



**Analytical Reference Materials**  
**Grob Test Mix**

**Catalog # 35000**

**Lot # A071903 & A065333**

110 Benner Circle Bellefonte, PA 16823-8812  
(814) 353-1300

FOR LABORATORY USE ONLY. READ MSDS PRIOR TO USE.

RAW MATERIAL TEST INFORMATION AVAILABLE UPON REQUEST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM



# Certificate of Analysis

110 Benner Circle  
Bellefonte, PA 16823-8812  
Tel: (800)356-1688  
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 35000 Lot No.: A065333  
Description : Grob Test Mix  
Expiration Date<sup>1</sup>: December 2011 Storage: Refrigerate

Elution Order	Compound	CAS #	Percent Purity <sup>2</sup>	Concentration <sup>3</sup> (weight/volume)	Percent Uncertainty <sup>4</sup>
1	2,3-Butanediol (+/-)	6982-25-8	99%	530.000 ug/ml	+/-0.04 %
2	n-Decane (C10)	124-18-5	99%	280.000 ug/ml	+/-0.04 %
3	1-Octanol	111-87-5	99%	360.000 ug/ml	+/-0.04 %
4	n-Undecane (C11)	1120-21-4	99%	290.000 ug/ml	+/-0.04 %
5	Nonanal	124-19-6	99%	400.000 ug/ml	+/-0.04 %
6	2,6-Dimethylphenol	576-26-1	99%	320.000 ug/ml	+/-0.04 %
7	2-ethylhexanoic acid	149-57-5	99%	380.000 ug/ml	+/-0.04 %
8	2,6-Dimethylaniline	87-62-7	99%	320.000 ug/ml	+/-0.04 %
9	Methyl decanoate (C10:0 FAME)	110-42-9	99%	420.000 ug/ml	+/-0.04 %
10	Methyl undecanoate (C11:0 FAME)	1731-86-8	99%	420.000 ug/ml	+/-0.04 %
11	Dicyclohexylamine	101-83-7	99%	310.000 ug/ml	+/-0.04 %
12	Methyl dodecanoate (C12:0 FAME)	111-82-0	99%	410.000 ug/ml	+/-0.04 %
Solvent:	Methylene Chloride	75-09-2	99%		

Column:  
30m x .25mm x .5um  
Rtx-5 (cat.#10238)

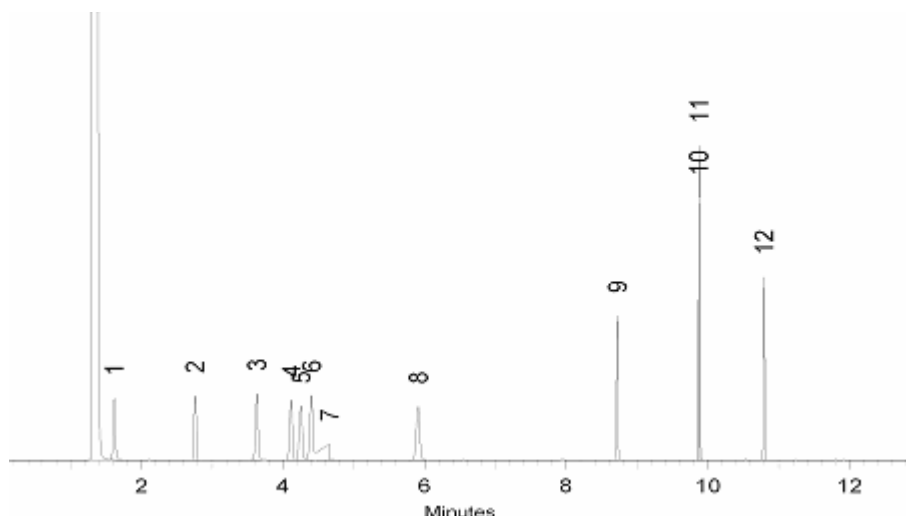
Carrier Gas:  
hydrogen @ 40 cm/sec.

Temp. Program:  
100°C (hold 6 min.) to 330°C  
@ 20°C/min.

Inj. Temp:  
250°C

Det. Temp:  
300°C

Det. Type:  
FID



*Sara Eyster*  
Sara Eyster QA Analyst

Date Passed: 01/30/2009 Balance: 1128353505

Manufactured under Restek's ISO 9001-2000  
Registered Quality System  
Certificate #FMB0397

- 1 Expiration date of the unopened ampule stored at recommended temperature.
- 2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using one or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
- 3 Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
- 4 Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.



