

RP-91 ATTACHMENT

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About this manual

This manual is designed to familiarize you with the principle of operation and structure of the RP-91 attachment (hereinafter referred to as the attachment), its design, performance characteristics and operating conditions. It contains information that will allow you to provide comprehensive and efficient utilization of the attachment's capabilities and also to properly operate the attachment.

Prior to studying this manual, you have to be acquainted with the operation manual for the RA-915+ Mercury Analyzer.

Please take the time to read this manual in order to attain the best results in operation and to gain the greatest benefit from the use of the RP-91 attachment.

This manual contains:

- List of important safety measures, warnings and precautions, which you should follow when operating with the attachment.
- Description of the attachment, its basic performance and analytical characteristics and operating conditions.
- Brief description of the physical foundations and principle of operation of the attachment and of its structure.
- Functional controls of the attachment, their purpose and operation.
- Procedures for preparation of the attachment for operation together with an RA-915+ analyzer.
- Brief description of the calibration and analysis procedures using an RA-915+ analyzer and the attachment together with a PC.
- List of main procedures for the attachment maintenance.
- Additional capabilities of the attachment and instructions for trouble shooting and removal.

Safety guidelines

Important safety precautions

Read these rules completely before starting operation with the attachment.

- Carefully study all the sections of this operation manual, the attachment design, and operational procedures.
- Connect the attachment to the RA-915+ analyzer properly.
- Do not allow the ingress of liquids on the case or inside the pump.
- Do not try to repair the attachment yourself.
- Call an authorized agent or certified service engineer in the following cases:
 - If the attachment does not operate properly;
 - If the attachment has fallen down;
 - If glass parts are cracked or broken;
 - If a liquid has got inside the pump.
- When measuring mercury concentration, it is necessary to follow instructions and documents stating safety regulations for operation in chemical laboratories and safety rules for operation with electric appliances.
- Keep with all the safety requirements described in the RA-915+ operation manual.

Personnel admitted to operate the device are recommended to undergo training at the Supplier premises or at a regional service center.

Note symbols



Cautions. Pay attention to these in order to avoid damage of the equipment.



Notes. They contain useful information, which will simplify handling.

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Introduction

Our compliments on the acquisition of the analytical system comprising an RA-915+ mercury analyzer and an RP-91 attachment, which essentially enhances the capabilities of the RA-915+ analyzer. The RP-91 attachment enables mercury determination in samples of potable and waste waters, sewage, surface run-offs, urine, blood, food, soil, etc., using the "cold vapor" technique.

After taking the attachment out of the package, make sure that you have got everything necessary for its operation.

1	Reaction vessel (impinger)	2
2	Power supply unit for the attachment	1
3	Case with a holder and air pump	1
4	Clamp	2
5	Connecting silicone tubes, Ø 5 mm	0.6 m
6	Rotameter (0.2 - 1 l/min)	1
7	Rotameter (1 - 10 l/min)	1
8	Absorption filter	2
9	Inlet fitting for multipath analytical cell	1
10	Outlet fitting for multipath analytical cell	1
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Application

The RP-91 attachment is a "generator of Hg vapor" which is produced by reducing Hg(II) into elemental state by means of the CV (cold vapor) technique. The RA-915+ mercury analyzer equipped with the RP-91 attachment is used for the determination of the mercury content in liquid samples and used for environmental monitoring, technological processes control, medicine and for scientific research.

The RP-91 attachment is designed to be part of a mercury analytical system, which enables determination of the mercury content in gaseous, liquid, and solid samples. The mercury analytical system comprises an RA-915+ analyzer, RP-91 attachment for determination of the mercury content in samples using the CV technique, and an RP-91C attachment for determination of the mercury content in liquid and solid complex-matrix samples using the pyrolysis technique. Basic analytical characteristics of the system are given in table below:

Subject	Detection limit	Sample parameters (flow rate, volume, weight)	Detection technique	Complete set
Ambient air	2 ng/m ³	20 l/min	Direct	RA-915+
Natural and industrial gases	2 - 500 ng/m ³	1 - 20 l/min		
Water	0.5 ng/l	20 ml	"Cold vapor" technique	RA-915+ RP-91
Urine	5 ng/l	1 ml		
Solid samples (soils, rocks)	0.5 µg/kg	0.2 g	Pyrolysis technique	RP-91C RA-915+
Biological samples (tissues, liver, etc.)	5 µg/kg	0.02 g		
Hair	20 µg/kg	0.01 g		
Oil and oil products	50 µg/kg	0.01 g		
Plants	2 µg/kg	0.05g		
Foodstuff	2 - 10 µg/kg	0.005 - 0.05 g		

