Extraction of Fat from Chocolate Using Accelerated Solvent Extraction

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Key Words
Accelerated solvent extraction, lipid extraction, Soxhlet, Mojonnier Method, Twisselmann Extraction, AOAC Method 922.06, DIN 12602, solvent evaporation, Rocket Evaporator

Introduction

Accelerated solvent extraction is a proven technique that speeds up the gravimetric fat determination of chocolate products and greatly reduces the amount of solvent used. The Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor system uses a combination of elevated temperature and pressure to increase the extraction kinetics, thus decreasing time and solvent consumption. Current methods for determining the fat content in chocolate are labor-intensive and require large amounts of solvent and time (Table 1). By using the Dionex ASE 350 system, extraction time is reduced to 18 min and solvent use is reduced to 20 mL per sample and has been demonstrated to produce superior results to current extraction techniques for fat in chocolate. Furthermore, the accelerated solvent extraction process is fully automated, making it possible to extract up to 24 samples unattended.

Table 1. Comparison of sample preparation techniques for fat extraction in chocolate.

	Dionex ASE 350 System	Mojonnier Method	Soxhlet	Twisselmann Extraction
Solvent per sample	20 mL	140 mL	>100 mL	>100 mL
Time per sample	18 min	2.5–3.5 hours	4 hours	2 hours

This Application Update expands the work performed in Thermo Scientific Application Note 344 by using the Dionex ASE 350 system and Thermo Scientific™ Rocket™ Evaporator. The Dionex ASE 350 system has replaced the Dionex ASE 200 system. The Rocket Evaporator eliminates the need for cumbersome nitrogen stream evaporation.

Equipment

- Dionex ASE 350 Accelerated Solvent Extractor system (P/N 083114, 120 V or 083146, 240 V)
- 10 mL Stainless Steel Extraction Cells (P/N 068087)
- Collection Vials, 60 mL (P/N 048784)
- Cellulose Filters (P/N 068093)
- Analytical Balance (to read to the nearest 0.0001 g or better)
- Mortar and pestle (Fisher Scientific)
- Rocket Evaporator (P/N 075904, 120 V or 082766, 240 V)
- Julabo® Recirculating Chiller, Model FL 601 (P/N 075905 (120 V) or 076364 (240 V))
- Thermo Scientific Dionex ASE Pucks (P/N 075910)

Drying Agents and Dispersants

- Thermo Scientific Dionex ASE Prep DE (diatomaceous earth) (P/N 062819)
- Ottawa Sand (Fisher Scientific)

Solvent

Petroleum Ether (pesticide grade or equivalent; Fisher Scientific)

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Extraction Conditions*	
Solvent:	Petroleum ether 100%
Oven Temperature:	125 °C
Static Time:	3 min
Static Cycles:	3
Flush Volume:	60%
Purge Time:	60 sec
Total Time for Extraction Per Sample:	18 min
Total Solvent Used for Extraction Per Sample:	20 mL

*Pressure is fixed at 1500 psi and does not need to be programmed in the method.

Samples

Baking chocolate and milk chocolate bars were purchased from a local grocery store.

Dionex ASE 350 Accelerated Solvent Extractor system

Sample Preparation

Baking Chocolate Bar

Finely grate the baking chocolate sample. Weigh out 1 g of the grated chocolate and grind with 2 g of Dionex ASE Prep DE using a mortar and pestle. Insert a cellulose filter into a 10 mL extraction cell and transfer the sample/Dionex ASE Prep DE mixture to the cell. Top off any void with Ottawa Sand.

Milk Chocolate Bar

Finely grate the milk chocolate sample. Weigh out 1 g of the grated chocolate and grind with 2 g of Dionex ASE Prep DE using a mortar and pestle. Insert a cellulose filter into a 10 mL extraction cell and transfer the sample/Dionex Prep DE mixture to the cell. Top off any void with Ottawa Sand.

Extraction Procedure

Weigh and label the appropriate number of collection vials and place in the Dionex ASE 350 system vial carousel. Place the loaded cells in the cell carousel. Set up the method described in the "Extraction Conditions" section and begin the extraction. When the extractions are complete, remove the collection vials and place into Dionex ASE Pucks (Figures 1 and 2). Load the Dionex ASE Pucks into the Rocket Evaporator (Figure 3 and 4) and run the appropriate preprogrammed evaporation method. When using petroleum ether, use Evaporation Method 1 for very low boiling point solvents. Note that samples can be removed from the Dionex ASE 350 system and added directly to the Rocket Evaporator. The Rocket Evaporator will evaporate the solvent to dryness and will stop automatically when all solvent is evaporated.

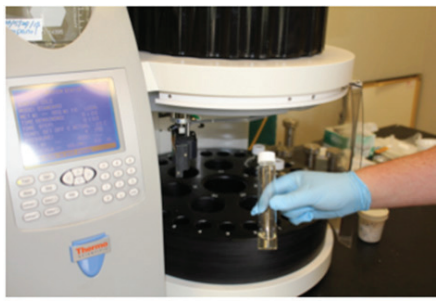

Figure 1. 60 mL vial containing extracts.

Figure 2. Dionex ASE Puck for three 60 mL collection vials.

Figure 3. Extracts are loaded into the Dionex ASE Puck.

Figure 4. Dionex ASE Pucks are loaded into Rocket Evaporator.

Results and Discussion

Application Note 344 demonstrated the extraction efficiency of the Dionex ASE 200 system versus an established method (Mojonnier). This update demonstrates that the Dionex ASE 350 system yields comparable performance to the Dionex ASE 200 system and that the Rocket Evaporator, streamlining the sample preparation workflow by eliminating the need for cumbersome nitrogen stream evaporation.

Table 2 shows the results of the extraction of fat from baking chocolate and milk chocolate using the Dionex ASE 350 system.

Table 2. % Fat* recovery from chocolate.

	Baking Chocolate	Milk Chocolate
Average (n=3)	52.27%	25.27%
Standard Deviation	0.03	0.01
%RSD	0.13	0.07
Label Claim	53%	26%

*% fat = (residue/sample weight) × 100%

Conclusion

The use of the Dionex ASE 350 system with the Rocket Evaporator automates the extraction of fat from chocolate. Accelerated solvent extraction eliminates much of the time and solvent required by conventional extraction methods, using 20 mL of solvent and requiring only 18 minutes per sample. The use of Dionex ASE Pucks and Rocket Evaporator eliminate the need for manual sample transfer and nitrogen stream evaporation. By using the Dionex ASE 350 system and Rocket Evaporator together, the sample preparation workflow for fat extraction in chocolate can be automated for increased productivity and throughput.

References

- Klöck, G., Noke, A, and Christiansen, T. (2013). Soxhlet or Twisselmann, and Evidential Report. Separation, volume number, issue number, pp. 32-33.
- Thermo Scientific Application Note 344. *Extraction of Fat from Chocolate Using Accelerated Solvent Extraction*. 2004.

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