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The Pressurized Solvent Extraction (PSE) of Dinitrotoluene (DNT) and Diphenylamine (DPA) from Explosives

Introduction

Explosive manufacturers require fast and accurate analytical methods to control the production of gunpowder. Presently, Soxhlet is used in the extraction of additives like dinitrotoluene and diphenylamine from explosive materials. Unfortunately, this extraction method is labor intensive and requires large volumes of organic solvent.

Pressurized solvent extraction is a new technique that reduces solvent consumption and sample preparation time. Solvent is pumped into an extraction vessel containing the sample and is heated and pressurized. The pressurized solvent at high temperature accelerates the extraction process by increasing the solubility of the analyte in the solvent and also increasing the kinetic rate of desorption of the analyte from the sample matrix.

The *fast* PSE is an automated system which processes six samples simultaneously. The parallel processing technology of the *fast* PSE dramatically increases sample throughput compared to Soxhlet and pressurized solvent extraction systems that employ serial processing. In addition to rapid extraction times, significant reduction in solvent consumption is achieved.

Pressurized solvent extraction can be used to replace Soxhlet and sonication techniques and is approved for use as EPA Method 3545A. This application describes a procedure to extract DNT and DPA from gunpowder using pressurized solvent extraction. Extracts can be obtained in 12 minutes resulting in significant time savings compared to the Soxhlet technique

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Equipment

- ✓ Applied Separations' *fast* PSE Pressurized Solvent Extractor
- ✓ 11 mL Extraction Vessels-Cat.#10625
 - Note: the *fast* PSE can run 6 samples simultaneously
- ✓ Analytical balance
- ✓ HPLC with UV, 280 nm

Solvents and Materials

- ✓ Methanol
- ✓ gunpowder R-5027
- ✓ Collection Vials (60mL for extract collection)- Cat. #10650
- ✓ Cellulose Filter Disks- Cat. #10711
- ✓ S/S Vessel Frits- Cat. #10710
- ✓ Ottawa Sand- Cat. #10584

Summary of Method

 <p>1. Prepare Sample</p>	 <p>2. Load Sample</p>
 <p>3. Run Sample</p>	 <p>4. Collect Extract</p>

**Applied
Separations**

930 Hamilton Street
Allentown, PA 18101
610-770-0900
610-740-5520 (FAX)

www.appliedseparations.com

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Procedure

Prepare Sample

Grind single base gunpowder to 100-200 mesh (150 μ m- 75 μ m).

Load Sample

Prepare the extraction vessels for analysis by placing a cellulose filter disk in the bottom opening followed by a 10 μ m s/s frit, and secure them in place with the retaining nut. Place funnel in top opening and pour the sample mixture through a funnel into the extraction vessel. Add clean Ottawa sand to within 1 cm of the top of the vessel's interior flange as directed by User's Manual.

Place the extraction vessel into the instrument as described in the *fast* PSE operator's manual. Ensure that the pump is primed and that the extraction solvent is Methanol. Place a pre-cleaned collection vial in the instrument for each sample, and program the instrument using the following parameters:

Extraction Conditions

*Program the following extraction parameters on the fast PSE
Program A Mode – 11 mL vessels*

Solvent:	Methanol
Temperature:	100 ° C
Pressure:	100 Bar
Static:	6 minutes
Solvent Module:	1*
Cycles:	1
Pause:	N=0
Flushing Program:	Solvent/gas/repeat flush: 1 min/2 min/0

***Note:** *If automatic solvent selection module is used, enter the appropriate position number (i.e. 2, 3, or 4).*

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Optimize the conditions as needed. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction vessel is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the samples. Any pressure in the range of 100 BAR should suffice. Once established, the same parameters should be used for all samples extracted for the same analysis type.

Collect Extract

Collect each extract in a clean 60mL vial. Allow the extract to cool after the extraction is complete.

Analysis

After extraction sequence is complete analyze samples via reversed phase HPLC with UV detection at 280nm.

Results

DNT and DPA Content of Gunpowder (R-5027)

	PSE (%)	Soxhlet (%)
DNT (dinitrotoluene)	4.38 ± 0.32	4.46 ± 0.17
DPA (diphenylamine)	1.06 ± 0.02	1.17 ± 0.08

Comparison of Soxhlet and PSE

	PSE	Soxhlet
Time	0.2 Hours	8 Hours
Solvent Consumption	15 mLs	50 mLs

Conclusion

Pressurized solvent extraction results compared closely to Soxhlet values. In addition, sample processing time was reduced from hours to minutes, and the solvent consumption was reduced from 50 mLs to 15 mLs using pressurized solvent extraction.

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References

US EPA Method 3545A – Pressurized Fluid Extraction

Kaziunas, A. and Vejrosta, J. “The Pressurized Solvent Extraction of Diphenylamine (DPA) from Explosives.” *Pittcon*. New Orleans, LA., 2000.

Safety

The use of organic solvents, elevated temperatures, and high pressures present potential safety concerns in the laboratory. Common sense laboratory practices can be employed to minimize these concerns. However, the following sections describe additional steps that should be taken.

Extraction vessels in the *fast* PSE oven are hot enough to burn unprotected skin. Allow the vessels to cool before removing them from the oven, or use appropriate protective equipment (e.g. insulated gloves or tongs) as recommended by the manufacturer.

During the gas purge step, some solvent vapors may exit through a vent port in the instrument. Connect this port to a fume hood or other means to prevent release of solvent vapors to the laboratory atmosphere. This precaution also applies to the removal of post extraction solvent from the collected extract.