

Sample Containers and Crucibles For Organic Analysis

LECO® 628, TruMac, TruSpec-Micro, 828, 928, and 832 Instrument Series

LECO Corporation; Saint Joseph, Michigan USA

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Instruments: FP628 **CN628 CHN628** FP828 **CN828 CHN828** TruSpec-Micro CHN **TruSpec-Micro CHNS** O-Module Tru<u>Mac-N</u> TruMac-CN TruMac-CNS FP928 **CN928 CNS928** C832 S832 SC832 Series (Including DR and HT Models) S-Module

Sample Containers:

502-338 - Small Quick-Cap Capsules 502-382 - Medium Quick-Cap Capsules 502-810 - Large Quick-Cap Capsules 502-040-100 - Small Tin Capsules 502-167 - Medium Tin Capsules 502-825 - Large Tin Capsules 502-186-100 - Small Tin Foil Cups 502-397-400 - Large Tin Foil Cups 528-203-250 (or 529-203) - Ceramic Combustion Boats 528-206 – Non-Porous Ceramic Combustion Boats 502-343 - Nickel Boat Liners 625-505-430 - Nickel Combustion Boats 501-571 - Copper Capsules 502-008 - Tin Plugs 502-293/206 - Silver Capsules (Micro) 601-963 - Tin Capsules (Micro)

Note: Click on an instrument or sample container type to view specific information regarding that instrument or container and its use.



Sample Containers and Crucibles For Organic Analysis

	Small Quick-Cap Capsules	Med. Quick-Cap Capsules	Large Quick-Cap Capsules	<u>Small Tin Capsules</u>	<u>Medium Tin</u> <u>Capsule</u>	<u>Large Tin Capsule</u>	Small Tin Foil Cups	<u>Large Tin Foil Cups</u>	<u>Ceramic</u> Combustion Boats	<u>Non-Porous</u> <u>Ceramic Boats</u>	Nickel Boat Liners	Nickel Combustion <u>Boats</u>	Copper Capsules	Tin Plugs	Silver Capsule (Micro)	<u>Tin Capsules</u> (Micro)
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Vertical Furnaces																
<u>FP628</u>																
N Determination	Х	Х	Х	Х	Х	Х	Х	Х					Х	Х		
<u>CN628</u>																
N Determination	Х	Х	Х	Х	Х	Х	Х	Х					Х	Х		
C, N Determination				Х	Х	Х	Х	Х					Х	Х		
<u>CHN628</u>																
N Determination	Х	Х	Х	Х	Х	Х	Х	Х					Х	Х		
C, H, N Determination				Х	Х	Х	Х	Х					Х	Х		
<u>FP828</u>																
N Determination	Х	Х	Х	Х	Х	Х	Х	Х					Х	Х		
<u>CN828</u>																
N Determination	Х	Х	Х	Х	Х	Х	Х	Х					Х	Х		
C, N Determination				Х	Х	Х	Х	Х					Х	Х		
<u>CHN828</u>																
N Determination	Х	Х	Х	Х	Х	Х	Х	Х					Х	Х		
C, H, N Determination				Х	Х	Х	Х	Х					Х	Х		
TruSpec- Micro																
C,H,N,S Determination															Х	
C,H,N Determination															Х	Х
O-Module (628/Micro)																
O Determination																Х
Horizontal Furnaces																
TruMac-N																
N Determination									Х	Х	Х	Х				
TruMac-CN																
C, N Determination									Х	Х	Х	X*				
TruMac-CNS																
C, N Determination									Х		Х	Χ*				
S Determination									Х							
FP928																
N Determination									Х	Х	Х	Х				
CN928																
C, N Determination									Х	Х	Х	Χ*				
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C, N Determination									Х		Х	X*				
S Determination							-		X					1		
<u>C832</u>																
C Determination					t t				Х	Х	Х					
<u>\$832</u>																
S Determination									Х	Х						
SC832 Series (HT, DR incl.)																
C Determination									Х	Х	Х					
C, S Determination									X	X						
<u>S-Module (628)</u>										·						
S Determination									Х	Х						
Note: Click on an instrum				1									4 i.e. e. 4 .e.			

Note: Click on an instrument or sample container type to view specific information regarding that instrument or container and its use.

* For use at temperatures not to exceed 1100 °C.

