

Petrochemical

2011 Applications

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Who are our Customers?

Petroleum



ExxonMobil

Syn crude

Schönbberger

ifp

GAZPROM

Chemicals



WACKER

EASTMAN

elf atochem
ATO

Innophos

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Products analyzed in Segment

Petroleum



Oil, Natural gas, gasoline, kerosene, diesel, Naphtha

Hydrocarbons, Sulfur,
Biomarkers, Aromatics, Alcohols

Bio-Fuels, Biodiesel, Bioethanol

glycerides, FAME, alcohols

Chemicals



Intermediates and final product

C2-C5 Alkenes, Aromatics, Amines,
Glycols, Acids, Solvents, Halogenated
compounds, Alcohols, Aldehydes

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Outline

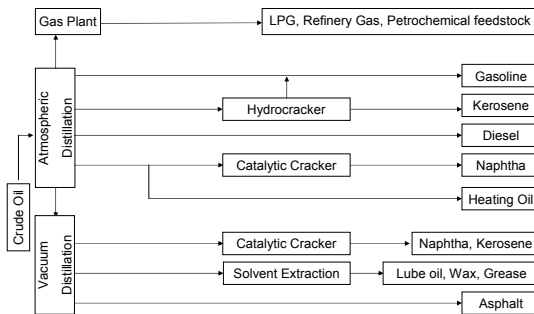
- MXT-1HT Sim Dist
- MXT-2887
- Rtx-DHA
- D3606 packed column set
- Rt-XL Sulfur packed column
 - And Siltek deactivation for sulfurs
- PLOT columns
- Appendix
 - Products for biodiesel and chemical analysis

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Refinery Process Map



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What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene /Toluene
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions
does Restek offer?

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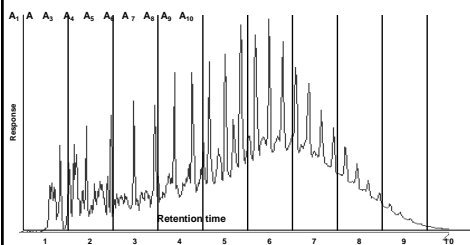
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HROM or Zymolene ANALYST 20M
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Simulated Distillation



- The chromatogram of the sample is divided into time slices
- The software determines the area count for each slice

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Challenges

- Columns Break (fused silica)
 - Need to recalibrate often (time)
 - Replace columns (price per analysis, time)
 - Reduced accuracy (reliability challenge)
- Columns loose stationary phase by bleed
 - Do not meet resolution requirements

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Needs for Accurate Simulated Distillation Analysis

- High temperature stable columns
- Low Bleed phases

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Sim Dist with fused silica: Big Problem area..

- FS Standard fused silica column polyimide coated, type Inferno..



Polyimide outside coating seriously damaged

This will happen with ALL fused silica columns

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Optimize strength/robustness of capillary Application of MXT Metal capillary tubing

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MXT Tubing is Extremely Strong



- Virtually unbreakable
 - Stainless steel with Siltek coated surface
- Can be bent / coiled to small radius
- Siltek surface stabilizes the stationary phase
- Sim Dist columns wound on standard 11-pin cage
- MXT columns can be cut with standard scoring wafer
 - Special tools also available

MXT columns provide reliability:
No column breakage possible!

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New, easy accessible column cage



MXT is nicely wound on the 11-pin cage:
Easy to install, handle and unwind..



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Simulated Distillation of Crude oil

Analysis: Simulated Distillation

C5-C110 D6352 High Temp

C5-C44 D2887

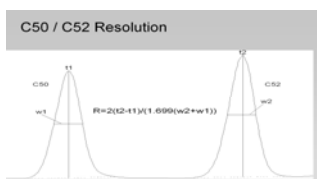
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ASTM Spec for High Temperature Sim Dist

