

RP-91C 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-91C 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-91C 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 structure, and operational procedures.
- Connect the attachment to an RA-915+ analyzer properly.
- Handle the thermal-chamber of the attachment carefully; it contains quartz components.
- To avoid burns, do not operate the attachment without the protective housing.
- Do not remove the attachment from the analyzer before it is properly cooled.



Caution! Atomizer cooling time is about 20 - 30 minutes.

- Do not allow liquids on the case or inside the pump and power supply units of the attachment.
- Do not try to repair the attachment by 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 or if its case is damaged;
 - If quartz parts of the thermal-chamber are cracked or broken;
 - If liquid has gotten inside the pump or power supply unit.
- When measuring mercury concentration, it is necessary to follow safety regulations for operation in chemical laboratories and safety rules for operation with electric appliances.

- Observe all the safety requirements described in the RA-915+ operation manual.
- Observe the following rules during any transportation of the device:
 - To avoid a strong vibration effect, do not place the device on a hard surface (baggage compartment, floor of a vehicle, etc.).
 - Place the device on shock-insulated surfaces (a seat, damping support, etc.).
 - Secure the device on a working surface to prevent its fall or incidental contact with other objects.

Individuals permitted to operate the device are recommended to undergo training at 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 composed of an RA-915+ mercury analyzer and an RP-91C pyrolysis attachment, which essentially enhances the capabilities of the RA-915+ analyzer. The RP-91C attachment enables mercury determination in complex-matrix samples, such as soils, rocks, blood, hair, foodstuff, oil products, etc., using the pyrolysis technique without chemical pretreatment of samples.

After taking the attachment out of the package, make sure that you have everything necessary for its operation. The attachment is packed in two boxes, which contain the following items:

Packing box № 1

1	Attachment pumping unit	1
2	Thermal chamber (atomizer) together with an optical unit and external analytical cell	1
3	Absorption charcoal filter	1
4	Rotameter holder	1
5	Rotameter for measuring air flow rate (0.2-1 l/min)	1
6	Gas tee-branch	1
7	Protective housing	1
8	Operation manual	1

Packing box № 2

1	Attachment power supply unit (for mains)	1
2	Wrench for securing the attachment on the RA-915+ analyzer	1
3	Injection unit (as an assembly)	2
4	Rotameter for measuring air flow rate (1 – 10 l/min)	1
5	Power supply cable	1
6	Silicone tube, m	1
7	Absorption charcoal filter (spare)	1
8	Thermal chamber window (spare)	2
9	Atomizer (spare)	1
10	Sample "boat" (spare)	2

Application

The RP-91C attachment is intended for decomposing a sample and for reducing the mercury from a bound state into an atomic state using the pyrolysis technique. The determination of the mercury released is made by the RA-915+ mercury analyzer.

The RA-915+ mercury analyzer equipped with the RP-91C attachment is used for the determination of the total mercury content in solid and liquid samples for environmental monitoring, technological process control, prospecting ore and oil/natural gas deposits, industrial sanitary inspection, and for scientific research. In most cases, analyses are performed without preliminary sample preparation.

The RP-91C 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 consists of an RA-915+ analyzer, RP-91 attachment for determination of the mercury content in liquid samples using the "cold vapor" technique, and an RP-91C attachment for determination of the mercury content in solid and liquid complex-matrix samples using the pyrolysis technique. Basic analytical characteristics of the system are given in the 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 technological 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-91C attachment

Rate of air flow through the gas line, l/min	from 0.4 to 4.0
Sample weight, mg	from 5 to 300

Main technical data and specifications

Power supply	220/110 V, 50/60 Hz
Power consumption	150 W
Nonstop operation time	8 hours
Size of the pumping unit, mm	340*240*120
Size of the power supply unit, mm	145*150*85
Weight, kg:	
Packing box № 1	5.5
Packing box №2	2.8

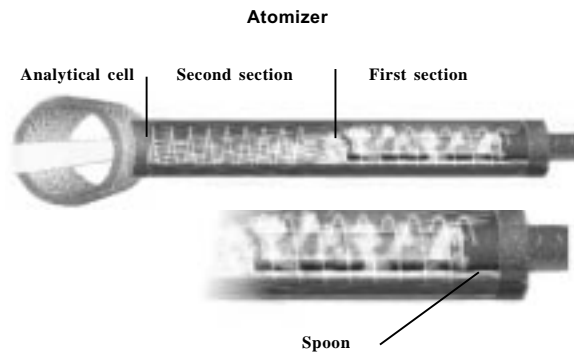
Attachment design and operation

The principle of operation of the RP-91C attachment is based on the thermal destruction of a sample matrix and the reduction of the bound mercury in the sample followed by the determination of the amount of elemental mercury formed thereby.

The atomizer consists of two sections, which are independently heated to 800 °C. A sample is placed into a sample "boat" and is then inserted into the first section of the atomizer.