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Vertical Furnace Instruments (TruSpec-Micro, 628, 828 Series) Includes: FP628, CN628, CHN628, FP828, CN828, CHN828, and TruSpec-Micro

Nitrogen and Protein Determination (FP628 and FP828)



FP628

FP828

Applicable sample containers:

- Quick-Cap Capsules (Gel Caps)
- Tin Capsules
- Tin Foil Cups
- Copper Capsules and Tin Plugs

Note: Click on a sample container type to view specific information regarding the container and its use.

> Carbon, Hydrogen, and Nitrogen Determination (CN628, CHN628, CN828, and CHN828)



CN628

CHN628

CN828



CHN828

Applicable sample containers:

- Quick-Cap Capsules (Gel Caps) (Nitrogen Determination ONLY)
- Tin Capsules
- Tin Foil Cups
- Copper Capsules and Tin Plugs



 Carbon, Hydrogen, Nitrogen, and Sulfur Determination (TruSpec-Micro)



Applicable sample containers:

- <u>Tin Capsules (Micro)</u> (For use with the TruSpec-Micro CHN)
- <u>Silver Capsules (Micro)</u> (For use with the TruSpec-Micro CHNS)

Note: Click on a sample container type to view specific information regarding the container and its use.

 Oxygen Determination (O-Module for CN628/CHN628 and TruSpec-Micro)



Applicable sample containers:

• Tin Capsules (Micro)



Horizontal Furnace Instruments (TruMac, 928, and 832 Series)

Includes: TruMac-N, TruMac-CN, TruMac-CNS, FP928, CN928, CNS928, C832, S832, and SC832 Series (including SC832DR and SC832HT)

> Nitrogen and Protein Determination (TruMac-N and FP928)



TruMac-N

FP928

Applicable sample containers:

- <u>Ceramic Combustion Boats</u>
- Non-Porous Ceramic Combustion Boats
- <u>Nickel Boat Liners</u>
- <u>Nickel Boats</u>

Note: Click on a sample container type to view specific information regarding the container and its use.

Carbon and Nitrogen Determination (TruMac-CN and CN928)



TruMac-CN

CN928

Applicable sample containers:

- <u>Ceramic Combustion Boats</u>
- Non-Porous Ceramic Combustion Boats
- <u>Nickel Boat Liners</u>
- <u>Nickel Boats</u> (For use at temperatures not to exceed 1100 °C)



 Carbon, Nitrogen and Sulfur Determination (TruMac-CNS and CNS928)



TruMac-CNS

CNS928

Applicable sample containers:

- <u>Ceramic Combustion Boats</u>
- <u>Nickel Boat Liners</u> (For Carbon and Nitrogen Determination ONLY)

Note: Click on a sample container type to view specific information regarding the container and its use.

 Carbon and Sulfur Determination (C832, S832, and SC832 Series, including DR and HT Models)



C832, S832 and SC832 Series

Applicable sample containers:

- <u>Ceramic Combustion Boats</u>
- Non-Porous Ceramic Combustion Boats
- <u>Nickel Boat Liners</u> (Carbon Determination Only)



> Sulfur Determination (S-Module for FP628/CN628/CHN628)



Applicable sample container(s):

- <u>Ceramic Combustion Boats</u>
- <u>Non-Porous Ceramic Combustion Boats</u>



Quik-Cap Capsules (Gel Caps)

Applicable Sample Type: Solid Samples ONLY

Applicable Instrument(s): 628/828 Series (Vertical Furnaces)

Part Number	Description	Dimensions (mm)	Sample Mass (mg)
502-338	Small Quik-Cap Capsules	7.3 D x 18.5 H	50-250
502-382	Medium Quik-Cap Capsules	8.2 D x 20.2 H	50-500
502-810	Large Quik-Cap Capsules	9.5 D x 22.0 H	50-1000



Considerations:

• Sample Type:

- Solid Samples Only
- Cannot be used with liquid samples or samples with a high moisture content (The capsules are made of a gelatin material that dissolves when in contact with liquids/high moisture levels)

• Elemental Determination:

- Can be used for nitrogen and protein determination
- Cannot be used for carbon determination because the capsules themselves contain a significant amount of carbon, which would result in a substantial carbon bias

• Ash Production:

- Quick-Cap Capsules combust completely, resulting in very little ash, therefore the furnace crucible (614-961-110 Porous Reticulated Crucible) does not fill up as quickly with combustion residue as it does when using tin capsules or tin foil cups. As a result, the furnace crucible typically does not need to be replaced as frequently.
- Note: It is important to understand that the required frequency of furnace crucible replacement is also dependent on the combustibility of the material being analyzed.

