

# Rapid Analysis of Soil Samples by Flame Atomic Absorption

Andrew Kavan - Elemental Scientific, USA

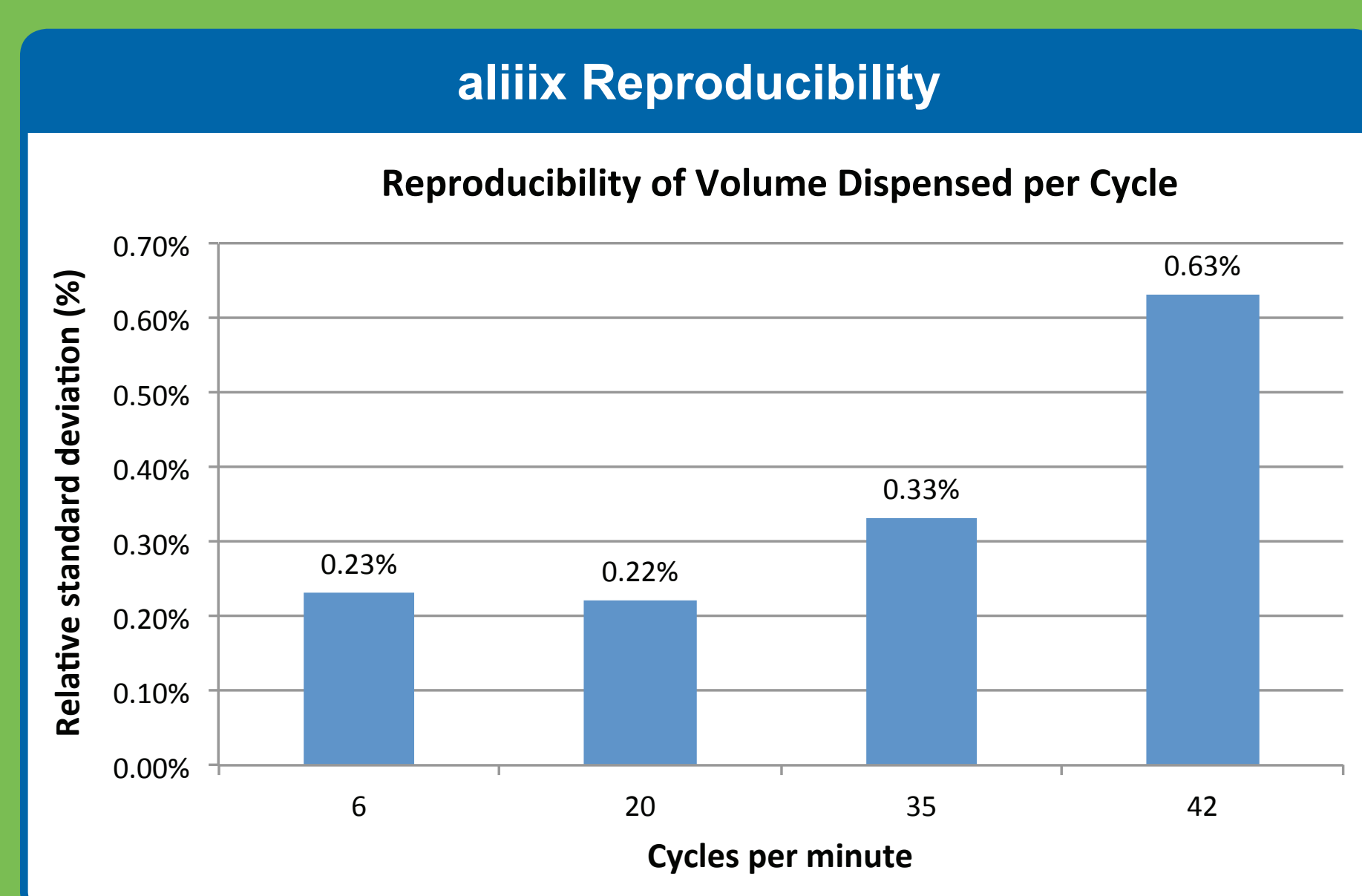
## Abstract

A new automated procedure for multi-element soil analysis by flame atomic absorption (FAA) is described. Two grams of soil are weighed and placed in a 125 mL plastic bottle for Mehlich extraction. The sample preparation procedure involves, 1) addition of 20 mL of Mehlich 3 extractant, 2) 5 minutes of mixing, and 3) filtration of extractant (Whatman grade 2 filter paper). One mL of sample filtrate is aliquoted into a vial for offline (20x) autodilution. The undiluted sample is measured for low concentration metals (Cu, Mn, Na and Zn) by FAA while the 1 mL fraction is autodiluted for the determination of higher concentration metals (Ca, Fe, K and Mg). After the first group of metals is