Additional parameters of the RP-91 attachment

Rate of air flow through the gas line, l/min	from 0.4 to 4.0
Sample volume, ml	from 1 to 20

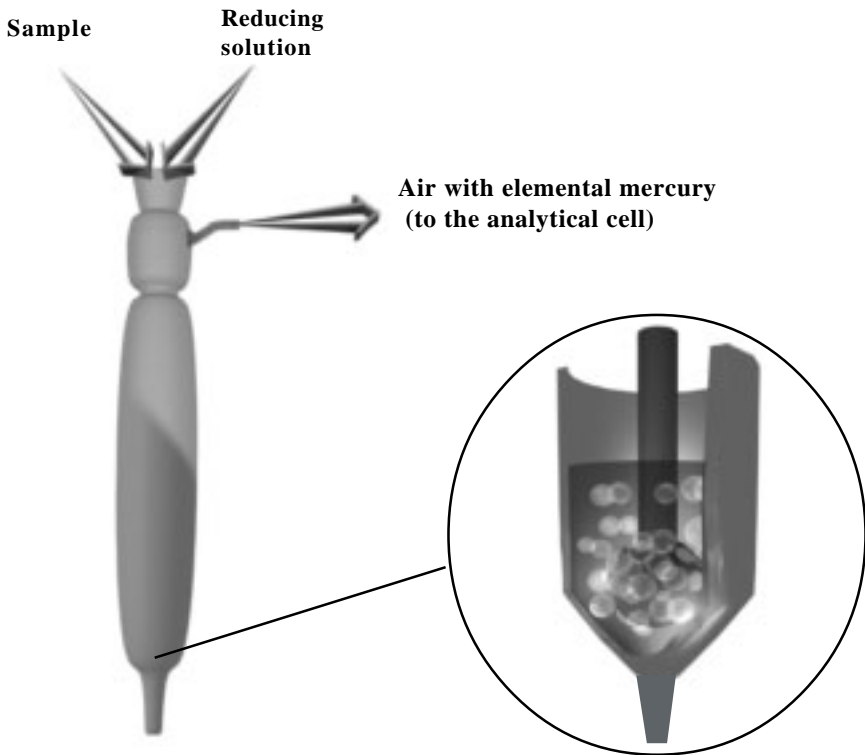
Main technical data and specifications

Power supply	220/110 V, 50/60 Hz
Power consumption	20 W
Nonstop operation time	8 hours
Size of the pumping unit, mm	340*240*120

Attachment design and operation

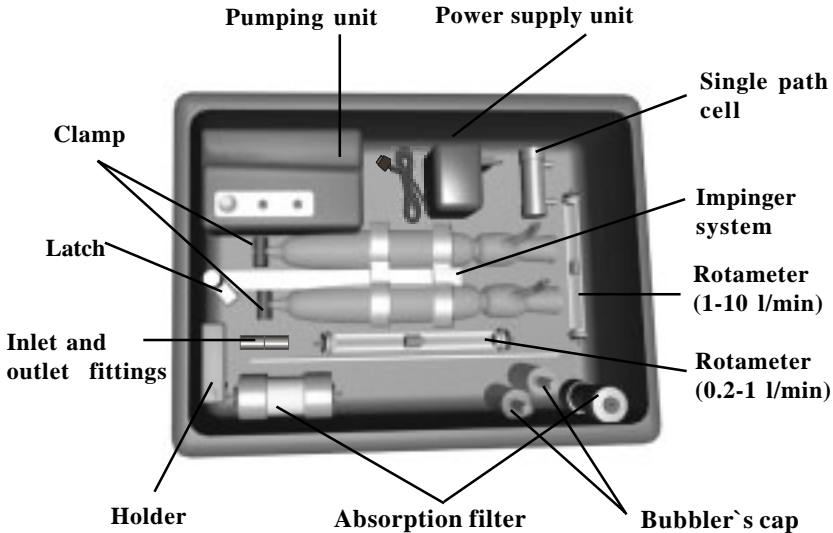
The principle of operation of the RP-91 attachment is based on the reduction of Hg(II) to the atomic state using a reducing solution and then transporting mercury atoms into an analytical cell by an air flow (the "cold vapor" technique). The mercury concentration is then measured by the RA-915+ atomic absorption mercury analyzer. The operational principle of a system with the RP-91 attachment for analysis of samples by the "cold vapor" technique is shown below.