## Chemical Standard Batch Sheet

Lot #: A065333

Catalog #: 35000	Target: 280 - 530 ug/ml		
Description: Grob Test Mix			
Solvent: Methylene Chloride	Solvent Lot: 084187		Final Volume: 500 ml

<b>Made by:</b> Michael Maye	<b>Date:</b> 1/19/2009 3:30:28PM		
<b>Tested by:</b> Diane Shaffer	<b>Date:</b> 1/21/2009 2:00:17PM		
Pass	<b>By:</b> Sara Eyster	<b>Date:</b> 1/30/2009 5:05:11PM	
<b>Packaged by:</b> Jessie Emel / Marty Rockey	<b>Date:</b> 1/21/2009 1:26:18PM	<b>No. Units:</b> 353	<b>Pkg Size:</b> 2 mL
<b>Balance Used:</b> ARMAMPPC1 XP205	<b>Serial #:</b> 1128353505		

<u>Compound</u>	<u>CAS</u>	<u>Storage Location</u>	<u>Lot #</u>	<u>Purity (</u>	<u>Target Conc(ug/ml)</u>	<u>Target</u>	<u>Actual</u>	<u>Calc Conc(ug/ml)</u>
Methyl decanoate (C10:0 FAME)	110-42-9	F0072	01305CH	0.99	420.00	210.00 mg	210.00 mg	420.00
Methyl undecanoate (C11:0 FAME)	1731-86-8	R0004	057K2624	0.99	420.00	210.00 mg	210.00 mg	420.00
Methyl dodecanoate (C12:0 FAME)	111-82-0	FA1B10D	01401EE	0.99	410.00	205.00 mg	205.00 mg	410.00
n-Decane (C10)	124-18-5	RA1C11D	08074MD	0.99	280.00	140.00 mg	140.00 mg	280.00
n-Undecane (C11)	1120-21-4	RA1C12B	06607KH	0.99	290.00	145.00 mg	145.00 mg	290.00
1-Octanol	111-87-5	RA1D10C	57052/1	0.99	360.00	180.00 mg	180.00 mg	360.00
Nonanal	124-19-6	RA1D10D	GF01	0.99	400.00	200.00 mg	200.00 mg	400.00
2,6-Dimethylphenol	576-26-1	RA1D11A	52802/1	0.99	320.00	160.00 mg	160.00 mg	320.00
Dicyclohexylamine	101-83-7	RA1D12A	11601HE	0.99	310.00	155.00 mg	155.00 mg	310.00
2,3-Butanediol (+/-)	6982-25-8	RA1G9C	07825EX	0.99	530.00	265.00 mg	265.00 mg	530.00
2,6-Dimethylaniline	87-62-7	RB1D5A	08206LE	0.99	320.00	160.00 mg	160.00 mg	320.00
2-ethylhexanoic acid	149-57-5	RB2H4D	01720MU	0.99	380.00	190.00 mg	190.00 mg	380.00



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FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 35000 Lot No.: A071903  
Description : Grob Test Mix  
Expiration Date<sup>1</sup>: November 2012 Storage: Refrigerate

Elution Order	Compound	CAS #	Percent Purity <sup>2</sup>	Concentration (weight/volume) <sup>3</sup>	% Uncertainty (95% C.L.; K=2) <sup>4</sup>
1	2,3-Butanediol (+/-)	6982-25-8	99%	530.000 ug/ml	+/-0.58 %
2	n-Decane (C10)	124-18-5	99%	280.000 ug/ml	+/-0.58 %
3	1-Octanol	111-87-5	99%	360.000 ug/ml	+/-0.58 %
4	n-Undecane (C11)	1120-21-4	99%	290.000 ug/ml	+/-0.58 %
5	Nonanal	124-19-6	99%	400.000 ug/ml	+/-0.58 %
6	2,6-Dimethylphenol	576-26-1	99%	320.000 ug/ml	+/-0.58 %
7	2-ethylhexanoic acid	149-57-5	99%	380.000 ug/ml	+/-0.58 %
8	2,6-Dimethylaniline	87-62-7	99%	320.000 ug/ml	+/-0.58 %
9	Methyl decanoate (C10:0 FAME)	110-42-9	99%	420.000 ug/ml	+/-0.58 %
10	Methyl undecanoate (C11:0 FAME)	1731-86-8	99%	420.000 ug/ml	+/-0.58 %
11	Dicyclohexylamine	101-83-7	99%	310.000 ug/ml	+/-0.58 %
12	Methyl dodecanoate (C12:0 FAME)	111-82-0	99%	410.000 ug/ml	+/-0.58 %
Solvent:	Methylene Chloride	75-09-2	99%		

## Column:

30m x .25mm x .25um  
Rtx-5 (cat.#10223)

## Carrier Gas:

hydrogen-constant pressure 10 psi.