analyzed, the second set of autodiluted samples are ready. Samples are introduced using a high throughput introduction system that utilizes an injection valve to rapidly load (vacuum) and inject (peripump) the samples into the flame. Using this fast sample introduction system, throughput is approximately 8 seconds per sample for each analyte. Combining offline autodilution and inline fast analysis provides accurate and precise data for 8 analytes in roughly 1min per sample. This procedure offers a low-cost, simple FAA alternative for soil analysis versus ICP.

## Sample Preparation



**Figure 1.** After soil is added to bottles, 20 mL of Mehlich 3 extracting solution is rapidly added by the aliix pump.



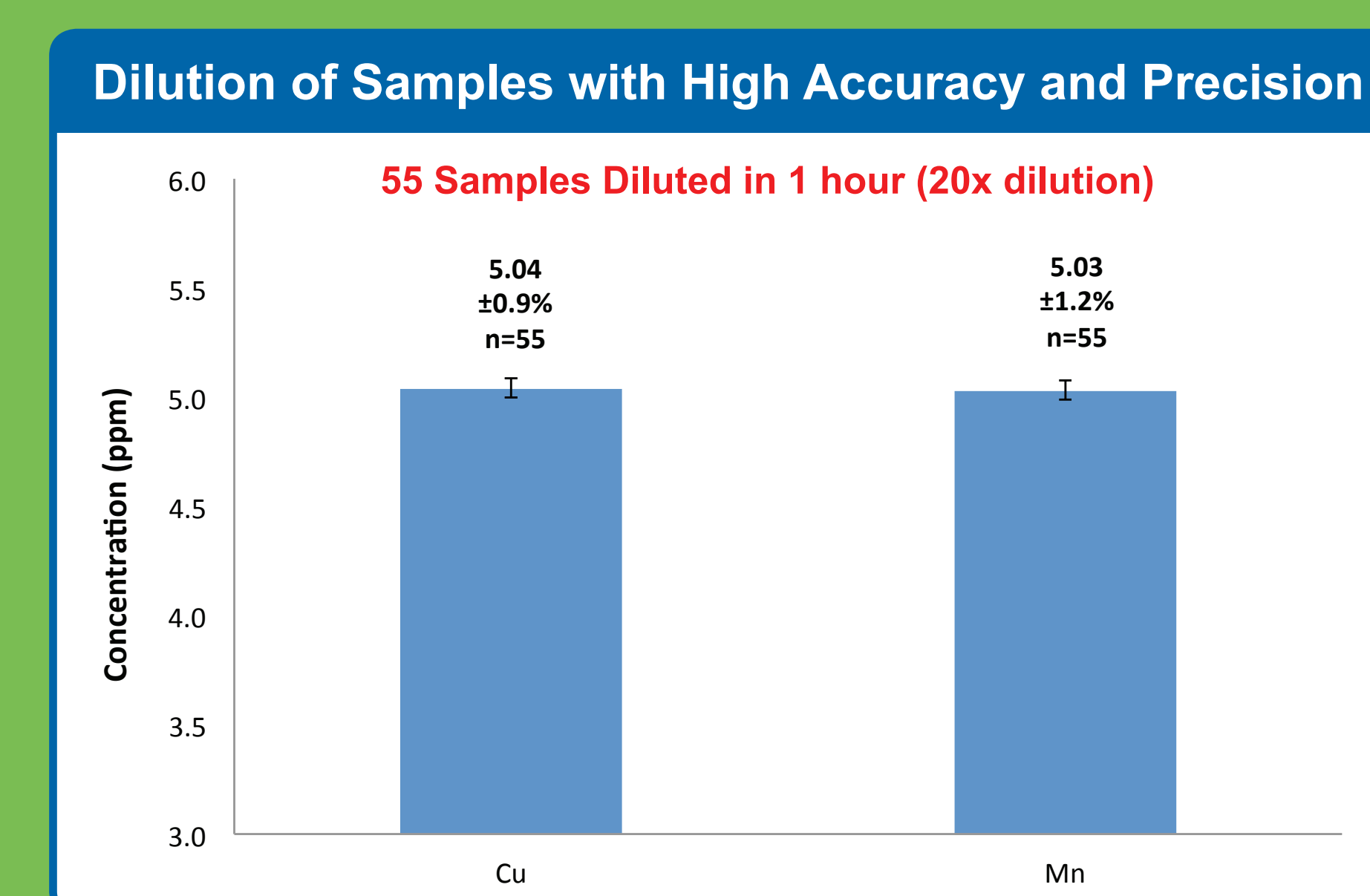
**Figure 2.** Effect of delivery speed on precision. 20mL dispensed each time from each syringe.