The reducing solution and the sample being analyzed are placed into the impinger. The elemental mercury, which is formed during continuous air pumping through the bubblers, reach analytical cell of the RA-915+ analyzer, where the mercury atoms are detected.



Appearance and functional controls

Appearance



Power supply unit

Supplies power to the pumping unit.

Single path cell

Uses when high concentration in samples is expected.

Impinger system

Consists of two bubblers. In the first bubbler mercury transfers from liquid to gaseous phase. The second bubbler is used for removal aqueous aerosols from the flow.

Rotameter

Uses for air pumping rate control.

Absorption filter

Absorbs mercury after the analytical cell.

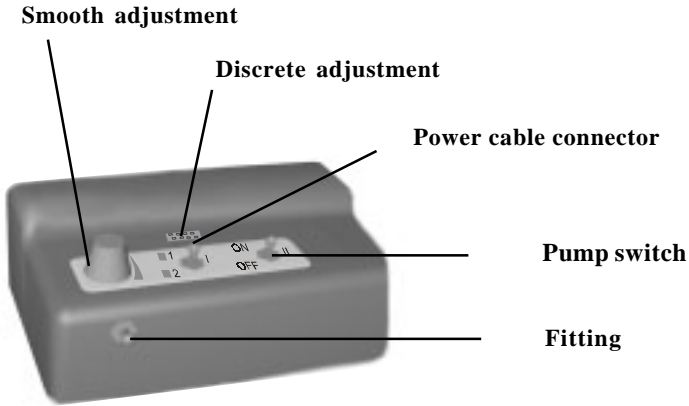
Holder

Unit is intended for installation of the impinger system and the rotameter.

Latch

Fixes the impinger system during transportation.

Pumping unit



Smooth adjustment.

Smooth adjustment the air pumping rate.

Discrete adjustment

Discretely sets the air pumping rate (rate I, rate II).

Power cable connector

Connects the pumping unit to the power supply unit.

Pump switch

Switches pump on/off.

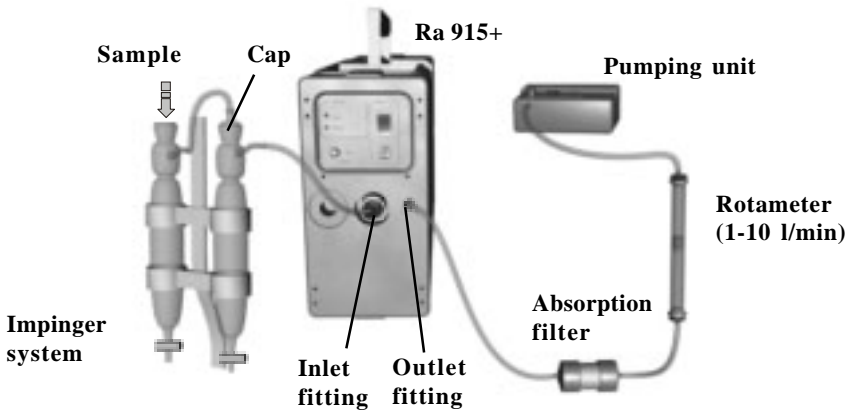
Fitting

Connects the pumping unit to the upper fitting of the rotameter.

