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A LabVIEW®-Based Software for the control of the AUTORAD Platform:

A fully automated multi-sequential flow injection analysis Lab-on-Valve (MSFIA-LOV) system for radiochemical analysis

Donato Barbesi, Víctor Vicente Vilas, Sylvain Millet, Miguel Sandow, Jean-Yves Colle, Laura Aldave de las Heras

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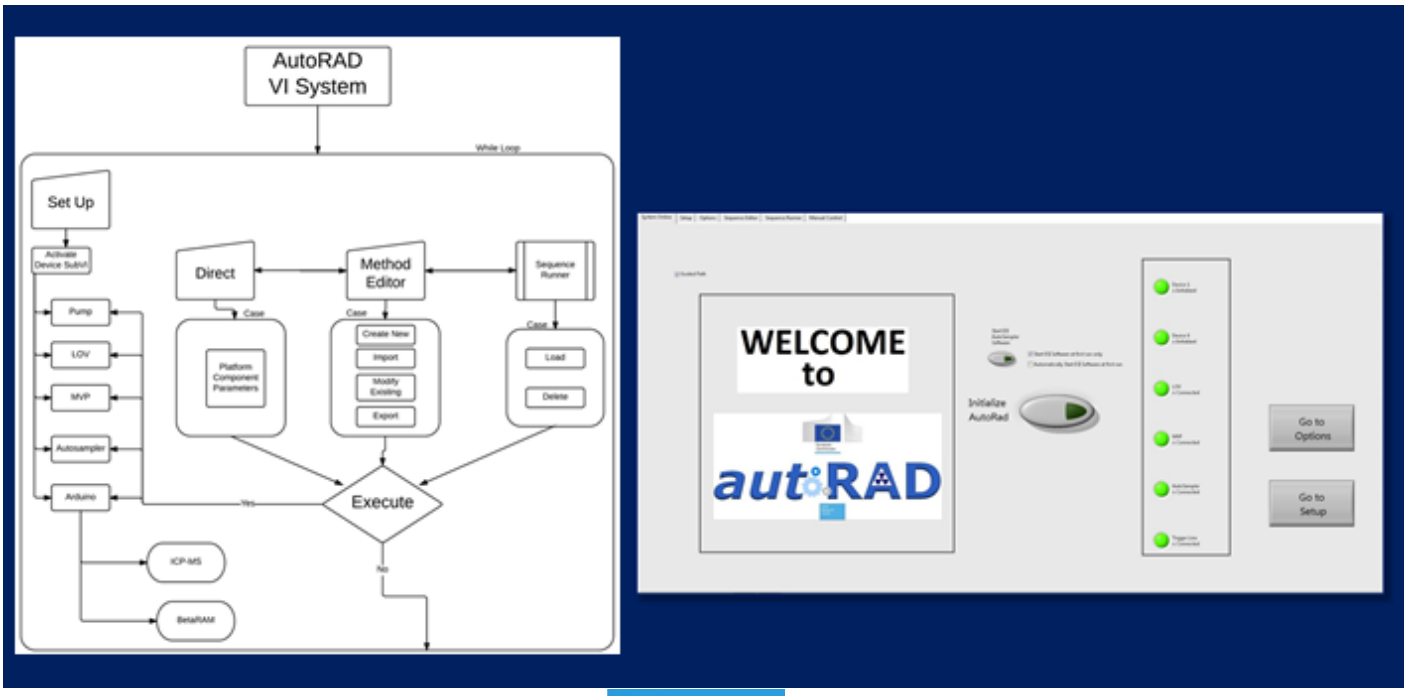

Joint Research Centre

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Abstract

A LabVIEW®-Based Software for the control of a fully automated multi-sequential flow injection analysis Lab-on-Valve (MSFIA-LOV) platform performing radiochemical analysis is reported. The analytical platform interfaces an Arduino®-based device triggering multiple detectors providing a flexible and fit for purpose choice of detection system. The AUTORAD platform is composed of a multisyringe pump, a Lab-on-Valve (LOV) manifold, a commutation valve, an autosampler and two detectors. The different analytical devices are interfaced to the PC running LabVIEW®/VIs using USB and RS232 data connection, both for sending commands and receiving confirmation or error responses. The AUTORAD platform has been successfully applied for the chemical separation and determination of strontium in certified reference materials.

1. Introduction

Integrated and automated analytical platforms capable of performing different steps of the analytical procedure are a current trend in analytical chemistry resulting in increased reproducibility and repeatability. During research and development phase changes in the system hardware are common requiring flexibility for the automation of such platforms [1].

In the nuclear field, the development of automated multi-purpose platforms to performing safe and fast determination of radionuclides represent a significant improvement when monitoring operations on-site or in situ are more effective than traditional sampling coupled to manual laboratory analyses. Flow analysis techniques are a major step forward in automating radiochemistry procedures that incorporate extraction and pre-concentration steps, allowing the development of rapid, sensitive and selective methods for the determination of hard to detect radionuclides such as ^{90}Sr, ^{99}Tc, ^{129}I and alpha emitters with high reproducibility. Even though the development and application of automated methods based on flow techniques present a great advance in the nuclear field, most of them are only partially automated and only few fully automated systems incorporate on-line detectors.

LabVIEW® is a software tool used to develop sophisticated systems both for the industry and research [2-6] using its intuitive graphical block diagram, which resembles a wired flow chart [7]. Typically different subVIs, each of them performing specific duties, are wired together to perform complex operations. Unlike text based programming languages which need long development and contribution from external experts for maintenance or expansion LabVIEW® allows easy hand-over to subsequent users.

