Thermo Scientific

TRACE GC Ultra UFM
Ultra Fast Module Device

Standard Operating Procedures

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Thermo
S C I E N T I F I C

Checkout of the Ultra Fast Module (UFM)

Chapter at a Glance…

Operating Procedures

⚠️ **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:		338 190 35
	Component	*Concentration*	
	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	
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:		338 190 35
	Component	*Concentration*	
	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	
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. Scrool to col. flow and set the flow value.

```
          LEFT CARRIER¹

 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<
```

3. 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.

4. 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.

5. Ignite the FID flame scrolling to Flame and pressing **ON**.

6. 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**.
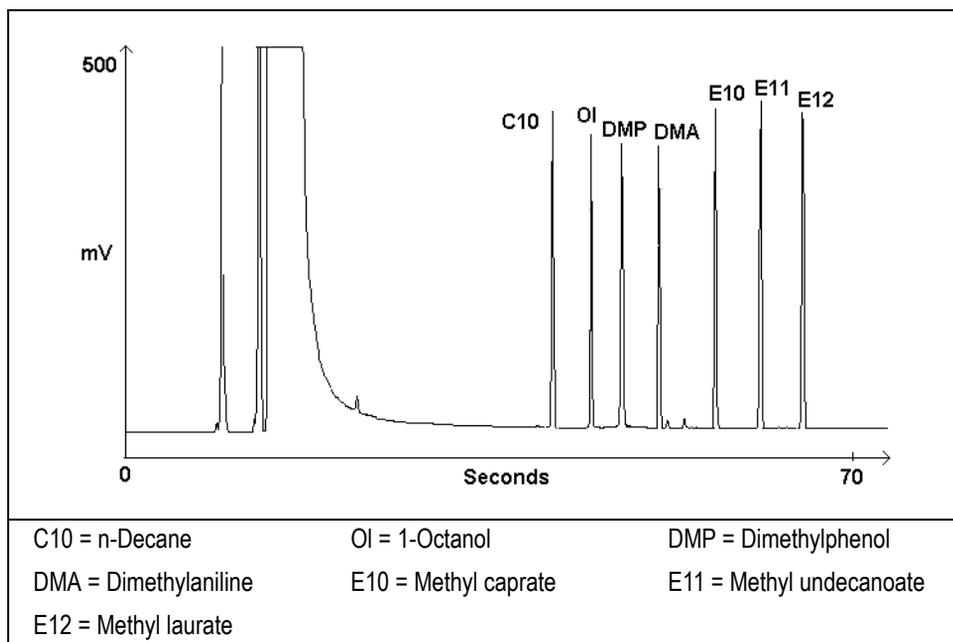
Baseline Acquisition and Analysis

🔍 **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*.

C10 = n-Decane	OI = 1-Octanol	DMP = Dimethylphenol
DMA = Dimethylaniline	E10 = Methyl caprate	E11 = Methyl undecanoate
E12 = Methyl laurate		

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 CARRIER¹

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.

