CEI-100 Capillary Electrophoresis Interface for ICP-MS

Product Warranty Statement

SD Acquisition, Inc., DBA CETAC Technologies ("CETAC"), warrants any CETAC unit manufactured or supplied by CETAC for a period beginning on the date of shipment and ending on the sooner to occur of: (a) the date that is twelve (12) months from the date of installation, or (b) the date that is thirteen (13) months from the date of shipment. Units found in the reasonable judgement of CETAC to be defective in material or workmanship will be repaired or replaced by CETAC without charge for parts and labor. CETAC reserves the right to change or improve the design of any unit without assuming any obligation to modify any unit previously manufactured.

This warranty does not cover any unit that has been subject to misuse, neglect, negligence, or accident. The warranty does not apply to any damage to the unit that is the result of improper installation or maintenance, or to any unit that has been operated or maintained in any way contrary to the instructions specified in the CETAC instruction and operation manual. Operation of the CETAC unit inside a laboratory fume hood is contra-indicated and will void the warranty. Any attempt to repair or alter any CETAC unit by anyone other than by CETAC authorized personnel or agents will void this warranty. If any non-CETAC component is installed in the CETAC manufactured unit without the approval of CETAC, the warranty will be voided. In addition, this warranty does not extend to repairs made necessary by the use of parts, accessories or fluids which are either incompatible with the unit or adversely affect its operation, performance or durability. CETAC'S obligation under this warranty is strictly and exclusively limited to repair or replacement of defective CETAC parts, and no claim of breach of warranty shall be cause for cancellation or recission of the contract of sale of any unit.

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Purchaser shall indemnify CETAC against any claim or liability which may be asserted as relates to the following: (i) the use to which any product supplied hereunder is put infringes the patent, copyright or other intellectual property rights of any third party; or (ii) any liability resulting from the failure by Purchaser to observe the terms of this Warranty.

Returned Product Procedures

Claims for shipment damage (evident or concealed) must be filed with the carrier by the buyer. CETAC must be notified within ninety (90) days of shipment of incorrect materials. No product may be returned, whether in warranty or out of warranty, without first obtaining approval from CETAC. No replacements will be provided nor repairs made for products returned without such approval. Any returned product must be accompanied by a return authorization number. The expense of returning the unit to CETAC for service will be paid by the buyer. The status of any product returned later than thirty (30) days after issuance of a return authorization number will be subject to review. Shipment of repaired products will generally be made forty-eight (48) hours after the receipt.

Products may not be returned which are contaminated by radioactive materials, infectious agents, or other materials constituting health hazards to CETAC employees.

Returned Product Warranty Determination

After CETAC'S examination, warranty or out of warranty status will be determined. If a warranted defect exists, the product will be repaired at no charge and shipped prepaid back to the buyer. If the buyer desires an airfreight return, the product will be shipped collect. Warranty repairs do not extend the original warranty period.

If an out of warranty defect exists, the buyer shall be notified of the repair cost. At such time the buyer must issue a valid purchase order to cover the cost of repair and freight, or authorize the products to be shipped back as is, at the buyer's expense. Failure to obtain a purchase order number approval within fifteen (15) days of notification will result in the products being returned as is, at the buyer's expense.

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REVISIONS

CETAC Technologies strives to provide the scientific community with an unparalleled combination of effective technology and continuing value. Modular upgrades for existing instruments will continue to be a prime consideration as designs progress.

CETAC Technologies reserves the right to revise this document and/or improve products described herein at any time without notice or obligation. Warranty registration entitles the named owner exclusively to manual change pages/new editions as they are published.

SAFETY

Instruments, accessories, components or other associated materials ***may not*** be returned to CETAC Technologies if contaminated with biohazard or radioactive materials, infectious agents, or any other materials and/or conditions that could constitute a health or injury hazard to CETAC employees. Call Customer Service and Support if there is any question or doubt relative to decontamination requirements.

CAUTION and WARNING statements, as applied in this document, shall be interpreted consistent with the following context: CAUTION applies only to potential property damage conditions; WARNING applies to potential personal injury conditions, in combination with or exclusive of potential property damage.

WARNING

The handling of organomercurial concentrates which may be used in the preparation of process standards presents a substantial (potentially lethal) safety hazard. Only an experienced, professionally trained organo-metallic chemist, knowledgeable and skilled specifically in the safe handling of organomercurials (using approved apparatus and approved protection measures in an approved facility) should attempt to prepare diluted organomercurial process standards from concentrates.