In recent years several published papers describe LabVIEW®-based software for the automation of specific analytical systems such as FIA [8], SIA [9] or CE [1]. Applications include automatic liquid handling, treatment, separation and, in some cases, detection of analyte of interest. Lab-on-Valve (LOV) devices are programmable, flow-based platforms with different coupling modes and high versatility [10]. Such devices allow the automatic separation and pre-concentration of the analytes prior to detection thus increasing reproducibility and repeatability [11, 12].

The AUTORAD platform is based on a LOV integrated into a multi-syringe flow injection analysis (MSFIA) system enhanced with bed injection (BI), and interfacing an Arduino®-based device triggering multiple detectors providing a flexible and fit for purpose choice of detection systems [13]. The choice of the detection technique depends on the analyte properties and desired feature of the system. The developed platform and software is able to work with ICP-MS and Radio flow detectors, thus improving the flexibility and allowing direct comparison between the two detectors. Drawbacks of a detection system, such as matrix effect or interferences, can be mitigated by using both detectors increasing the versatility of the platform.

Radio flow detection is a versatile and high sensitive technique suitable for different applications [14-17]. The radioactivity detected is directly proportional to the concentration of the analyte. Online ß detection allows full automation of the proposed system allowing detection of beta emitters over a large working range. ICP-MS has shown applicability for quick and accurate determination of long-lived and environmental relevant radionuclides. The working principles are described elsewhere [17-19].

This report describes the development and implementation of LabVIEW®-Based software for the automatic control of the AUTORAD platform. The possibilities of the resulting control platform are demonstrated by successfully applying for the chemical separation and determination of strontium in certified reference materials.

2. Configuration of the hardware

The Lab-on-Valve (LOV) was fabricated in house from methacrylate and includes eight integrated 16 mm length microchannels. Seven microchannels with 1.5 mm i.d. and the column channel having 3.2 mm i.d.. The system is mounted on a Cheminert Selection Valve C25 (Valco Instruments Co. Inc.). A schematic description of the AUTORAD platform is shown in Figure 1 [13].

The central port of the valve in the system is connected to a 10 mL glass syringe (Hamilton Company Inc. Nevada, USA) via a 10 mL holding coil. The syringe is mounted in a MicroLab 600 syringe pump (Hamilton Company Inc. Nevada, USA). The syringe head has a three-way valve enabling multicommutation schemes (in: reservoir; out: system). The extraction resin (Sr-Resin, particle size 100-150 µm, 36 mg) is located at channel 5 of the LOV, a glass fiber filter contains the resin within the LOV channel allowing solution flow. An autosampler (ESI, Omaha, Nebraska, USA) is connected to port 8 to allow quick processing of real samples. The peripheral port configuration was: port 7, eluent; port 1, resin suspension; port 2, washing solution; port 4, waste (Figure 1). Port 5 is connected to a commutation valve (Hamilton Company Inc. Nevada, USA) which drives the flow coming from the LOV system in the desired way (on: detector; off: waste; flush: resin exchange).

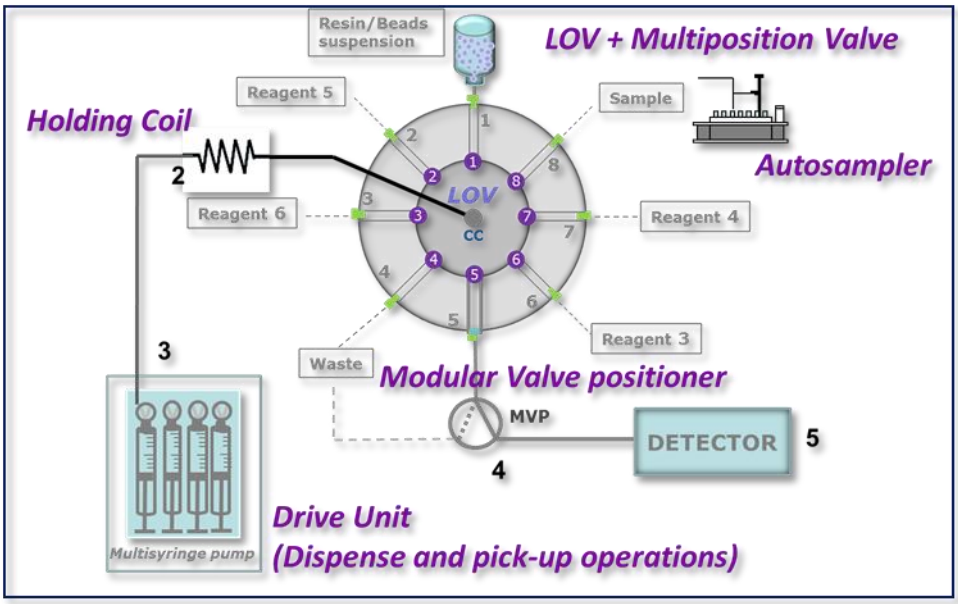

Figure 1: Peripheral port configuration

ICP-MS measurements were carried out in a double focusing sector field ICP-MS (Element 2, Thermo Finnigan MAT GmbH, Bremen, Germany). Element 2 is equipped with a self-aspiring micro-concentric nebulizer, a PC3 compact Peltier Cooled inlet system which incorporates the ESI cyclonic spray chamber, a Fassel torch and a 27 MHz generator. Isotopic measurements were carried out using only electric scanning (E-scanning), at low (M/ΔM = 300) resolution settings. The instrument is placed in a clean room facility class 1000. Instrumental settings and optimised measurement parameters for Sr isotopes are given in Table 1. Typical sensitivity at low resolution is 1.2-1.5x10^6 cps per µg L^{-1} of strontium.

