Applied Radiation and Isotopes xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Applied Radiation and Isotopes

journal homepage: www.elsevier.com/locate/apradiso

Qualification of a precision pattern dispenser

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HIGHLIGHTS

- A precision dispenser was developed in order to automate source preparation.
- Flexible to dispense pre-set amounts of liquid in different kinds of sample holders.
- Different spiking patterns (size, amount per spot) can be dispensed on air filters.
- Qualification proved repeatability < 1 %, trueness < 2% in range of 20 μL to 4 mL.
- A series of 1100 sources was produced with excellent repeatability and trueness.

ARTICLE INFO

Keywords:
Source preparation
Automatic dispensing
Spiking
Qualification

ABSTRACT

In order to automate sample preparation processes, a precision pattern dispenser was designed to reproducibly dispense radioactive solutions at pre-defined positions. It is composed of an automatic liquid sample handling unit coupled to an XYZ table. Qualification tests were conducted to evaluate the performance of the instrument and to assess the compliance with the requirements, in particular trueness (< 2%) and repeatability (< 1%). The instrument allows preparing sources in different source holders and on air filters, in a fast and accurate way.

1. Introduction ensure

Many laboratories prepare different types of radioactive sources that can be used as reference materials or as samples in intercomparisons or proficiency tests (Dean et al., 2017; Lourenço et al., 2014). The reference materials can be used to calibrate measurement set-ups and check their performance (Larijani et al., 2017; Mejuto et al., 2014; Shakhashiro et al., 2012). Intercomparisons and proficiency tests are excellent tools to assess the analytical performance of laboratories in general (ISO/IEC 17025, 2005) and environmental monitoring laboratories in particular (Benedik, 2013; Wershofen et al., 2008). Specifically for air monitoring laboratories, air filters can be prepared by spiking blank filters with radioactive solutions using different source preparation techniques (Monsanglant-Louvet et al., 2015; Ceccatelli et al., 2010).

The European Commission Joint Research Centre (JRC) is on a regular basis preparing radioactive sources that are used as calibration standards or reference sources in different applications and is organising interlaboratory comparisons (Wätjen et al., 2008; Sobiech-Matura et al., 2017) for laboratories monitoring radioactivity in the environment (Wätjen et al., 2010; Merešová et al., 2014; Jobbágy et al., 2014 ; 2015), e.g. by measurement of air filters (Wätjen et al., 2006; Máté et al., 2016). Often a series of samples, having the same composition and analyte content, need to be produced for these purposes. Good source preparation is a key element to come to successful results. In order to make this process more efficient and accurate, an instrument to dispense radioactive solutions, called precision pattern dispenser, was developed and installed at JRC-Geel.

The most important requirement of the instrument is to accurately dispense predefined amounts of radioactive solutions in an automated way at certain predefined positions. These known amounts can be dispensed on different sizes and shapes of blank filters or in ampoules or custom-made source holders (Van Ammel et al., 2016a). The instrument is described and the results of the qualification tests are presented.

2. The instrument

The precision pattern dispenser is a modular instrument built by

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http://dx.doi.org/10.1016/j.apradiso.2017.06.019
Received 10 March 2017; Received in revised form 9 June 2017; Accepted 9 June 2017
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Fig. 1. Ampoules filling station of the automatic dispensing robot, with different items suspended on the horizontal arm (solvent reservoir, waste vial and wash vial, support for the source tray containing 5 mL ampoules).

SampleQ, Belgium. The core of the instrument is a liquid handling unit. It is a commercially available automatic dispensing system PAL HTC-XT, produced by CTC Analytics AG, Switzerland controlled by Chronos software (LEAP Technologies, USA). The handling unit (Fig. 1) has a movable tower in which syringes of different volumes (25 µL to 5 mL) can be mounted. The amount of liquid to be dispensed can be chosen within the volume range of 20 µL to 5 mL. The movements of the tower are executed smoothly to avoid any spillage of liquid. The tower of the dispenser can move over different modules that are suspended on the main horizontal arm. These modules can include: solvent reservoirs (100 mL), waste and wash vials (20 mL), supports in which source trays that can contain different types of ampoules or other source holders can be mounted. In the current set-up, one module containing a waste and a wash vial and two modules for three solvent reservoirs each are suspended. All reservoirs and vials used are closed with septa to prevent evaporation before, during and after dispensing. The septa can be easily replaced, if deemed necessary.