## Temp. Program:

40°C (hold 2 min.) to 330°C  
@ 10°C/min. (hold 10 min.)

## Inj. Temp:

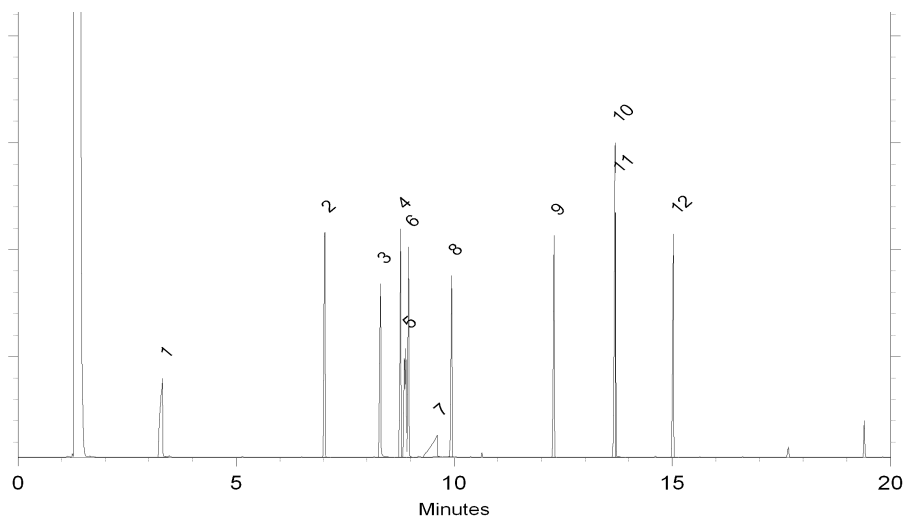
250°C

## Det. Temp:

330°C

## Det. Type:

FID



- 1 Expiration date of the unopened ampule stored at recommended temperature.
- 2A Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest whole number. In addition to detectors listed above, chemical identity and purity are confirmed using one or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
- 2B Compounds with a listed purity of less than 99% have been weight corrected to compensate for impurities.
- 2C Compounds with a listed purity of less than 99% may be salts, derivatives, or hydrates. The listed purity is actually a correction factor that was used to calculate the amount of compound necessary to achieve the desired concentration of the parent compound.
- 2D Purity of isomeric compounds is reported as the sum of the isomers. Value is rounded to the nearest whole number after summation.
- 3 Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
- 4 Uncertainties determined using repeatability and reproducibility data for balances and glassware from measurement systems analysis methodology, balance and glassware tolerances, raw material purity, and, where applicable, eccentricity and linearity values from an accredited calibration laboratory.



## Chemical Standard Batch Sheet

Lot #: A071903

Catalog #: 35000	Target: 280 - 530 ug/ml		
Description: Grob Test Mix			
Solvent: Methylene Chloride	Solvent Lot: 094665		Final Volume: 1,000 ml

<b>Made by:</b> Michael Maye	<b>Date:</b> 12/17/2009 12:30:26PM		
<b>Tested by:</b> Adam Clark	<b>Date:</b> 12/18/2009 10:54:21AM		
Pass	<b>By:</b> Adam Clark	<b>Date:</b> 12/23/2009 1:44:08P	
<b>Packaged by:</b> Jessie Emel / Joe Conway	<b>Date:</b> 12/18/2009 8:52:35/	<b>No. Units:</b> 658	<b>Pkg Size:</b> 2 mL
<b>Balance Used:</b> ARMAMPPC1 XP205	<b>Serial #:</b> 1128353505		

