Automation during the concentration of water samples for analysis

Category: Rotary Evaporator
Whitepaper published December 2020

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Automation during the concentration of radionuclides and heavy metals in water samples

Water is our most important beverage and represents the major uptake route (incorporation route) for chemotoxic and radiotoxic substances. For this reason, water constituents are monitored extensively, which requires high efforts. Radionuclides and heavy metals are analyzed in surface and drinking water, groundwater and wastewater, as well as in rainwater, primarily as part of nuclear power plant monitoring. The activity concentrations of the β-emitters tritium, ^{14}C and ^{90}Sr, the β-emitting isotopes of uranium, plutonium, americium and curium and of γ-emitters such as ^{137}Cs or ^{60}Co, are monitored continuously. The detection limits to be maintained during their analysis frequently require concentration, often performed after a radiochemical purification, to suppress interference in the various measuring techniques, whether radiometry, mass spectrometry or some other technique. Radionuclides are determined in aqueous samples for research purposes as well. The detection limit requirements are usually even higher in this case.

Objective: Automating the concentration of water samples using a large-scale rotary evaporator with an automatic module

Evaporation is a suitable method for concentrating radionuclides and heavy metals from large volumes of aqueous samples. Alternatively, sorbents or flocculating agents can be used. These are only marginally suitable, however, when several chemically very different radionuclides such as ^{137}Cs, ^{90}Sr and plutonium isotopes, have to be analyzed together in a sample. Gentle concentration by evaporation in open vessels is a proven process, but no longer practicable and time-intensive for sample volumes greater than a few liters. Use of a largescale rotary evaporator is efficient, starting at sample volumes of about 10 liters. An additional advantage is that simple distillation is sufficient as purification for tritium, which is present in aqueous samples as HTO (tritiated water) and therefore the distillate accumulating in the condenser of a rotary evaporator is used directly for the subsequent activity measurement (in the case of tritium, using liquid scintillation counting).

Conventional evaporators have the disadvantage that, depending on the volume, the evaporation flask must be refilled manually at relatively short time intervals. As a result, personnel must be transferred permanently during normal work time, which in turn leads to interruption of other tasks. Outside of operating times, conventional rotary evaporators must be shut down. The evaporation of large volumes of samples is therefore greatly extended.

The use of an automated Hei-VAP Industrial large-scale evaporator with the automatic module Distimatic from Heidolph, can remove the limitations of conventional rotary evaporators. The sample concentrate level in the evaporation flask is determined via a capacitive fill-level sensor. When the sensor detects too little media, water is automatically refilled through a PTFE tube from a sample storage tank. When the storage tank is empty, this is detected by the automatic module and operation is stopped. The system is switched off automatically. For this reason, the evaporator can also be run overnight. With suitably dimensioned storage tanks, operation on the weekend is also possible.

In the present case the customer wished to replace the conventional large-scale evaporator setup in use with the automated system, in order to increase the efficiency.

Method and results: Successful automation of rainwater evaporation with 4,6 l/h

The goal was to evaporate samples of up to 70 liters of rainwater to a quantity of about 1 liter within one day. The pH of the rainwater samples was initially adjusted to pH 1 with nitric acid and pre-filtered with a 63 µm filter before the evaporation process.

Fig. 1 shows the test setup. The Distimatic Industrial Platinum 8 Package was used for this test. With the recirculation chiller set to 5 °C, rotational speed of 115 rpm and a vacuum value of 30 mbar at 65 °C heating bath temperature a distillation rate of 4,6 l/h could be achieved. Fig. 2 shows the rainwater concentrate at the end of the process.

The customer's requirements were met – the volume of sample could even be increased. About one technician work hour is saved per workday in comparison with previously operated conventional rotary evaporators. Because the device can be run overnight, the entire evaporation process is completed within a much shorter time than with the previously used conventional largescale evaporator.

Fig. 1: Hei-VAP Industrial with Distimatic test setup

Fig. 2: Concentrated rainwater

Conclusion: Heidolph's Distimatic Industrial Platinum 8 Package meets the challenge

The example shows that Heidolph products enable the automation of rainwater evaporation. The efficiency could be increased significantly by establishing a continuous workflow over night and weekends.

All in all, the same results can therefore be achieved with a rather small number of personnel within a rather short time as with a conventional evaporator with an otherwise identical technical design.

For any technical questions, application support etc. please contact us under:

EUROPE / AFRICA / MIDDLE EAST / ASIA PACIFIC / SOUTH AMERICA
Heidolph Instruments GmbH & Co. KG
Walpersdorfer Str. 12
D-91126 Schwabach
Phone: +49 9122 9920-0
Fax: +49 9122 9920-0

sales@heidolph.de
www.heidolph.com

NORTH AMERICA
Heidolph North America
1235 N Mittel Blvd. Suite B
Wood Dale IL 60191

Phone: +1 224-265-9600
Fax: +1 224 265-9611

custserv@heidolph.com
www.heidolphna.com

heidolph
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