

Sample Preparation Guide for Elemental Analysis

This booklet provides instruction for preparing the sample for elemental analysis.

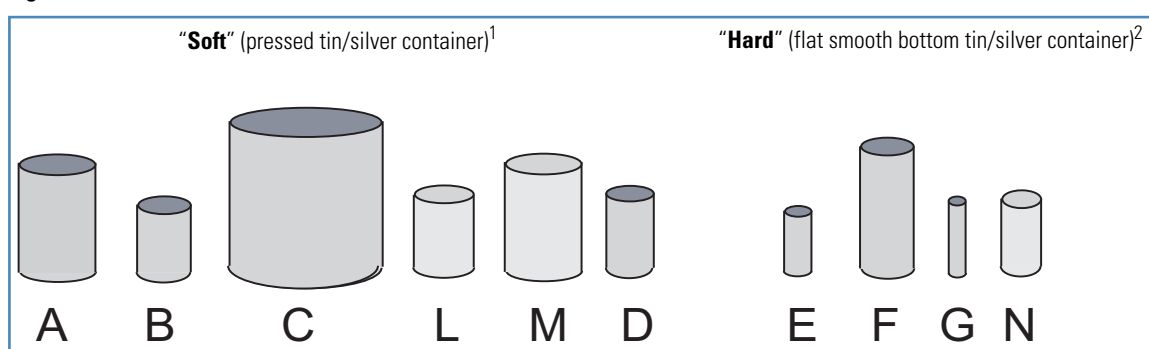
Contents

- OEA Tin and Silver Containers
- Containers Cleaning Procedure
- Pre-treatment of Heterogeneous Material
- Particular Techniques of Sampling in Nitrogen Determination
- Sealing Device for Liquid Samples
- Analytical Standards and Reference Materials

OEA Tin and Silver Containers

Figure 1 shows the containers used for elemental analysis.

Figure 1. Tin and Silver Containers



1. "Soft" containers are obtained by pressing light metal foil.
2. "Hard" tin containers are heavier than the "Soft" ones and allow hermetic sealing. For this reason the "Hard" tin containers are obtained by moulding ultrapure tin sheets.

The containers used for elemental analysis are detailed in [Table 1](#).

Table 1. Tin and Silver Containers (Sheet 1 of 2)

Part Number	Type	Description	Data
240 06400	A	Tin containers for solid substances (Set of 100) Determination of N / CN in solid and non volatile liquid samples. The cleaning procedure is recommended. Closing containers using tweezers	Internal diameter: 5.0 mm Height: 8.0 mm Thickness: 0.02 mm Weight: 22 mg
240 06410 (Megabox)	A	Tin containers for solid substances (Set of 1000) Determination of N / CN in solid and non volatile liquid samples. The cleaning procedure is recommended. Closing containers using tweezers	Internal diameter: 5.0 mm Height: 8.0 mm Thickness: 0.02 mm Weight: 22 mg
240 05300	B	Tin containers for solid substances (Set of 100) Determination of CHNS / N / CN in solid samples. The cleaning procedure is recommended. Closing containers using tweezers	Internal diameter: 3.6 mm Height: 5 – 6 mm Thickness: 0.02 mm Weight: 8.5 mg
252 08000	C	Tin containers for N/Protein and N/Brew analysis (Set of 100) Recommended for N/Proteins and N/Brew determination. Closing containers using tweezers or Sealing Device	Internal diameter: 10.0 mm Height: 10.0 mm
240 08810	D	Ultrapure tin containers for liquid substances (Set of 100) Nitrogen analysis for liquid and volatile liquid substances. Closing containers using tweezers or Sealing Device (P/N 252 09010)	Internal diameter: 2.88 mm Height: 6.0 mm Weight: 24 mg

Table 1. Tin and Silver Containers (Sheet 2 of 2)