<u>Compound</u>	<u>CAS</u>	<u>Storage Location</u>	<u>Lot #</u>	<u>Purity (</u>	<u>Target Conc(ug/ml)</u>	<u>Target</u>	<u>Actual</u>	<u>Calc Conc(ug/ml)</u>
Methyl decanoate (C10:0 FAME)	110-42-9	F0072	61796LJ	0.99	420.00	420.00 mg	420.00 mg	420.00
Methyl undecanoate (C11:0 FAME)	1731-86-8	R0004	057K2624	0.99	420.00	420.00 mg	420.00 mg	420.00
Methyl dodecanoate (C12:0 FAME)	111-82-0	R0005	1375564	0.99	410.00	410.00 mg	410.00 mg	410.00
Dicyclohexylamine	101-83-7	R0287	11601HE	0.99	310.00	310.00 mg	310.00 mg	310.00
2,3-Butanediol (+/-)	6982-25-8	R0383	07825EX	0.99	530.00	530.00 mg	530.00 mg	530.00
n-Decane (C10)	124-18-5	R0531	69396PJ	0.99	280.00	280.00 mg	280.00 mg	280.00
n-Undecane (C11)	1120-21-4	R0533	06607KH	0.99	290.00	290.00 mg	290.00 mg	290.00
1-Octanol	111-87-5	R0652	57052/1	0.99	360.00	360.00 mg	360.00 mg	360.00
Nonanal	124-19-6	R0653	GF01	0.99	400.00	400.00 mg	400.00 mg	400.00
2,6-Dimethylphenol	576-26-1	R0654	52802/1	0.99	320.00	320.00 mg	320.00 mg	320.00
2,6-Dimethylaniline	87-62-7	R0851	08206LE	0.99	320.00	320.00 mg	320.00 mg	320.00
2-ethylhexanoic acid	149-57-5	R1884	01720MU	0.99	380.00	380.00 mg	380.00 mg	380.00

# QA Report: Grob Test Mix (Cat.#35000)

<u>COMPONENT</u>	Runs of Lot # A071903						Runs of Lot # A065333						%D MEAN	<b>P/F</b>
	Run #1	Run #2	Run #3	AVG	STD DEV	% RSD	Run #1	Run #2	Run #3	AVG	STD DEV	% RSD		
2,3-Butanediol (+/-)	697392	699243	703246	699960	2992	0.43	695730	713783	712593	707369	10097	1.43	-1.06	PASS
n-Decane (C10)	787698	785289	792025	788337	3413	0.43	781858	802774	798331	794321	11020	1.39	-0.76	PASS
1-Octanol	827999	828136	832900	829678	2791	0.34	824140	841035	838749	834641	9166	1.10	-0.60	PASS
n-Undecane (C11)	825852	823837	829951	826547	3116	0.38	818970	837895	834302	830389	10051	1.21	-0.46	PASS
Nonanal	366898	366928	370802	368209	2245	0.61	349373	376569	353826	359923	14587	4.05	2.25	PASS
2,6-Dimethylphenol	433901	434988	436433	435107	1270	0.29	406082	394132	413872	404695	9943	2.46	6.99	PASS
2-Ethylhexanoic acid	829383	828173	832935	830164	2475	0.30	819239	836305	833346	829630	9120	1.10	0.06	PASS
2,6-Dimethylaniline	454707	480128	489497	474777	18002	3.79	464809	491897	483956	480221	13925	2.90	-1.15	PASS
Methyl decanoate (C10:0 FAME)	839153	837436	842834	839808	2758	0.33	779693	796590	792865	789716	8878	1.12	5.96	PASS
Methyl undecanoate (C11:0) & Dicyclohexylamine	909307	908375	912819	910167	2343	0.26	901285	915884	914577	910582	8078	0.89	-0.05	PASS
Methyl dodecanoate (C12:0 FAME)	1736268	1735866	1744320	1738818	4769	0.27	1724128	1748039	1748721	1740296	14006	0.80	-0.09	PASS

Does your GROB Test Mix look like this ( On an Rtx-5 type Column ) ?

OR WORSE !

a Dirty active - NON-deactivated liner may even show NO sample peaks at the Ng level