Online radioactivity detection was achieved using a ß-RAM 5 flow detector (LabLogic Systems Ltd, Sheffield, UK). This model possesses an internal cocktail pump and can be fitted with flow cells of various types and sizes. All experiments within this work were

made using a 500 µL coiled Teflon flow cell placed in a fixed geometry between two photomultiplier tubes. Samples coming from the LOV are directed to the detector and mixed with the LSC Cocktail. The cocktail flow rate was set to 2 mL/min. The detector counting parameters were controlled using LAURA 4.2.8 software (LabLogic Systems Ltd, Sheffield, UK) run on a desktop PC and connected to the detector via USB. Selection and triggering of the detector was performed via the developed software and an Arduino® microprocessor[20] .

For the preparation of all solutions, high-purity water (18.2 MΩ cm) from a Miliq-Element system designed for ultratrace analysis (Millipore, Milford, MA) was used. Nitric acid, suprapur grade from Merck (Darmstadt, Germany), was purified using a quartz sub-boiling distillation unit. Both the water purification system and the sub-boiling distillation unit were operated in a clean room. Natural element standards were obtained from CPI international (Amsterdam, The Netherlands) as 1000 µg ml-1 stock standard solutions and a 86Sr standard solution of 10 mg L^{-1} (ESI, Omaha, Nebraska, USA), were used. A carrier-free radiostrontium standard solution containing 2000 Bq ml^{-1} was purchased from Eckert & Ziegler. Stock and working standards were prepared with Milli-Q deionized water or HNO_3 1%.

Table 1: HR-ICP-MS instrument settings and scanning conditions

Sample introduction system and instrumental operating conditions	
Nebuliser	0.1 ml min^{-1}, self-aspiration mode
Spray chamber	PC^3 Peltier cooler
Sampling cone	Nickel
Skimmer cone	Nickel
Rf Power	1250
Plasma gas flow rate (L/min)	15.5
Auxiliary gas flow rate (L/min)	0.8
Nebuliser gas flow rate (L/min)	1.0-1.2
Measurement conditions	
Resolution (10 % valley definition)	Low, M/ΔM = 300
Acquisition mode	E-Scan
Magnet settling time (s)	0.300/0.0200
Magnet mass	82.914
Mass Range (amu)	
^{86}Sr	85.766-86.052
^{87}Sr	86.764 – 87.053
^{90}Sr	89.757 – 90.057

Table 1: cont…

Search window (%)	100
Integration window (%)	80
Sample time (s)	0.01
Sample per peak	20
Segment duration	0.2
Detection mode	EScan
Run & passes	160 x 1
Dead time correction (ns)	12

3. Programming-LabVIEW Software Interface

Since the programme has to control the different hardware devices maintaining a user-friendly interface, all the actions required from the user are selected from drop-down menus; thus avoiding typing errors. Moreover all subVIs and devices must be coordinated to perform all the analytical steps before determination. This section shows the programming description of the main blocks, figures indicate operation performed by each subVI. The schematic flow chart of the AUTORAD VI system is represented in Figure 2.

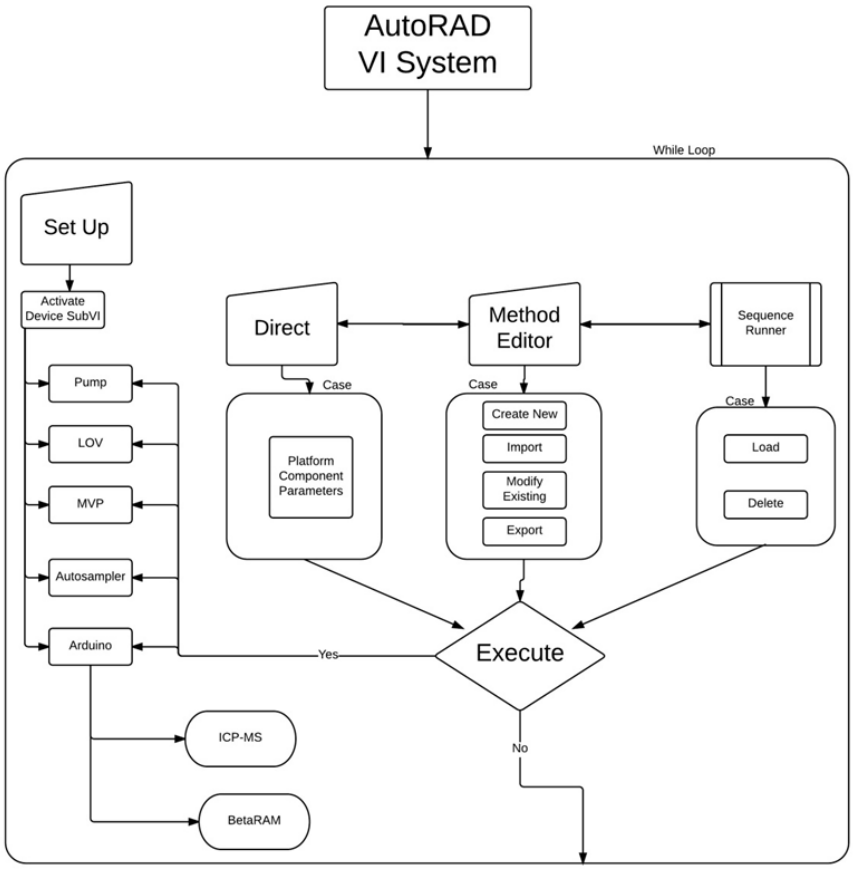

Figure 2: Schematic Flow Chart of the AUTORAD VI System

3.1 Operational Software

The software has been written with the SubVI function "Tab Control", which allows the user to access different features without exiting the program. As in the case structure, tab structures execute actions depending on the user input. The welcome tab serves as a crossroad and allows the initialization of the different devices. Ready devices are shown with a green light. In the set-up tab, selection of the devices to be used regarding the desired procedure can be performed.

