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An Automated, On-Line Rapid Chemistry System

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AN AUTOMATED, ON-LINE RAPID CHEMISTRY SYSTEM

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Abstract

We have developed an Automated Chromatographic Chemical Element Separation System (ACCESS) and an Automated Injection System (AIS) for rapid and reproducible ion-exchange separations of various chemical species. The system is modular to allow for many different separation configurations. Two high-precision chromatography pumps, LDC's, provide constant solvent pressure throughout the system. An additional pump, and a 6-way valve, permit the use of a total of up to eight different eluants in succession. The system is controlled by an IBM PC running a BASIC Program. Chemical yield with the system has been determined to be 80 ± 9 %

We have developed the Automated Chromatographic Chemical Element Separation System (ACCESS) and an Automated Injection System (AIS). This apparatus was designed for repeated high precision chemical separations of short lived radionuclides produced at the Lawrence Berkeley Laboratory 88-Inch Cyclotron.

These computer-controlled ion chromatography and injection systems are designed to provide a rapid and reproducible separation using establish-ed methods such as α-hydroxy-isobutyrate elutions from cation exchange resins or bis(2-ethylhexyl)-ortho-phos-phoric acid reverse phase extraction chromatography. The system is modular, and can be used in either an off-line (manual injection) or on-line (AIS interfaced) configuration. ACCESS[1]-AIS[2] is controlled by an IBM PC running a BASIC program. An analog to digital and digital to analog interface (Data Translation DT-2801) board in the PC provides 16 digital I/O ports, 8 ADC inputs and 2 DAC outputs. With the digital outputs, we control two chemically inert pumps (Cheminert Kel-F Precision Pumps, model number CMP-1VK), another pump (Eldex Laboratories Dual Piston Pump, Kel-F Heads, model number AA-100-S), a 6-way valve (Eldex Laboratories Selector Valve, model number 1200), and the pneumatically actuated slider valves used to direct the flow of solution through the system and the AIS. Chemical fractions of separated elements are collected with a fraction collector (Gilson Scientific, model number FC-203), which is controlled by the PC through a RS-232C serial interface. Figure 1 is a schematic of the ACCESS control-system.

Solvents are introduced by controlling the positions of pneumatic actuators (Rainin Instruments Tefzel Actuators, model number 201-57) which control slider valves (Rainin Instruments Tefzel 4-way slider valves, model number 201-52). The sliders are chemically inert materials such as Delrin and the tubing between the valves is Teflon. Bores in the slider are positioned such that the solvents are forced to follow different paths as dictated by the user. The Teflon tubing has an inside diameter (i.d.) of 0.5 mm. The tubing used to supply the N_2 to the pneumatic actuators is also Teflon with an i.d. of 0.8 mm. Teflon was chosen over other materials because of its relative resistance to aqueous chemical attack and the ease with which it can be connected to the system. All of the Teflon tubes are as short as possible to minimize dead volume and hence increase resolution and speed. The separation columns are constructed from precision-bore glass tubing and chemically inert column end fittings (Rainin Instruments Tube End Fittings, 1/16-inch outside diameter, model number 200-00). This permits optimiza-tion of the separation by varying the column length and solvent flow rates. A typical 2-cm length x 2 mm i.d. column has a free column volume of about 5 drops (13 μL/drop). The entire system has a void volume of about 50 drops, but is dependent on the configuration in use. Figure 2 is a schematic of the ACCESS configuration for a chromatographic separation employing a single column and the AIS.

Radioactivity is introduced into the system either by the off-line injection loop, or by AIS. The AIS is a pneuma-tically actuated device for automated collection of the products from a He/KCl gas jet transport system and injection of these products into ACCESS. Figure 3 is a schematic of the AIS. The reaction products from bombardments at the 88-Inch Cyclotron at Lawrence Berkeley Laboratory are transported out of the target system [3] by KCl aerosols carried in He. The jet deposits the activity laden aerosols in one of two small collection sites on the surface of the Delrin bar (see Figure 3). An advantage of ACCESS-AIS is that while chemistry is being performed on one sample, another is being collected. After a preset collection time, the Delrin bar is pneumatically positioned so that the aerosol deposit is in-line with one of the two Delrin plugs (see Figure 3) which have solvent lines leading to and from ACCESS. The aerosol deposit is dissolved in the solvent and transported into ACCESS. The Delrin bar is pneuma-tically secured against o-rings by two plungers at the bottom so that solvent leakage is avoided. A vacuum pump connection at the collection site is perpendicular to the direction of the incoming He jet.

