

Thermo Scientific

TRACE GC Ultra UFM

Ultra Fast Module Device

Standard Operating Procedures

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HOME



TRACE™ GC Ultra UFM - Chechout of the Ultra Fast Module - Standard Operating Procedures

May 2007 Edition

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Checkout of the Ultra Fast Module (UFM)

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NOTE!

The Standard Operating Procedures here described are related **ONLY** to the column modules listed in this manual. Operating parameters and analytical results for column modules not mentioned in the list will be described in specific dedicated documentation included with the specific module.

SOP Number: TE P0420/05/E - 16 Sept 2005

Scope

The Standard Operating Procedure (SOP) described in this chapter are a series of instructions, operations and test criteria derived from our quality policy procedures used for final testing of the Ultra Fast Module (UFM).

The SOP has been developed to test and verify instrument complete analytical performance after the installation has been completed. This will help you as a guideline, to check if your UFM continues to perform according to the original checkout testing specifications carried out in the factory premises.

However, these tests alone cannot define if the device is not performing according to the original specifications.

The checkout is carried out injecting a standard solution into the column under analytical conditions set according to the injector and detector hardware provided with the GC. Before starting the test checkout, refer to the Parts Referenced and the Analytical Condition required.



NOTE

Each SOP has a proper Registration and Revision Number (e.g. PO407/01/E - 27 March 2002), according to our Quality Management policy.

Preliminary Operations

Before starting checkout, perform the following preliminary operations sequentially:

1. Gas Supply Connections

Connect the gas supplies following the instructions reported in Chapter 4 of the TRACE GC Ultra Site Preparation and Installation Manual.

2. Data Handling Connections

Connect your data handling following the instructions reported in Chapter 5 of the TRACE GC Ultra Site Preparation and Installation Manual.

3. Liner and Septum Installation

— When S/SL injector is used, install the glass liner following the following the instructions reported in Chapter 6 of the TRACE GC Ultra Operating Manual.

— When PTV injector is used, install the silcosteel liner following the instructions reported in Chapter 12 of the TRACE GC Ultra Operating Manual.

4. Column Installation

— When the S/SL injector is used, install the UFM column module in the S/SL-FID configuration as described in Chapter 4 of the UFM Device Instruction Manual.

— When the PTV injector is used, install the UFM column module in the PTV-FID configuration as described in Chapter 4 of the UFM Device Instruction Manual.

5. Column Leak Check

Perform the column leak check following the he instructions reported in Chapter 15 of the TRACE GC Ultra Operating Manual.

6. UFM Calibration

Perform the calibration of the UFM device following the instructions reported in Chapter 2 of the UFM Device Instruction Manual.

7. Column Evaluation

For pressure controlled injector (such as Programmable Temperature Vaporizing Injector), the indirect control of the column flow by automatic pressure programming relies on the calculation and pre-determination of a *column constant*. The *column evaluation* is an automatic measurement of the column resistance that determines the *column constant*.



NOTE Column evaluation assumes there are no leaks in the column connection or gas plumbing lines.

Perform column evaluation following the instruction reported in Chapter 2 of the UFM Device Instruction Manual.

Parts Referenced for S/SL Injector

Table 0-1. FID-S/SL Parts Referenced

Part	Description	Part Number																
Glass Liner	3 mm ID for split injection	453 200 31																
Liner Seal	Graphite seal for glass liner	290 334 06																
Septum	Standard septum for S/SL injector	313 032 11																
Syringe	10 µl size; 50 mm needle length	365 005 25																
Test Mixture	Seven components in Methylene Chloride: <table border="0" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="text-align: left;"><i>Component</i></th> <th style="text-align: right;"><i>Concentration</i></th> </tr> </thead> <tbody> <tr> <td>n-Decane</td> <td style="text-align: right;">0.14 mg/mL</td> </tr> <tr> <td>Octanol</td> <td style="text-align: right;">0.18 mg/mL</td> </tr> <tr> <td>2,6-Dimethylphenol</td> <td style="text-align: right;">0.16 mg/mL</td> </tr> <tr> <td>2,6-Dimethylaniline</td> <td style="text-align: right;">0.16 mg/mL</td> </tr> <tr> <td>Methyl caprate (C10:0 FAME)</td> <td style="text-align: right;">0.21 mg/mL</td> </tr> <tr> <td>Methyl undecanoate (C11:0 FAME)</td> <td style="text-align: right;">0.21 mg/mL</td> </tr> <tr> <td>Methyl laurate (C12:0 FAME)</td> <td style="text-align: right;">0.20 mg/mL</td> </tr> </tbody> </table>	<i>Component</i>	<i>Concentration</i>	n-Decane	0.14 mg/mL	Octanol	0.18 mg/mL	2,6-Dimethylphenol	0.16 mg/mL	2,6-Dimethylaniline	0.16 mg/mL	Methyl caprate (C10:0 FAME)	0.21 mg/mL	Methyl undecanoate (C11:0 FAME)	0.21 mg/mL	Methyl laurate (C12:0 FAME)	0.20 mg/mL	338 190 35
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Methyl laurate (C12:0 FAME)	0.20 mg/mL																	
Gases	Chromatographic-grade purity																	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing Integrator																	