• Atmospheric Influence:

- Quick-Cap Capsules are left open for analysis, allowing any atmosphere that may be present in the top portion of the capsule to be purged from the capsule within the purge chamber. This decreases the possibility of a nitrogen bias, and eliminates the need for correcting nitrogen results using an atmospheric blank.
- Recommendation(s):
 - The Large Quik-Cap Capsules (502-801) are a good choice for low-density materials, non-homogenous materials or materials with low nitrogen concentrations. The larger capsule size allows for an increased sample mass which is often required for these types of materials.



Tin Capsules

Applicable Sample Type: Solid and Liquid Samples

Applicable Instrument(s): 628/828 Series (Vertical Furnaces)

Part Number	Description	Dimensions (mm)	Sample Mass (mg)
502-040-100	Small Tin Capsules	6.4 D x 15.9 H	50-400
502-167	Medium Tin Capsules	8.7 D x 19.1 H	50-750
502-825	Large Tin Capsules	9.1 D x 21 H	50-1000



Considerations:

- Sample Type:
 - Can be used with both solid and liquid samples
- Elemental Determination:
 - The 502-040-100 Small Tin Capsules and 502-167 Medium Tin Capsules can be used for carbon, hydrogen, and nitrogen determination
 - The 502-825 Large Tin Capsules are for total nitrogen determination ONLY

• Ash Production:

- Tin Capsules oxidize completely, forming tin oxide, which results in a relatively high ash load. Therefore, the furnace crucible (614-961-110 Porous Reticulated Crucible) fills up more quickly with combustion residue than it does when using Quik-Cap capsules. As a result, the furnace crucible may need to be replaced more frequently.
- Note: It is important to understand that the required frequency of furnace crucible replacement is also dependent on the combustibility of the material being analyzed.

• Atmospheric Influence:

- Tin Capsules should NOT be sealed shut in order to avoid trapping atmosphere in the capsule with the sample.
- Leaving the capsule open allows any atmosphere that may be present in the top portion of the capsule to be purged from the capsule when in the purge chamber. This decreases the possibility of a nitrogen bias, and eliminates the need for correcting nitrogen results using an atmospheric blank.

• Recommendation(s):

- The Large Tin Capsules (502-825) are a good choice for low-density materials, nonhomogenous materials, or materials with low nitrogen concentrations. The larger capsule size allows for an increased sample mass, for both solid and liquid samples, which is often required for these types of materials.
- Note: Larger sample masses may result in an increase in ash buildup, requiring more frequent furnace crucible changes. Larger aqueous sample masses may result in stress fractures in the furnace crucible.



Tin Foil Cups

Applicable Sample Type: Solid Samples (and Liquid Hydrocarbons)

Applicable Instrument(s): 628/828 Series (Vertical Furnaces)

Part Number	Description	Dimensions (mm)	Sample Mass (mg)
502-186-100	Small Tin Foil Cups	35.6 L x 35.6 W	50-350
502-397-400	Large Tin Foil Cups	45 L x 45 W	50-1000



Considerations:

• Sample Type:

- Typically not recommended for use with aqueous solutions because liquids have a tendency to wick up the sides of the foil cup due to capillary action. This can result in sample loss when the foil cup is sealed, as some of the solution may be squeezed out of the cup when it is twisted to seal it.
- Liquid hydrocarbons can be analyzed utilizing Tin Foil Cups with the addition of a combustion aid, such as LECO 501-427 Com-Aid[™] for liquids, which prevents the oil from wicking up the sides of the foil cup.

• Elemental Determination:

• Can be used for carbon, hydrogen, and nitrogen determination

• Ash Production:

- Tin Foil Cups oxidize completely, forming tin oxide, which results in a relatively high ash load. Therefore, the furnace crucible (614-961-110 Porous Reticulated Crucible) fills up more quickly with combustion residue than it does when using Quik-Cap capsules, but not as quickly as when using Tin Capsules. As a result, the furnace crucible may need to be replaced more frequently.
- Note: It is important to understand that the required frequency of furnace crucible replacement is also dependent on the combustibility of the material being analyzed.

• Atmospheric Influence:

- Some atmosphere will be trapped with the sample when it is encapsulated in the tin foil cup. This can cause biased nitrogen results at low nitrogen concentrations, therefore an atmospheric blank should be determined and used to correct for the trapped atmosphere.
- An atmospheric blank can be determined by analyzing a non-nitrogen containing material [such as reagent grade sucrose (ground), baked-off Com-Aid for Liquids, or HP Graphite (outgassed)] several times using a similar mass of the material to the mass of the samples being analyzed. The average nitrogen value obtained is considered the atmospheric blank and can be automatically compensated for using the instrument's software. (Refer to the operator's instruction manual for details regarding the setting of an atmospheric blank).