The AIS has been successfully interfaced with ACCESS and experiments have been performed to determine chemical yield through the AIS. The yield was determined using 4.8 m ^{221}Fr recoils from a ^{225}Ra/^{225}Ac source in-line with our He/KCl transport system. For yield normalization, the ^{221}Fr laden KCl aerosols were transported via a polyvinyl chloride capillary (2.0 mm i.d.) and collected on Ta foils for counting by alpha-pulse height analysis with a surface barrier detector. The He/KCl was then directed into AIS and collected for various time intervals. The collected KCl was positioned in-line with ACCESS by the AIS and solvent flow initiated. The sample was completely dissolved in about 60 λ of H_2O and deposited on a Ta foil for drying and activity determination. Several experiments were performed and the average yield, corrected for the decay of the ^{221}Fr, was determined to be 80 ± 9 %. Because the KCl can deposit anywhere on the Ta foil in the initial activity determination and only a 5 mm space is provided in AIS, it was necessary to install a capillary guide (see Figure 3) to ensure that the activity was deposited into the AIS properly. Any misalignment of the capillary will significantly reduce the efficiency of the system.

ACCESS has been used in several experiments, including the identification of the previously unknown ^{253}Md [4], and study of the fission properties of ^{261}Lr [5]. In both cases, ACCESS separated the elements by α-hydroxy-isobutyrate elution.

The chemical yield through ACCESS is dependent on the chemistry used in the separation and the time (for short-lived species). The chemically inert surfaces of ACCESS provide nearly 100% transport efficiency of the activity. Using the lanthanides ^{171}Tm, ^{152}Eu, and ^{166m}Ho, we observed separation factors that were consistent with previous work [6, 7]. The resolution of our measurements was improved using smaller drops (13 μL vs. 45 μL).

The implementation of ACCESS-AIS maximizes the use of accelerator time and allows for the collection of as much data as possible during the course of one experiment. This system also reduces the labor intensive nature and increases the reproducibility of repeated rapid manual chemical separations.

Acknowledgements

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Figure Captions

Figure 1. Automated control-system diagram for ACCESS.

Figure 2. Schematic of ACCESS. The boxes with dashed and solid lines represent 4-way valves. When the slider is in the open position, solvent is directed via the solid path through the valve. When the slider is in the closed position, solvent is directed via the dashed path.

Figure 3. Schematic of AIS. Activity is being collected from the He/KCl jet on the right side of the Delrin bar while the previously collected sample is being dissolved and washed into ACCESS in the left. The silicon detector is positioned to allow for monitoring of the sample for unexpected alpha activity from our target system.