Parts Referenced for PTV Injector

Table 0-2. FID-PTV Parts Referenced

Part	Description	Part Number																
Liner	Silcosteel 2 mm ID	453 220 46																
Liner Seal	Graphite seal for silcosteel liner	290 034 17																
Septum	Standard septum for PTV injector (set of 10)	313 132 25																
Syringe	10 µl size; 50 mm needle length	365 005 25																
Test Mixture	Seven components in Methylene Chloride: <table border="0" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="text-align: left;"><i>Component</i></th> <th style="text-align: right;"><i>Concentration</i></th> </tr> </thead> <tbody> <tr> <td>n-Decane</td> <td style="text-align: right;">0.14 mg/mL</td> </tr> <tr> <td>Octanol</td> <td style="text-align: right;">0.18 mg/mL</td> </tr> <tr> <td>2,6-Dimethylphenol</td> <td style="text-align: right;">0.16 mg/mL</td> </tr> <tr> <td>2,6-Dimethylaniline</td> <td style="text-align: right;">0.16 mg/mL</td> </tr> <tr> <td>Methyl caprate (C10:0 FAME)</td> <td style="text-align: right;">0.21 mg/mL</td> </tr> <tr> <td>Methyl undecanoate (C11:0 FAME)</td> <td style="text-align: right;">0.21 mg/mL</td> </tr> <tr> <td>Methyl laurate (C12:0 FAME)</td> <td style="text-align: right;">0.20 mg/mL</td> </tr> </tbody> </table>	<i>Component</i>	<i>Concentration</i>	n-Decane	0.14 mg/mL	Octanol	0.18 mg/mL	2,6-Dimethylphenol	0.16 mg/mL	2,6-Dimethylaniline	0.16 mg/mL	Methyl caprate (C10:0 FAME)	0.21 mg/mL	Methyl undecanoate (C11:0 FAME)	0.21 mg/mL	Methyl laurate (C12:0 FAME)	0.20 mg/mL	338 190 35
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Methyl laurate (C12:0 FAME)	0.20 mg/mL																	
Gases	Chromatographic-grade purity																	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing Integrator																	

Analytical Conditions Required for S/SL Split Injection

Table 0-3. FID-S/SL Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 0.5 ml/min Constant Flow (*) Hydrogen = 35 mL/min Air = 350 mL/min Make-up Gas: Nitrogen = 30 mL/min
Oven UFM	Initial Temperature = 50 °C Initial Time = 0.1 minute Ramp 1 = 180 °C/minute Final Temperature = 250 °C Final Time = 0.3 minute Block Temperature = 260 °C
Injector	Operating Mode = Split Temperature = 250 °C Split Flow = 150 ml/min (*)
Detector	Base Temperature = 260 °C Detector Signal Range = 10 ⁰
Injected Volume	1 µl + needle of Test Mixture
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 100 Hz

(*) For specific analytical conditions refer to the Column Factory Report provided with the UFM column.

Analytical Conditions Required for PTV CT Split Injection

Table 0-4. FID-PTV Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 0.5 ml/min Constant Flow (*) Hydrogen = 35 mL/min Air = 350 mL/min Make-up Gas: Nitrogen = 30 mL/min
Oven UFM	Initial Temperature = 50 °C Initial Time = 0.1 minute Ramp 1 = 180 °C/minute Final Temperature = 250 °C Final Time = 0.3 minute Block Temperature = 260 °C
Injector	Operating Mode = CT Split Temperature = 250 °C Split Flow = 150 ml/min (*)
Detector	Base Temperature = 260 °C Detector Signal Range = 10 ⁰
Injected Volume	1 µl + needle of Test Mixture
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 100 Hz

(*) For specific analytical conditions refer to the Column Factory Report provided with the UFM column.