aliix Model	60 samples	120 samples
50x1	86 sec	171 sec
50x3	29 sec	57 sec

**Table 1.** Compares the time needed to fill every vessel with Mehlich 3 extractant for each aliix model for 60 samples and 120 samples.

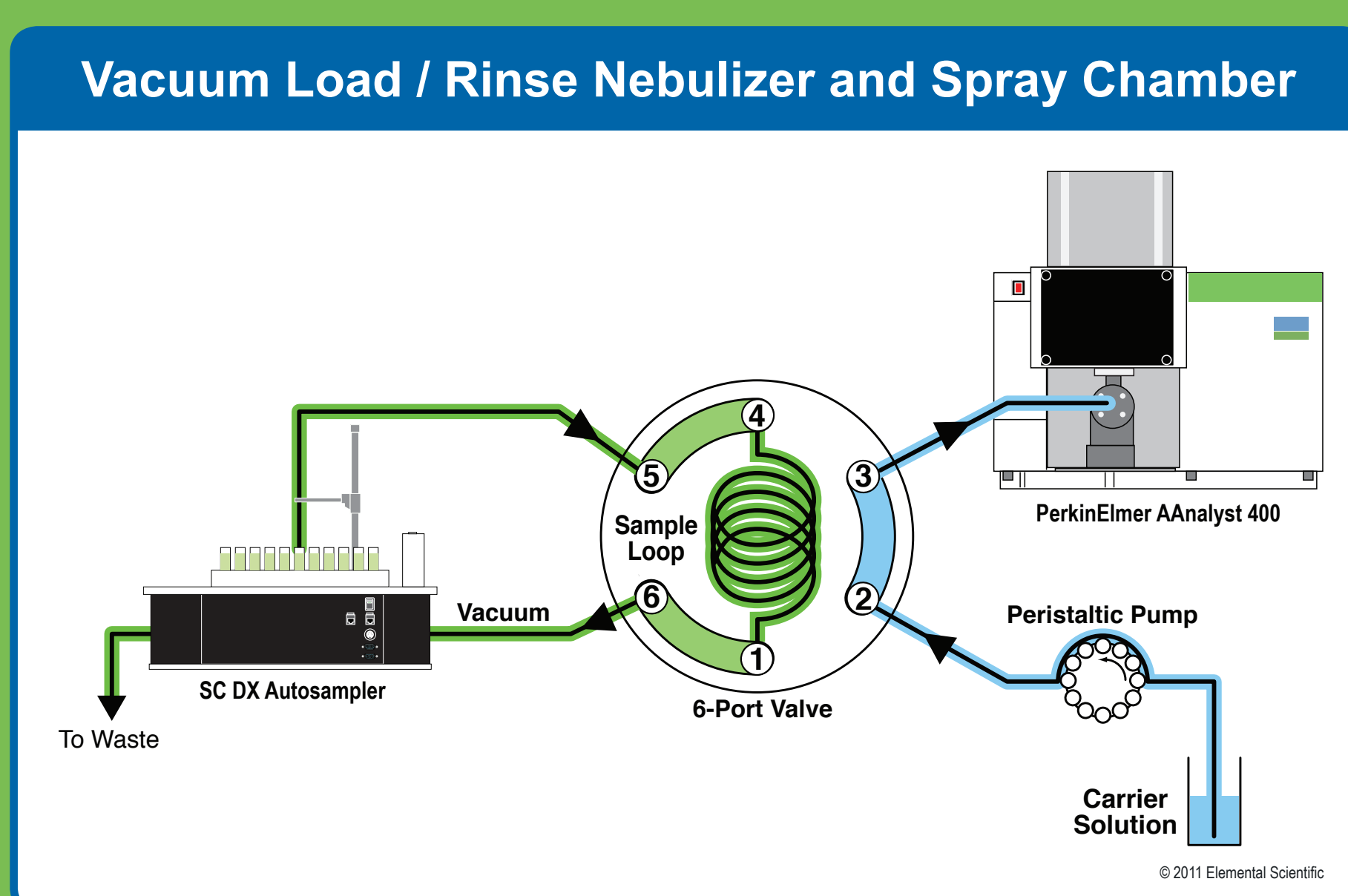
**Figure 3.** Samples are then placed into the vorso rack and mixed for 5 minutes.