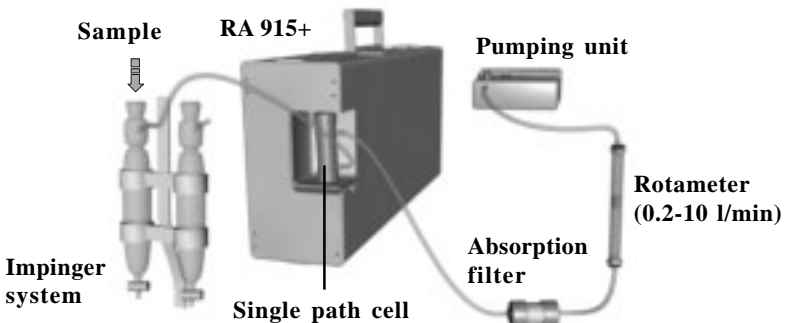
Pre-operational procedures

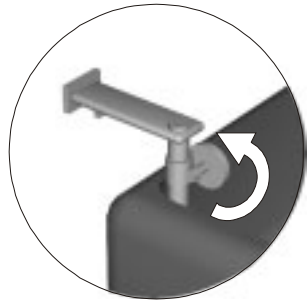
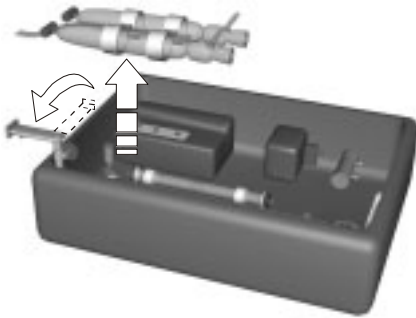
This section contains instructions for preparing the RP-91 attachment for operation. For detailed instructions on preparing the RA-915+ analyzer for operation, please refer to the RA-915+ operation manual.

- 1 Prior to starting operation, make an external examination of the attachment to be sure that there are no mechanical damages. Do not use if the unit is damaged. Call your regional dealer for replacement or repair of the attachment.
- 2 Prepare the RP-91 attachment for operation as shown in the diagram below depending on the using cell:
 - a) The multipath cell;

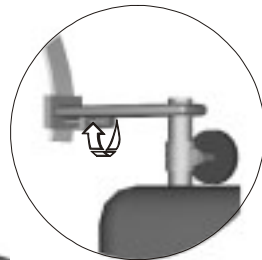
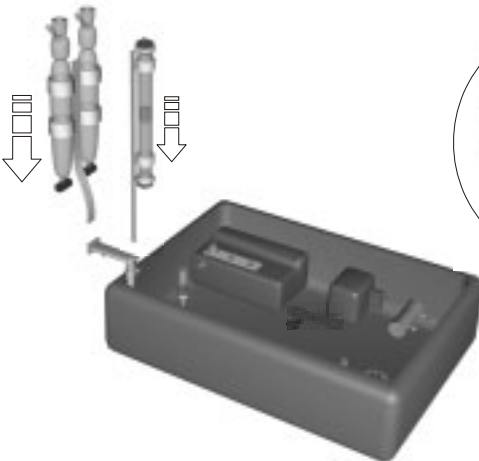


- b) The single path cell.





- Set the holder in the working position.
- Loosen the latch and take the impinger system out of the box.
- Place the impinger system and the rotameter into the holder and fasten in the vertical position.



- Insert the inlet and outlet fittings into the inlet and outlet of the analyzer RA-915+.
 - Connect the analytical cell inlet to the first bubbler and bubblers with each other by the silicone tubes.
 - Connect the analytical cell outlet to the rotameter, absorption filter and the pump by the silicone tubes.
 - Connect the attachment power supply unit to the connector at the attachment and to an a.c. mains.
 - On the toggle switch located at the pumping unit.
- 3 Set the airflow rate using the controls for its smooth and discrete adjustment, checking the flow rate by the rotameter (4 l/min).
 - 4 Check the air-tightness of the air duct by pinching the air tube which connects the analytical cell and the gaimpinger system. If the air flow rate, read out on the rotameter, exceeds 1 l/min, check the tubes and their connections.

Operational procedures

This section contains basic instructions for operation of the RA-915+ analyzer and RP-91 attachment with a PC. For more detailed instructions refer to the "Mercury analyzer RA-915+. User`s manual. Operation with a PC".

- 1 Make sure, that device is on and is connected to PC. Start running the RA-915+ program.
- 2 Select the *LIQUID* mode from the MAIN MENU. The **Liquid analysis** window will appear.
- 3 Go to the **Graph. Liquid analysis** and start scrolling by the RUN button at the tool bar.
- 4 Now the RA-915+ and RP-91 analytical system is ready for operation.

