INL/EXT-13-28796

Evaluate Disposable Sample Chips and Complete the Evaluation of Sample Accuracy Through Evaluation of Chip Filling Methods

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April 2013

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The INL is a U.S. Department of Energy National Laboratory
operated by Battelle Energy Alliance

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Prepared for the
U.S. Department of Energy
Office of Nuclear Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

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In FY-12, the glass sample chips were filled by capillary action when they were dipped into the solutions being sampled either by hand or by the robotic sampling system. Issues were noted at the end of FY-12 that many of the channels were not completely filling (10-15% of the 10 µl channels and up to 40% of the 2 µl channels). Therefore, testing was conducted in FY-13 on filling the glass sample chips by use of a flow-through header (Table 1). Results indicate flow through filling is better at completely filling the chip channels, although some amount of solution can be lost when the chip is removed from the filling header.

Table 1. Flow through filling versus dipping of glass chips.

	Glass Chips Flow Fill (UV-VIS) 1 g/L solution		Glass Chips Flow Fill (UV-VIS) 100 g/L solution		Glass Chips Dip Fill (UV-VIS) 100 g/L solution	
	2µl	10µl	2µl	10µl	2µl	10µl
% channels not filled	~1-5%	~1-5%	~1-5%	~1-5%	40%	10-15%
%RSD*	12.9%	11.4%	12.6%	6.7%	13.2%	2.75%
# channels	12	12	12	12	20	20

*does not include channels noted as not full

Several prototype "disposable" sample chips were made by embedding SS capillary tubing into a plastic framework (Figure 1). Although this design will allow capillary filling, the design limits the volume of the sample to 2 µl.

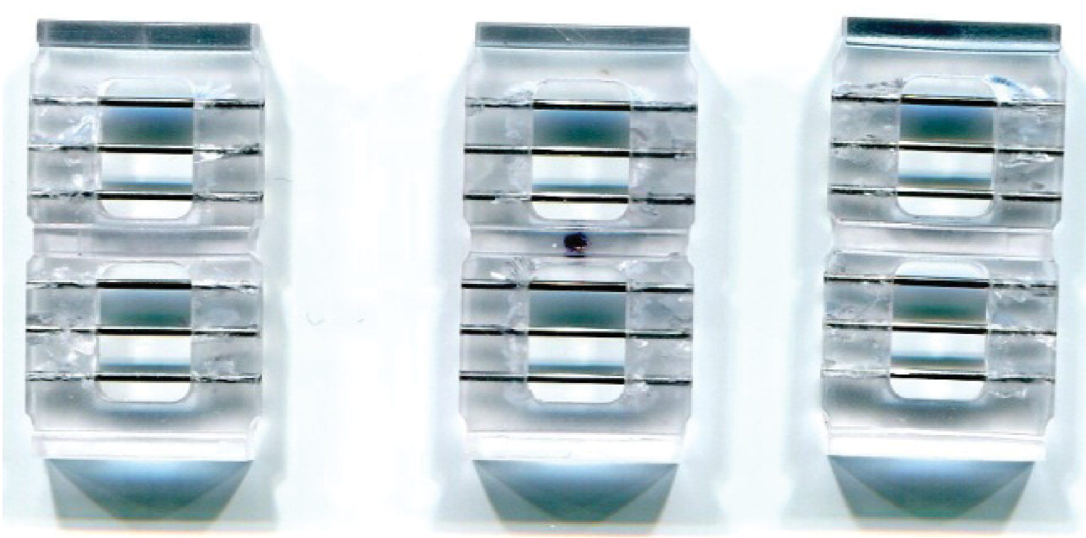

Figure 1. Prototype disposable sample chips estimated to cost <$15 each.

The results of some of the testing with these chips are in Table 2.

Table 2. Plastic chip dip and flow-through filling tests

	Plastic chip w/ SS channels – Flow Fill – UV VIS 1 g/L solution	Plastic chip w/ SS channels – Dip Fill – UV VIS 1 g/L solution	Plastic chip w/ SS channels – Flow Fill – UV VIS 100 g/L solution	Plastic chip w/ SS channels – Dip Fill – UV VIS 100 g/L solution
	2µl	2µl	2µl	2µl
%RSD	34.1%	44.6%	14.8%	79.01%
# channels	36	36	36	36

Since the sample channels are stainless steel, it is not possible to visually determine if the channels are completely filled. The results in Table 2 indicate that flow-through filling provided less relative standard deviation in the results than by dip filling. However, even with flow-through filling the standard deviation is still too high. This seems to indicate, that a slightly larger sample size is needed to reduce the variation in the analyses. Therefore, different chip designs were conceptualized (Figure 2) that had a minimum 10 µl sample size.