After the five minute mixing, samples are filtered using Whatman grade 2 filter paper.

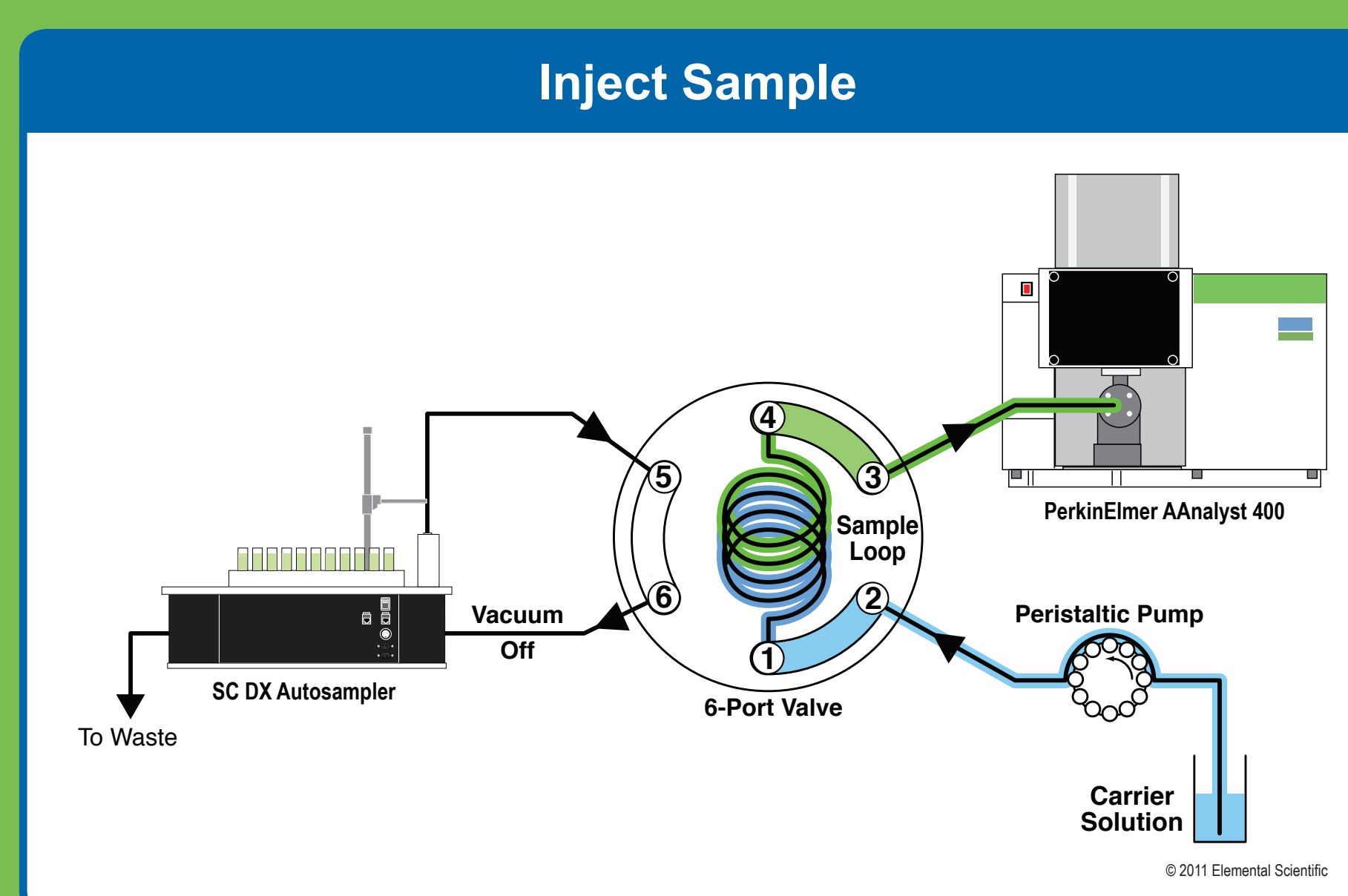


**Figure 4.** 100 ppm samples were autodiluted (20x) 55 times and analyzed by ICP.

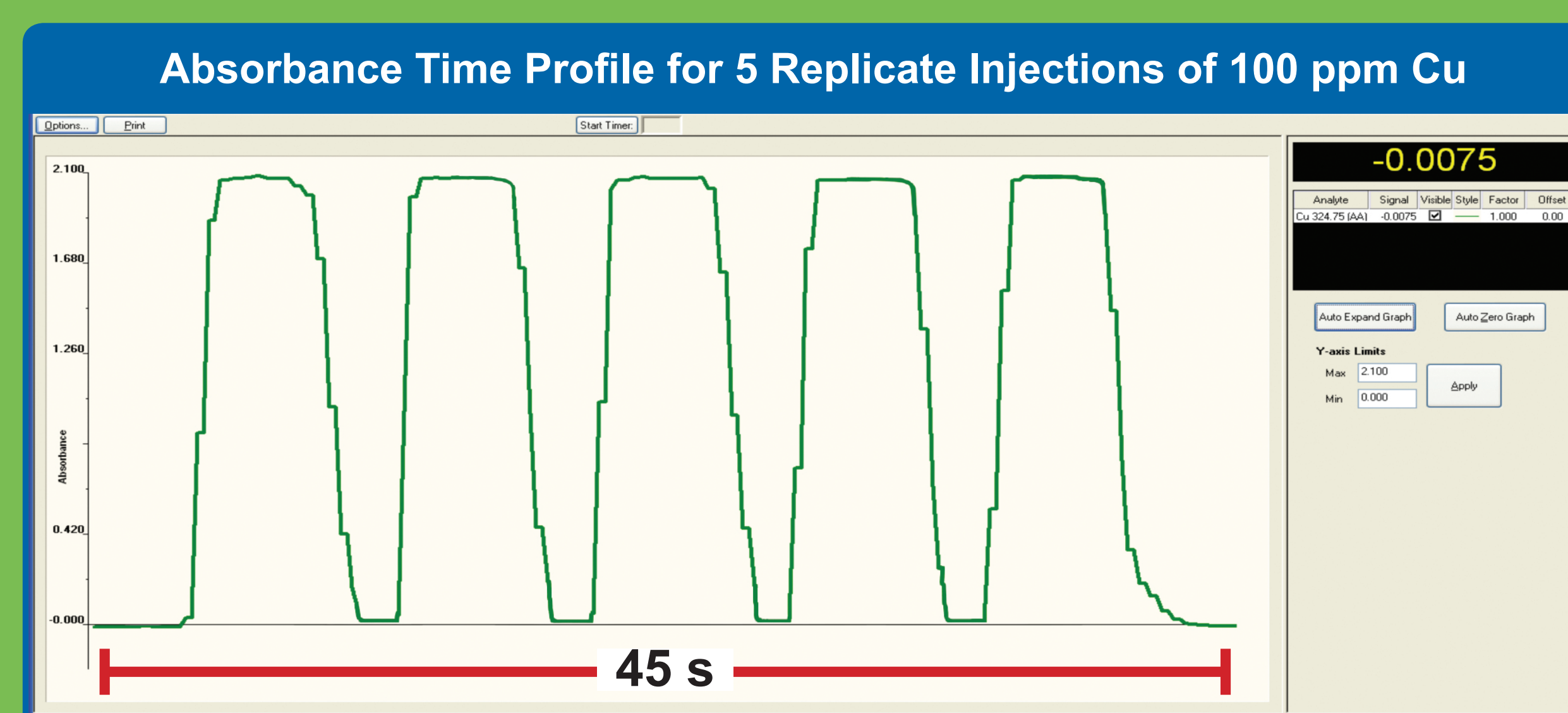