Main operation stages

Preliminary stage

- Introduce the known amount of the reducer (SnCl_2 or NaBH_4) into the bubbler (not less than 3 ml). The deviation of the analytical signal in the window **Graph. Liquid analysis** should not exceed 100. If the deviation level is larger, this means that the reducing solution is contaminated with mercury.



- To purify the reducer, introduce it into the bubbler or into any other vessel and blow pure air through it for 10 min. Upon completion of this procedure, check the purity of the reducing solution once more. Repeat the procedure, if necessary.*
- Go to window **Table. Liquid analysis**. Enter the name of the Table.

Calibration

Obtaining the calibration points

The analyzer can be calibrated in two ways:

- Calibration by a single standard sample (Method A);
- Calibration by several standard samples (Method B).

- 1 Fill in the first line in the column **Description** of the window **Table. Liquid analysis** according to the identification of the standard sample.



To mark a measurement as a blank one, point the mouse cursor at the necessary line and double click the left button of the mouse. Select the BLANK entry from the menu opened. The blank sample marker BLANK will appear in the Description column.

- 2 Introduce the known amount of the reducing solution into the bubbler (in proportion of 2 ml of the reducing solution per 5 ml of the blank sample).
- 3 Using the pipette, take the known amount of the blank sample (5 ml of the sample per 2 ml of the reducing solution).
- 4 Go to the **Integration** window, click the RUN button on the tool bar and immediately introduce the blank sample into the bubbler.
- 5 Upon completion of the analytical peak (or after 60 seconds), click the END button in the **Integration** window. The integration results will appear in the **Integration** window, and they will be automatically recorded into the table.
- 6 Loosen the clamp and pour out the contents of bubbler. Wash the bubbler by distilled water.
- 7 Enter the volume of the blank sample into the column V, ml in the window **Table. Liquid analysis**. Thereby you will obtain one blank measurement point.
- 8 Fill in the last free line in the column Description in the window **Table. Liquid analysis** according to the requirements of description for a standard sample.



To mark a measurement as a standard sample analysis, place the mouse cursor at the necessary line and double click the left button. Select the STANDARD item from the menu opened. The standard sample marker Std_ will appear in the Description column. Enter the mercury concentration in the standard sample directly after double underlining without a space. For example, if you use a standard sample solution with a concentration of 1000 ng/l, the appropriate entry in the table will read: Std_1000.

- 9 Introduce the known amount of the reducing solution into the bubbler (in proportion of 2 ml of the reducing solution per 5 ml of the blank sample).



The deviation of the analytical signal should not exceed 100. If the deviation level is larger, this means that the reducing solution is contaminated with mercury, and it is necessary to purify the solution.

- 10 Using the pipette, take the known amount of the standard sample.
- 11 Go to the **Integration** window, click the RUN button on the tool bar and immediately introduce the standard solution into the bubbler.
- 12 Upon completion of the analytical peak (or after 60 seconds), click the END button in the **Integration** window. The integration results will appear in the **Integration** window, and they will be automatically put into the table.
- 13 Enter the volume of the taken standard sample into column M, ml in window **Table. Liquid analysis**. Thereby you obtain one calibration point.
- 14 To obtain additional calibration points follow the instructions for paragraphs 7 - 12.
 - **Method A:** enter the known, but different amount of the same concentration standard sample into the bubbler.
 - **Method B:** enter the known amount of a different concentration standard sample into the bubbler.



It is recommended to use 3 - 4 calibration points for plotting a calibration graph.

Plotting the calibration graph

- 1 Mark the lines, which are necessary for plotting the calibration graph, in the window **Table. Liquid analysis**.
- 2 Click the CALIBRATION GRAPH button on the tool bar. The **Calibration graph** window will appear, in which a calibration graph will be plotted according to the selected points.



Previous and updated calibration coefficients should not differ much. Otherwise, check the correctness of the calibration and make sure that the standards used are valid.

- 3 Click the APPLY button on the tool bar and then click the EXIT button. The following warning will appear:

Save calibration coefficients ?

Yes No

If you select **Yes**, the given calibration coefficients will be saved permanently. If you select **No**, they will be saved for the current run only. Upon completion of the calibration, you can start measuring.

Measuring procedure

Before the measurements you should treat (digest if needed) the sample to convert all mercury forms in the ionic form and prepare a blank sample by the same technique used.

