Application Note - *Easy*PREP Sample Handler for oil dilutions.

Introduction:

The present application note evaluates the liquid handling performance of the ***Easy*PREP Sample Handler** for automated dilution of oil samples. Accuracy and precision of the system are evaluated through dilution of samples and preparation of calibration curve standards from a stock standard. Samples are analyzed on a Spectro Arcos ICP-AES.

Sample Type:

Multi-element standard containing 21 elements at 900ppm in oil (**CONOSTAN**® S-21 at 900ppm), diluted using PremiSolv ICP solvent.

Supplies and Reagent:

1) ***Easy*PREP Sample Handler*** (010-400-001)
2) 50ml ***Digi*TUBE**s* (010-500-264)
3) PremiSolv ICP solvent* (150-700-00x)
4) Stock multi-element solution **CONOSTAN**® S-21 at 90ppm* (150-021-0xx)
5) Spectro Arcos ICP-AES
6) Cyclonic spray chamber, baffled* (020-071-031)
7) Concentric nebulizer, C2,* (020-060-006)
8) Torch, 1.8mm quartz injector* (020-050-090)

* Manufactured by **SCP SCIENCE**

Sample Handler Settings:

Airgap up (µl)	50
Airgap %	70
Small volume pickup (µl/s)	300
Small volume dispense (µl/s)	700
Volume threshold (µl)	2000
Airgap down(µl)	5
Offset (µl)	0
Large volume pickup (µl/s)	300
Large volume dispense (µl/s)	700

ICP-AES Settings:

RF Power (L/min)	1550
Coolant flow (L/min)	15
Auxiliary flow (L/min)	2.0
Nebulizer flow (L/min)	0.60
Sample intake (ml/min)	1

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Sample Preparation Procedure:

In a first step, the 900ppm stock standard was diluted 1:10 by weight using PremiSolv as diluent. The 90 ppm working standard was then diluted both by weight (manually) and by volume (Sample Handler). Three replicates at two dilution factors were prepared and analyzed by ICP-AES to verify both the accuracy and precision of the automated dilution, and to draw a comparison with the established protocol of diluting oil samples by weight.

In a second step, the ***Easy*PREP Sample Handler** performance at preparing calibration curves from a stock standard was evaluated using again the S-21 at 90ppm and the PremiSolv ICP solvent as diluent. Finally, the capacity of the Sample Handler to remove cross-contamination was evaluated, using PremiSolv and heptane as rinsing solutions.

Results and Discussion:

1- Automated vs Manual dilution

Precision and accuracy of automated dilution was evaluated by diluting the S-21 multi-element standard at concentrations of 2.25 and 9ppm, followed by analysis by radial ICP-OES. Repeatability of three sample replicates and sample recovery are seen in Table 1.

Table 1a: Sample results, 9ppm dilution

		Manual			Automated		
Element	**λ**	Average	%RSD	%Rec	Average	%RSD	%Rec
Al	308.215	8.92	1.41	99	9.09	0.86	101
Ba	230.424	8.72	0.79	97	8.84	0.46	98
Ca	315.887	8.91	0.82	99	9.02	0.58	100
Cd	228.802	8.82	0.83	98	8.9	0.49	99
Cr	283.563	8.81	0.85	98	8.88	0.54	99
Cu	324.754	8.89	0.89	99	8.95	0.72	99
Fe	238.204	8.80	0.84	98	8.86	0.53	98
Mg	279.553	8.76	0.29	97	8.79	0.26	98
Mn	260.569	8.64	0.62	96	8.67	0.27	96
Na	589.592	8.90	1.01	99	8.96	0.88	100
Ni	221.648	9.01	1.34	100	9.13	0.19	101
Pb	220.353	8.86	0.84	98	8.96	0.52	100
Sn	189.991	8.93	0.79	99	9.07	0.47	101
Ti	336.121	8.93	0.11	99	8.95	0.77	99
Zn	206.200	8.91	0.79	99	9.03	0.56	100

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Table 1b: Sample results, 2.25ppm dilution

		Manual			Automated		
Element	**λ**	Average	%RSD	%Rec	Average	%RSD	%Rec
Al	308.215	2.207	0.52	98	2.202	0.30	98
Ba	230.424	2.167	0.54	96	2.160	1.17	96
Ca	315.887	2.194	0.51	98	2.176	0.85	97
Cd	228.802	2.167	0.69	96	2.169	0.74	96
Cr	283.563	2.227	0.62	99	2.208	0.68	98
Cu	324.754	2.212	0.74	98	2.185	1.22	97
Fe	238.204	2.273	0.59	101	2.234	0.77	99
Mg	279.553	2.226	0.77	99	2.209	1.16	98
Mn	260.569	2.254	0.68	100	2.260	0.75	100
Na	589.592	2.334	0.77	104	2.312	0.74	103
Ni	221.648	2.292	0.56	102	2.287	0.65	102
Pb	220.353	2.163	0.66	96	2.139	0.82	95
Sn	189.991	2.180	0.64	97	2.114	0.95	94
Ti	336.121	2.174	0.80	97	2.114	1.36	94
Zn	206.200	2.202	0.46	98	2.209	0.04	98

