampler or manual injector wires.

If you have one or two thermocouples, plug them into the connectors on the board as shown in Figure V-1. NOTE: Unless you ordered the 15 additional analog channels, they, along with the thermocouple inputs will not be enabled.

Liquid

The unit is shipped with pure isopropanol in the cell to prevent bacterial growth. The Inlet/Outlet ports are plugged with standard 10-32 column plugs. To test that the BI-MwA is communicating with the PC in the next section, you do not have to attach anything else to the BI-MwA.

However, if you wish, you can attach tubing either stainless steel or PEEK tubing in order to push a liquid through the system. See Section IV for the Plumbing Connections.

Software: Installing the USB/MwA Drivers

Follow these instructions to install these special drivers (ftdibus.inf and ftdiport.inf).

1. Turn on the PC and BI-MwA, if you haven't done so already.
2. Attach the USB cable provided to both the PC and the BI-MwA.
3. Note the window referring to new hardware found. Click the **Next** command button. Insert CD provided by Brookhaven with USB driver, BI-MwATASW, and any application software you purchased such as BI-ZPMwA or BI-ACOMP.
4. Click "Search for suitable driver for my device (recommended)."
5. Check the "CD-ROM drives" box.
6. Click the **Next** command button.
7. When Windows finds the file ftdibus.inf, click the **Next** command button.
8. Wait until this part of the installation is finished.
9. Click the **Finish** command button.
10. Note the window referring to new hardware found. This refers to a virtual port. Click the **Next** command button.
11. When Windows finds the file ftdiport.inf, click the **Next** command button.
12. Repeat steps #4 to #9 to install ftdiport.inf.

If you were successful, you should be returned to the desktop.

Software: Installing the BI-MwATASW Test and Analysis Software

The BI-MwATASW Test and Analysis Software will be supplied by and at the discretion of Brookhaven or its representatives. Follow instructions to install the software. If successful, you will see "BI-MwA Test and Analysis Software" in the program menu (submenu "Brookhaven Instruments Corp. (Win32).) Alternatively, the default software path is C:\BICw32\MwATASw\MwATASw32.exe.

Software: Running the BI-MwATASW Test and Analysis Software

Double click the icon labeled BI-MwATASW on your computer screen. You should see a window like the one in Figure VIII-2.

The sole purpose of this software during installation is to show that the BI-MwA and PC are communicating. If they are, you will see the statement "Status: Connected" in the Title Bar. If the statement is "Status: Disconnected", then there is a problem and you should review all the steps in this Section VIII. Contact the factory if you still cannot establish communication.

Click on the Start button in the upper left-hand corner of the display. Note the serial number of the instrument just below the Start button. It should match the number on the silvery ID tag on the instrument. Note the temperature displays. Of the seven, red, CCD peaks, the one furthest to the right corresponds to the 35° scattering angle and the one furthest to the left corresponds to the 145° scattering angle.

The BI-MwATASW software has many other specialized uses reserved for Brookhaven personnel and authorized representatives. Please leave it on your computer.

Figure VIII-2: Main Window of BI-MwATASW software.

Appendix A: Clearing Blockages

If liquid (with or without solute or polymer) dries inside the instrument, the signal stability will suffer. The signal will drift continuously. You need to slowly flush the system with a solvent that will dissolve whatever has dried onto the walls of the tubing and cell. This may take many hours depending on the solubility of the dried material in the liquid you use to dissolve it. Use a syringe pump such as the BI-SPump. Alternatively, flush both inlet and outlet with 2-10 mL of solvent many, many times throughout the day.

If solvent will not dislodge the dried particles or polymer, use 0.1 M nitric acid. Wear gloves and safety goggles. You may have to repeat this several times to get even a low flow rate going. Since the blockage is often near the beginning of the internal tubing, it often helps to flush the instrument in the opposite direction. In other words, by injecting solvent or acid into the outlet port, the dried material near the inlet port may dissolve or become dislodged more easily.

If the instrument is left with water, bacteria may grow and adsorb onto the walls of the system. When flushed initially, the signal may drift for a long time just as if salt or polymer had dried in the system. This will also require slow flushing with alcohol and then water to remove it.

You can determine when the instrument is stable by monitoring the signal as a function of time using one the stability graph of the BI-ZPMwA software (optional) or the BI-MwATASW software (standard).

Appendix B: COM Port: Re-flashing the Firmware

In addition to providing a standardized serial port, the dedicated microcontroller can be re-programmed from an external computer through the COM port. This may be done to add certain features. Should this become necessary, follow these instructions:

Instructions for Re-Flashing the Firmware

The dedicated microcontroller can be re-programmed from an external computer through the COM port. First, install the WSD software that will be provided with the mwa.hex file if needed . Then follow these instructions:

1. Attach the provided RS-232 cable from the computer to the BI-MwA.
2. Locate the program/normal running switch on the auxiliary analog input board.
3. Put the "Prog" and "Norm" switch into the "Prog" position.
4. Press and hold the RESET button for at least three seconds.
5. Double click the WSD icon to run the software.

WARNING: This next step is very important. If omitted, the data in flash memory will also be deleted.

6. Click "Configuration".
7. Click "Erase the CODE memory only." Click O.K.
8. Click "Download".
9. Browse to find the MWA.HEX file.
10. Click OK.
11. When finished, put the "Prog" and "Norm" switch into the "Norm" position.
12. Press and hold the RESET button for at least three seconds.

If you were successful in re-flashing the memory, use the BI-MwATASW software as described in SECTION VIII of the BI-MwA Installation Manual. Then proceed to use your application software. Contact the factory if you have any questions.