- 1 Go to the window: **Table. Liquid analysis.**
- 2 Fill in the last free line in the column **Description** in the window **Table. Liquid analysis.**



It is recommended to enter additional data on the sample type in the description column. For example, sample 1 lake Erie, bottom sediments.

- 3 Introduce the known amount of reducing solution into the impinger system.
- 4 Using the pipette, take the known amount of a sample.
- 5 Go to the **Integration** window, click the START button on the tool bar and immediately inject the sample into the impinger system.



To prevent foaming add antifoam agent.

- 6 Upon completion of the analytical peak (or after 60 seconds), click the END button in the **Integration** window. The integration results will appear in the **Integration** window, and they will be automatically recorded into the table.
- 7 Loosen the clamp and pour out the contents of bubbler. Wash the bubbler by distilled water.
- 8 Enter the sample volume in column V, ml in the window **Table. Liquid analysis.**

Calculation of final results

To calculate the final results, select the lines, which you want to process. Click the CALCULATION button on the tool bar. The value of the area/maximum will be evaluated in the concentration units for the selected lines in accordance with the last calibration. Calculation result will be recorded into the Concentration column.



When liquid is found in the second bubbler, stop pumping, remove the liquid and wash by distilled water.

Maintenance

Preventive maintenance of the attachment should be performed at the working place every quarter, and it includes:

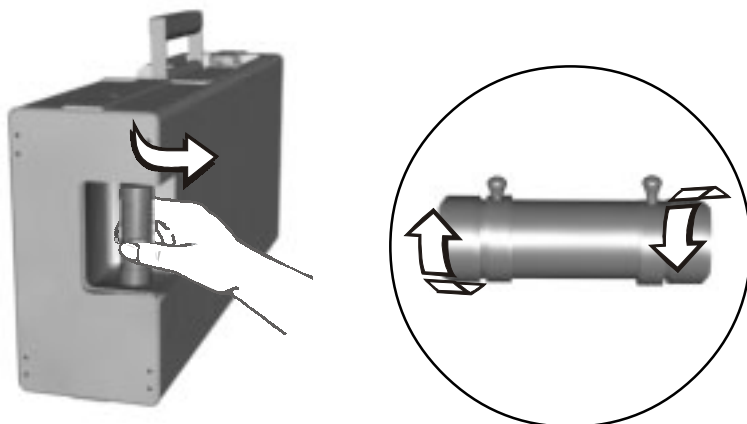
- Checking the air tubes for air-tightness.
- Checking the condition of the electrical cable.
- Dismantling and cleaning of the single-path analytical cell and its quartz windows.



Dismantling and cleaning of the multi-path cell should be performed at the service center or at the manufacturer.

Dismantling and cleaning of the single-path cell

- 1 Remove the single-path cell from the auxiliary compartment of the RA-915+.
- 2 Disconnect the silicone tubes from the cell.
- 3 Unscrew the cell windows.



- 4 Clean quartz windows of the cell by a cotton wool plug wetted with a solvent.
- 5 Clean the cell by a cotton wool plug wetted with a solvent.
- 6 Assembling of the single-path cell is performed in the reverse order.

Storage rules

For short term storage, keep the attachment indoors at an ambient temperature of 5 - 40 °C and relative humidity lower than 80 % at 25 °C. The ambient air should not contain any corrosive impurities. For long-term storage, place the RP-91 attachment into a polyethylene case and seal it hermetically by heating. Store the attachment indoors at an ambient temperature from - 50 °C to + 50 °C and relative humidity lower than 98% at 35 °C.

Appendix

Troubleshooting

The RP-91 attachment should be repaired only at a special service center or at the manufacturer.

However, prior to contacting the service center, check the troubleshooting table below.

Fault symptom	Possible cause	Remedial measure
When the attachment is on, the air pump does not function.	Power cable is out of order.	Repair the power cable.
When the attachment is on, the air pump does not provide a flow rate of 4 l/min.	<ul style="list-style-type: none"> - Air tube connections out of order or there is a leak. - Rotameter floater sealing. - The absorption filter is blocked up. 	<ul style="list-style-type: none"> - Check the tube connections - Dismantle the rotameter and wash it out with ethyl alcohol - Replace the absorption filter with a spare one.
Ingress of liquid into the single-path analytical cel.	Air flow is to high.	Dismantle the single-path analytical cell and clean it up together with its quartz windows.