NOTE

SD Acquisition, Inc., DBA CETAC Technologies assumes no liability for the handling of organomercurial concentrates or the preparation, handling, or use of diluted organomercurial process standards. Instead, CETAC Technologies recommends use of appropriate standard reference materials to validate sample preparation (dissolution/digestion) and use of inorganic mercury standards for instrument calibration.

All user-serviceable components are specifically identified in this document as such; the balance shall be assumed to require the expertise of a factory service technician/engineer for adjustment, repair,

replacement, modification, etc. Others not so qualified and performing these actions shall do so at their own risk. Furthermore, never operate the instrument without first reading and understanding the *CEI-100 Capillary Electrophoresis Interface for ICP-MS Operator Manual* and ensuring that it is operated safely and properly.

ORIGINAL PACKAGING

Retain original factory packaging for moves and factory return shipments. Shipping in anything other than the original fitted foam and container can result in incidental damage from which the purchaser will not be protected under warranty.

WARNING

Under all conditions the user must observe safe laboratory procedures during the operation of this product.

Contents

Contents

Preface

The *CEI-100 Capillary Electrophoresis Interface for ICP-MS Operator's Manual* provides an overview and explains the theory of operation of the CEI-100. It also supplies CEI-100 installation and operation information, technical specification data about the systems and sub-systems, and it details troubleshooting and maintenance procedures.

Who Should Read This Book

The primary audience for the *CEI-100 Capillary Electrophoresis Interface for ICP-MS Operator's Manual* consists of laboratory managers, chemists, technicians, and field-service engineers. To use this manual (and product) safely and effectively, at least a general knowledge of chemistry, electronic or laboratory equipment, and basic chemical handling procedures are required.

How to Use This Book

The *CEI-100 Operator's Manual* contains 7 sections. Read the sections sequentially the first time. Thereafter, refer to the sections separately as needed.

Conventions Used in This Book

This book uses certain conventions to distinguish different types of information. This section describes these conventions.

Instructions

All step-by-step instructions are numbered and in bold, as in the following example.

1 Flushing the CE Capillary.

Many numbered instructions are followed by more detailed explanations.

Notes

Notes contain a reminder about the effect of particular actions. They are indicated as follows:

> **Note:**
>
> This example shows how a note is displayed.

Cautions

Cautions indicate situations that require immediate attention to prevent harm to the CEI-100 system. Cautions are indicated as follows:

CAUTION

This example shows how a caution is displayed.

Warnings

Warnings indicate situations that could cause bodily harm. Warnings are indicated as follows:

WARNING

This example shows how a warning is displayed.

Where to Go for More Information

In addition to the *CEI-100 Capillary Electrophoresis Interface for ICP-MS Operator's Manual*, the analyst can refer to:

CETAC Technologies Customer Service and Support:

Tel: (800) 369-2822 (USA only)