Part Number	Type	Description	Data
240 08820	E	Ultrapure tin containers for liquid substances (Set of 100) Trace analysis of CHNS (solid and liquid samples, volatile or not volatile substances). Closing containers using Sealing Device (P/N 252 09010)	Internal diameter: 2.0 mm Height: 4.5 mm Weight: 26 mg
240 07200	F	Ultrapure tin containers for liquid substances (Set of 50) Nitrogen analysis for liquid and volatile liquid substances. Used only for Nitrogen traces in volatile compounds. Closing containers using tweezers or Sealing Device (P/N 252 09010)	Internal diameter: 3.5 mm Height: 9.0 mm Weight: 90 mg
240 07000	G	Ultrapure tin capillary containers for liquid substances (Set of 50) CHNS analysis for volatile liquids. Closing containers using Sealing Device (P/N 252 0900)	Internal diameter: 1.06 mm Height: 5 – 6 mm Weight: 10 mg
252 08015	H	Tin Disks 30 mm diameter (Set of 100) Use Sealing Device (P/N 252 09010)	---
252 08018	I	Tin Disks 35 mm diameter (Set of 100) Use Sealing Device (P/N 252 09010)	---
240 05400	L	Silver containers for solid substances (Set of 100) Oxygen determination in solid samples. Closing containers using tweezers	Internal diameter: 4.0 mm Height: 5 - 6 mm Weight: 8 mg
240 05410 (Megabox)	L	Silver containers for solid substances (Set of 1000) Oxygen determination in solid samples. Closing containers using tweezers	Internal diameter: 4.0 mm Height: 5 - 6 mm Weight: 8 mg
240 05405	M	"Soft" Silver containers (Set of 100) Recommended for Total Organic Carbon (TOC) determination according to the Thermo Scientific method. See Kit for Total Organic Carbon in Solid Sample (P/N 190 14128) in the FlashSmart Consumables and Spare Parts catalog	Internal diameter: 5.0 mm Height: 8.0 mm
240 05100	N	Silver containers for liquid substances (Set of 100) Oxygen determination in liquid samples. Closing containers using Sealing Device (P/N 252 09010)	Internal diameter: 1.5 mm Height: 5.0 mm Thickness: 0.5 mm Weight: 90 mg

This section provides the instructions for cleaning the containers.

❖ **Cleaning Tin Containers for CHNS, Nitrogen and NC Determination**

This procedure is absolutely necessary for **B** and **F** containers type. **D**, **E**, **G** and **H** containers type can be used without pre-treatment but the cleaning procedure is recommended.

Proceed as follows:

1. Place in a clean beaker one or more tin containers sets.
2. Wash twice with CCl_4 (carbon tetrachloride), then with acetone and discard the excess of solvent.
3. Finally wash three times with distilled water and placed the beaker with containers in the oven for 1 hour at 110 °C to ensure complete dryness.

This procedure is recommended for all applications and it is essential for trace determination (element concentration less than 1%).

❖ **Silver Containers for Oxygen Determination**

The Silver containers (**A** and **C** type) may be oxidized to Ag_2O due to the presence of Oxygen in the air.

1. It is possible to reduce the amount of oxidation by heating the silver containers to 350 – 400 °C until the Ag_2O decomposes (when yellowish color on the container surface disappear is a good indication of total decomposition of the oxide, in some cases up to 1 hour at 400°C is required).
2. The heating can be made directly using a muffle.
3. It is necessary to keep the silver containers in tightly closed vessel or under an inert gas environment (Nitrogen, for example).

This section details the sample preparation.

One of the most important parameter in quantitative determination is the extent of sample homogenization obtained prior to analysis.

To ensure the complete homogenization of different kinds of materials proceed as follows:

1. Dry and not too tough materials

Materials like grain, dry bread, coal, peat, plant tissue, and so on.

Grind the material in a cutting mill as much as possible.

If it is not fine enough for analysis, mix the ground material and grind a small portion of it in a mortar mill.

2. Dry mixture of hard, soft and tough materials

Materials like rock-containing soil, shale, mining slags, and so on.

Pass the material first through a crushing mill which crushes the hard material, then, if necessary, through a cutting and if the further treatment is necessary, use a homogenizer or grind in a mortar mill or a mortar.

3. Dry or wet soft materials

Materials like meat, sausages, fish meat, and so on.

Grind into knives mill with liquid Nitrogen.

In this condition the material will become brittle allowing easier powder formation.

The sample homogenization obtained with some techniques may facilitate the weight procedure and allows to obtain representative results.

Table 2 shows a list of **homogenization tools** used according to the sample nature:

Table 2. Homogeneous Tool

Tool	Sample Nature
Mortar (Agatha or Porcelain)	Coals, soils
Coffee Mill	Cereals (barley, meal, and so on)
Hammer Mill	Texture, paper, and so on.
Ball Mill	Solid fuels as coals, cokes, lignites, and so on.
Ball Mill + Liquid Nitrogen	Hard materials as meat, fresh vegetables, tissue, and so on.
Scissors, Blades	Leaves, fibers, plastic products, and so on.