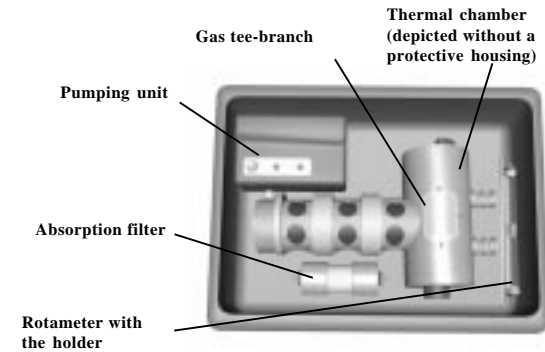
NB All of the mercury compounds dissociate at temperature not higher than 600 °C.

When a sample is inserted, nonvolatile mercury compounds dissociate directly in the first section, and readily volatile compounds, for example, mercury organic compounds, can be evaporated during heating and remain undissociated. Organic substances, which are present in the sample matrix, being heated, produce a large amount of smoke and other compounds, which could give rise to strong background absorption of the mercury resonance radiation. All the gaseous products formed are transported into the second section of the atomizer by a carrier gas (commonly, by air), where all of the residual mercury compounds are completely dissociated, and smoke and other interfering compounds are burnt up to form, mainly, water and carbon dioxide. After the atomizer, the gas flow enters the analytical cell of the attachment. The effect of the residual mixture compounds is eliminated due to the inherent selectivity of the RA-915+ analyzer.

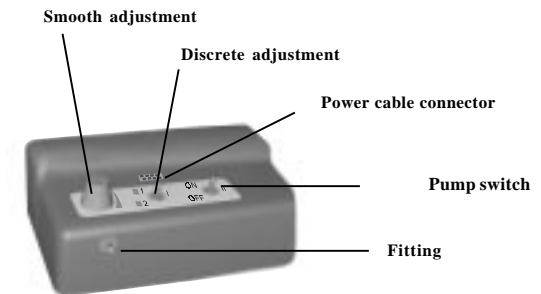


Appearance and functional controls

Appearance



Pumping unit



Smooth adjustment

Smoothly adjusts 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.

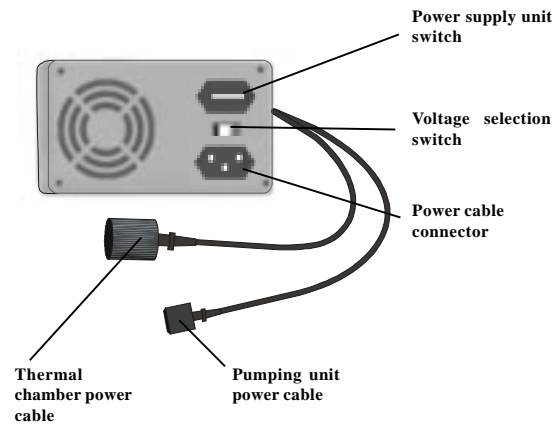
Pump switch

Switches pump on/off.

Fitting

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

Power supply unit



Power cable connector

Connects the power cable.

Power supply unit switch

Switches on and off the power supply unit.

Voltage selection switch

Switches 110 or 220 V a.c.

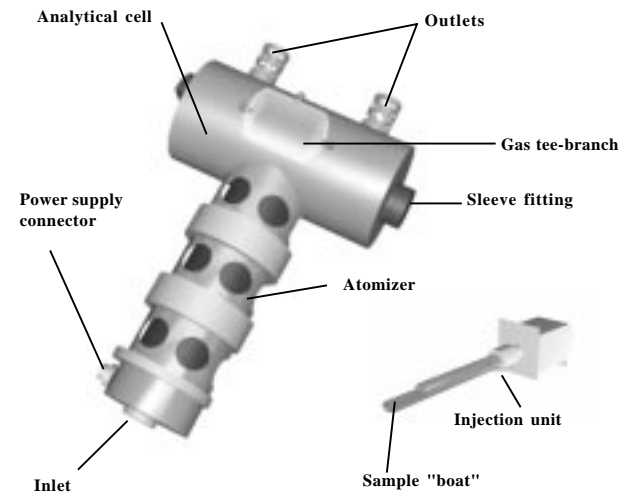
Thermal chamber power cable

Power supply to the thermal chamber.

Pumping unit power cable

Power supply to the pumping unit.

Thermal chamber with an injection unit



Sleeve fitting

Attaches the thermal chamber to an RA-915+ analyzer.

Atomizer

Converts all bound mercury to elemental form.

Inlet

Point for insertion of a sample by the sample "boat" of the injection unit.

Power supply connector

Connection of the power supply cable for operation with a.c. mains.

Analytical cell

Keeps mercury vapor in the analytical beam.