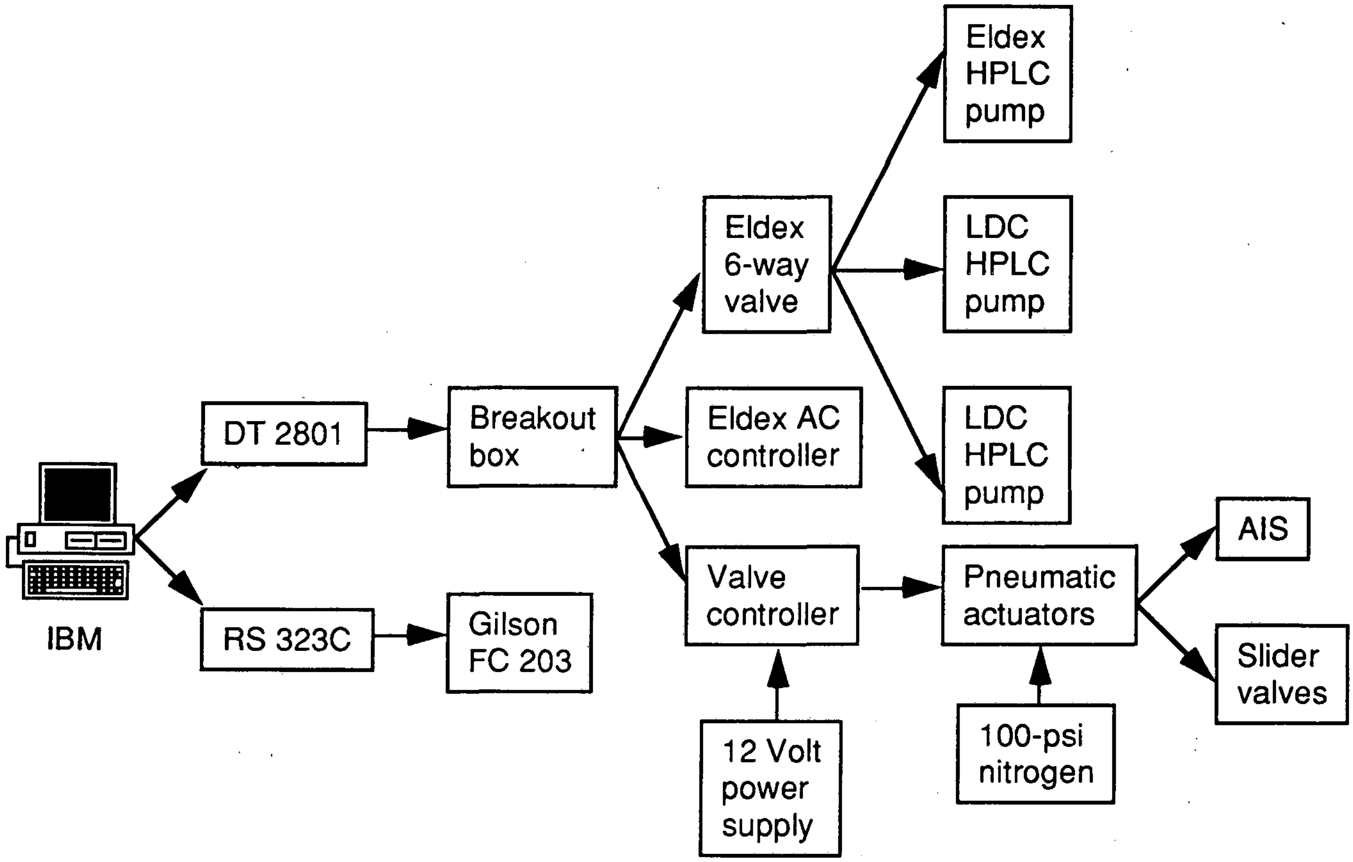
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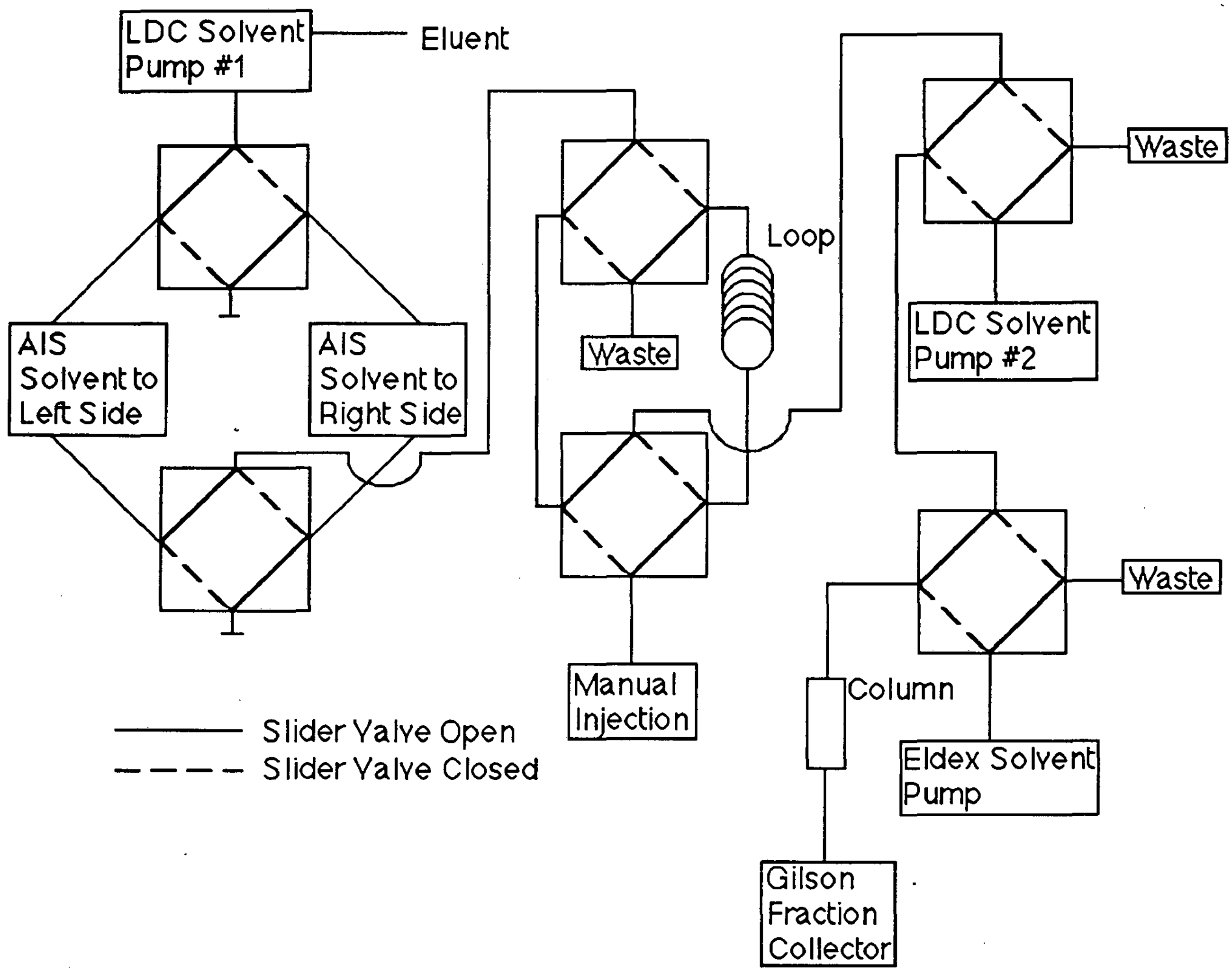
Figure 1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Gilson Fraction Collector
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Slider Valve Closed