Tel: (402) 733-2829

Fax: (402) 733-1932

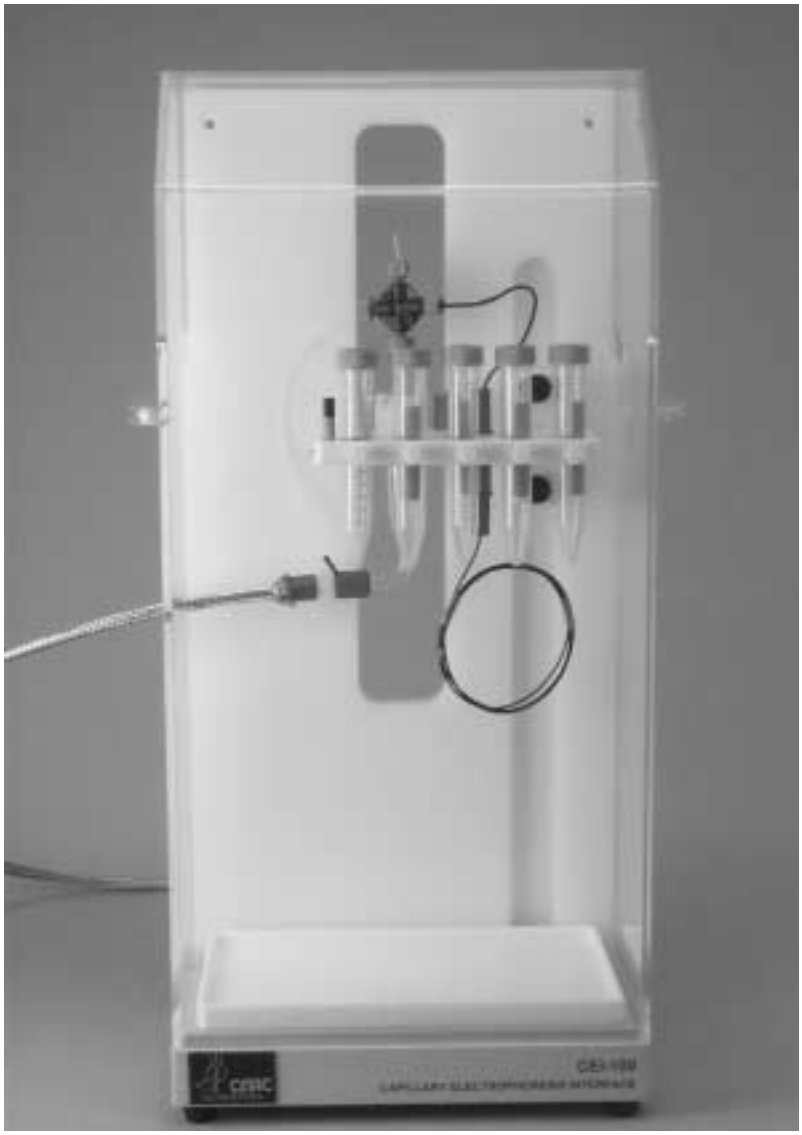
eMail: custserv@cetac.com

I. Introduction

The use of capillary electrophoresis (CE) as a high resolution separation technique with an inductively coupled plasma - mass spectrometer (ICP-MS) as a sensitive element specific detector is of growing interest for speciation research. A dedicated interface between a CE system and an ICP-MS instrument is necessary to preserve the separation capabilities of the CE technique.

The design of the CETAC CEI-100 capillary electrophoresis interface overcomes a major disadvantage of previous designs: degradation of species separation by nebulizer suction. Also, the interface is designed to provide effective analyte transport to the ICP-MS instrument and a stable electrical connection. The main component of the CEI-100 interface is a low flow concentric nebulizer, which is specially designed for CE-ICP-MS coupling.

References for recent publications referring to the CEI-100 interface are given in Appendix I.



Front view with cover.

CEI-100 without cover.

II. Components of the CEI-100 Interface

Component	***Number***
Low-flow concentric nebulizer	1
Nebulizer gas line	1
Crosspiece, including	1
Platinum electrode	
Fitting for CE capillary	
Fitting for buffer line	
Connection to nebulizer	
Support stand	1
Transparent cover	1
Vials for buffer / make-up liquid	5
Syringe kit	1
Spray chamber	1
Transfer line and fittings to ICP-MS	1
Waste line to peristaltic pump	1
Grounding cable to CE system	1

III. Assembly of the Interface

Refer to the numbered items in Figures 1 and 2. Descriptive photographs are also included.

To begin assembly, place the low-flow nebulizer (2) into the support plate (1) of the stand and secure it with the thumbscrew (9). Then connect the crosspiece (3) with the fitting to the rear of the nebulizer. The grounding cable (4) attached to the crosspiece is plugged into the jack (5). Opposite the cable is the line (6) for the buffer / make up liquid. Place this line into the vial (7) with the buffer / make up liquid. The vial marker (8) can be used for measuring the liquid level and the thumbscrews (14) for securing the vials into the support plate.

A second grounding cable (18) attaches to the bottom of the jack (5) under the support plate (1). The free end of this cable (18) then attaches to the ground of the host CE system.

Remove the protective cap from the nebulizer end and insert the nebulizer (2) into the spray chamber (10). The transfer line (11) consists of a Teflon®[1] tube (4 mm i.d.) and a metal sleeve to lessen electrostatic effects on the dry sample aerosol. Connect the transfer line (11) to the spray chamber and the nebulizer gas line (12) to the side of the nebulizer (2).

The level of the entire support plate (1) of the stand can be changed by loosening two thumbscrews (13), and adjusting the plate position up or down in the groove. A threaded fitting (15) serves as a holder for the crosspiece in case it is removed from the nebulizer. By connecting a waste line (16) to the barbed end of that fitting (15), the crosspiece can be flushed separately (e. g. by a peristaltic pump).

Note:

Numbered items may again be referenced later in this manual.

[1] Teflon is a registered trademark of E.I. du Pont de Nemours.