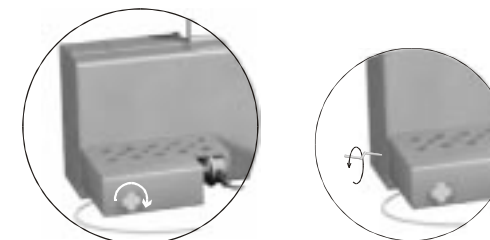
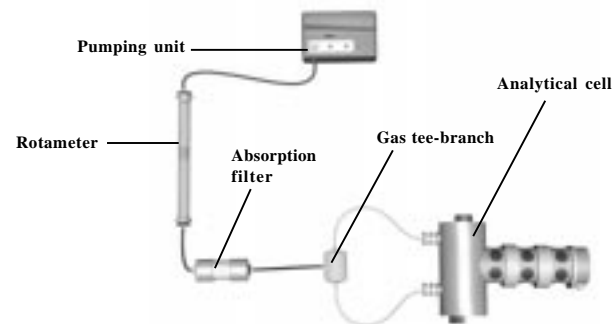
Injection unit/sample "boat"

Introduces a sample in the atomizer.

Pre-operational procedures

This section contains instructions for preparing the RP-91C 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. Never switch on the attachment on your own if your attachment has external damages. Call your regional dealer for replacement or repair of the defective attachment.
- 2 Prepare the RP-91C attachment for operation as shown in the diagram below:



- vertically install the holder with the rotameter in the bracket of the pump box and connect its upper union with the pump using the silicone tube;
- connect the lower union of the rotameter with the absorption filter using silicone tubing;
- connect the power supply unit to the connector on the control unit with the cable;
- connect the power supply unit to the connector on the thermal chamber with the cable (align the red marks);
- mount the protective housing of the thermal chamber;
- mount and secure the thermal chamber in the external analytical cell port of the RA-915+ analyzer using the wrench from the kit;
- connect the outlets of the external analytical cell to the gas tee-branch and the gas tee-branch to the absorption filter using silicon tubes.

- 3 Prepare the RA-915+ analyzer for operation and check its serviceability.
- 4 Set the RA-915+ analyzer's optical bridge handle into position I.
- 5 Connect the power supply unit to an a.c. mains.
- 6 Switch on the attachment power supply unit.
- 7 Set the discrete adjustment of the flow rate in position II on the pumping unit. Using the smooth flow rate adjustment valve, set the air flow rate to 1 l/min, checking it with the rotameter.



The operating mode of the atomizer is attained after 30 - 40 minutes. Check the heating coils of the atomizer visually: the coils should be heated to a red incandescence.

- 8 Check the seal of the gas duct by pinching the silicone tube that connects the gas tee-branch to the absorption filter. If the air flow rate, read out on the rotameter, exceeds 0.2 l/min, check the tubes and their connections.

Operational procedures

This section contains basic instructions for operation of the RA-915+ analyzer and RP-91C 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 the device is on and is connected to a PC. Start running the RA-915+ program.
- 2 Select the *COMPLEX* mode from the MAIN MENU. The **Complex sample analysis** window will appear.
- 3 Go to the **Graph** window on the tool bar **Complex sample analysis** and start scrolling by clicking on the RUN button on the tool bar.
- 4 Click the STATISTICS button on the tool bar. The root-mean-square deviation (RSD) value will appear.
- 5 If the RSD value is less than 15, the RA-915+ and RP-91C analytical system is ready for operation, otherwise it is necessary to stop the operation and to clean the windows of the analytical cell of the attachment (refer to the Maintenance section).

Main operation stages

Preliminary stage

- Start running the RA-915+ program. Select the *COMPLEX* mode from the MAIN MENU. The **Complex sample analysis** window will appear.
- Go to the **Graph** window on the tool bar **Complex sample analysis** and start acquiring data by clicking on the RUN button on the tool bar.
- Go to window: **Table. Complex sample analysis**. Fill in the name of the Table.
- Clean up the sample boat of the injection unit. Go to the **Integration** window, click the START button at the tool bar and immediately insert the empty injection unit into the atomizer for 1 minute and then take it out.

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. Complex sample analysis** according to the identification of the standard sample.




In the description column, the standard sample is marked as Std___. Enter the mercury concentration ($\mu\text{g}/\text{kg}$, ppb) in the standard sample after double clicking on the description column. For example, when using a standard sample with a concentration of 1000 $\mu\text{g}/\text{kg}$, enter Std__1000 in the description column.


- 2 Put the known amount of a standard sample in the injection unit's sample "boat". The accuracy of the analytical balance should be ± 0.5 mg.



When making an analysis, the sample "boat" should be at the room temperature.