As soon as the software is running, the Hamilton® Connect_0.vi (Hamilton Company Inc. Nevada, USA) sets up an IP connection between the computer and the syringe pumps, which are wired together over a Daisy Chain. To be able to communicate with the devices (LOV, the autosampler, the commutation valve as well as the in-house build Trigger Line), VISA Configure Serial Port Node initializes a serial connection. Interfacing with the devices occurs via RS232 or USB, configured with company specific values. The Master loop then waits for further user action. Figure 3 show the software welcome tab.

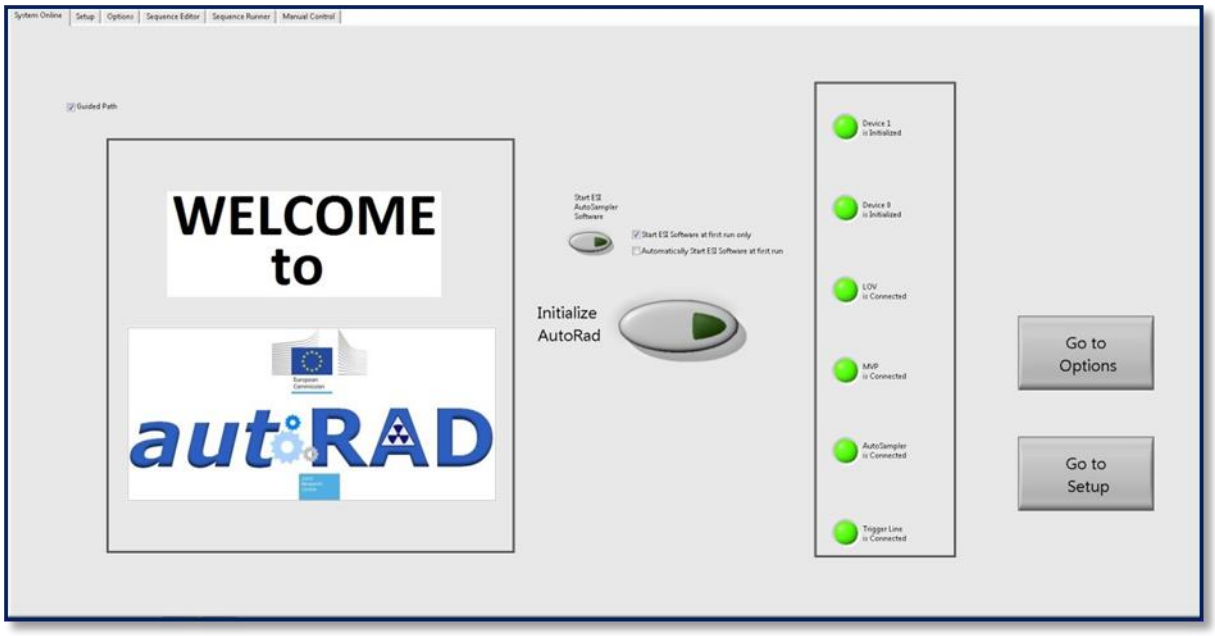

Figure 3: AUTORAD Main window graphical user interface, welcome menu

3.2 Programming for daily operation

Depending on the specific requirements, the user is able to select three different tabs "Direct", "Method Editor" or "Sequence Runner" corresponding to software cases. In the Editor tab, the user can create, modify or run single sequences whereas the Sequence Runner will allow programming of multiple sequences. Clearly, the direct control allows the operator to perform single steps. The commands are chosen from a dropdown menu, making the software robust and user-friendly.