Figure 2

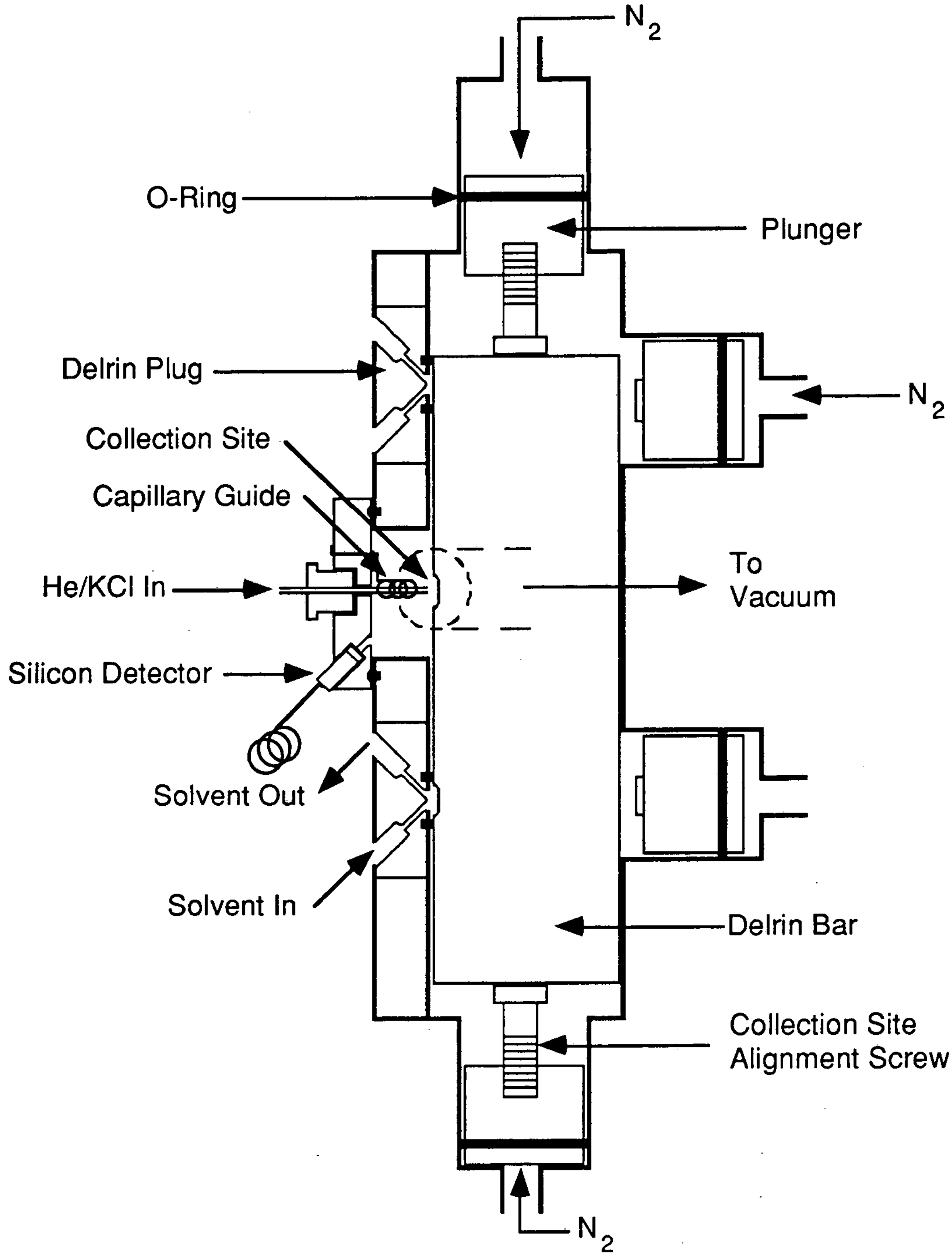
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Figure 3