

Extraction of Fat from Chocolate Using Accelerated Solvent Extraction (ASE[®])

INTRODUCTION

Accelerated Solvent Extraction (ASE) is a new way to speed up gravimetric fat determination of chocolate products and greatly reduce the amount of solvent used. The ASE 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. For example, the Mojonnier ether extraction method takes 2–3 h and over 110 mL of solvent and requires the laboratory technician to be present for most of the extraction. Using ASE, extraction time is reduced to 18 min and solvent use to 20 mL. ASE has been shown to produce comparable if not better results than the current methods. Furthermore, the ASE process is fully automated, making it possible to extract up to 24 samples unattended.

In this application note, fat is extracted from baking chocolate, milk chocolate, and cocoa powder using the ASE 200 Accelerated Solvent Extractor. The results are compared to those of the Mojonnier method (AOAC Method 922.06).

EQUIPMENT

ASE 200 Accelerated Solvent Extractor with 11-mL stainless steel extraction cells (P/N 048765)
Cellulose Filters (P/N 049458)
Collection Vials, 40 mL (P/N 048783)
Analytical balance (to read to the nearest 0.0001 g or better)
Mortar and pestle (Fisher Scientific or equivalent)
N-EVAP[®] solvent evaporator (Organomation Associates or equivalent)
Forced air oven

REAGENT

ASE Prep DE (diatomaceous earth) (P/N 062819)

SOLVENT

Petroleum ether (pesticide grade or equivalent; Fisher Scientific)

EXTRACTION CONDITIONS

Solvent:	Petroleum ether 100%
Temperature:	125 °C
Pressure:	1500 psi
Cell Heatup Time:	6 min
Static Time:	3 min
Flush Volume:	60%
Purge Time:	60 s
Cycles:	3
Total Time:	18 min
Total Solvent:	20 mL

SAMPLES

Hershey's® baking chocolate bars, milk chocolate bars, and cocoa powder were purchased from a local food store.

SAMPLE PREP

Baking Chocolate Bar

Finely grate the baking chocolate sample. Weigh out 1 g of the grated chocolate and grind with 2 g of ASE Prep DE using a mortar and pestle. Insert a cellulose filter into an 11-mL extraction cell and transfer the sample/ASE Prep DE mixture to the cell.

Milk Chocolate Bar

Finely grate the milk chocolate sample. Weigh out 1 g of the grated chocolate and grind with 2 g of ASE Prep DE using a mortar and pestle. Insert a cellulose filter into an 11-mL extraction cell and transfer the sample/ASE Prep DE mixture to the cell.

Cocoa Powder

Insert a cellulose filter into an 11-mL extraction cell. Weigh out 1 g of ASE Prep DE into the cell and tare. Weigh out 1 g of cocoa powder onto the ASE Prep DE bed. Carefully mix the cocoa powder and the ASE Prep DE together in the extraction cell using a spatula. (Mixing the cocoa powder with a mortar and pestle causes a slight loss of sample.)

EXTRACTION PROCEDURE

Weigh and label the appropriate number of collection vials and place in the ASE 200 vial carousel. Place the loaded cells in the ASE 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 evaporate the solvent under a nitrogen stream using an N-EVAP or equivalent. Dry each sample in an oven at 102 °C for 30 min. Let these vials cool to room temperature and reweigh.

RESULTS AND DISCUSSION

Tables 1 and 2 show the results of ASE and Mojonnier extractions of fat from baking chocolate and milk chocolate, respectively. The methods show equivalent recoveries in both cases. However, the Mojonnier extractions took approximately 2.5 h and 140 mL of solvent versus approximately 18 min and 20 mL of solvent for ASE.

**Table 1. Baking Chocolate % Fat* Recovery
ASE vs Mojonnier Method (n = 3)**

	ASE	Mojonnier
Average	52.80	51.69
SD	0.35	0.26
RSD	0.67	0.50

*%fat = (residue/sample wt.) × 100

**Table 2. Milk Chocolate % Fat* Recovery
ASE vs Mojonnier Method (n = 3)**

	ASE	Mojonnier
Average	31.80	32.34
SD	0.32	0.33
RSD	1.02	1.02

*%fat = (residue/sample wt.) × 100

Table 3 compares results from ASE and Mojonnier extractions of fat from cocoa powder; again, the recoveries are equivalent. The Mojonnier extraction took approximately 3.5 h and 140 mL of solvents. The cocoa powder required a centrifugation step after each addition of the solvent to achieve a clear separation of the phases. The ASE 200 method took 18 min and 20 mL of solvent.

CONCLUSION

ASE effectively extracts fat from chocolate, producing results comparable to the traditional extraction method. Furthermore, ASE eliminates much of the time and solvent required by the Mojonnier method, using 20 mL of solvent and requiring only 18 min.

SUPPLIERS

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 Organomation Associates, Inc., 266 River Road West, Berlin, MA 01503 USA, Tel: 888-838-7300, www.organomation.com.

**Table 3. Cocoa Powder % Fat* Recovery
 ASE vs Mojonnier Method (n = 3)**

	ASE	Mojonnier
Average	11.82	11.52
SD	0.12	0.15
RSD	1.01	1.33

*%fat = (residue/sample wt.) × 100



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 N-EVAP is a registered trademark of Organomation Associates.
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 * Designed, developed, and manufactured under an NSAI registered ISO 9001 Quality System.