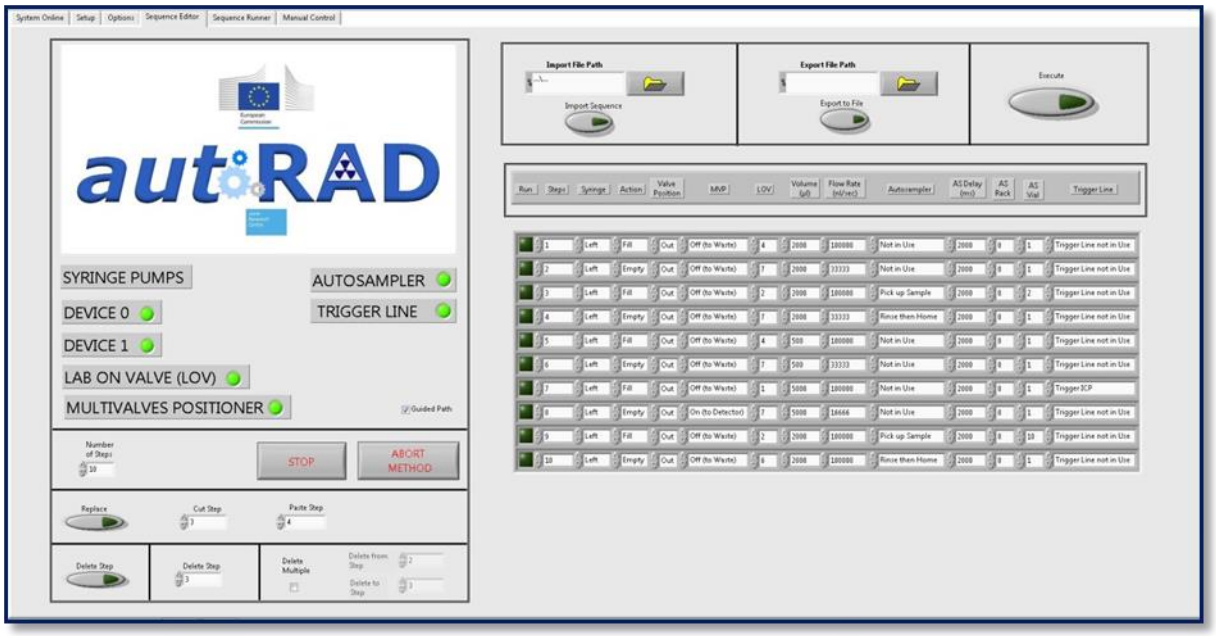

Figure 4: Graphical user interface (GUI), method editor tab

The Figure 4 shows the operation panel in the "Method Editor" tab. The left-hand part of the screen displays the connected devices information and the editing possibilities, such as Delete and Replace. On the right administrational buttons are integrated. The task of "Method Editor" is to create, modify, import or export as well as running a work sequence. The data contained in the cluster can be easily accessed and ordered with functions such as Unbundle by Name. Modification of the data or execution through the devices can so be achieved. Figure 5 shows the operation panel in the Sequence Runner" allowing multiple sequences.

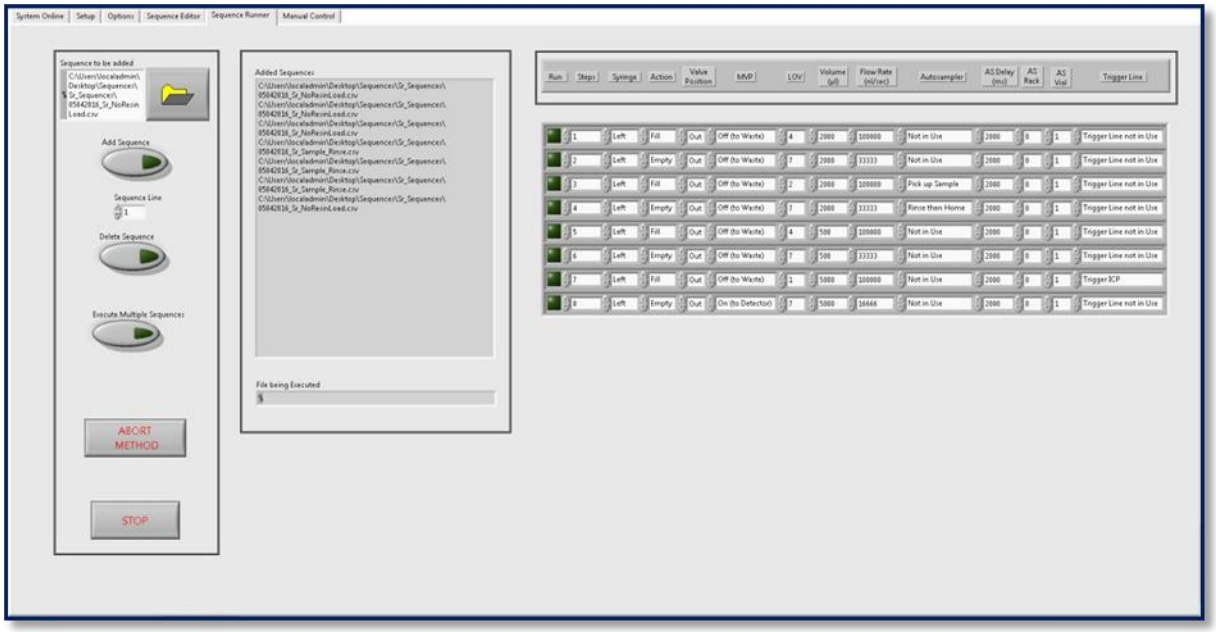

Figure 5: Graphical user interface, Sequence Runner tab

LabVIEW offers different possibilities to arrange and duplicate data. When simplicity is requested, the array function is a good choice to store and reproduce data. Arrays clone the input Object.

While Arrays are only able to store one Data Type, Clusters can contain multiple Data Types (e.g. Boolean, String, Arithmetic) or even whole Arrays. They are more powerful, yet as easy as Arrays. The clear advantage of using Clusters is that being single objects, they are easy and safe to operate with, making them the ideal tool to shift big amounts of data through the complex graphical block diagram. Combined with Enums (=enumerated values), standing for an operation governed by the devices, a user-friendly Menu is created. The user defines the number of steps in the GUI, and then configures the devices operation. The Cluster is situated immediately under the buttons. The Figure 6 shows an application of a cluster.

