



Elimination of Excess Water for Method 1664A/B by Automated Solid Phase Extraction Utilizing the SPE-DEX[®] 1000/3000XL Controller v2.2

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Introduction

The purpose of this application note is to introduce a modification to the preprogrammed methods on the Horizon Technology SPE-DEX 1000/3000XL Controller with application firmware version 2.2. The methods are utilized in the solid phase extraction (SPE) process to extract samples for EPA Method 1664AB and fulfill QC requirements.

EPA Method 1664 is a performance based method which is used to find the total n-Hexanes extractable material (HEM) and the silica-gel treated n-Hexanes extractable material (SGT-HEM) within a sample. The method specifically outlines the steps needed to perform the liquid-liquid extraction (LLE) and details the process to determine if an alternative method may be used.

The SPE-DEX 1000 and 3000XL Automated Extraction Systems were specifically designed to automatically extract oil and grease from a wide range of clean and dirty aqueous samples using the EPA 1664A or 1664B method. The Speed-Vap III Solvent Evaporation System allows for consistent and gentle heating and air flow over the sample to prevent the loss of volatile compounds. The Solvent Trap Recovery System is designed to recover up to 70% of the n-Hexanes solvent vapors for reuse or disposal. The Horizon Technology Pacific Premium disks were designed to obtain the accuracy and consistency of data needed to comply with method 1664A/B QC specifications.

In software version 2.2, four extraction methods come preloaded: 26, 27, 28, and 29. The first two methods were developed for use with a 47 mm disk and the last two, for use with the 100 mm disk. It has been noted that, upon the completion of these methods, some water is eluted into the collection vessel. The methods introduced within this application note are meant to replace methods 26 and 28 and eliminate the presence of water in the collection vessel in most circumstances.



Horizon Technology Pacific™ Premium SPE Disk



Horizon Technology Speed-Vap™ Evaporation System, SPE-DEX[®] 3000XL Automated Extractor System, Controller v2.2, and Solvent Trap™ Solvent Recovery System.

Instrumentation

- Horizon Technology
 - SPE-DEX[®] 3000XL with Controller v2.2
 - Speed-Vap™ III Evaporation System
 - Solvent Trap™ Recovery System
 - Pacific™ Premium SPE Disks (both 47 mm and 100 mm)
 - Oil & Grease Standard Containing 4 mg/mL Hexadecane and 4 mg/mL Stearic Acid
- Aluminum Weight Pans, 105 mm
- Mettler AE 200 (Balance)
- Silica Gel Sorbent Material
- Glass Wool
- Glass Funnel

Method Summary

1. Obtain eight 1-Liter volumes of DI water.
2. Acidify each with 1:1 Hydrochloric Acid (until pH < 2).
3. Add 5 mL of Oil and Grease Standard to each bottle (total concentration of 40 ppm).
4. Extract four samples using the SPE-DEX 3000XL with 47 mm Pacific Premium SPE Disks and the method in Table 1.
5. Extract four samples using the SPE-DEX 3000XL with 100 mm Pacific Premium SPE Disks and the method in Table 2.
6. Pre-weigh sixteen aluminum pans and add one extract to each of eight of them.
7. Use the Speed-Vap III Evaporation System to evaporate each extract.
8. Weigh each extract's pan and calculate HEM recovery (nominally 40 mg).
9. For the determination of SGT-HEM reconstitute each extract using n-Hexanes.
10. Place glass wool in glass funnel's downspout.

Table 1: Modified Method for 47 mm Disks.

| | Time | | Time |
|----------------------|--------------|------------------------|--------------|
| Prewet Hexane | | Prewet Methanol | |
| Dispense | 6 sec | Dispense | 6 sec |
| Saturate | 1 sec | Saturate | 1 sec |
| Soak | 30 sec | Soak | 30 sec |
| Drain | 3 sec | Drain | 3 sec |
| Air Dry | 3:00 min | | |
| Solvent | Rinse | Soak | Elute |
| 1 Hexane | 4 sec | 45 sec | 45 sec |
| 2 Hexane | 2 sec | 45 sec | 45 sec |
| 3 Hexane | 2 sec | 45 sec | 45 sec |
| Methanol | | | |
| 4 Discard | 2 sec | 10 sec | 2:30 min |
| 5 Hexane | 2 sec | 0 sec | 0 sec |
| Methanol | | | |
| 6 Discard | 0 sec | 10 sec | 2:30 min |
| 7 Hexane | 2 sec | 45 sec | 30 sec |
| 8 Hexane | 2 sec | 45 sec | 30 sec |
| 9 Hexane | 2 sec | 45 sec | 30 sec |
| 10 Hexane | 2 sec | 45 sec | 1:00 min |