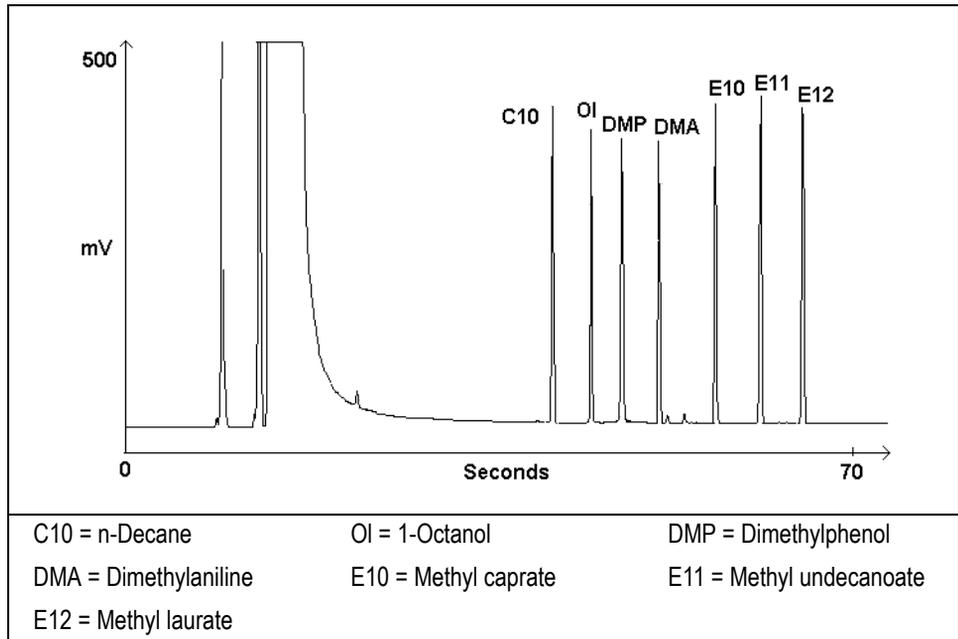8. In the FID Detector Signal Control Table, scroll to Auto zero? and turn it **YES**.

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.*

C10 = n-Decane	OI = 1-Octanol	DMP = Dimethylphenol
DMA = Dimethylaniline	E10 = Methyl caprate	E11 = Methyl undecanoate
E12 = Methyl laurate		

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-Octane/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-Octane/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	
Acceptance Values	**Baseline Parameters** *(10V Full Scale)*	
	Noise (μV)	< 2 000
	Analytical Results *(10V Full Scale) - Area Counts (μVs)*	
	Components	> 50 000 for each component
	Area Count Ratio Calculated as 1-Octane/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 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	
Acceptance Values	**Baseline Parameters** *(Acquisition Frequency = 100 Hz)*	
	Noise (Counts)	< 2 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 500 000 for each component
	Area Count Ratio Calculated as 1-Octane/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 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	
Column Part Number	**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:

$$TZ = \frac{t_{RB} - t_{RA}}{W_{0,5(A)} + W_{0.5(B)}} - 1$$
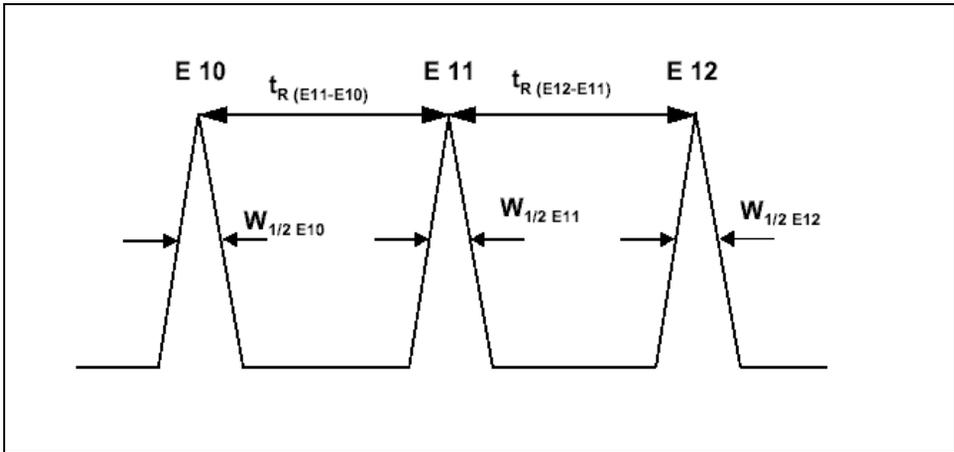
- where for the calculation of E 10 - E 11:

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.

Use http://www.thermo.com/com/cda/resources/resource_detail/1,,12512,00.html address for products information.

Use http://www.gc-gcms-customersupport.com/WebPage/Share/Default.aspx address to contact your local Thermo Fisher Scientific office or affiliate GC-GC/MS Customer Support.