OPERATING PROCEDURE

UFM Checkout with FID-S/SL in Split Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Oven > Enable UFM	Yes
Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant flow mode. If not, scroll to **Flow** mode, press **MODE/TYPE** to access the selection menu, then select **con flow**. Scroll to **col. flow** and set the flow value.

LEFT CARRIER ¹		
Col.flow	0.5	0.5
Pressure	200	
Flow mode	con flow<	

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Oven UFM Control Table. Set the column temperature program required.

OVEN (UFM)		
Temp	50.0	50.0
Initial Time		0.10
Ramp 1		180
Final temp		250
Final time 1		0.30
Ramp 2		Off
Block temp.	260	260<

- Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table. Set the required temperature *Temp* setpoint. Verify to operate in **Split** mode. If not, scroll to *Mode*, press **MODE/TYPE** to access the selection menu, then select *Split*.

LEFT INLET (S/SL) ¹		
Temp	250	250
Pressure	200	
Mode:		Split
Total flow		(155.5)
Split flow	150.0	150.0<
Split ratio		300

1. These settings could also be for a left/right inlet.

- Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FID Detector Control Table. Set the required temperature *Base Temp* and the detector gases *H2*, *Air* and *Mkup* required setpoints.

LEFT DETECTOR (FID) ¹		
Flame		Off
Base temp	260	260
Signal pA		(5.5)
Ign.thresh		2.0
Flameout retry		Off
H2	35	35
Air	350	350
Mkup N2	30	30<

1. These settings could also be for a right detector.

- Ignite the FID flame scrolling to *Flame* and pressing **ON**.
- Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the flame-on background offset. Scroll to *Range* and set the electrometer amplifier input range required.

LEFT SIGNAL (FID) ¹	
Output	(1000)
Offset	5500
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline Comp	Off

1. These settings could also be for a right signal.

7. Activate your Data System and set the parameters required for the checkout.
8. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

Baseline Acquisition and Analysis



NOTE

Refer to the Acceptance Values reported in the Table 0-5 according to the data handling in use.

9. With the GC in Stand-by/Prep Run condition, activate the data system for two minutes to evaluate your baseline in isothermal condition.
10. After the baseline evaluation has been completed, set-up the data system to acquire a single run.
11. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 1.1*.

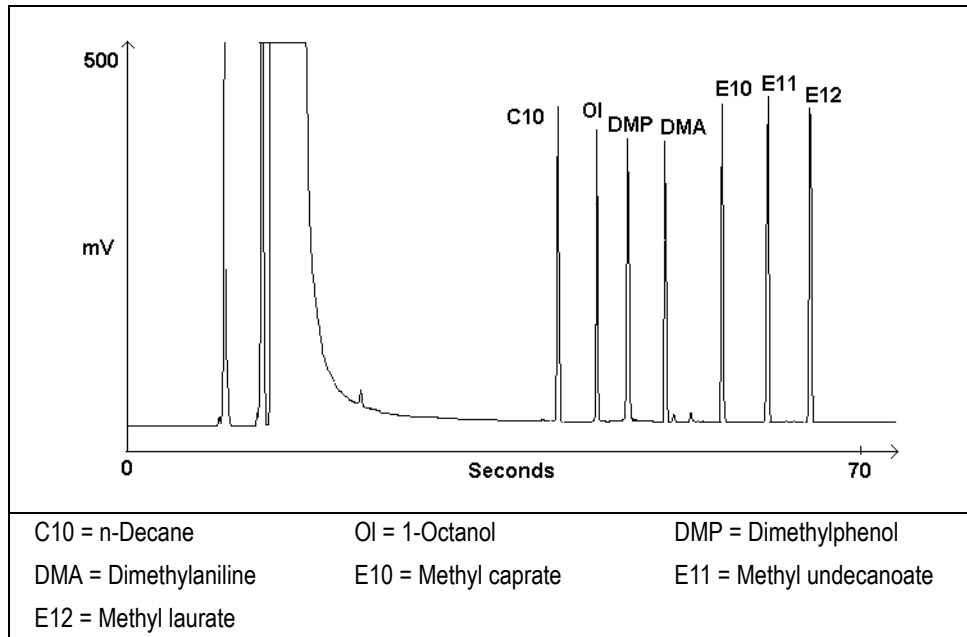


Figure 0-1. FID-S/SL Chromatogram of UFM Test Mix

12. The functionality checkout of the column is considered positive if the obtained data are in compliance to the acceptance criteria reported in Tables 0-5 and 0-6.
13. If these criteria are not met, repeat the test.