Table 2: Modified Method for 100 mm Disks.

| | Time | | Time |
|----------------------|--------------|------------------------|--------------|
| Prewet Hexane | | Prewet Methanol | |
| Dispense | 10 sec | Dispense | 10 sec |
| Saturate | 1 sec | Saturate | 1 sec |
| Soak | 1:00 min | Soak | 1:00 min |
| Drain | 1:00 min | Drain | 5 sec |
| Air Dry | 3:00 min | | |
| Solvent | Rinse | Soak | Elute |
| 1 Hexane | 8 sec | 45 sec | 45 sec |
| 2 Hexane | 7 sec | 45 sec | 45 sec |
| 3 Hexane | 7 sec | 45 sec | 30 sec |
| Methanol | | | |
| 4 Discard | 4 sec | 10 sec | 2:30 min |
| 5 Hexane | 2 sec | 0 sec | 0 sec |
| Methanol | | | |
| 6 Discard | 0 sec | 10 sec | 2:30 min |
| 7 Hexane | 4 sec | 45 sec | 30 sec |
| 8 Hexane | 4 sec | 45 sec | 30 sec |
| 9 Hexane | 4 sec | 45 sec | 30 sec |
| 10 Hexane | 4 sec | 45 sec | 1 min |

- Rinse Silica Gel Sorbent, glass wool and funnel with n-Hexanes and discard rinsate.
- Pass reconstituted extract through the funnel into clean pre-weighed pan making sure to rinse the HEM pan thoroughly (use clean wool, Silica Gel, and pan for each extract).
- Use the Speed-Vap III Evaporation System to evaporate each extract again.
- Weigh each extract and calculate the SGT-HEM recovery (nominally 20 mg).

Results

Table 3: Results of 40 ppm spikes on 47 mm Disk

| | Initial Wt (g) | Final Wt (g) | Recovery (%) |
|----------------|----------------|------------------|--------------|
| HEM | 6.2451 | 6.2849 | 99.5 |
| | 6.3172 | 6.3569 | 99.3 |
| | 6.2475 | 6.2874 | 99.8 |
| | 6.1153 | 6.1549 | 99.0 |
| | Average | | 99.4 |
| | | Deviation | 0.3 |
| SGT-HEM | 6.2991 | 6.3189 | 99.0 |
| | 6.2464 | 6.2665 | 100.5 |
| | 6.2064 | 6.2263 | 99.5 |
| | 6.1257 | 6.1457 | 100.0 |
| | Average | | 99.7 |
| | | Deviation | 0.6 |

Table 4: Results of 40 ppm spikes on 100 mm Disk

| | Initial Wt (g) | Final Wt (g) | Recovery (%) |
|----------------|----------------|------------------|--------------|
| HEM | 6.2217 | 6.2620 | 100.7 |
| | 6.3100 | 6.3498 | 99.5 |
| | 6.3047 | 6.3446 | 99.7 |
| | 6.2967 | 6.3365 | 99.5 |
| | Average | | 99.9 |
| | | Deviation | 0.6 |
| SGT-HEM | 6.2518 | 6.2708 | 95.0 |
| | 6.2363 | 6.2557 | 97.0 |
| | 6.2244 | 6.2436 | 96.0 |
| | 6.2235 | 6.2436 | 100.5 |
| | Average | | 97.1 |
| | | Deviation | 2.4 |

Conclusions

Using the methods outlined above, the Horizon Technology SPE-DEX 1000/3000XL Automated Extraction System fulfills the necessary requirements put forth within the EPA 1664A or 1664B method. At the same time they will eliminate the elution of water into the collection vessel and allow for reduced labor and solvent cost, faster turnaround times, and improvements on both precision and accuracy.

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