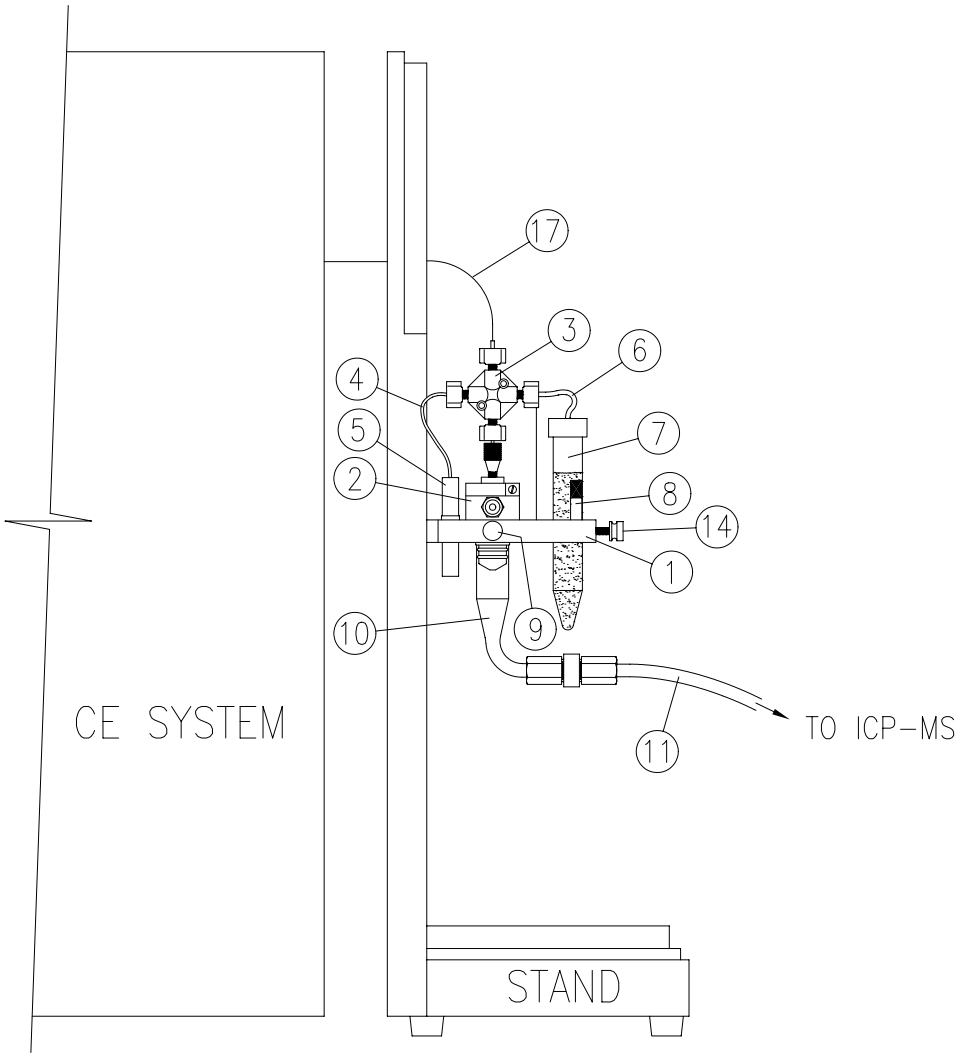


Figure 1. Side view.