OPERATING PROCEDURE

UFM Checkout with FID-PTV in Split Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Oven > Enable UFM	Yes
Right inlet	PTV
Right carrier	He (helium)
Right detector	FID

1. Use **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant flow mode. If not, scroll to **Flow mode**, press **MODE/TYPE** to access the selection menu, then select **con flow**. Scroll to **col. flow** and set the flow value.

RIGHT CARRIER ¹		
Col. flow	0.5	0.5
Pressure	200	
Flow mode	con flow	<

2. Use **OVEN** to display the Oven UFM Control Table. Set the column temperature program required.

OVEN (UFM) ¹		
Temp	50.0	50.0
Initial Time		0.30
Ramp 1		180
Final temp		250
Final time 1		0.30
Ramp 2		Off
Block temp.	260	260<

3. Use **RIGHT INLET** to display the appropriate Programmable Temperature Vaporizing Injector Control Table. Set the required temperature *Temp* setpoint. Verify to operate in **CT Split** mode. If not, scroll to *Mode*, press **MODE/TYPE** to access the selection menu, then select **CT Split**.

RIGHT INLET (PTV)		
Temp	250	250
Pressure	200	
Mode:	CT Split	
Total flow	(155.5)	
Split flow	150.0	150.0<
Split ratio	300	

4. Use **RIGHT DETECTOR** to display the appropriate FID Detector Control Table. Set the required temperature *Base Temp* and the detector gases *H2*, *Air* and *Mkup* required setpoints.

RIGHT DETECTOR (FID) ¹		
Flame		Off
Base temp	260	260
Signal pA		(5.5)
Ign.thresh		2.0
Flameout retry		Off
H2	35	35
Air	350	350
Mkup N2	30	30<

5. Ignite the FID flame scrolling to *Flame* and pressing **ON**.
6. Use **RIGHT SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the flame-on background offset. Scroll to *Range* and set the electrometer amplifier input range required.

RIGHT SIGNAL (FID) ¹	
Output	(1000)
Offset	5500
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline Comp	Off

1. These settings could also be for a right signal.

7. Activate your Data System and set the parameters required for the checkout.
8. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

Baseline Acquisition and Analysis



NOTE

Refer to the Acceptance Values reported in the Table 0-5 according to the data handling in use.

9. With the GC in Stand-by/Prep Run condition, activate the data system for two minutes to evaluate your baseline in isothermal condition.
10. After the baseline evaluation has been completed, set-up the data system to acquire a single run.
11. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 1.2*.