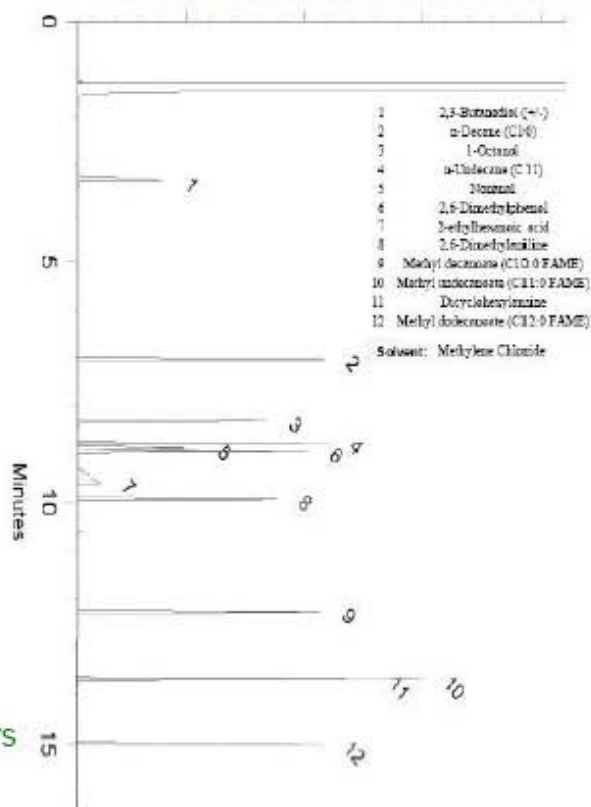
Go to Dianostics Page for explanation

A GOOD GROB COLUMN TEST

Siilcotek provides a RE-deactivation service for "used" liners  
- restores to NEW condition

Guaranteed 99+% Inert

99+% inert Guaranteed





# some GROB Tests - DIAGNOSTICS

Chromatogram showing detector response over time. The x-axis represents time in minutes, and the y-axis represents detector response. The chromatogram shows several peaks, with some labeled with 'X' marks. The peaks are identified as follows: 1. Solvent tail flashback or too large an injection - 2. Symptomatic of Direct injection and flashback (hard to avoid unless sample is <0.1ul) 3. Butanediol some lost ??? 4. If tailing ? - ineffective silane treatment (if used as an alternative) but other "polar" peaks can be lost completely 5. Decane pretty good 6. Often the case even with faulty liner - but other peaks can still be problematic 7. Octanal (oxidation O2 in carrier maybe) 8. 26-DMP 9. 26-DMA DMP/DMA ratio - can indicate acid/base nature of glass used 10. Octanoic acid (not unusual on nonpolar columns w/o perfect deactivation) 11. Interfacial tension problem (solubility) 12. "Bump" can be in between injection trapping of back flash stuff onto column into septum or injector body 13. Varies with time between ??? 14. Hooks on most peaks suggest a minor fitting issue (dead volume) - becomes much more pronounced as sample size decreases 15. Last 3 peaks FAMES ??? 16. Normally are near perfect!

solvent tail flashback or too large an injection -

symptomatic of Direct injection and flashback (hard to avoid unless sample is <0.1ul)

butanediol some lost ???

if tailing ? - ineffective silane treatment (if used as an alternative)  
but other "polar" peaks can be lost completely

decane pretty good

often the case even with faulty liner

- but other peaks can still be problematic

octanal (oxidation O2 in carrier maybe)

26-DMP

26-DMA DMP/DMA ratio

- can indicate acid/base nature of glass used

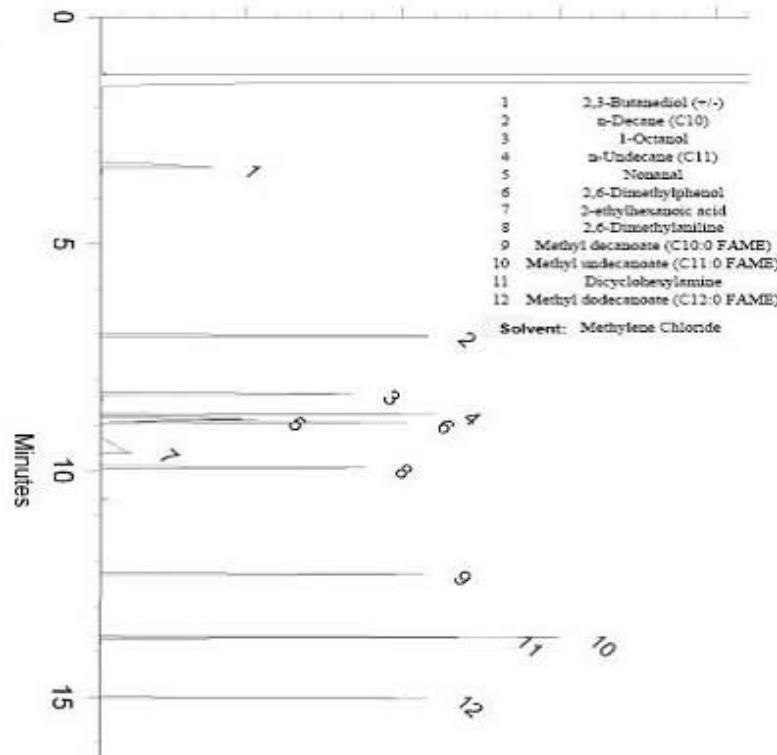
octanoic acid (not unusual on nonpolar columns w/o perfect deactivation)  
interfacial tension problem (solubility)