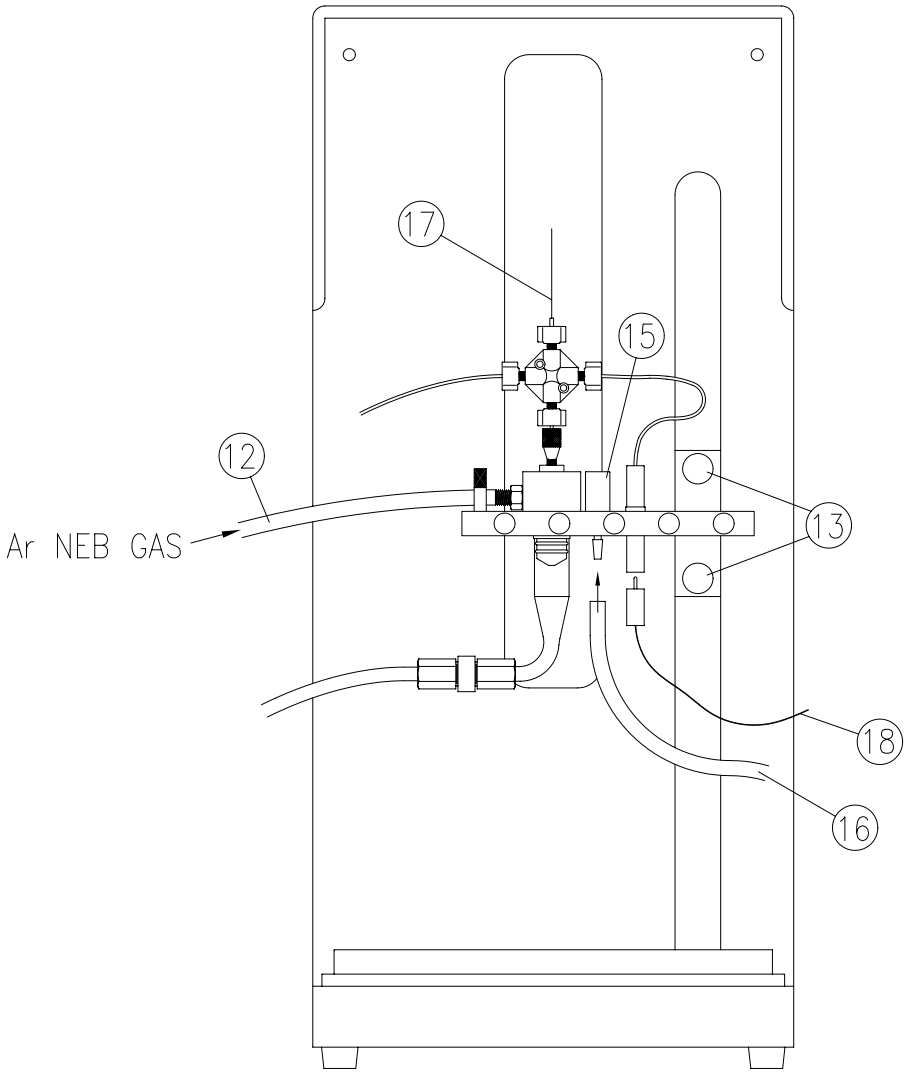


Figure 2. Front view.

Insertion of nebulizer into spray chamber.

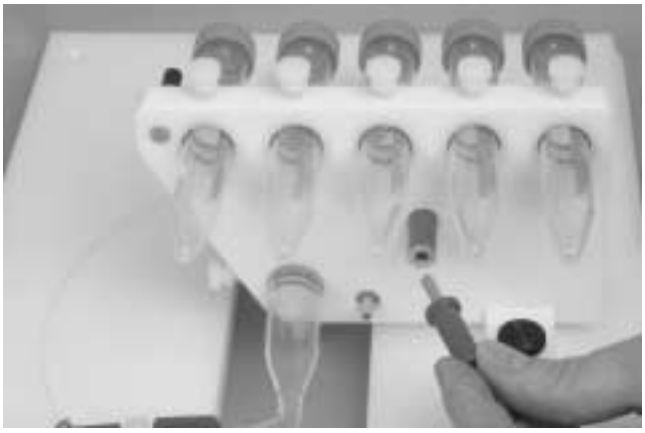
Thumbscrew to secure nebulizer.

Nebulizer gas line connection.

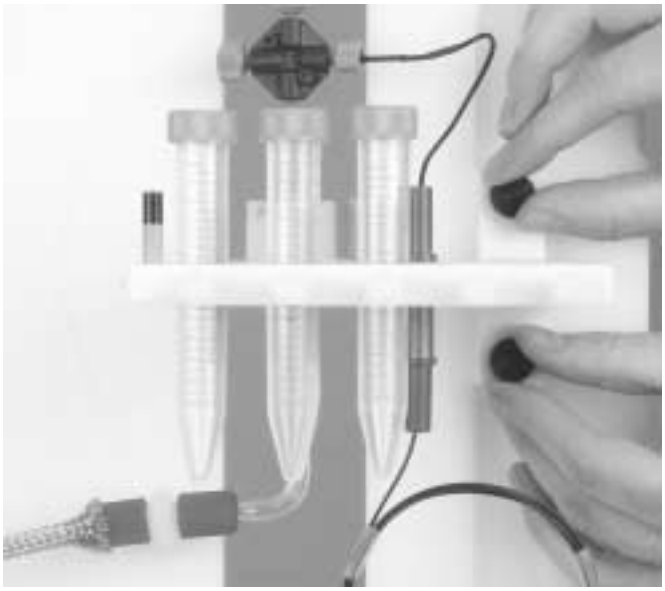
Top view.

Top view, ground cable connection.

Top view, make up liquid.



Bottom view, ground connection.



Height adjustment.

Installation of the CEI-100 Interface

Place the CEI-100 interface box close to the CE system as it is shown in Figure 1. The CE capillary (17) should pass through the slit on the back of the CEI-100 interface stand so that the length of the CE capillary (17) outside the CEI-100 box is as short as possible.