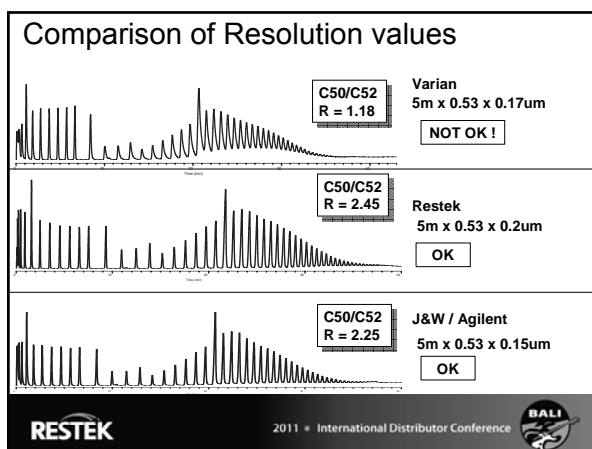
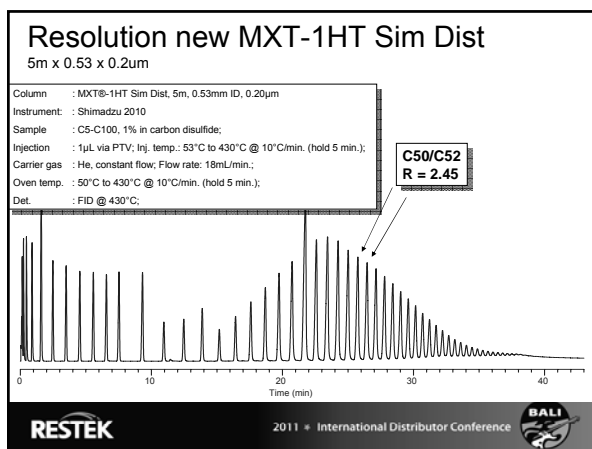
- In order to be approved for ASTM D86 and 6352, 7169 and 7500:
 - Resolution between C50 and C52 > 2.0, running under ASTM conditions



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Resolution values

The higher resolution value indicates the Restek MXT-1HT Sim Dist:

- Has a smoother coated surface of the PDMS phase over the length of the column
- The higher resolution value allows for a longer column lifetime

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Column Stability / Lifetime

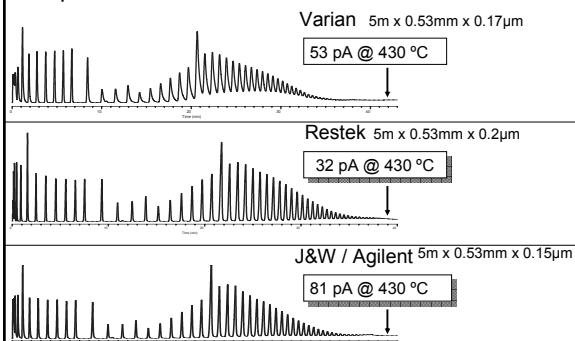
- Column lifetime in Sim Dist applications are also in direct proportional with the level of the column bleed
- The LOWER the bleed, The LONGER the life time

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Comparison of Bleed values



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Performance-ASTM D7169

- Customer feedback on Beta testing, done by Joaquin Lubkowitz, Separation systems.
- Authority in petro – analysis field and ASTM

S Separation Systems, Inc.
Experts In Gas Chromatography

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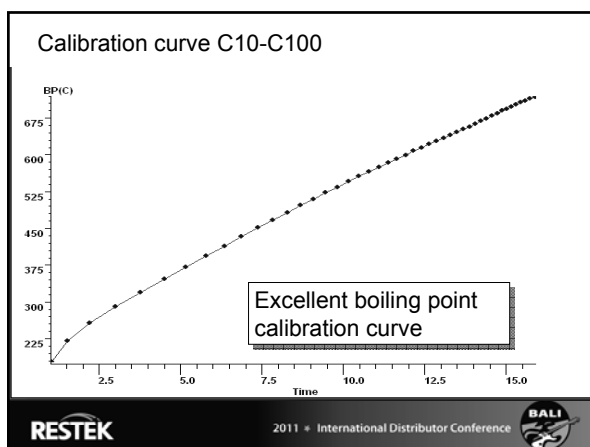
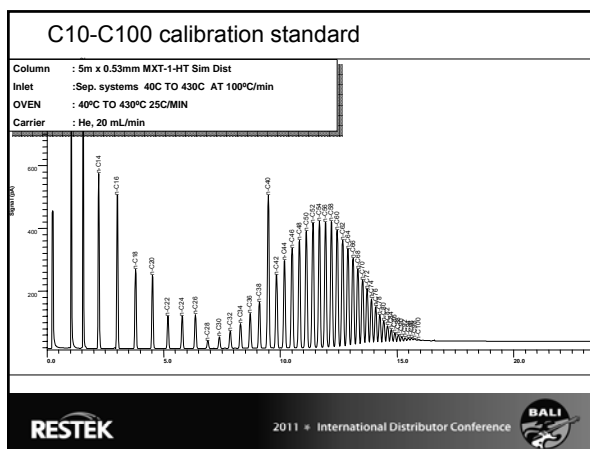
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Column Cutting

For installation in a PTV or on-column injector, the column inlet must be well-cut..

