

Automation during the Concentration of Radionuclides in Water Samples

Radionuclides are routinely analyzed in surface and groundwater, drinking, and other types of water. Concentration must usually be carried out beforehand. Concentration by evaporation is a simple but proven and reliable method in this case. The use of a fully automated rotary evaporator is very useful especially with large sample volumes.

Water is our most important "food" and represents a major uptake route (incorporation route) for chemotoxic and radiotoxic substances. For this reason, water constituents are monitored extensively and with high effort. Radionuclides are analyzed in surface and drinking water, groundwater and wastewater, as well as in rainwater and seawater primarily as part of nuclear power plant monitoring. The activity concentrations of the beta-emitters, tritium, ^{14}C , and ^{90}Sr , the α -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.

Concentration by evaporation is a suitable method for concentrating radionuclides 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 must 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 large-scale 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, 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.

Use of the automated large-scale evaporator Laborata 20 control automatic from Heidolph can remove these limitations of conventional rotary evaporators. The sample concentrate level in the evaporation flask is determined indirectly via an integrated weight sensor in the evaporator's drive head. When a minimum value, easily set by the user, is reached, water is automatically refilled through a PTFE tube from a sample storage tank until a preset maximum level is reached. If the storage tank is empty, this is detected by the evaporator and operation is stopped. For this reason, the evaporator can also be run overnight. With suitably dimensioned storage tanks, operation on the weekend is also possible. 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.

In the case of water samples, a heating bath temperature of 80 °C at an operating pressure of about 130 mbar has proven successful. The daily throughput of the evaporator under these conditions is about 50 L.

The best optimization in the author's field was achieved during the concentration of monthly rainwater samples, which are analyzed continuously for their radionuclide content in relation to

radioecological problems and the volumes of which can reach several hundred liters in months of heavy precipitation. (These large sample volumes are necessary for detecting plutonium isotopes and ⁹⁰Sr, which originate predominantly from the fallout of aboveground nuclear weapons testing in the early 1960s.) About 1 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 large-scale evaporator. The pH of the rainwater samples is initially adjusted to pH 1 with nitric acid. Due to the concentration process, the nitric acid concentration in the evaporation flask rises to 65% in the final concentration phase. Because of the chemical-resistant design of the evaporator, especially of the two-stage vacuum pump, however, the evaporator can be operated under these operating conditions as well.

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