## Analysis



**Figure 5.** Vacuum load sample into valve loop located near the AA spray chamber (1s).



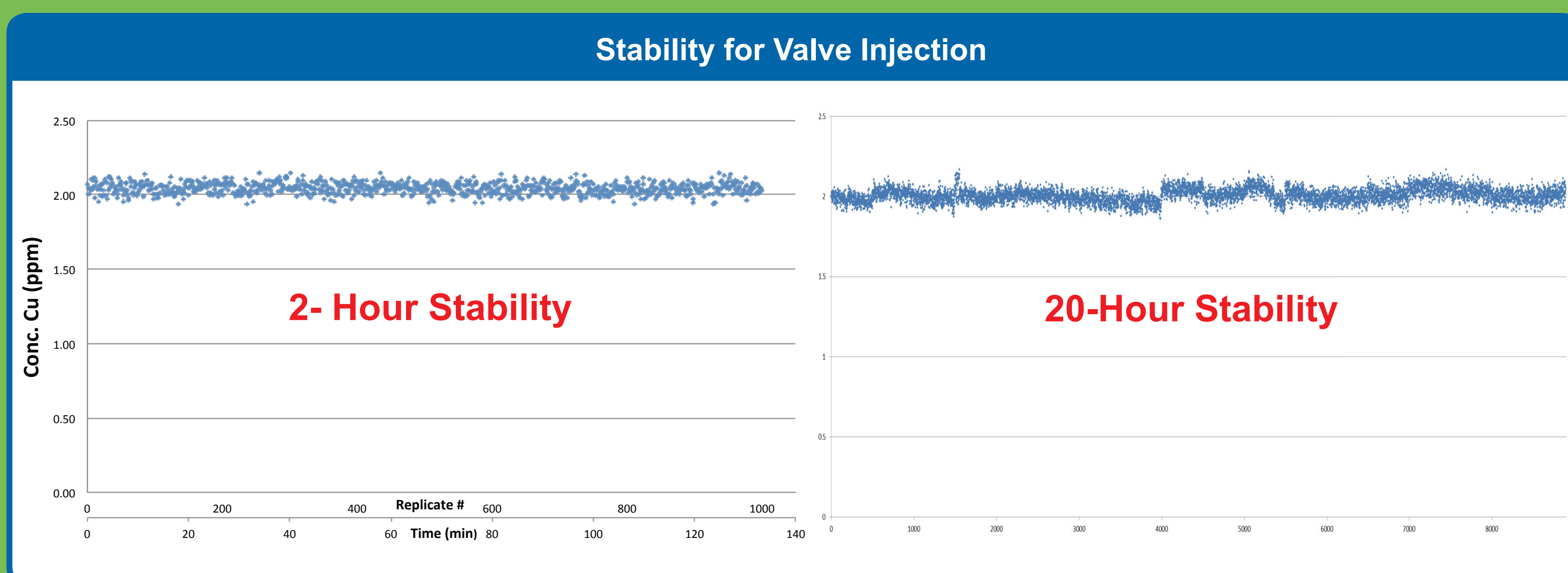
**Figure 6.** Inject sample aliquot into a peristaltic pump-driven carrier liquid at 5 mL/min (2s) and read absorbance (2s).



**Figure 7.** Injecting the Sample: After the loop is loaded, the valve switches to inject. The sample is pushed by the carrier solution into the spray chamber and is analyzed by the instrument.

