

CSN004 Version 4 April 2020



# **Customer Support Note 004**

# **Bake-out method for UNITY and autosamplers**

Disclaimer: It is vital that this Customer Support Note is read carefully before proceeding and that any instructions contained within the document are followed closely. Markes International will not accept responsibility for any damage done to instrumentation or personnel if any instructions within this document are not followed exactly. Any ongoing warranty or contract may be voided if failure to follow these instructions results in damage to the instrumentation. If anything is unclear, you must clarify the details with a Markes representative before proceeding.

This document details the procedure designed to bake-out the thermal desorption (TD) system and clean the system flow path.

# **1. Maverick software**

#### **1.1 TD method**

Set up the TD method as shown below, and save it as the 'Bake-out method' for future use. The software shown is version 5.x.x, but the parameters will be the same for each type of software.

**NOTE:** If your tubes or cold trap contain Chromosorb or Porapak, then the temperature will need to be reduced to avoid damaging the sorbents.

Pre-Desorption	Tube/Samp	ole desorption	Tra	ap Settings
Purge				
1.0	Trap In Line	Split Ratios →	Split On	
200 Flow Path Temp 5.0 Minimum Carrier Pressure 0.1 GC Cycle Time		??? No Split ???	Inlet Outlet Total	Split Ratio Calculator

Figure 1: Method panel showing 'Pre-Desorption' tab.



Pre-Desorption		Tube/Sample desorption	Trap Settings	
Tube Desorb Time 1 10.0	Temp 1 320	🔲 Trap In Line	✓ Split On	

Figure 2: Method panel showing 'Tube/Sample Desorption' tab.

Pre-Desorption	Tube/Sample desorption		Trap Settings
Trap Desorb			
Pre-Trap Fire Purge/min 1.0 Trap Low /ºC 30 Heating Rate ºC/s Tra MAX MAX 32	ap High /ºC	Trap Hold /min  5.0	☑ Split On

Figure 3: Method panel showing 'Trap Settings'.

#### 1.2 Set the gas flows

The desorption and split flows will need to be set to 50 mL/min. If your TD system has MFCs then you can set this in the method panels above. Alternatively, you can set these manually using a flowmeter and the needle valves.

#### **1.3** Link the method

If using a UNITY 1 or UNITY 2, please select the 'Link Method' icon on the toolbar:

File	Edit	View	Link	Instrument	Window	Help
	2	8	<b>e</b>	a 🛍 🕨	<del>ب</del> ه اهر	

Figure 4: 'Link Method' icon in the toolbar.

If using an ULTRA or TD100, then this step is unnecessary.

#### **1.4 Build the sequence**

Add a sample tube to run and select the 'Bake-out method' created above. Select the tick-box next to the 'Recycle' option, outlined in red. This will run the method continuously until you stop the sequence.

🕼 New Sequence (Modified)						
Recycle	Recycle Stop after 1 Recycle:					
ſ		Se	equence Builder			
Set		TYPE M	lethod			

Figure 5: Sequence builder.

#### **1.5** Set up the system

Load an empty or blank sample tube into the system. Set up the GC(–MS) software to run at least 40 recycles, preferably overnight. This will allow the system to bake-out and remove contamination overnight.

# 2. MIC software

#### 2.1 TD method

Set up the TD method as shown below, and save it as the 'Bake-out method' for future use. The software shown is version 2.x.x, but the parameters will be the same for each type of software.

**NOTE:** If your tubes or cold trap contain Chromosorb or Porapak, then the temperature will need to be reduced to avoid damaging the sorbents.

General			
Apply presets for:		Default	~
Standby split on	Flow (mL/min)		50 🖯
Flow path temperature (°C)		Ì	200
		U	
C Overap			
GC cycle time (min)			0.1
Minimum annia anna (ani)		ĺ	
Minimum carrier pressure (psi)		Į	
Pre-desorption			
Prenume	Prepurge time (min)	ĺ	Ê 1.0 ₽
• Hepuige	r repuige unie (min)	l	
Trap In line	Trap flow (mL/min)		50
Split on	Split flow (mL/min)	ſ	50
		l	

Figure 6: Method panel showing 'General presets' and 'Pre-Desorption' tabs.

Tube desorption		
Desorb time 1 (min)		10.0
Desorb temperature 1 (°C)		320
Trap in line	Trap flow (mL/min)	50
☑ Split on	Split flow (mL/min)	50



Trap settings		
C Desold trap		
Trap purge time (min)		1.0
Trap purge flow (mL/min)		50
Trap low temperature (°C)		30
Elevated trap purge		
Elevated trap purge temperature (°C) Trap heating rate (°C/s)		<ul> <li>₹ 25</li> <li>₹</li> <li>MAX ~ ₹</li> </ul>
Trap high temperature (°C)		325
Trap desorb time (min)		5.0
Desorb split on	Split flow (mL/min)	50

Figure 8: Method panel showing 'Trap Settings'.

# 2.2 Set the gas flows

The desorption and split flows will need to be set to 50 mL/min. If your TD system has MFCs, then you can set this in the method panels above. Alternatively, you can set these manually using a flowmeter and the needle valves.

## 2.3 Set up the system

Load an empty or blank sample tube into the system. Set up the GC(–MS) software to run at least 40 recycles, preferably overnight. This will allow the system to bake-out and remove contamination overnight.

### 2.4 Build the sequence

Add a sample tube to run and select the 'Bake-out method' created above. Press 'Play', then tick 'Run sequence a total of', and select 'Continuous'. This will run the method continuously until you stop the sequence.

Edit	Live					
► I t	) 📂 💾	<b>5</b> (	≝ 1≑ 🖊	<b>#</b>   <b>v</b>   <b>v</b>		
	Sample Ty	ре	Comment	Method	Tube	M Run Sequence
1	Sample	~		TD - Bake Out	1	•#• Kun Sequence
						Run sequence a total of 1 times © Continuous Delay Sequence Stat 02/09/2019 v 12:32 v Priority Sequence Start after current sample © Start after current sequence
						OK Cancel

Figure 9: Sequence builder.

#### For all technical support queries, please contact Markes International.

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