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MXT Column Cutting

- A special tube “scorer” has been developed..



Makes a perfect “cut” required for
“on-Column injection”..

For 0.25 - 0.53mm MXT

tubing scorer : 20523
replacement wheel: 20522



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Carrier gas considerations Avoid introduction of oxidizers

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Impact of Oxygen/Moisture

- Increase of bleed
- Retention times drop fast
- Peaks will broaden
- Peaks will tail

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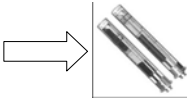
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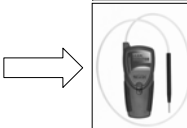
Carrier Gas purification

- Degradation reaction of stationary phase + water/oxygen develops exponentially with higher temperature..

Need: Pure gases



Need: Elimination of leaks



Also use graphite ferrules for temperatures above 360°C



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Simulated Distillation of Gasoline Range Hydrocarbons

Analysis: Simulated Distillation

C5-C110 D6352 High Temp

C5-C44 D2887

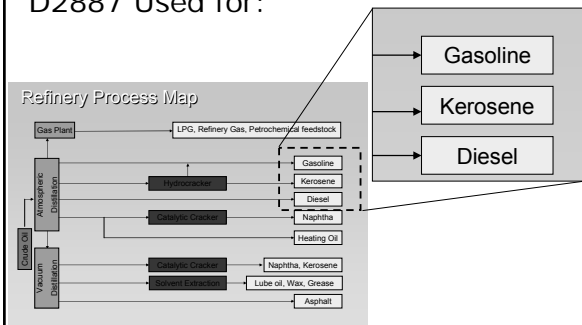
Also Called “gasoline-range” Sim Dist

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D2887 Used for:



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Gasoline Range Sim Dist

- C5-C44 D2887
- Column: 10m x 0.53mm x 2.65µm Rtx-2887 (Fused silica or MXT)

Uses a THICK film because the sample is injected on the column with no dilution;

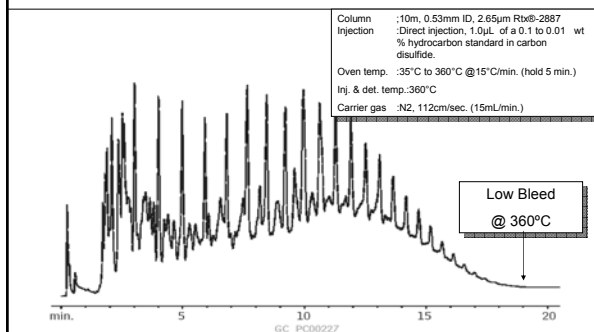
Need LOADABILITY !!

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D-2887 Sim Dist



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Restek New Generation MXT Sim Dist Columns

- Features: Low bleeding, Temperature Stable, Better Efficiency
- Benefit: Longer Column Lifetime, Less Replacement, less calibrations, Saving \$\$\$\$\$

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Product information for MXT Sim Dist

- Sim Dist sales companion + presentation
- Restek Refined brochure PCFL1195A: <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Simdist brochure PCFL1201A: <http://www.restek.com/restek/images/external/PCFL1201A.pdf>
- Applications see: <http://www.restek.com/restek/class/5004088.asp>; and http://www.restek.com/info_chromatograms.asp
- Technical Literature: http://www.restek.com/aol_lit_main.asp
- Article: Tighten Control of Distillation Processes with the New MXT®-1HT Sim Dist Column : http://www.restek.com/aol_petro_A022.asp



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- Benzene /Toluene
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions
does Restek offer?

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What products come from most refineries?

- Gasoline
- Diesel
- Jet Fuel
- Refinery Gas

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Analysis: Gasoline

Detailed Hydrocarbon Analysis (DHA) D6730

- Column: 100m x 0.25mm x 0.50um Rtx-DHA 100
- Tuning column: 5m x 0.25mm x 1.0um Rtx-5
- Tuning column is for adjusting selectivity for aromatics

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Biggest Challenges for Customers Running ASTM D6730

- Have good peak shape for alcohols
- Have sufficient theoretical plates to separate the complex samples
- Coupling of “tuning” capillary
- Analysis time too long, wish for faster methods
- Reproducibility of column-column quality parameters

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Why do the Refiners Run DHA Analysis?

- At the end a certain gasoline must have a certain octane number..
- It is very important for the refiner to monitor the blending process carefully to avoid too much yield.