Analysis of liquid samples adsorbed on Chromosorb W. This method is used for analysis of:

- Urine
- Any water solution type
- Physiological liquids
- Blood
- Wine
- Fuels

We used the container type **F** where we introduced one or two tips of spatula of Chromosorb W (80/100 mesh, CWR 100g, P/N 338 026 00). This is an inert material consisting of diatom earths mixed with sodium carbonate.

This substance does not give blank values, consequently the weight should not be recorded.

After obtaining the tare, we introduce the liquid sample by syringe.

The container was sealed using tweezers and quickly weighed.

Water or solvent (non-nitrogen containing solvent) loss did not affect the analytical results.

The Sealing Device, P/N 205 03002, allows the analysis of hygroscopic solid compounds and volatile substances without gain or loss of weight. See Figure 2 on page 4.

Figure 2. Sealing Device



The samples are introduced in the container using a spatula, syringe or a metal needle (for viscous samples).

We use thin container for liquids or tin capillaries depending on the viscosity of the liquid sample, then the container is placed in the sealing device slide.

Before closing, purge the container with a oxygen or helium flow.

The container is tightly closed and accurately weighted using an electronic balance.

For high vapor pressure liquids, using tin capillaries (E type), the treatment with inert gas is not necessary because the vapor pressure is sufficiently high to displace the atmospheric gases.

This section details the analytical standards and the reference materials. See [Table 3](#), [Table 4](#), [Table 5](#), [Table 6](#), and [Table 7](#).

Table 3. Analytical Standards

Part Number	Analytical Standard	Quantity
338 36700	Acetanilide	2 g
338 40022	Aspartic Acid	2 g
338 40023	Aspartic Acid	10 g
338 24400	Atropine	2 g
338 35210	BBOT [2,5 bis (5-tert-butyl-1-benzoxazol-2-yl) thiophene]	2 g
338 22500	Benzoic Acid	2 g
338 22400	Cyclohexanone 2,4-dinitrophenylhydrazone	1 g
338 21951	Fluorene	1 g
338 35220	dl-Methionine	25 g
338 35430	Imidazole	1 g
338 40018	L-Cystine	2 g
338 40019	Nicotinamide	2 g
338 40037	Polyethylene	1 g
338 40038	Polystyrene	1 g
338 25100	Sulphanilamide	2 g
338 40036	Tocophenol Nicotinate	1 g
338 40001	Urea	2 g
338 40002	Urea 1	10 g

Table 4. Reference Materials

Part Number	Analytical Standard	Quantity
338 35240	Lubricant Reference Material	2 g
338 40024	Pasta Reference Material	10 g
338 40027	Pasta Reference Material for Sulfur determination (FPD)	2 g
338 40025	Soil NC Reference Material	5 g
338 40026	Soil NCS Reference Material	2 g
338 19040	OEA FPD Reference Solutions kit	(4 flasks)

Table 5. Analytical Standards Acceptance Values

Standard	N%	C%	H%	S%	O%
Acetanilide	10.36	71.09	6.71		11.84
Aspartic Acid	10.52	36.09	5.30		48.08
Atropine	4.84	70.56	8.01		16.59
BBOT	6.51	72.53	6.09	7.44	7.43
Benzoic Acid		68.85	4.95		26.20
Cyclohexanone 2-4 DNPH	20.14	51.79	5.07		23.00
Fluorene		93.94	6.06		
dl-Methionine	9.39	40.25	7.43	21.49	21.45
Imidazole	41.15	52.93	5.92		
L-Cystine	11.66	29.99	5.03	26.69	26.63
Nicotinamide	22.94	59.01	4.95		13.10
Polyethylene		85.71	14.47		
Polystyrene		91.95	7.84		
Sulphanilamide	16.27	41.84	4.68	18.62	18.58
Tocophenol Nicotinate	2.61	78.46	9.97		8.96
Urea	46.65	20.00	6.71		26.64

Table 6. Reference Materials Acceptance Values

Standard	N%	C%	H%	S%
Lubricant (batch n° 244870)	1.12	82.32	13.82	2.54
Pasta (batch n° 235380)	2.227			
Pasta for OEA/FPD (batch n° 174182)				0.136
Soil NC (batch n° 133317)	0.21	2.29		
Soil NCS (batch n° 133317)	0.21	2.29		0.023

Table 7. OEA/FPD Reference Solutions Kit

Kit
10 ppm of Sulphur (2 mL flask)
50 ppm of Sulphur (2 mL flask)
100 ppm of Sulphur (2 mL flask)
500 ppm of Sulphur (2 mL flask)