## INTRODUCTION

The analysis of volatile samples can be done quite easily on the Exeter Analytical CE-440 analyzers without sacrificing accuracy or precision. Liquid samples are simply injected into sample containers and the containers are sealed. Very volatile samples, in particular petroleum products, tend to creep once placed in the containers. Small portions of the sample may end up above the seal. This requires slight changes to the normal liquid handling procedure. By following this procedure for handling volatile liquids, 440 users can now be assured of quality results.

## EQUIPMENT

| | | |
|---|---|---|
| | Exeter Analytical Capsule Sealer | P/N 313-00012 |
| | Tin Capsules; 6x2.9 mm | P/N 6703-0418 |
| | Nickel Sleeves | P/N 6703-0499 |
| | Syringe, 10 ml | P/N 318-00010 |
| or | Micropipette & Plunger (Pkg 100) | P/N 318-00041 |

## PROCEDURE

Place a tin capsule in a sturdy holder made of grease-free metal. Place the micropipette or syringe, with plunger fully inserted, into the liquid sample. Pull back on the plunger to draw liquid into the pipette or syringe. Approximately 2.5 ul will give you the correct sample weight. Withdraw the micropipette or syringe from the liquid sample and insert it into the tin capsule so that the tip is resting on the bottom of the capsule. Be sure not to touch the inside walls of the tin capsule with the micropipette or syringe at any time during this procedure as this may lead to a poor seal.

Press plunger down until the liquid is in the capsule. Pull the micro pipette or syringe out of the capsule, again without touching the inside walls, and seal the capsule immediately with the Capsule Sealer. Wash the capsule in acetone to remove any liquid that has gotten above the seal. Place the capsule aside for 1 minute to let the acetone evaporate. Weigh the capsule on a microbalance. Once stabilized, if a weight change is observed over a short period of time, either seal the capsule with more force, or dispose of the capsule and repeat the sample loading procedure. Clean the syringe or dispose of the micropipette and clean the plunger with methanol and a dry tissue.

Place the sealed capsule inside a nickel sleeve and proceed with the sample injection, or place the sample in the proper position on the HA-64 wheel. Figure 1 contains data for a volatile liquid analysis, indicating the type of precision that shoud be seen.

Figure 1: Attainable accuracy and precision on volatile samples

| **Sample Run** | **%C** | **%H** |
|---|---|---|
| 1 | 86.90 | 13.07 |
| 2 | 87.05 | 13.14 |
| 3 | 86.94 | 13.15 |
| 4 | 86.94 | 13.14 |
| 5 | 87.00 | 13.12 |
| 6 | 86.90 | 13.12 |
| 7 | 86.68 | 13.06 |
| 8 | 86.84 | 13.07 |
| 9 | 86.75 | 13.03 |
| 10 | 87.01 | 13.13 |
| | | |
| **Mean Values** | **86.90** | **13.10** |

| | |
|---|---|
| Combustion Temperature | 980$^{o}$C |
| Reduction Temperature | 650$^{o}$C |
| Oven Temperature | 80$^{o}$C |
| Combustion Temperature | 30 seconds |
| Purge Time | 30 seconds |
| Weighing Capsules | High Purity Tin |
| Calibration Standard | O.A.S. Acetanilide |