The interface transfer line (11) is connected directly to the torch of the ICP-MS instrument. The torch connection adapter depends on the torch style used for a particular ICP-MS instrument. For example, ICP-MS torches with a 12/5 glass male joint can be interfaced with a corresponding female joint adapter (See Figure 3). The adapter is then simply connected to the end of the interface transfer line (11).

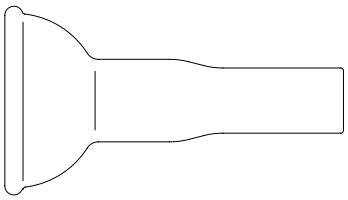


Figure 3. 12/5 glass female adapter.

The transfer line and metal sleeve may be shortened to an optimal length.

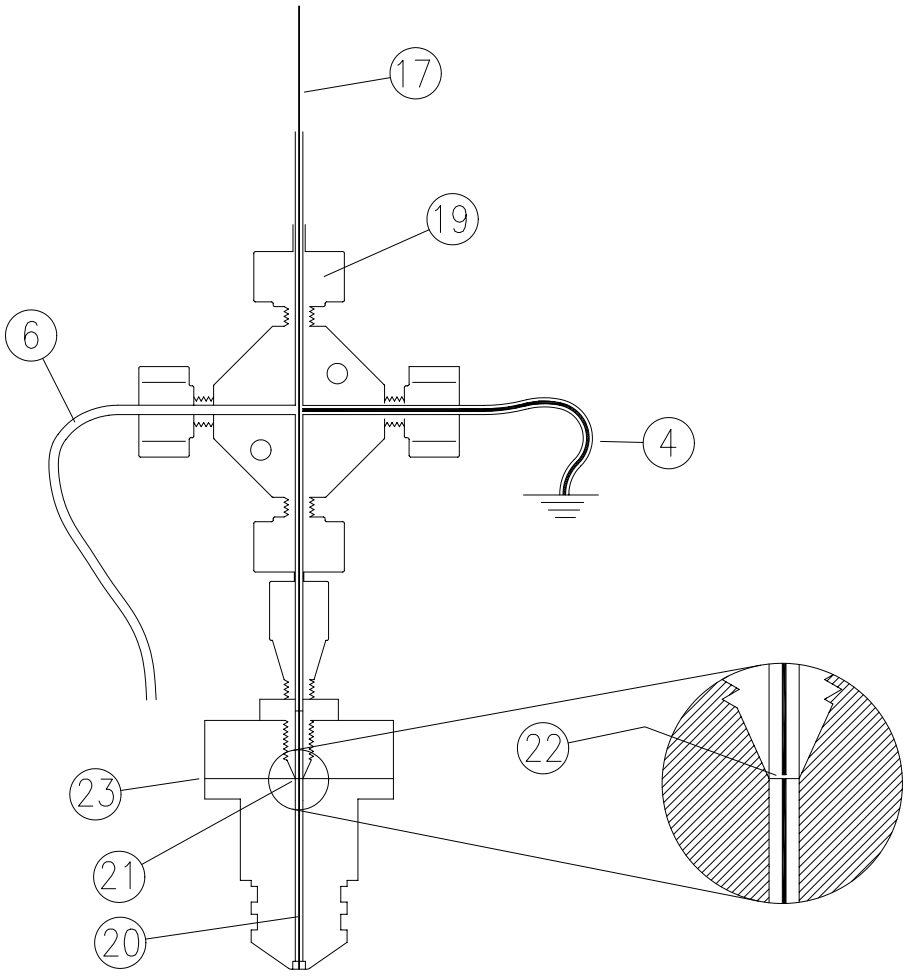


Figure 4. Crosspiece / Nebulizer assembly.

Figure 4 shows a detailed view of the nebulizer and the crosspiece assembly. Insert the CE capillary (17) through fitting (19) down to the beginning of the nebulizer capillary (20) at point (21) in the nebulizer fitting. Between the nebulizer capillary and CE capillary should be a small gap (22) to avoid excessive dead volume and band broadening. Tightly screw the fitting (19) to secure the CE capillary to the crosspiece.

To avoid any hydrodynamic flows, the inlet and outlet of the CE capillary must be on the same level. Therefore, note the inlet level outside of the CEI-100 box and adjust the outlet level by adjusting the height of the support plate (1). The outlet level is at point (23) as it is shown in Figure 4.

The liquid levels must also be at the same level. Therefore adjust the liquid level of the buffer / make up liquid to the same level of the liquid in the sample inlet vial (CE system) by adjusting the vial (7) of the make up liquid.

