Supercritical Fluid Application Notes



Extraction of Oil from Oilseeds using Supercritical Fluid

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Introduction

The determination of oil in oilseeds is traditionally accomplished by soxhlet extraction with organic solvents. The solvent-based method remains the most commonly employed method because it is similar to the industrial



solvent extraction process for seed oil.

Alternative techniques using supercritical carbon dioxide to extract seed oil in the laboratory and process operations eliminate the use, exposure to, and disposal of hazardous solvents.

Equipment

- ✓ Applied Separations' *Spe-ed*TMSFE Supercritical Extraction System
- ✓ Modifier pump

Materials

- ✓ Spe-ed Matrix (Cat. #7950)
- ✓ Spe-ed Wool (Cat. #7953)
- ✓ Carbon dioxide welding or industrial grade with dip tube

Method

Weigh 2g of ground oilseeds to an accuracy of ± 0.1 mg. Place a wad of *Spe-ed* Wool into

an extraction vessel and pour the prepared sample into the vessel using a funnel, then place a wad of *Spe-ed* Wool on top. Compress the sample with a tamping rod, fill the void volume with *Spe-ed* Matrix, then seal the vessel. Install the vessel into the *Spe-ed* SFE. Place a pre-dried and preweighed collection vial containing a wad of *Spe-ed* Wool on the discharge tube. Extract sample according to the specified extraction conditions. Remove collection

Extraction Conditions #1

(For to process scale extractable oil with results comparable to AOCS method for determining oil content using the Butt extraction apparatus.)

vial and dry residual moisture from oil to

constant weight (AOAC Method ca 2d –

Extraction vessel: 24mL
Sample: 2g
Pressure: 7500 psi
Temperature: 100°C
Valve temperature: 120°C
CO₂ Flow Rate: 6L/min (gas)

Collection: 60mL pre-weighed vial

Dynamic time: 30 minutes

Extraction Conditions #2

(For total oil content with results comparable to exhaustive extraction methods.)

 $\begin{array}{lll} Extraction \ vessel: & 24mL \\ Sample: & 2g \\ Pressure: & 7500 \ psi \\ Temperature: & 100^{\circ}C \\ Valve \ temperature: & 120^{\circ}C \\ CO_2 \ Flow \ Rate: & 3L/min \ (gas) \\ Ethanol \ Flow \ Rate: & 0.9mL/min \ (liq.) \\ \end{array}$

Collection: 60mL pre-weighed vial

Dynamic time: 30 minutes



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Results

Percent oil – SFE vs Solvent Extraction

	Soyflakes		Canola		Wet	Milled
					Corn	Germ
	SFE	Soxhlet	SFE	Soxhlet	SFE	Soxhlet
%	20.62	20.52	39.82	40.51	48.93	50.43
Oil						
Av.						
SD	0.20	0.15	0.46	0.45	0.45	1.34
RSD	0.98	0.75	1.17	1.12	0.93	2.66

Conclusion

The supercritical carbon dioxide extraction of seed oils offers a viable alternative to solvent-based procedures. The techniques can be applied to high and low oil content seeds containing varying amounts of moisture. The accuracy and precision of the results were comparable to the standard method while extraction times were reduced. In addition, the use of hazardous solvents was eliminated.

References

Taylor, King, JAOCS, Vol. 70, No. 4 (April 1993)

AOCS Official Method AM 3-96 Oil in Oilseeds: Supercritical Fluid Extraction Method