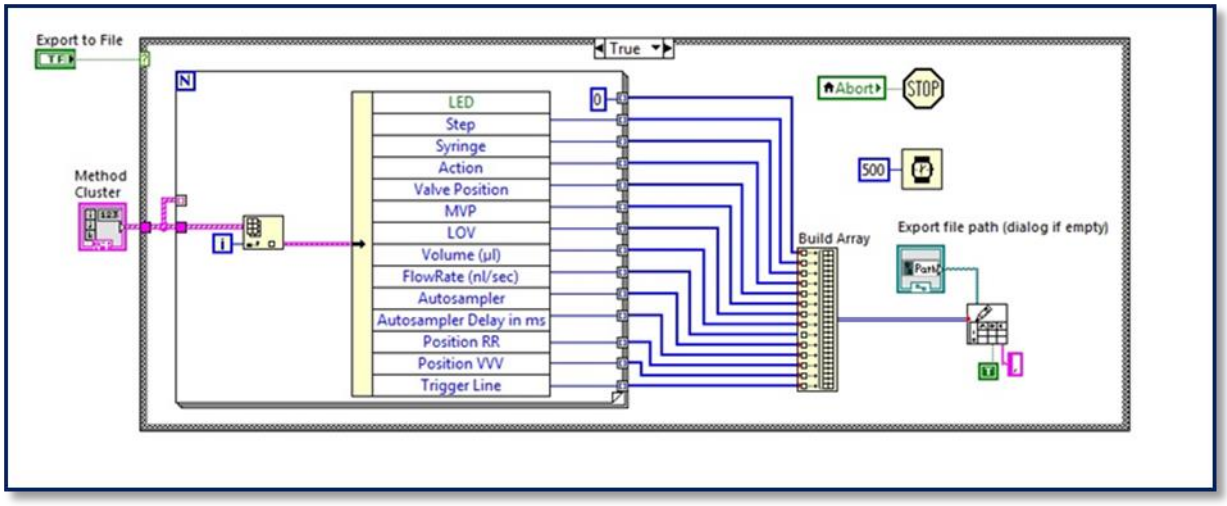

Figure 6: Application of a Cluster

Reordering a step is achieved with Unbundle Cluster function and Insert Into Array Function, applied on the respecting steps.

The Export Button triggers the cluster to be input into a .csv file, which is then easily storable and accessible with various programs. The steps are ordered by category with Index Array Node, exported with Build Array Node into various Arrays and Export to Spreadsheet Node.

When "Execute" is pressed, the unbundled data is input to a sequential frame, containing the subVIs representing the devices, as schematically represented in Figure 7 reflecting the block diagram.

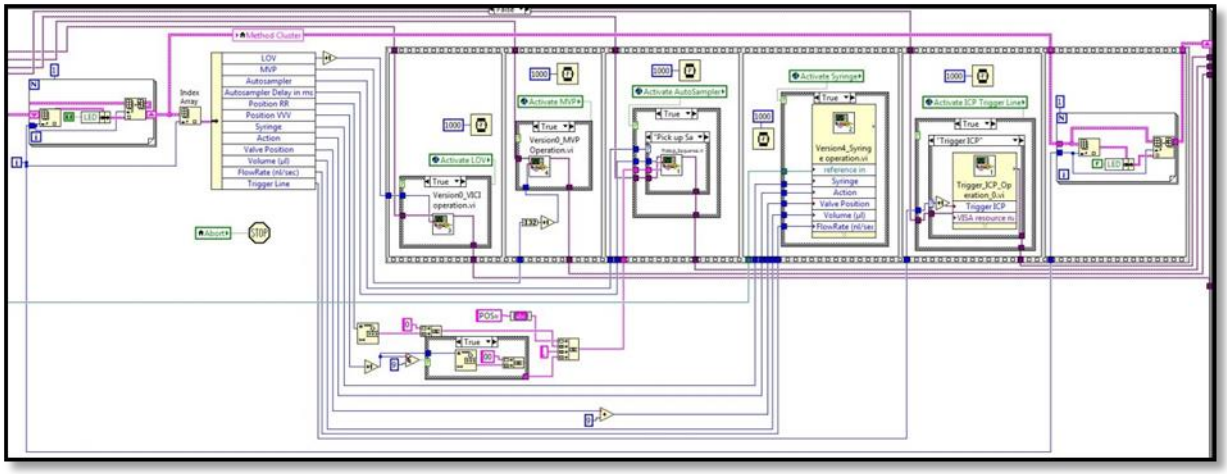

Figure 7: Block Diagram

3.3 User friendliness

The programme is user-friendly providing "Tip Strips", i.e brief descriptions of what is behind different objects and blocks. It is highly adaptable to changes for every interfaced device preventing offsetting (Figure 8).

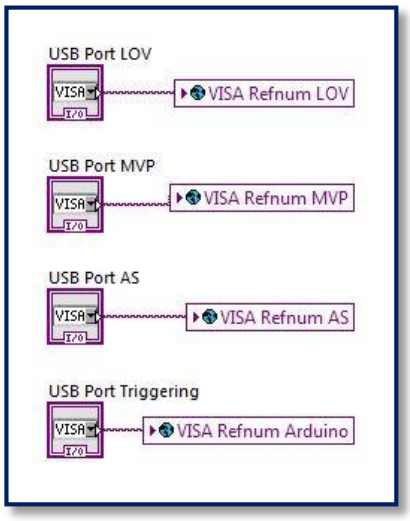

Figure 8: AUTORAD adaptable ports

In order to avoid common errors governing the flow rate and syringe volume working ranges due to adaptability to different models and suppliers, an algorithm has been implemented. If needed it asks the user to replace the critical value with an acceptable one. Programmatically this is achieved with the "Unbundling by Name" function and replacing the value (Figure 9).