Different octane numbers are available

- Accurate DHA analysis can save a lot of money ..



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Important Quality Parameter for DHA Analysis

- The column must not only elute hydrocarbons
- Also need to elute **alcohols**



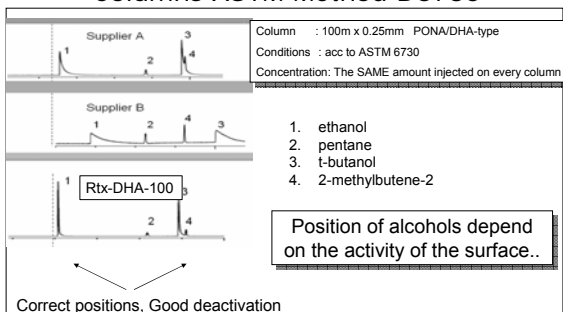
This is a big challenge and that's why
Rtx-DHA 100 columns are preferred

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Elution of Alcohols on the DHA-type Columns ASTM Method D6730



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Impact of a Tailing Alcohol Peak

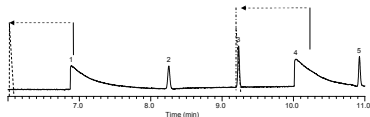
Supplier 1 type DHA column

Oven: 5 °C → 8.23 min., 22°C/min → 48 °C

24160U

1. ethanol
2. C5
3. 2 methyl butene 2
4. t butanol

Alcohol peaks are retained by column
activity / adsorption;
They elute at wrong position..



Risk: Misidentification and possible wrong data

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Specifications DHA column: for AstmD6730

Theoretical plates, n, pentane at 35°C;	40000 to 50000
Retention factor, k, pentane at 35°C	0.45 to 0.50
Resolution, R, t-butanol and 2-methylbutene-2 at 35°C	3.25 to 5.25
Peak symmetry, t-butanol at 35°C	>1.0 to <5.0

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Chromatogram showing peaks for ethanol, t-butanol, 2-methyl-butene-2, and pentane. The x-axis is time in minutes (0.00 to 16.00) and the y-axis is signal in pA (-10.00 to 130.00).

Compound	Approximate Retention Time (min)	Approximate Signal (pA)
ethanol	7.5	125
t-butanol	9.5	105
2-methyl-butene-2	9.8	70
pentane	8.8	40

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TEMP PROGRAM: 35°C Isothermal			
INJ TYPE: Split		INJ TEMP: 250°C	
DET TYPE: FID		DET TEMP: 300°C	
LINEAR VELOCITY: 27 cm/sec (Helium)		SPLIT FLOW: 230 mL/min	
Max Cal: 3500			
Retention Time	Name	Area	Height
7.79	ethanol	213288	127.793
9.16	C5	79783	41.430
9.51	1 butanol	226500	108.583
9.80	2 methylbutane 2	162390	77.061
			Width @ 50%
			0.024
			0.028
			0.030
			0.031
Max Cal: 3500			
Partition Ratio (K) C5: 0.48		Resolution (1 butanol / 2 methylbutane 2): 5.63	
Theoretical Plates (C5): 589,096		Asymmetry (1 butanol): 1.38	
Plates per meter (C5): 5,918			
		Column Bleed (pA): 9.9	

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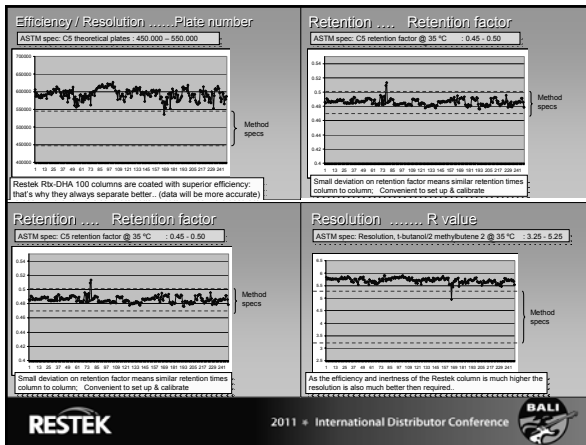
Reproducibility of Rtx-DHA-100 Columns

Following graphs show the typical performance of the chronological production of 250 Rtx-DHA 100 columns

- Efficiency /Resolution
 - Retention
 - Inertness
- } Set by ASTM
- Bleed

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ASTM D 6730 Requires a "tuned" Column...