"Bump" can be in between injection trapping of back flash stuff onto column into septum or injector body  
varies with time between ???

A GOOD Column - GROB Test

hooks on most peaks suggest a minor fitting issue (dead volume)  
- becomes much more pronounced as sample size decreases

last 3 peaks FAMES ???  
normally are near perfect!



## Other Hints ...

tailing and/or reduced decane

... dirt or septum particles in liner

graphite ferrule specs in column ends - check ... break off 10mm from column end

loss of peaks ??? ...

a bad : active or contaminated liner - all peaks can be lost

- replace with SILTEK deactivated liner

SilcoTEK provides a RE-deactivation Service to restore inertness to >99% Guaranteed!

**CHROMALYTIC** +61(0)3 9762 2034  
**ECHnology** Pty Ltd

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Importers & Manufacturers  
www.chromtech.net.au

Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 ... in AUSTRALIA

Sensitive, selective detection and identification of a wide variety of chemical species is a necessity in many applications including chemical warfare agents (CWAs), explosives, environmental pollutants, as well as other toxic industrial compounds and materials (TICs/TIMs). In many instances, field-deployable sensors are preferred because valuable time, resources and chemical information are conserved by performing analyses directly on-site, rather than retrieving samples to be studied at a later time in the laboratory. The ICx family of mass spectrometers have been developed to fulfill this need. Based on Cylindrical Ion Trap (CIT) technology, the products are compact and transportable. One widely applicable sample introduction method supported is a Low Thermal Mass Gas Chromatograph (LTM-GC). Performance characterization of the LTM-GC-equipped product using the diagnostic Grob mixture is presented. It has been designed to perform rapid separations without significantly compromising performance.

In order to assess the conditioning of the chromatographic system and separation capabilities over a wide range of temperature regimes, Grob mixture (Sigma Aldrich, St. Louis, MO) was used. This diagnostic mixture contains various classes of organic components including hydrocarbons, fatty acid methyl esters (FAMES), acids, bases and alcohols<sup>1,2</sup> (Table 1). The resulting elution order and chromatographic peak shapes are indicative of column type and age/condition. Periodic analysis of the Grob mixture allows for convenient assessment of the columns deterioration over the period of use.

