Analysis: ‘Radium (Ra-226) in water’

This method describes the determination of radium (Ra-226) in water solutions working without interferences from other radium isotopes or radionuclides. For solid samples special chemical treatment is necessary.

Basics

The method of Ra-226 determination uses the α–radiation from its progenies Rn-222, Po-218 and Po-214 called emanation-method. Progenies are transferred to a scintillation chamber and measured with high efficiency. Chemical concentration of Ra-226 is reached by precipitation from greater volumes.

Method

The method includes the following steps:

1. Precipitation of Ra-226 as barium(lead)-sulfate by addition of barium and lead carrier solution and sulphuric acid.
2. Dissolve of the precipitation by forming a complex compound and transfer of solution to a emanation tube.
3. Build-up phase of Rn-222
4. Transfer of the Rn-222 to a scintillation chamber under the use of a emanation stand with a following delay time to reach equilibrium between Rn-222 and its progenies.
5. Measurement of the scintillation chamber

Method description

Sampling

To avoid adsorption the sample should have pH = 1 by adding 10 ml 14 mol/l nitric acid per liter sample. In laboratory the samples are filtrated.

Sample treatment

1. Precipitation

To a 1 liter sample the following chemicals are added:

- 5 ml citric acid (1 mol/l)
- about 10 drops methylen-red indicator
- concentrated ammonia solution until indicator turns yellow
- 3 ml barium carrier (barium nitrate 0,043 mol/l)
- 2 ml lead carrier (lead nitrate 0,48 mol/l)

The solution is heated to boiling. From the hot solution the sulfates are precipitated by adding 2,5 ml sulphuric acid (9 mol/l). Wait overnight and decant from the solution. The precipitation is to be washed with distilled water and centrifuged two times. Leave the precipitation in the centrifuge glass.

2. Dissolve

The precipitation is dissolved with 5 ml Na_4EDTA (0,5 mol/l) by use of a water bath in the heat and quantitatively transferred to a de-emanation tube. The end volume of the solution in the tube is about 25 ml.

3. Buildup phase

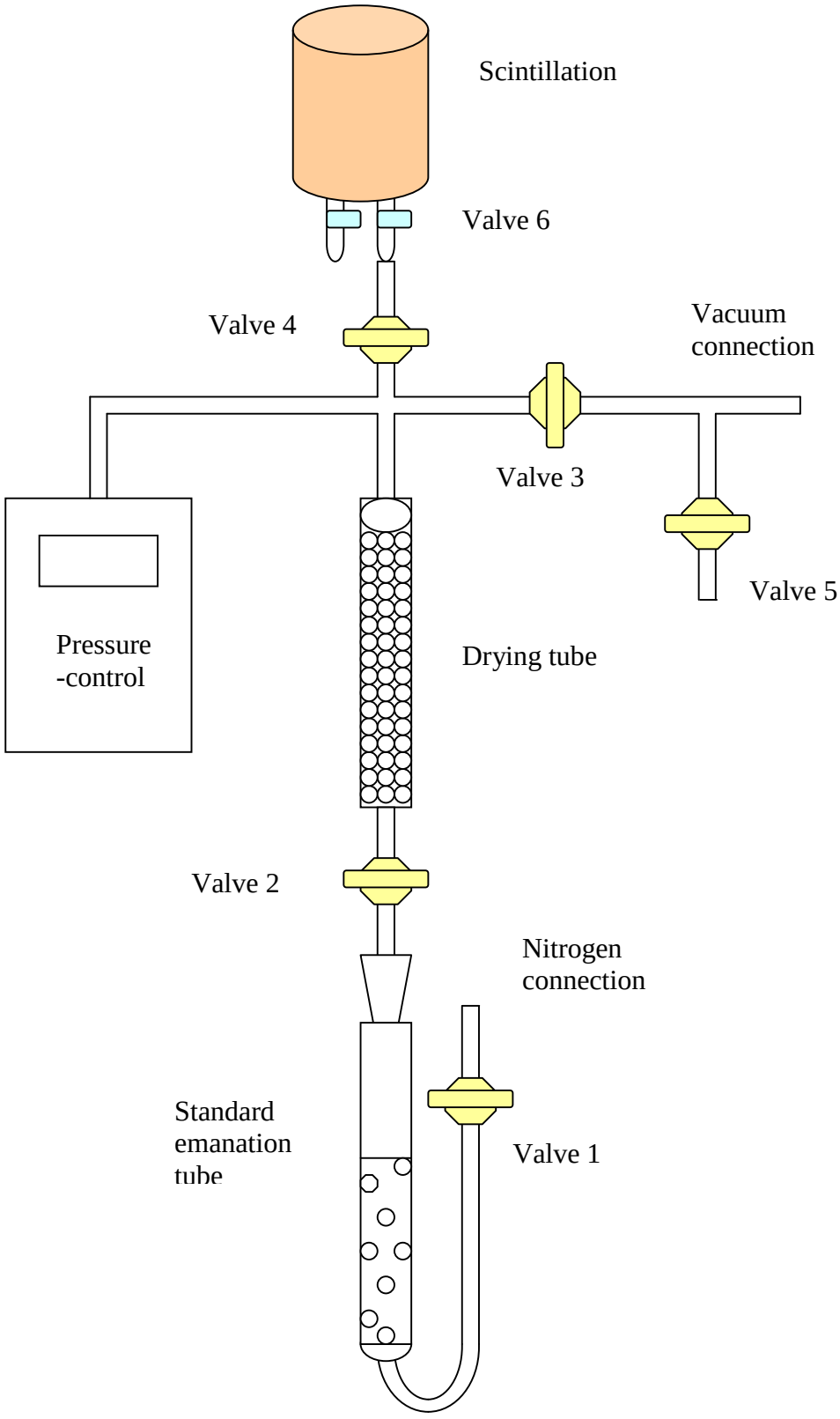
The solution is de-emanated completely from Rn-222 by bubbling with nitrogen for about 5 min. At the end the tube is set under vacuum with a pump. The end-point of the procedure is the date/time ‘First de-emanation after sample preparation’. This is equal to the start point of Rn-222 buildup. After 7 days about 70 %, after 14 days about 90 % equilibrium are reached.

4. Transfer of Rn-222

Attach a scintillation chamber as well as the sample tube to the emanation-stand. With the vacuum pump on open valves 3, 4, 6. When the control instrument shows constant pressure close valve 3. Slowly open valve 2 until pressure equalisation visualised by the instrument. Pressure value will reach about 100 mbar. Very carefully open valve 1 and let nitrogen bubble through the liquid. Useful is a needle valve for slow gas transport. Prevent the drying tube from contamination with foam. At pressure equalisation close all valves with valve 6 being the first. The end-point of the procedure is the date/time ‘De-emanation into scintillation chamber’.

5. Measurement

Measure the chamber by use of the analysis ‘Ra-226 in liquid’ of the counter program. Ensure that a well nitrogen flushed chamber is used for analysis. Before each analysis the actual chamber background can be determined by an overnight measurement.


Scintillation
Valve 6
Valve 4
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connection
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