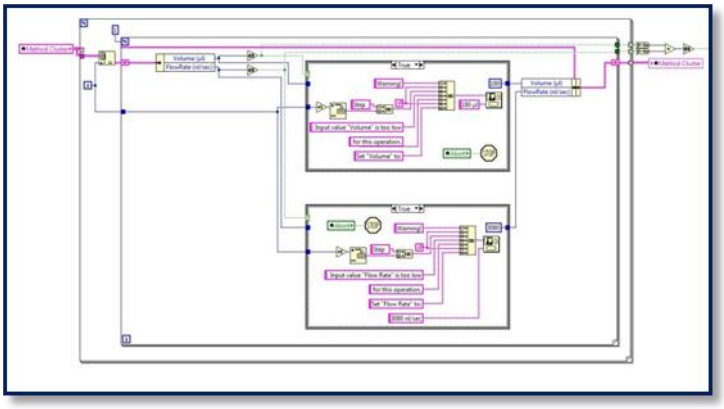

Figure 9: Monitoring algorithm

When starting the software, a specific order has to be followed to ensure a safe initialisation of all connected devices. To respect this "Guided Path" algorithm hides the tabs buttons, suggesting a clear way to the desired end procedure.

4. Application to the Determination of Strontium Isotopes

The software described here has been tested for the determination of strontium in standard samples at ultra-trace levels. Radiostrontium is of high importance in nuclear waste management, decommissioning, toxicology and environmental monitoring issues. ^{90}Sr and its daughter product ^{90}Y are both pure β emitters, so determination of ^{90}Sr by β counting requires separation from matrix constituents and interfering radionuclides prior to analysis. High specific activity and isobaric interferences represent a challenge in the spectrometric determination of ^{90}Sr. The analysis of ^{90}Sr is highly important, but due to the time consuming chemical separation, assumptions for the ^{90}Sr/^{137}Cs activity correlation is usually taken for its estimation [21].

Measurements on ^{86}Sr were performed using the ICP-MS detector, whereas the ^{90}Sr determination was carried out with the ß-RAM 5 flow detector. The Sr concentrations were 47.9 and 9.85 pg g^{-1}, respectively. The complete operational sequence for strontium isolation, pre-concentration and on-line detection is listed in Table 2, and summarised as follows:

1. Loading of resin: the column is automatically loaded with resin. First, resin is loaded into the HC from the resin reservoir (port 1) which contains a saturated solution of the resin and dispensed at port 5 with V-off (to waste) to fill the column.
2. Conditioning of Sr-Resin: the CC is connected to port 3 to aspirate 2 mL of 4 mol L^{-1} HNO_3 into the HC. Then it moves to port 5 and the HNO_3 is propelled toward the column at a flow rate of 6 mL min^{-1}. V is deactivated (V-off, to waste).
3. Sample loading: once the column is ready, 1 mL of standard or sample (port 8) is dispensed toward the column (port 5) at a flow rate of 1 mL min^{-1}.
4. Elimination of interferences: the CC is connected to port 3 to aspirate 0.5 mL of 4 mol L^{-1} HNO_3 into the HC. Then it moves to port 5 and the HNO_3 is propelled toward the column at a flow rate of 1 mL min^{-1}.
5. Elution of Sr: at this point all the strontium retained on the column is eluted. 5 mL of MilliQ water as eluent (port 7) are loaded into the HC and V is activated (V-

on) to propel the eluent through the column (port 5) at a flow rate of 1 mL min^{-1} to the detection system.

6. Change of sample: in order to avoid memory effects, 1 mL of the new sample is aspirated (port 8), and 2 mL are discarded toward waste (port 4).
7. Change of the resin: applicable when required depending on the sample matrix. The column is regenerated by replacing the resin automatically. First, the old resin is loaded into the HC and sent to waste (port 4), then new resin is loaded into the HC from the resin reservoir (port 1) which contains a saturated solution of the resin and dispensed at port 5 with V-off (to waste) to fill the column.

Table 2: Automated procedure for Sr separation, pre-concentration and detection

	Flow Rate (ml/min)	LOV Position	MVP
Resin loading			
(a) Loading beads into HC	6	1	Off
(b) Filling the column	1	5	Off
Conditioning of Sr-Resin			
(a) Loading 2 mL of 4 mol L−1 HNO3 into HC	6	3	Off
(b) Rinsing 2 mL on the column	1	5	Off
Sample loading			
(a) Loading 1 mL sample into HC	6	8	Off
(b) Rinsing 1 mL on the column	1	5	Off
Elimination of interferences			
(a) Loading 0.5 mL of 4 mol L−1	6	3	Off
(b) Rinsing 0.5 mL on the column 2 3 Off	1	5	Off
Elution of strontium			
(a) Loading 5 mL of MilliQ	6	7	Off
(b) Rinsing on the column	1	5	On
Change of sample			
(a) Loading 1 mL of new sample into HC	6	8	Off
(b) Discarding 2 mL to the waste	6	4	Off
Resin replacing			
(a) Loading old resin into HC	6	5	Off
(b) Discarding old resin	6	4	Off
(c) Loading new resin into HC	6	1	Off
(d) Filling the column	1	5	Off

The analytical performance of the AUTORAD platform was evaluated by considering the linearity, linear range, limit of detection (LOD) and repeatability. Figure 10 shows the corresponding ^{86}Sr elution profiles using ICP-MS as well as the relationships between the peak area and ^{86}Sr concentration. The linear regression was calculated using the least squares linear regression method. A fit for purpose curve is obtained which does not introduce an extra uncertainty component. The LOD was calculated by means of repeated measurements of the blank and according to Currie [22] and is 2 pg g^{-1}. The repeatability of the method, based on the relative standard deviation of the peak area

calculated on the basis of three repetitions is always less than 4 % in this concentration interval (10 to 120 pg g^{-1}).