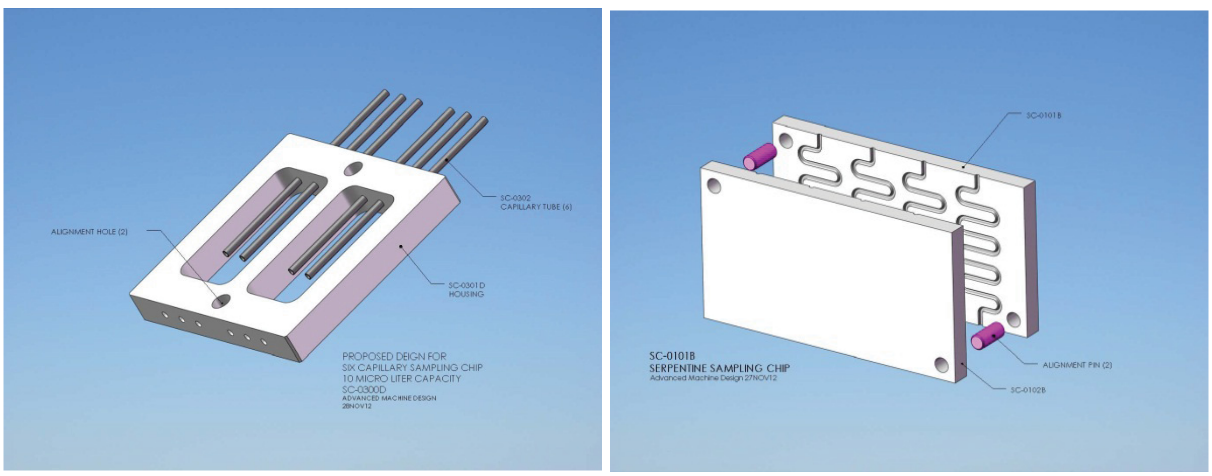

Figure 2. 10 µl disposable chip designs

It was decided that since the flow-through filling generated more consistently filled chips, that the channels in these chips could be a slightly larger diameter (do not have to fill with capillary action but have to hold the solution in the channels when disconnected from the fill station. To get the 10 µl size the flow-through loading and unloading stations on the robotic system would have to be slightly redesigned (Figure 3). Funding was not available to have the chips made and the loading/unloading station modified.

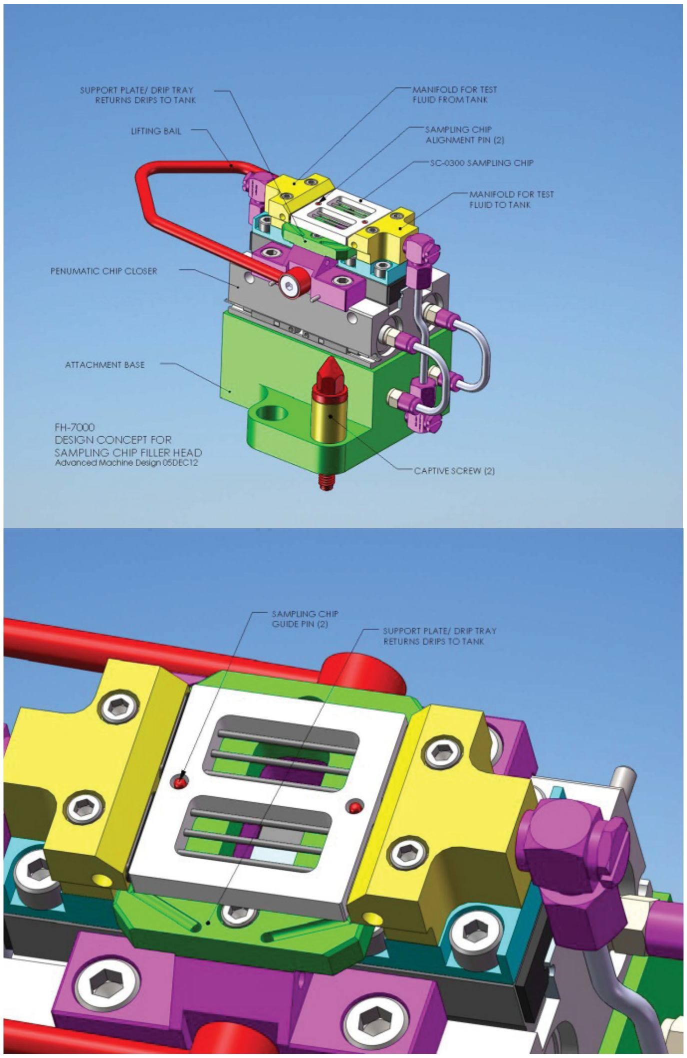

Figure 3. Conceptual design of 10 µl chip loading/unloading station.

Additional testing with both the glass and 2 µl prototype chips was completed using an ICP-OES (instead of the UV-Vis previously used. To better evaluate the effectiveness of the robotic system an ICP-MS will be used in replace of the UV-Vis. Chips were filled by flow through primarily (with one dipped). Tests were conducted with 100 g/L $Ho(NO_3)_3$ and with a stock La solution. After the chip was filled it was placed by hand in the spare sample unloading header and transported to the sample unloading system attached to the ICP-OES. After analysis was complete, the process was repeated once with the same chip (i.e. two runs). Results from the ICP-OES are still being evaluated and will be reported in the end of year report.