

Non-Volatile Methylene Chloride Extract

Purpose: To determine the non-volatile residue remaining after evaporation of the methylene chloride extract.

A. Apparatus:

1. Extractor-Goldfisch, Bailey-Walker or equivalent.
2. Desiccator.
3. Oven, forced draft recommended.
4. Steam bath.
5. Balance, 1.0 mg. sensitivity.

B. Reagents:

1. Methylene chloride, anhydrous (dichloromethane), ACS grade.
2. Anhydrous calcium sulfate (Drierite) or silica gel.
3. Diethyl ether, anhydrous (ethyl ether), ACS grade.

C. Preparation of Sample:

1. Use Method 1.0.

D. Procedure:

1. Weigh 2.000g. of sample into a paper extraction thimble, Alundum crucible (porosity RA 360), or cup made of Whatman #1 filter paper. (Note 1)
2. Place sample and container in the extractor. Assemble apparatus and extract with methylene chloride for 20 hr. (Note 2). If residue is to be weighed in the extraction flask, tare flask before assembling.
3. Quantitatively transfer extract to tared beaker or other suitable container such as an aluminum dish of suitable dimensions with a minimum quantity of methylene chloride. Evaporate the methylene chloride on a steam bath under suitable fume hood. Avoid bringing to a boil.

Non-Volatile Methylene Chloride Extract

4. When last traces of methylene chloride have disappeared, place container in hot air oven at $110^{\circ} \pm 2^{\circ}\text{C}$ until two successive weighings taken at 1½ hr. intervals differ by no more than 1 mg. Sample should be cooled to room temperature in desiccator containing drying agent before weighing. The dried residue is the non-volatile methylene chloride extract.

E. Calculation:

$$\% \text{ non-volatile methylene chloride extract} = \frac{\text{Wt. of residue}}{\text{Wt. of sample}} \times 100$$

F. Statistics:

TBD

G. Notes:

1. In the case of spices with low bulk density such as sage, it may be necessary to reduce sample size to accommodate certain extractors.
2. To determine non-volatile ether extract follow above procedure, substituting diethyl ether (anhydrous) for dichloromethane. Observe extreme caution due to flammability of diethyl ether and avoid splattering of extract due to the low boiling point of diethyl ether during evaporation. Methylene chloride is the more efficient and solvent of choice for black and white pepper for piperine extraction.

H. Reference:

AOAC Official Methods of Analysis (1995) 43.1.08 (940.29).
 JAOAC 72,89 (1989).