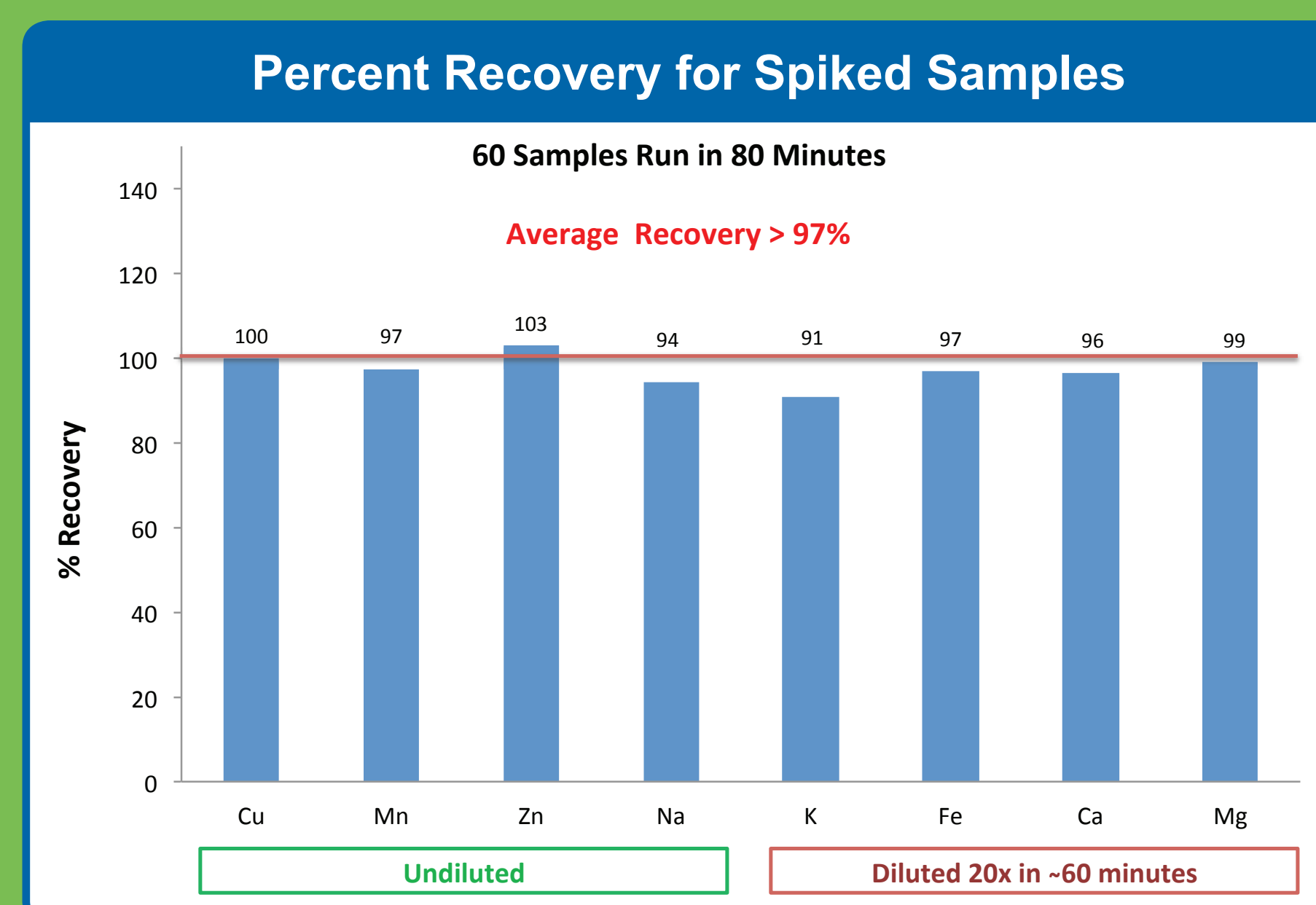
Concentration	Time	Cu 324.75 (ppm)
Cal Blank	1:37:43 PM	0.00
1 ppm	1:37:51 PM	1.00
2 ppm	1:37:59 PM	2.00
6 ppm	1:38:07 PM	6.00
Blank	1:38:15 PM	0.01 *
Blank	1:38:24 PM	-0.00
Blank	1:38:33 PM	-0.01
2 ppm	1:38:41 PM	2.04
2 ppm	1:38:50 PM	2.03
2 ppm	1:38:58 PM	2.06

**Table 8.** Flame AA Analysis of 10 Solutions (Calibration, 3 Blanks and 3 Samples) in 75 seconds with > 600x washout between samples.



**Figure 9.** 1000 samples of 2 ppm Cu in 2 hours and 17 minutes (8.2 s/sample). Mean: 2.00

n = 8931, Average = 2.01 ppm, RSD = 2.1% over 20 hours



**Figure 10.** Compares the spiked recoveries for each sample. Average recovery is 97%. The set of samples is analyzed multiple times, once for each element. CRM is NAPT Program 2012-110.

## Summary

- aliix pump rapidly and accurately dispenses 20 mL of Mehlich 3 solution.
- Vorso rack mixes solution.
- prepFAST Offline automatically dilutes samples within the analytical range of the instrument.
- Valve injection offers several advantages over self-aspiration for Flame AA, including:
  - ✓ Higher throughput by a factor of 2x to 3x, depending upon integration time
  - ✓ Less sample consumption
  - ✓ Excellent spike recoveries



**Elemental Scientific**

FASTFlame.com | prepFAST.com | icpms.com | sales@icpms.com