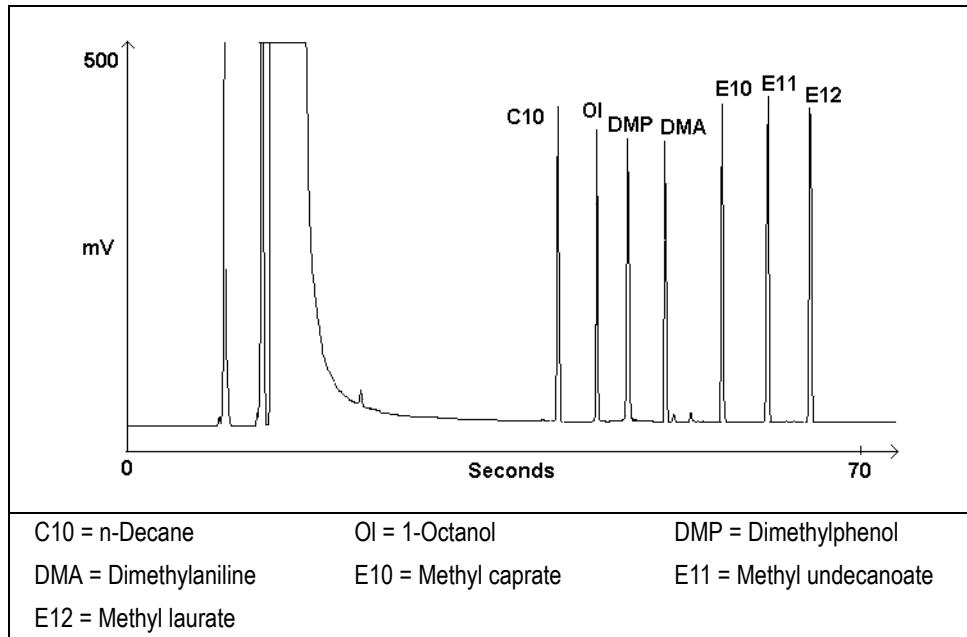





Figure 0-2. FID-PTV Chromatogram of UFM Test Mix


12. The functionality checkout of the column is considered positive if the obtained data are in compliance to the acceptance criteria reported in Tables 0-5 and 0-6.
13. If these criteria are not met, repeat the test.


Table 0-5. FID-S/SL or PTV Acceptance Criteria

CHROM-CARD				
Acceptance Values	Baseline Parameters		Analog (1V Full Scale)	Digital (10V Full Scale)
	Noise (μV)		< 200	< 2 000
	Analytical Results		Analog (1V Full Scale) Area Counts (0.1 μVs)	Digital (10V Full Scale) Area Counts (0.1 μVs)
	Components		> 50 000 for each component	> 500 000 for each component
	Area Count Ratio Calculated as 1-Octano/Decane		1 ± 0.1	1 ± 0.1
	Area Count Ratio Calculated as Dimethylphenol/Decane		1 ± 0.1	1 ± 0.1
	Area Count Ratio Calculated as Dimethylanilina/Decane		1 ± 0.1	1 ± 0.1
	Using Chrom-Card, connected to the TRACE GC Ultra digital output, refer to Analog or Digital Acceptance Values according to the TRACE Signal Time (0 or 1) set in Chrom-Card WCC.INI Configuration.			

Computing-integrator (e.g. ChromJet)	
	When a Computing-integrator (e.g. ChromJet) is used, the peak area counts will result to be 5 times lower than the peak area counts obtained by using Analog Chrom-Card .

CHROMQUEST		
Acceptance Values	Baseline Parameters (1V Full Scale)	
	Noise (μV)	< 200
	Analytical Results (1V Full Scale) - Area Counts (0.01 μVs)	
	Components	> 500 000 for each component
	Area Count Ratio Calculated as 1-Octano/Decane	1 ± 0.1
	Area Count Ratio Calculated as Dimethylphenol/Decane	1 ± 0.1
	Area Count Ratio Calculated as Dimethylanilina/Decane	1 ± 0.1
	Using Chrom-Quest, connected to the TRACE GC Ultra digital output, and using a range of zero, set in the ChromQuest configuration page, a multiplier of 10^{-8} in V scale (or 10^{-5} in mV scale) in order to have equivalency with TRACE GC Ultra displayed signal output. Different range values need a change of the multiplier used in ChromQuest.	

ATLAS	
Baseline Parameters (10V Full Scale)	
Acceptance Values	Noise (µV) < 2 000
Analytical Results (10V Full Scale) - Area Counts (µVs)	
Acceptance Values	Components > 50 000 for each component
Acceptance Values	Area Count Ratio Calculated as 1-Octano/Decane 1 ± 0.1
Acceptance Values	Area Count Ratio Calculated as Dimethylphenol/Decane 1 ± 0.1
Acceptance Values	Area Count Ratio Calculated as Dimethylanilina/Decane 1 ± 0.1
	Using Atlas, connected to the TRACE GC Ultra digital output, make sure that the area counts are expressed in µVs. Refer to the “Report Table Properties”

XCALIBUR	
Baseline Parameters (Acquisition Frequency = 100 Hz)	
Acceptance Values	Noise (Counts) < 2 000
Analytical Results Area Counts (Cts*s)	
Acceptance Values	Components > 500 000 for each component
Acceptance Values	Area Count Ratio Calculated as 1-Octano/Decane 1 ± 0.1
Acceptance Values	Area Count Ratio Calculated as Dimethylphenol/Decane 1 ± 0.1
Acceptance Values	Area Count Ratio Calculated as Dimethylanilina/Decane 1 ± 0.1
	Using Xcalibur, connected to the TRACE GC Ultra digital output, make sure that the acquisition frequency is set to 10 Hz

Analytical Acceptance Comments	
1	When the make-up gas is not used, the acceptance values will result to be 2.5 times lower than the values reported in Table 0-5.
2	When helium is used as make-up gas, the acceptance values will result to be 10 times lower than the values reported in Table 0-5.

