Rapid Determination of Oil/Fat Utilizing High Temperature Solvent Extraction
(For XT10, XT10I, XT15, XT15I)

DEFINITION
This method determines crude fat by extracting with petroleum ether. The compounds extracted are predominantly triacylglycerides. Small amounts of other lipids along with minor components having some solubility in petroleum ether are also extracted.

SCOPE
This method is applicable to solid processed foods with up to 100% fat.

APPARATUS
1. Analytical Balance—capable of weighing 0.1 mg.
2. Oven—capable of maintaining a temperature of 102 ± 2°C.
3. Extraction instrument—capable of extracting at 90 ± 2.0°C (XT10, XT15, XT20, ANKOM Technology).
4. Filter Bags—constructed from chemically inert and heat resistant filter media, capable of being heat sealed closed and able to retain 1 micron particles while permitting solution penetration (XT4, ANKOM Technology).
5. Heat sealer—sufficient for sealing the Filter Bags closed to ensure complete closure (1915, ANKOM Technology).
6. Desiccator pouch—collapsible sealable pouch with desiccant inside that enables the removal of air from around the Filter Bags (MoistureStop weigh pouch, ANKOM Technology).
7. Marking pen—solvent resistant (F08, ANKOM Technology).

REAGENTS
1. Petroleum Ether (B.P. 35-65° C). Depending on the method used, other solvents such as hexanes and ethyl ether may be used (see Notes, *Caution*).

PROCEDURE
1. Place a labeled Filter Bag on the balance and zero balance.
2. Weigh 1-2 g of sample into the bag and record the weight (W_1).
3. Heat-seal the Filter Bag closed within 4mm of the top to encapsulate the sample.
4. It is good practice to include a blank bag periodically, especially when changing solvents, in order to measure any effect on the Filter Bag.
5. If a meat sample has an expected value of >15% or a plant sample has a value >25%, place the sample bags in tared weigh pans (W_4) during the oven drying procedure in order to capture fat that may migrate out of bag during heating.
6. Place samples in the drying oven for 3 hrs (see Numbered Notes, 1).
7. Cool dried samples in desiccant pouch, weigh and record weight (W_2).
8. Place sample bags into bag holder or carousel and place in extractor.
9. Select the extraction time and proceed according to the extraction instrument's instructions.
10. When the extraction process is complete place samples in the oven for 15-30 min (see Numbered Notes, 2).
11. Cool in Desiccant Pouch and weigh (W_3).

CALCULATION

$$\% \text{ Crude Fat} = \frac{100 \ (W_2 - W_3)}{W_1}$$

Where: W_1 = Original weight of sample.
W_2 = Weight of pre-dried sample with the Filter Bag.
W_3 = Weight of dried sample and Filter Bag after extraction.

For meat samples > 15% and plant samples > 25%:

$$\% \text{ Crude Fat} = \frac{100 \ ((W_2 - W_4) - W_3)}{W_1}$$

Where: W_4 = Weight of the weigh pan.

PRECISION
Results of the collaborative study (see Table 1.) indicates the precision (S_r, RSD_r, r) that the analyst should use as a benchmark for evaluation replication in the same laboratory.

NOTES, *Caution*
Most fat solvents are extremely flammable. Avoid static electricity and use in fume hood.

Table 1. A summary of the statistical analysis of the international collaborative study of the Filter Bag Technique for the proposed Official AOCS Method, "Rapid Determination of Oil/Fat Utilizing High Temperature Solvent Extraction" is presented in this table. Included in the summary is a comparison of the Filter Bag Technique with the results of analysis by AOCS certified laboratories using official AOCS or AOAC methods

Sample ID	oat meal	brownie mix	soybean A	canola	soybean meal	corn A	poultry starter	cattle feed	pig starter	alfalfa	cat food	cookies	bkft cereal	tortilla chips
Number of laboratories	12	12	11	9	12	12	11	10	11	11	12	11	12	12
Number of replicates	24	24	22	18	24	24	22	20	22	22	24	22	24	24
Filter Bag Average, % Crude Fat	5.8	8.7	20.9	39.0	1.6	3.3	3.3	3.2	5.6	2.4	6.3	22.7	2.3	19.9
Certified Labs Average[a]	5.7	8.7	21.1	39.7	1.6	3.6	3.5	3.0	5.5	2.2	6.2	23.1	2.3	20.0
AOCS Am 2-93 Avg. for Oilseeds			21.9	44.8		3.6								
S_r	0.36	0.20	0.35	0.23	0.14	0.31	0.24	0.18	0.20	0.39	0.27	0.20	0.26	0.39
S_R	0.54	0.31	0.63	0.68	0.27	0.42	0.42	0.20	0.28	0.50	0.30	0.20	0.36	0.48
RSD_r, %	6.2	2.3	1.7	0.6	8.5	9.5	7.3	5.6	3.6	16.1	4.2	0.9	11.4	2.0
RSD_R, %	9.4	3.5	3.0	1.7	16.3	12.7	12.6	6.1	5.0	20.7	4.7	0.9	15.7	2.4
r	0.99	0.56	0.98	0.65	0.39	0.88	0.68	0.51	0.56	1.08	0.75	0.56	0.72	1.09
R	1.52	0.86	1.76	1.90	0.75	1.18	1.16	0.55	0.78	1.39	0.83	0.56	1.00	1.35
HORRAT	3.05	1.21	1.19	0.76	4.38	3.87	3.77	1.83	1.61	5.92	1.56	0.35	4.45	0.95

Sample ID	dog food	crackers	turkey	ham	beef ground	chicken breast	soybean B	safflower	potato chips	hot dog	sausage	corn B	cheese curls	corn silage
Number of laboratories	12	10	12	9	11	11	11	9	11	12	11	12	12	12
Number of replicates	24	20	24	18	22	22	22	18	22	24	22	24	24	24
Filter Bag Average, % Crude Fat	6.8	23.8	3.2	11.6	23.8	2.8	19.4	22.5	32.0	39.5	25.7	3.4	30.6	2.3
Certified Labs Average[a]	6.9	24.0	3.2	11.3	23.5	2.7	19.7	23.2	32.0	39.0	25.0	3.7	30.5	2.3
AOCS Am 2-93 Avg. for Oilseeds							20.8	24.7						
S_r	0.35	0.23	0.21	0.30	0.24	0.33	0.38	0.53	0.48	0.35	0.34	0.39	0.48	0.23
S_R	0.35	0.23	0.34	0.30	0.36	0.33	0.62	0.83	0.52	0.59	0.51	0.41	0.69	0.51
RSD_r, %	5.23	0.96	6.57	2.59	1.01	11.89	1.97	2.36	1.49	0.89	1.33	11.48	1.59	9.87
RSD_R, %	5.23	0.96	10.84	2.59	1.49	11.89	3.19	3.69	1.61	1.49	1.98	11.93	2.27	22.45
r	0.99	0.64	0.58	0.84	0.67	0.94	1.07	1.49	1.34	0.98	0.96	1.10	1.36	0.63
R	0.99	0.64	0.96	0.84	0.99	0.94	1.73	2.33	1.45	1.65	1.43	1.14	1.94	1.44
HORRAT	1.74	0.39	3.22	0.94	0.6	3.47	1.25	1.48	0.68	0.65	0.81	3.59	0.95	6.36

[a] AOCS Official Methods Ba 3-38, AOAC 920.39 or equivalent