The source trays are custom-made to hold the different types of source holders. They fit into the tray support module which is suspended on the main horizontal arm. Parameters influencing the dispensing process can be set in the control panel of the dispensing system in order to obtain good dispensing. Some of these settings need to be adapted according to the syringe that is used.

The Chronos software reads in the settings from the control panel and controls all the movements of the tower between the different items suspended on the arm as well as the movements of the plunger of the syringe it contains. Some of the default settings in the control panel can still be adjusted in the Chronos software to dispense the liquid in a smooth and accurate way.

As one of the applications will be the spiking of air filters, a custom-made XYZ table to support the filters is coupled to the dispensing unit. The combination of both makes it possible to dispense pre-set amounts of liquid, specified as volumes or masses, onto pre-defined positions on supported filters.

The XYZ table serves to support and fix the filters (with maximum dimensions of 0.60 m by 0.60 m), on which solution can be dispensed. In order to support the filters well, metallic strings are stretched over the whole area of the table every 5 cm. The filter can be fixed and stretched using metallic bars of different length, which in turn are clamped on the magnetic bars positioned on the outer side of the filter (Fig. 2). The filter can be centred on the table using the marks on the bottom of the table. Under the metallic strings an adsorbing paper can be introduced to collect eventual spilling.

To steer the XYZ table and the dispensing unit together, software called Protocol Generator was developed by Flamac, a division of SIM, Belgium. In the software a model of a filter can be created with a certain

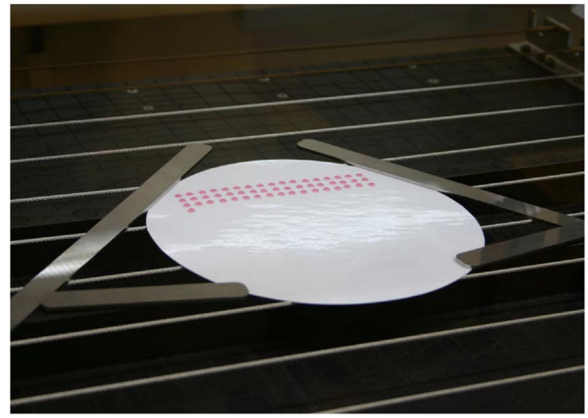

Fig. 2. The XYZ-table with mounted filter of 150 mm diameter.

width and length for a rectangular filter or a diameter for a circular filter. Also the thickness of the filter can be introduced. A dispensing pattern is created in the active area, being smaller than the filter size. Two kinds of patterns, rectangular or circular, can be generated independent of the shape of the filter. The pattern is a set of predefined positions where a chosen amount of liquid has to be dispensed by the device. Different sizes of filters together with their active area as well as the patterns to be dispensed and combinations of filters and patterns can be stored.

Once a combination of a filter and a dispensing pattern, called protocol, is generated in the software, it can be sent to the Chronos software (controlling the dispensing robot) and the motor controlling the XYZ table in order to execute the dispensing according to the inserted protocol. All relevant data of instrument settings – dispensing positions, volumes etc. – are stored electronically and can be easily retrieved.

The dispensed liquids are often solutions of hydrochloric or nitric acid, therefore all contact materials are resistant to acidic solutions. This contact is limited to the used reservoirs, vials and syringes. Only these parts need to be replaced when the solution to be dispensed is changed. Another advantage of this method of dispensing is the small dead volume contained in the needle of the syringe used, resulting in a relatively small amount of waste generated compared to other liquid handling systems.

The whole mechanical set-up, i.e. the dispensing robot and the XYZ table, is placed in a protective Plexiglas enclosure. The enclosure protects the set-up from external impacts and protects the operator during the mechanical movements, but allows a visual surveillance during operation. The enclosure can be opened from the front so that all parts are accessible when items like syringes, reservoirs, vials, source holders or filters need to be changed or when service is needed.

3. Qualification procedure

The properties described in the previous section were the subject of the qualification. Two key performance parameters examined were the repeatability (precision under repeatability conditions), and trueness (deviation of the actually dispensed amount of liquid from the pre-set amount programmed). For both criteria upper limits were required. The repeatability for any dispensed volume within the volume range shall be better than 1%, whereas the trueness, the bias between the set volume and test results, shall be better than 2%.

Both parameters were examined using six different syringes with a nominal volume of 50 µL, 100 µL, 250 µL, 500 µL and 1 mL and 5 mL. For each syringe, tests were carried out by dispensing 20%, 40%, 60% and 80% of its nominal volume. For each tested volume, ten new, empty 5 mL glass ampoules were tarred. The pre-set volume of demineralised water was dispensed once into each of the ampoules. As

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