Finally, make a connection between the grounding cable (4) in the jack (5) and the ground of the host CE system to close the electrical circuit. Use the second grounding cable (18) attached to the bottom of the jack (5) to make this connection.

IV. Testing and Operating the CEI-100 Interface

The low flow nebulizer used in the CEI-100 interface must be operated in the self-aspiration mode. This is necessary to avoid any suction or backpressure effects on the CE capillary. The suggested argon nebulizer gas flow range is between 0.9 and 1.2 L / min. This range results in a liquid uptake rate between 5 and 10 µL / min. To test the self-aspiration, remove the crosspiece (3) from the nebulizer (2) and connect the line (6) directly to the nebulizer. With the ICP-MS instrument and nebulizer gas on, insert the line (6) into a vial with tuning solution and monitor the corresponding tune elements. The nebulizer should introduce the tune solution by self-aspiration and a stable signal of the tuning elements can be monitored. To measure the liquid uptake rate, line (6) should be dry (aspirate air). Then insert line (6) into the tuning solution and allow it to self-aspirate. Remove line (6) immediately from the tuning solution (aspirate air again). Note the time when the signal(s) from the tuning element(s) begin to rise. This is the moment when the tune solution has reached the nebulizer. Then record the time when the signal(s) drop to a background level. From the time difference and the volume of the line, the liquid flow rate can be calculated:

$$\text{Flow rate } (\mu L / \text{min}) = (0.16 \cdot 3.14 \cdot l) / t$$

The i.d. of the tube is 0.8 mm. The length l of the tube must be measured in mm and the time difference in minutes.

The crosspiece must then be reconnected to the nebulizer. Rinse the line (6) with the syringe, remove all remaining liquid, and reconnect the line (6) to the crosspiece. If all connections are sealed properly and all liquid is removed from the crosspiece and line, then insert the line into the vial (7) with the buffer / make up liquid. The nebulizer should self-aspirate the buffer into the crosspiece. An ICP-MS tuning element may be added to the buffer to monitor the nebulization. If the nebulizer does not self-aspirate, then all of the liquid may not have been removed. If so, connect the syringe to the line (6) and drain the liquid out. Do this procedure several times if necessary.

The buffer / make up liquid should have the same pH as the electrolyte in the CE capillary (17) to ensure stable electrophoretic conditions. However, the buffer concentration should be not higher than 10 to 15 mmol/L. Otherwise, a salt deposit may form at the nebulizer orifice and clog the nebulizer.

After the CE capillary (17) is prepared for separation, the stability of the current should be tested. Increase the voltage within a time ramp of 30 s up to 30 kV and monitor the current. If the current is stable, the electrophoretic conditions are stable and the electrophoresis can be started.

Finally, the position of the CE capillary (17) has to be checked. Inject a sample containing a single analyte such as Rb (100 µg/L) and start the electrophoresis. The sample should produce a sharp peak with a width around 5 s with little or no tailing. If there is a broad peak with strong tailing, the CE capillary (17) has to be repositioned. Open the fitting (19), position the CE capillary (17) slightly up or down, retighten the fitting (19) again, and check the peak shape. Repeat this procedure several times until you have an optimal peak shape.

V. Helpful Suggestions

Flushing the CE capillary

With the CEI-100 interface, all operations of the CE system such as conditioning, flushing (with up to 1 bar pressure), and sample injection can be carried out while the ICP-MS instrument is operating. When the CE system is flushed or conditioned, a higher amount of liquid than during electrophoresis arrives at the interface. Due to the low flow rate of the nebulizer, not all the liquid can be effectively nebulized. Therefore, it is necessary to insert a waiting step after each flushing (or conditioning) step to allow the entire system to stabilize before the next operation. The waiting step should take approximately the same time as the flushing step.

Composition of the Make Up Liquid

The make up liquid is usually the same as the CE system buffer. For ICP-MS, it is suggested to work with buffers which are more completely consumed in the plasma to lessen salt deposits on the ICP-MS sampling cones. Certain organic buffers or ammonium nitrate can be used, but at the same pH as in the CE capillary.

Replace the Make Up Liquid

Replacement of the make up liquid by another requires approximately 10 minutes after the line (6) is inserted into a different vial. Replacement can be performed more quickly if the make up liquid in the line (6) and crosspiece is removed with the syringe. Again, all liquid must be removed for proper self-aspiration of the new make up liquid.