Tin Foil Cup Considerations Continued ightarrow

- Advantage(s):
 - The wide opening of Tin Foil Cups facilitates easy sample addition and weighing.
- Recommendation(s):
 - The Large Tin Foil Cups (502-397-400) are a good choice for low-density materials, non-homogenous materials, or materials with low carbon, hydrogen, or nitrogen concentrations. The larger cup size allows for an increased sample mass which is often required for these types of materials.
 - It is important that care is taken when sealing the Large Tin Foil Cups to ensure that the resulting shape of the sealed cup is not too wide or too long to drop properly from the autoloader. If the sealed cup is not shaped properly, it can get stuck in the autoloader and won't drop into the purge chamber.
 - Note: A larger sample mass may result in an increase in ash buildup, requiring more frequent furnace crucible changes.

Sample Containers and Crucibles For Organic Analysis



Combustion Boats and Boat Liners

Applicable Sample Type: Solid (Ceramic Boats), Liquid (Non-Porous Ceramic Boats and Nickel Boats & Boat Liners)

Applicable Instrument(s): 832, 928, and TruMac Series (Horizontal Furnaces)

Part Number	Description	Sample Mass (g*)
528-203-250 (or 529-203)	Ceramic Boats	0.2 – 3.0
528-206	Non-Porous Ceramic Boats	0.2 – 3.0
625-505-430	Nickel Boats	0.2 – 3.0
502-343	Nickel Boat Liners	0.2 – 3.0

*FP928 and TruMac-N: 1.0 – 3.0 g, 1.0g nominal / CN928 and TruMac-CN: 0.5 – 1.0 g, 0.5g nominal / CNS928 and TruMac-CNS: 0.2 – 0.3 g, 0.2g nominal.



Considerations:

- Sample Type:
 - Ceramic Combustion Boats are mainly recommended for use with solid samples. They are typically not recommended for analysis of liquid samples without the use of a Nickel Boat Liner, as they are made of a porous material. Liquid samples will soak into and through the crucible itself, resulting in sample loss.
 - Ceramic Combustion Boats can be utilized for analysis of some liquid samples (such as hydrocarbons), without using Nickel Boat Liners, with the addition of a combustion aid, such as LECO 501-427 Com-Aid™, or 502-321 Com-Cat™, which prevents the liquid sample from soaking into the crucible.
 - The Non-Porous Ceramic Combustion Boats were specifically designed for Total Organic Carbon (TOC) determination in soil and rock samples requiring acid treatment, as the acid treatment step can be performed directly in the boat. This eliminates the need to transfer an acid treated sample into a combustion boat, and therefore avoids possible sample loss that may occur during sample transfer. These boats prevent the acid solution from soaking into or through the boat itself; therefore, ensuring that all of the acid solution has mixed completely with the sample material. The Non-Porous Combustion Boats are expected to be re-usable for multiple analyses (Refer to the Recommendations section below for specific details).



Combustion Boat and Boat Liner Considerations Continued ightarrow

- The Non-Porous Ceramic Combustion Boats may also be used for other liquid applications. They prevent the sample from soaking into or through the boat; therefore, preventing sample loss.
- The Nickel Combustion Boats and Ceramic Combustion Boats with Nickel Boat Liners can be used with liquid samples.

• Elemental Determination:

- Ceramic Combustion Boats can be used for carbon, nitrogen, and sulfur determination.
- The Non-Porous Ceramic Combustion Boats can be used for Total Organic Carbon (TOC) determination.
- The Non-Porous Ceramic Combustion Boats can be used for Carbon, Nitrogen and Sulfur determination.
- Nickel Boats and Nickel Boat Liners cannot be used for sulfur determination. The presence of nickel, during sulfur determination in a resistance furnace, results in competing reactions which may result in the conversion of SO₂ to SO₃, therefore decreasing analyte recovery.

• Instrument Specifications:

- Nickel Boats (625-505-430) cannot be used in the 832 series. They can ONLY be used in the 928 and TruMac series due to furnace temperature requirements. Nickel Boats cannot be used with furnace temperatures exceeding 1100 °C because they will start to deform, soften or possibly melt.
- Nickel Boat Liners (502-343) can be used with furnace temperatures exceeding 1100 °C (typically not to exceed 1450 °C), as they are a single-use container.