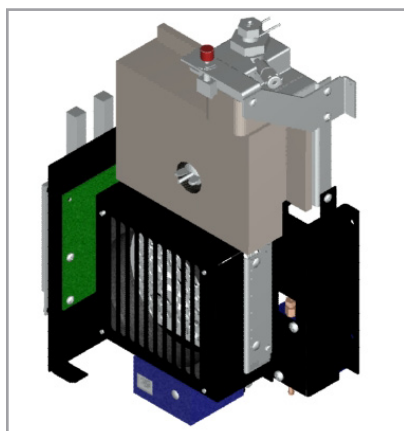


Figure 1: Low Thermal Mass Gas Chromatograph

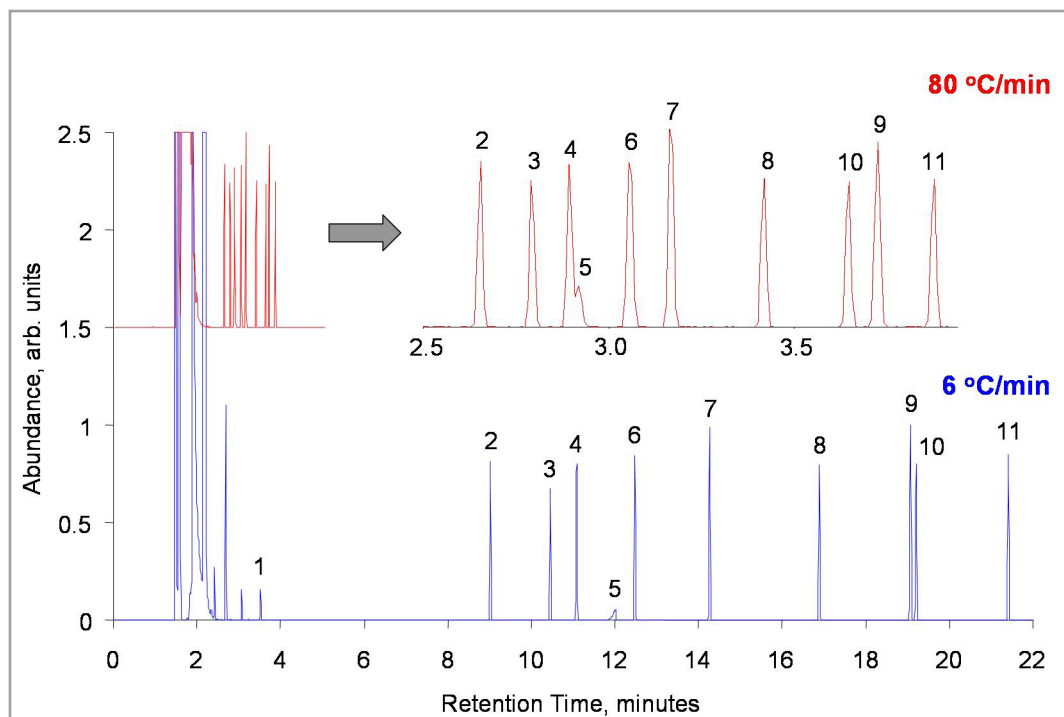
ANALYTE CLASS	APPRAISAL FUNCTION
Hydrocarbon (n-dodecane)	Proper column installation, system integrity
FAMES (methyl undecanoate)	Column separation efficiency, number of theoretical plates
Acid (2-ethylhexanoic acid), Bases (N-cyclohexanamine)	Active sites or underivatized surfaces
Alcohol (1-octanol)	Active sites in column or injector

Table 1: Grob mixture components and diagnostic function

The total ion current plot obtained for 1  $\mu$ L injection of Grob mix standard is shown in Figure 2. The blue total ion chromatogram (bottom) shows the results of a separation of the Grob mixture components using a conventional temperature program, i.e. with a column temperature ramp of 6°C/min. It is clear that the column is performing well in that all the components are present and the individual peaks are sharp and symmetrical. The only peak that is not sharply defined is that of 2-ethylhexanoic acid which is to be expected due to the non-polar nature of the Rtx-1MS column. The hydrocarbon and FAMES peaks show the column is properly installed and the peaks resulting from the alcohol components indicate that there are no significant active sites within the injector or column.

Also shown in Figure 2 is a Grob analysis performed with an accelerated temperature profile. The data is shown as the red total ion chromatogram on the same timescale as the previous, slower separation (top left). In addition, an expanded view of the Grob peaks is provided (top right). The low-thermal mass column allowed for a temperature ramp of 80°C/min which provided baseline separation for all but two of the components. Even with such a rapid separation, the peaks appear symmetrical indicating good separation efficiency. As in the conventional separation, the distorted shape of the 2-ethylhexanoic acid peak can be attributed to the use of a non-polar stationary phase (Rtx-1MS).

These data represent typical results.



**Figure 2:** Total ion current traces for injections of Grob test mixture on the ICx LTM-GC/MS with GC temperature ramps of 6°C/min (blue) and 80°C/min (red)

GC column: Rtx-1MS, 30 m  
0.25 mm i.d.  
0.25  $\mu$ m df

Carrier Gas: He, 1 mL/min  
15:1 split ratio

#### Grob Mixture Components:

- 1) 2,3-butanediol
- 2) n-decane
- 3) 1-octanol
- 4) 2,6-diphenylphenol
- 5) 2-ethylhexanoic acid
- 6) 2,6-dimethylbenzenamine
- 7) n-dodecane
- 8) methyl decanoate
- 9) N-cyclohexanamine
- 10) methyl undecanoate
- 11) methyl decanoate

#### References

1. Grob, Jr., K., G. Grob, and K. Grob, Comprehensive, Standardized Quality Test for Glass Capillary Columns, J. Chrom., 156, 1-20, 1978.
2. Grob, K., G. Grob, and K. Grob, Jr., Testing Capillary Gas Chromatographic Columns, J. Chrom. 219, 13-20, 1981.