DIONEX

Application Note 350

Now sold under the Thermo Scientific brand

Thermo SCIENTIFIC

Extraction of Zearalenone from Wheat and Corn by Accelerated Solvent Extraction (ASE®)

INTRODUCTION

Zearalenone (ZON) is a mycotoxin produced by the Fusarium fungus. ZON can be found in a wide variety of plants and soils, and can have negative health effects on animal husbandry and humans. Traditional methods for extracting ZON from soils or animal feed include wrist shaking or blending. These methods normally take 30–60 min per sample with constant lab technician attendance. Because of the time-consuming nature of these traditional extraction techniques, many sample prep labs experience large bottlenecks that hinder the flow of samples to the analytical lab.

Accelerated Solvent Extraction (ASE) is a proven extraction technology that not only helps to eliminate these bottlenecks by decreasing the extraction time, but requires far less technician attendance because it is an automated system. ASE uses increased temperatures to speed up the extraction process, while incorporating high pressure to maintain the solvents in their liquid states at these elevated temperatures. Because of the increased temperatures and pressures, ASE can perform extractions in less than half the time traditional extraction methods require and can do these extractions using very small amounts of solvent.

EQUIPMENT

Dionex ASE 200 Accelerated Solvent Extractor with Solvent Controller (P/N 048765)

22-mL stainless steel extraction cells (P/N 048764)

Dionex Cellulose Filters (P/N 049458)

Dionex Collection Vials, 60 mL (P/N 048784)

Analytical Balance (to read to nearest 0.0001 g or better)

Sand (Ottawa Standard, Fisher Scientific, Cat. No. S23-3)

Laboratory grinder or blender (Fisher Scientific)

Tyler Sieve 0.5 mm (Fisher Scientific)

PTFE Syringe Filter 0.45 µm (Fisher Scientific)

REAGENTS

Dionex ASE Prep DE (P/N 062819)

SOLVENTS

Methanol

Acetonitrile

(All solvents are pesticide-grade or equivalent and available from Fisher Scientific.)

EXTRACTION CONDITIONS

Solvent:	50% methanol, 50% acetonitrile
Temperature:	80 °C
Pressure:	1500 psi
Heatup time:	5 min
Static time:	5 min
Static cycles:	2
Flush:	75%
Purge:	100 s
Total extraction time:	15 min
Volume of solvent used:	25–35 mL

SAMPLE PREPARATION

Grind samples using a laboratory grinder to a powder that can pass through a 0.5-mm sieve. Weigh 5 g of the sample powder into a small beaker and mix thoroughly with 3 g of ASE Prep DE. Mixing the sample with ASE Prep DE ensures a porous mixture that allows the solvent to flow easily through the sample. Add the sample mixture to a 22-mL stainless steel extraction cell containing a cellulose filter. Fill any void volume with Ottawa sand and screw on the end cap.

EXTRACTION PROCEDURE

Place the cells onto the ASE 200. Label the appropriate number of collection vials and place these into the extractor. Set up the method suggested above and begin the extraction. When the extraction is complete, the extract can be diluted to any desired final volume or concentrated for samples containing low levels of contamination. Finally, filter a portion of the extract into an autosampler vial through a 0.45-µm PFTE filter and analyze using LC-MS.[1]

RESULTS AND DISCUSSION

Sample preparation is critical to good recoveries. It is important to grind the samples to a uniform particle size to ensure proper permeation of the solvent into the matrix. Proficiency tests were performed using corn and wheat samples spiked with a ZON standard at 400 ng/g, which showed that MeOH-ACN(1:1) at 80 °C using a 5-min static cycle was optimum for quantitatively extracting all of the ZON from the sample. Because ZON reference material is still not commercially available, two samples used in an international proficiency study were analyzed to evaluate the parameters chosen for the ASE instrument. The samples were extracted in triplicate for both matrices. The results, shown in Table 1, indicate that ASE can provide better results than traditional methods for the extraction of ZON from wheat and corn.

Table 1. Results of Extraction of ZON from Wheat and Corn Using ASE

Sample	Target Value (ng/g)	Average Recovery (ng/g) n = 3	Percent Recovery	Percent RSD
Wheat	112	132	118	5.2
Corn	285	305	107	2.2

CONCLUSIONS

These results confirm that ASE is comparable to traditional extraction methods for the extraction of Zearalenone from wheat and corn. The extraction times of traditional extraction methods usually range from 30 to 60 min per sample, and require large amounts of solvent and constant technician attendance. ASE reduces the extraction time to ~15 min per sample and uses only 25–35 mL of solvent. In addition, the ASE 200 can automatically extract up to 24 samples sequentially without user intervention.

ACKNOWLEDGEMENT

We would like to acknowledge the work of Lea Pallaroni and Christoph von Holst from the European Commission, Institute for Reference Materials and Measurements, Retieseweg, Belgium.

SUPPLIER

Fisher Scientific, 2000 Park Lane, Pittsburgh, PA 15275-1126 USA, Tel: 800-766-7000, www.fishersci.com.

REFERENCES

1. Pallaroni, L.; Holst, C. Determination of Zearalenone from Wheat and Corn by Pressurized Liquid Extraction and Liquid Chromatography-Electrospray Mass Spectrometry. *J. Chromatogr., A* **2003**, *993*, 39–45.

Dionex Corporation
1228 Titan Way
P.O. Box 3603
Sunnyvale, CA
94088-3603
(408) 737-0700

Dionex Corporation
Salt Lake City Technical Center
1515 West 2200 South, Suite A
Salt Lake City, UT
84119-1484
(801) 972-9292

Dionex U.S. Regional Offices
Sunnyvale, CA (408) 737-8522
Westmont, IL (630) 789-3660
Houston, TX (281) 847-5652
Atlanta, GA (770) 432-8100
Marlton, NJ (856) 596-06009

Dionex International Subsidiaries
Austria (01) 616 51 25 *Belgium* (03) 353 42 94 *Canada* (905) 844-9650 *China* (852) 2428 3282 *Denmark* 36 36 90 90
France 01 39 30 01 10 *Germany* 06126-991-0 *Italy* (06) 66 51 50 52 *Japan* (06) 6885-1213 *Korea 82 2 2653 2580*
The Netherlands (0161) 43 43 03 *Switzerland* (062) 205 99 66 *United Kingdom* (01276) 691722

LPN 1609 PDF 4/04