- 3 Enter the sample weight in the sample "boat" in the column: **M, mg** of the window: **Table. Complex sample analysis**.
- 4 Go to the **Integration** window, click the START button on the tool bar, and immediately insert the sample "boat" with a sample into the atomizer.
- 5 Upon completion of the atomization peak (or after 30 seconds), click the END button on the tool bar of the **Integration** window. The integration results will appear in the window, and they will be automatically entered into the table. Thereby you will obtain one calibration point. Put out the sample "boat" and remove the rest of the sample.
- 6 To obtain additional calibration points follow the instructions for paragraphs 1 - 5.
 - **Method A:** put the known, but different amount of the same concentration standard sample in the sample "boat".
 - **Method B:** put the known amount of a different concentration standard sample in the sample "boat".

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

 *If warning "LOW RADIATION" appears during an analysis, it may mean that the background absorption either exceeds the permissible value (in this case, the obtained result should be considered as invalid), or lies outside the measurement range (in this case, the sample weight should be reduced).*

Plotting the calibration graph

- 1 Select the lines, which are necessary for plotting the calibration graph, in the window: **Table. Complex sample analysis** (To select: highlight with cursor, shift, down arrow key.)
- 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

- 1 Go to the window: **Table. Complex sample analysis**.
- 2 Fill in the lines in the column **Description** of the window: **Table. Complex sample analysis**.

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

- 3 Put the known amount of a sample in the injection unit.

 *Mass of mercury in the sample should be less than 1 µg.*

- 4 Enter the sample weight in the column: **M, mg** of the window: **Table. Complex sample analysis**.
- 5 Go to the **Integration** window, click the START button on the tool bar, and immediately insert the sample "boat" with the sample into the atomizer.
- 6 Upon completion of the atomization process (or after 30 seconds), click the END button on the tool bar of the **Integration** window. The integration results will appear in the **Integration** window, and they will be automatically put into the table. Put out the sample "boat" and remove the rest of the sample.

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 results will be entered into the **Concentration** column (C, ng/g).

Maintenance

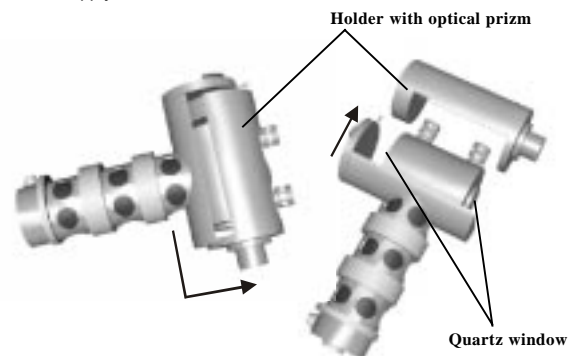
Preventive maintenance of the attachment should be performed at the work place, and it includes:

- Checking the gas path tubing for tightness;
- Checking the condition of the cables.

During quarterly maintenance procedures, or as required, it is necessary to remove and clean the quartz windows of the analytical cell of the attachment.



All the maintenance procedures should be performed with the power supply switched off.



- 1 Disconnect the silicone tubes from the gas tee-branch (do not remove the silicone tubes from the atomizer cell to avoid accidental breakage of the quartz unions of the cell).
- 2 Loosen the white teflon clamp and remove the protective housing.
- 3 Screw out a long screw near the clamp (up to the stop), carefully remove the atomizer with the cell from the base unit and take it off the holder (the holder with an optical prism should remain fixed on the base unit).
- 4 Carefully unscrew the clamps of the quartz windows and remove the windows.
- 5 Clean the quartz windows using cotton wetted with a solvent.
- 6 Clean the quartz cell using cotton wetted with a solvent.

Atomizer assembling

Atomizer reassembly is performed in the reverse order.

Checking the gas path for tight seal

The gas path is checked for seal tightness by pinching the silicone tube, which connects the gas tee-branch to the absorption filter. If the flow rate, read out on the rotameter, exceeds 0.2 l/min, it is necessary to check the tightness of the tubes and their connections.

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-91C 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-91C 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 switch on	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 1 l/min	<ul style="list-style-type: none"> - Air tubes connections out of order - Rotameter floater sealing - The absorption filter of the RP-91C attachment is blocked up 	<ul style="list-style-type: none"> - Check the sealings of the tubes connections and remove the troubles - Dismantle the rotameter and wash it out with ethyl alcohol - Replace the filter by a spare one
When the software start running, warning " LOW RADIATION " is displayed in the Graph window. When the RA-915+ optical switch is set to positions II or III, this warning disappears.	Contamination of the cell windows	Dismantle the atomizer and clean the cell windows.
Atomizer heating coils are not heated	<ul style="list-style-type: none"> - Atomizer power cable is out of order - Thermal chamber heater is out of order 	<ul style="list-style-type: none"> - Repair power cable - Replace the heater by a spare one. To this effect, screw out four screws, which fix the thermal chamber in the atomizer unit, and replace the heater.