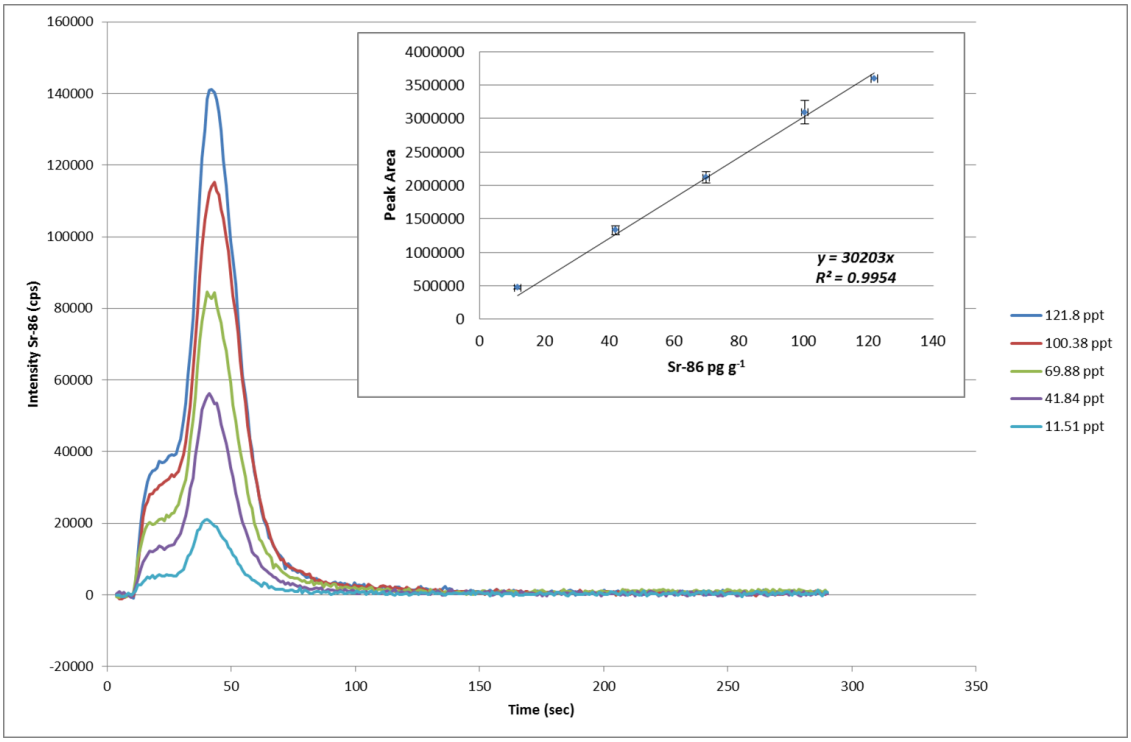

Figure 10: Sr elution peaks using ICP-MS as detector

Figure 11 shows the ^{90}Sr profile using the the BetaRAM 5 radio flow detector demonstrating the feasibility of AUTORAD software for the separation and simultaneous detection of ^{90}Sr in aqueous samples. However, the optimal counting window for ^{90}Sr, with the β-RAM flow scintillation analyser, and the determination of several counting parameters have to be optimised. The stopped-flow technique improving method sensitivity by extending, indefinitely, the residence time of the largest part of the sample zone within the flow cell, allowing a statistically meaningful number of counts to accumulate before the sample is permitted to exit the detector, is being investigated.

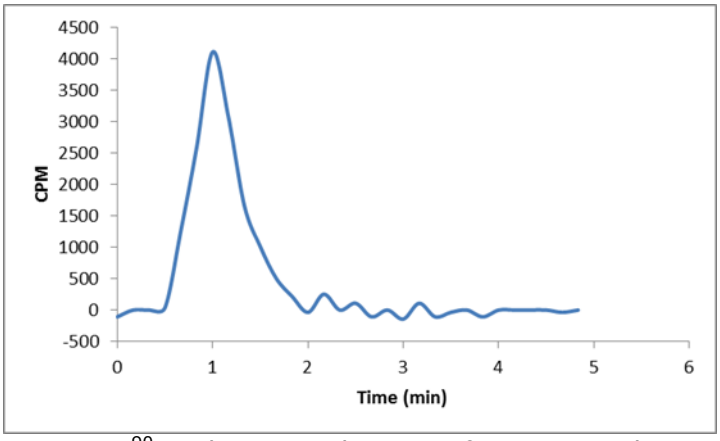

Figure 11: ^{90}Sr elution peaks using ß-RAM 5 as detector

Conclusion

A fully automated AUTORAD platform operated by lab-made LabVIEW®-based software has been developed and implemented. Its applicability to real samples was tested with Sr standard samples. The platform is versatile and is able to operate with different detectors. Simple and logical structure of the software makes it robust, user-friendly and suitable for on-site measurements.

The AUTORAD platform is a multi-purpose tool for performing safe, faster, less labour intensive and expected to be used as a powerful and convenient tool for the chemical separation and measurement of radioisotopes allowing monitoring operations where at-site or in situ measurements are more effective than traditional sampling and manual laboratory analyses.

Nuclear decommissioning, nuclear site remediation and monitoring R&D need measurement technology improvements to optimise and reduce intrusive sampling applications as they are costly, time consuming and can lead to workers' radiation exposure.

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List of abbreviations and definitions

CE: Capillary Electrophoresis
FIA: Flow Injection Analysis
GUI: Graphical User Interface
ICP-MS: Inductively Coupled Plasma Mass Spectrometry
LSC: Liquid Scintillation Counting
LOD: Limit of Detection
LOV: Lab-on-Valve
MSFIA: Multi-sequential Flow Injection Analysis
PC: Personal Computer
SIA: Sequential Injection Analysis
USB: Universal Serial Bus
VI: Virtual Instrument

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JRC Mission

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