Another method for replacing the make up liquid is to turn off the nebulizer gas and disconnect the crosspiece from the nebulizer. Then connect the crosspiece to the fitting (15) and flush the crosspiece with the new make up liquid with a syringe. Keep the syringe connected to the line (6) and reconnect the crosspiece to the nebulizer. Disconnect the syringe, insert the line (6) into the new make up liquid, and then

restart the nebulizer gas. This procedure can also be applied when the nebulizer has to be flushed to remove a clog.

Cleaning the System

After finishing sample measurements, the crosspiece and nebulizer assembly has to be carefully cleaned. Replace the make up liquid with deionized water and flush the assembly for 30 minutes. Also flush the CE capillary (fused silica) with water. Never leave an alkaline buffer in the system, as this could damage the polyimide coating of the CE capillary and the nebulizer capillary.

Clean the nebulizer tip by rinsing with deionized water from the outside after disconnecting the spray chamber.

Depending on the buffer, a deposit of the buffer salts can occur in the spray chamber. Therefore, clean the spray chamber after use with deionized water.

Connection of crosspiece to flush port.

Connection of waste line to bottom of flush port.

VI. Troubleshooting

Current and Nebulization Unstable

If the current and/or nebulization are unstable, often an air bubble in the crosspiece is the reason. Usually the nebulization becomes stable again when the bubble has passed through the nebulizer. It is recommended to degas the make up liquid.

If the current increases up to 35 µA or higher, air bubbles can again occur, due to the increased heating. Then use a lower concentration of the CE buffer.

Current Unstable

- If the current is unstable, there could be an air bubble in the CE capillary. Flush the CE capillary with buffer.
- The position of the CE capillary is not correct; reposition the CE capillary as described previously (page 16).
- The CE capillary may be broken. In this case, the nebulizer will have to be replaced. (Contact CETAC Customer Service.)

Nebulization Unstable

- There may be a salt deposit at the nebulizer tip. Clean the nebulizer tip as previously described (page 18).
- The nebulizer tip is broken or the polyimide coating is degraded. When using strong alkaline buffers or conditioning the CE capillary with sodium hydroxide solution, the polyimide coating of the nebulizer capillary can degrade. In this case, the nebulizer has to be replaced. (Contact CETAC Customer Service.)
- Fitting for nebulizer gas (12) is not secure; seal or replace the fitting.

- Nebulizer is clogged; clean nebulizer (see page 18) or replace nebulizer. (Contact CETAC Customer Service.)

Nebulizer Does Not Self-Aspirate

- The line (6) and crosspiece are not dry; liquid drops are inside. Wait until all drops are nebulized; usually after a short time the self-aspiration starts. If not, remove the liquid with the syringe or push air via an empty syringe into the line (6) and crosspiece. In this case air will also be introduced into the CE capillary; therefore, it is necessary to flush the CE capillary after the nebulization has restarted.

- The CE capillary is too close to the nebulizer capillary. Loosen the fitting (19), lift the CE capillary slightly, and retighten the fitting. Remove any liquid from the line (6) and crosspiece. When the self-aspiration starts, the CE capillary can again move closer to the nebulizer capillary.

- Nebulizer is clogged. Clean nebulizer or replace nebulizer. (Contact CETAC Customer Service.)

Nebulizer is Clogged

Due to the small i.d. of the nebulizer capillary, the capillary can be blocked by particles, salt crystals or a salt deposit on the nebulizer tip.

The most common reason for clogging is salt deposition at the nebulizer tip due to high buffer concentrations. In this case, nebulization becomes unstable before the nebulizer is clogged. This salt deposit can easily be removed as described previously (page 18), and this procedure should be performed daily.

To avoid blockage of the nebulizer capillary by particles, all solutions such as buffer and make up liquid should be filtered by a 0.45 µm filter.

Clogging of the nebulizer capillary by salt crystals can occur if the interface is operated for a long time and the nebulizer and crosspiece are not flushed with deionized water. It is recommended to clean the nebulizer daily.

If the nebulizer capillary is blocked by salt crystals or particles, disconnect the crosspiece and connect the make up liquid line (6) directly to the nebulizer. With a syringe, pull the liquid out of the line, then push air or new make up liquid into the nebulizer. Do this procedure several times until the self-aspiration restarts and the nebulization is stable.

If the nebulizer capillary cannot be unblocked or the tip of the nebulizer capillary is damaged, the nebulizer may have to be replaced.

VII. Appendix

[1] D. Schaumlöffel, A. Prange, Fres. J. Anal. Chem., 1999, 364, 452-456.

[2] A. Prange, D. Schaumlöffel, J. Anal. At. Spectrom., 1999, 14, 1329-1332.