Table 0-6. Specific Analytical Acceptance Criteria

UFM Columns	Analytical Results	
	Trennzahl (TZ) E10 - E11	Trennzahl (TZ) E11 - E12
UFM C0000 0010 403	> 8	> 7.5
UFM C0000 0070 401	> 6	> 5.5
UFM C0000 0070 901	> 5	> 4.5
UFM C0000 1010 401	> 10	> 9.5
UFM C0000 1010 501	> 10	> 9.5
UFM C0000 1010 503	> 9.5	> 9
UFM C0000 1010 601	> 10	> 9.5
UFM C0000 1010 606	> 9.5	> 9
UFM C0000 1010 703	> 9.5	> 9
UFM C0000 1070 401	> 6.5	> 6
UFM C0000 1070 404	> 6.5	> 6
UFM C0000 1070 904	> 5.5	> 5
UFM C0000 2010 006	> 9	> 8.5
UFM C0000 2010 207	> 9	> 8.5
UFM C0000 2010 601	> 11	> 10.5
UFM C0000 2010 701	> 11	> 10.5
UFM C0000 2030 306	> 7	> 6.5
UFM C0000 2030 603	> 7	> 6.5
UFM C0000 2070 414	> 3.5	> 3
UFM C0000 3030 303	> 7.5	> 7
UFM C0000 3030 603	> 7.5	> 7
UFM C0010 0000 000	> 9	> 8.5
UFM C0020 0000 000	> 9.5	> 9
UFM C0030 0000 000	> 10	> 9.5
UFM C0040 0000 000	> 10	> 9.5

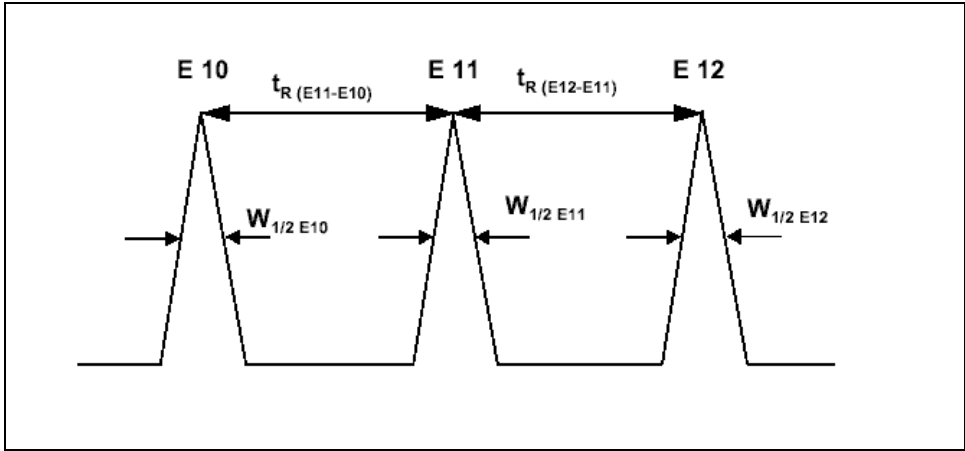
See the appendix [Separation Number \(Trennzahl TZ\) Calculation](#)

Appendix A

Separation Number (Trennzahl TZ) Calculation

The separation efficiency of the column express its capability degree to separate two members of an homologous series according to the following relationship:

$$TZ = \frac{t_{RB} \angle t_{RA}}{W_{0,5(A)} + W_{0,5(B)}} \angle 1$$



- where for the calculation of E 10 - E 11:

t_{RA}	Retention time of E10
t_{RB}	Retention time of E11
$W_{0,5(A)}$	Peak width of E10 at half height
$W_{0,5(B)}$	Peak width of E11 at half height

- where for the calculation of E 11 - E 12:

t_{RA}	Retention time of E11
t_{RB}	Retention time of E12
$W_{0,5(A)}$	Peak width of E11 at half height
$W_{0,5(B)}$	Peak width of E12 at half height

Appendix B

Customer Communications

Thermo Fisher Scientific provides comprehensive technical assistance worldwide and is dedicated to the quality of our customer relationships and services.

How To Contact Us

This appendix contains contact information for Thermo Fisher Scientific office. Use the list reported in *Customer Communication* to contact your local Thermo Fisher Scientific office or affiliate.

Thermo Electron S.p.A
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