As seen in the Figure 1 below, automated (v/v) and manual (wt/wt) dilutions of the S-21 multi-element standard at 2.25 and 9ppm are both efficient in obtaining accurate diluted samples. Accuracy is not affected when using aliquot volumes as low as 0.125ml (Figure 2). Precision is within tolerance and better than 1.5% for the three sample replicates, a result of both the sample preparation error and the analytical capabilities of the ICP-OES.

Figure 1:

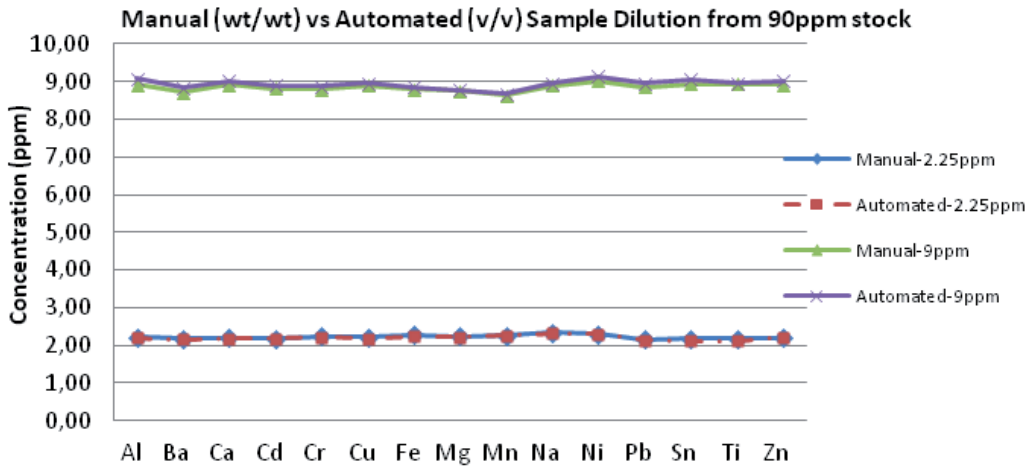

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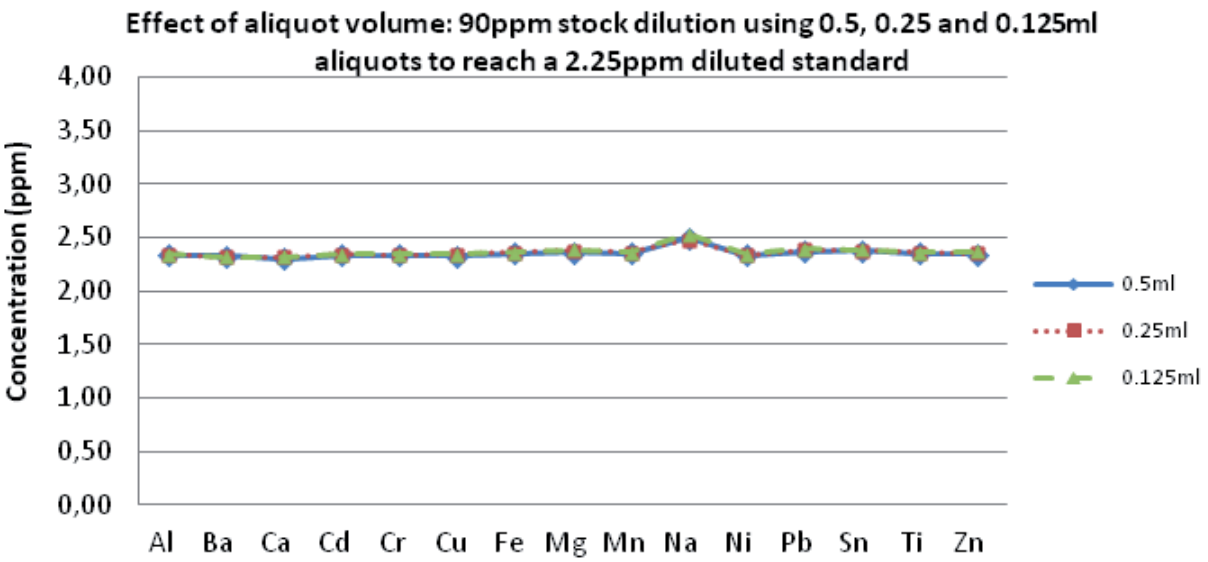
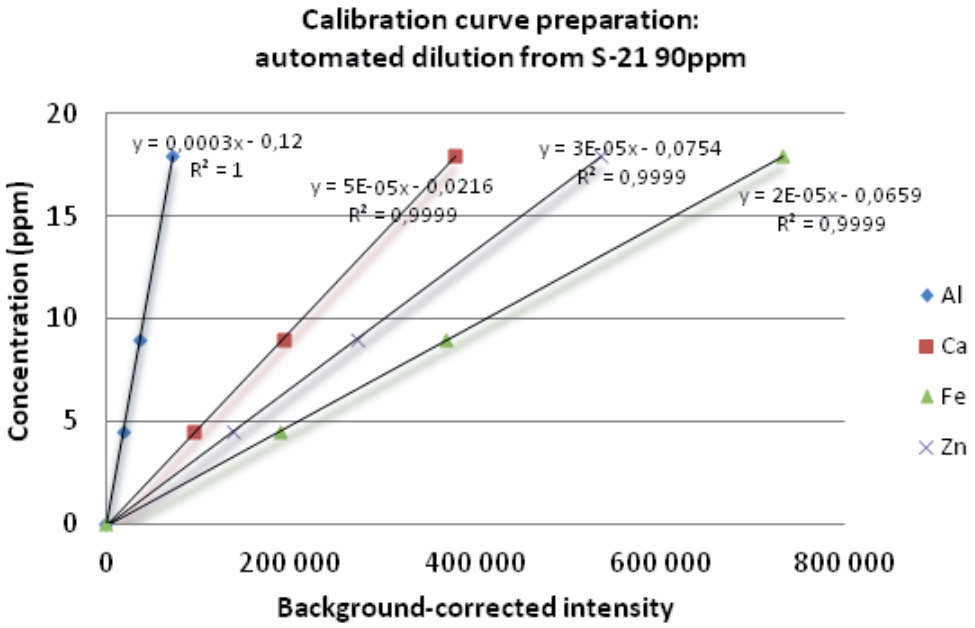
Figure 2:



2- Calibration curve preparation:

A series of calibration standards were prepared using again the S-21 90ppm and the PremiSolv ICP solvent as diluent. Linearity of the obtained regression demonstrates the precision of the liquid dispensing abilities of the Sample Handler.

Figure 3:



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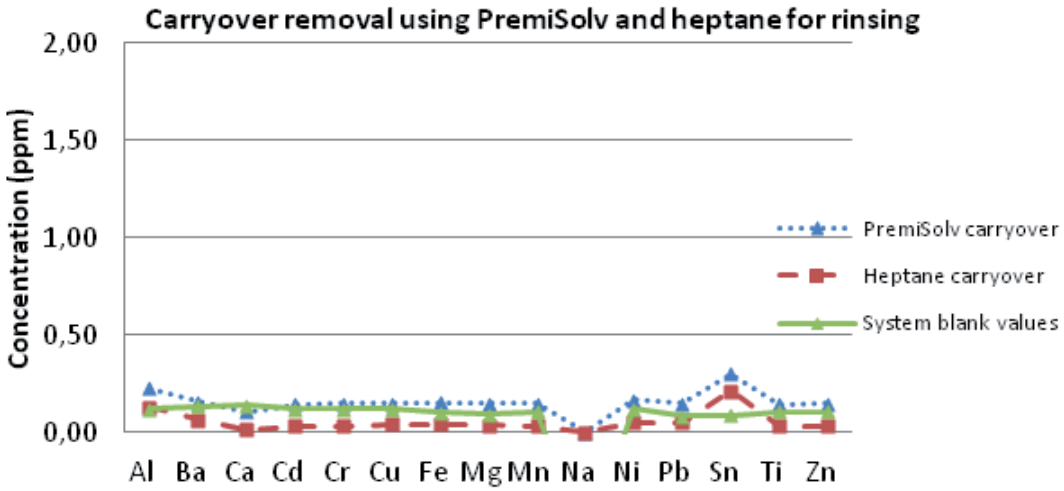
Finally, blank values before and after contact with the 90ppm stock standard are shown in Figure 4. Two solutions were used to rinse the tip of the probe, PremiSolv and heptane, with rinsing volumes of 10ml. Heptane is shown to be an efficient rinsing solution during oil handling.

Figure 4:



Conclusion:

*Easy*PREP Sample Handler provides an automated way to successfully dilute oil samples. Accuracy of sample results was maintained with the volume/volume dilution.

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