• Atmospheric Influence:

• Combustion Boats and Boat Liners are open vessels, which allows atmosphere to be purged from the boats when in the purge chamber. This decreases the possibility of a nitrogen bias, and eliminates the need for correcting nitrogen results using an atmospheric blank.

• Advantage(s):

• The wide opening of the Combustion Boats and Boat Liners facilitates easy sample addition and weighing.

• Recommendation(s):

- LECO recommends that Ceramic Combustion Boats be baked in a muffle furnace at 1000 °C for a minimum of 40 minutes prior to use. Once the ceramic combustion boats have cooled, they should be transferred to a desiccator for storage. If the ceramic combustion boats are not used within twenty-four hours, they should be re-baked. After baking, ceramic combustion boats should be handled with clean tongs only; do not use fingers.
- Ceramic Combustion Boats are good choice for low-density materials, non-homogenous materials, or materials with low elemental concentrations. The large size of the boats are designed to hold an increased sample mass, which is often required for these types of materials.
- When using the Non-Porous Ceramic Combustion Boats with an autoloader, a sample cool time of 15 seconds should be used in the software method parameters (Cornerstone 3.1.X or greater), and a lining of ~60 g of 501-608 Quartz Wool should be added to the bottom of the bucket to cushion the impact of the boat when it drops into the bucket. Both practices will extend the boat's lifetime.
- The Non-Porous Ceramic Combustion Boats may be reused for multiple analyses (typically 5 to 7 analyses), depending on the nature of the samples and if proper cleaning and handling is utilized; however, they should be visually inspected for any signs of damage or cracking prior to each reuse, and if any damage or cracking is observed, they should be discarded.



Copper Capsule and Tin Plugs

Applicable Sample Type: Hydrocarbons (per ASTM D5291)

Applicable Instrument(s): 628/828 Series (Vertical Furnaces)

Part Number	Description	Dimensions (mm)
501-571	Copper Capsule (Seal w/502-008 Tin Plug)	5.7 D x 17.8 H
502-008	Tin Plugs (for Sealing 501-571 Copper Capsule)	



Considerations:

- Sample Type:
 - Can be used with liquid samples
 - Typically used for analyzing hydrocarbon samples, following ASTM Procedure D5291

• Elemental Determination:

- Can be used for carbon, hydrogen and nitrogen determination
- Ash Production:
 - Copper Capsules and Tin Plugs oxidize completely, forming oxides, which results in a relatively high ash load. Therefore, the furnace crucible (614-961-110 Porous Reticulated Crucible) fills up quickly with combustion residue and as a result will need to be replaced frequently.
 - Note: It is important to understand that the required frequency of furnace crucible replacement is also dependent on the combustibility of the material being analyzed.

• Atmospheric Influence:

- Some atmosphere may be trapped in the Copper Capsule with the sample when it is sealed closed using a Tin Plug. This may cause biased nitrogen results at low nitrogen concentrations, therefore an atmospheric blank may need to be determined and used to correct for the trapped atmosphere.
- An atmospheric blank can be determined by analyzing a non-nitrogen containing material (such as: Paraffin Oil without nitrogen) several times using a similar mass of the material to the mass of the samples being analyzed. The average nitrogen value obtained is considered the atmospheric blank and can be automatically compensated for using the instrument's software. (Refer to the operator's instruction manual for details regarding the setting of an atmospheric blank).



Micro Capsules

Applicable Sample Type: Mainly Solid Samples (Some Liquids)

Applicable Instrument(s): TruSpec-Micro (Vertical Furnace)

Part Number	Description	Dimensions (mm)	Sample Mass (mg)
502-293/206	Micro Silver Capsules	4.0 D X 3.2 H	1-2
601-963	Micro Tin Capsules	3.5 D X 4.0 H	1-2



Considerations:

- Sample Type:
 - Used mainly with solid samples
 - Can be used with some liquid samples (with the use of LECO 502-283 Sorbit® absorbent pads)

• Elemental Determination:

- Silver Capsules should be used for carbon, hydrogen, nitrogen, and sulfur determination.
- Tin Capsules should be used for carbon, hydrogen, and nitrogen determination.
- Tin Capsules cannot be used for sulfur determination due to the potential reaction between tin and sulfur to form tin sulfide which does not decompose in a resistance style furnace at temperatures below 1280 °C, resulting in decreased analyte recovery