ASTM wants general purpose methods

Columns from different suppliers will show small differences in selectivity..

By coupling a short piece of Rtx-5, every column can be "tuned" to fulfill ASTM specs on selectivity..

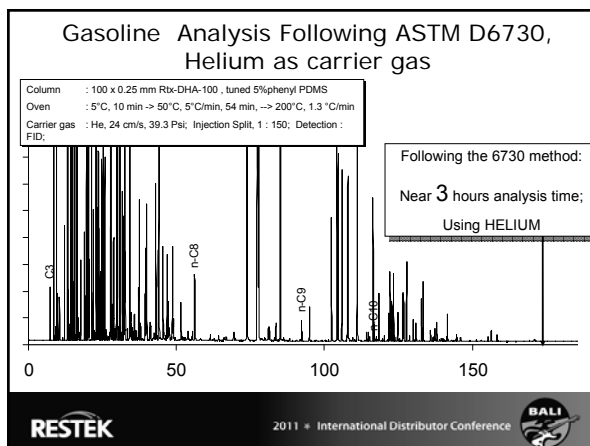
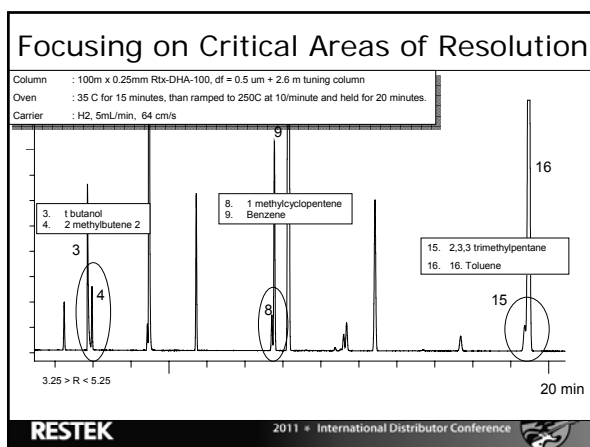
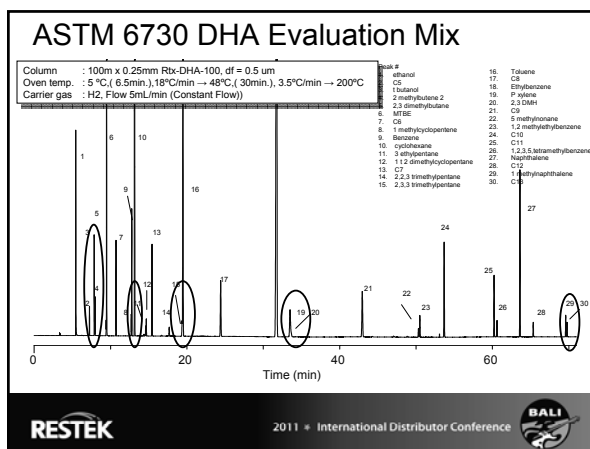
Tuning pre-column: Rtx-5, 2-5 meters
 Length to be determined empirically

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There will be always a wish from customers to SPEED UP the analysis

Using Helium and different temperature program

Using Hydrogen

Using shorter, smaller ID columns



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Using Helium and a different program

Using the same column, same GC

Column : 100m x 0.25mm Rtx-DHA-100, df = 0.5 μ m coupled with
2.6m x 0.25mm Rtx-5DHA tuning column, df = 1.0 μ m

- Carrier gas : He, Flow 2.3 mL/min (Constant Flow), 28 cm/s

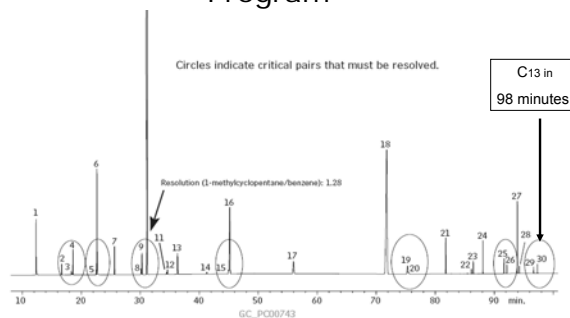
- Oven temp. : 5 $^{\circ}$ C \rightarrow 15min., 5 $^{\circ}$ C/min \rightarrow 50 $^{\circ}$ C, 50min.
8 $^{\circ}$ C/min \rightarrow 200 $^{\circ}$ C, 10 min



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Using Helium and a Different Program



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Speeding up the Application using HYDROGEN Carrier Gas

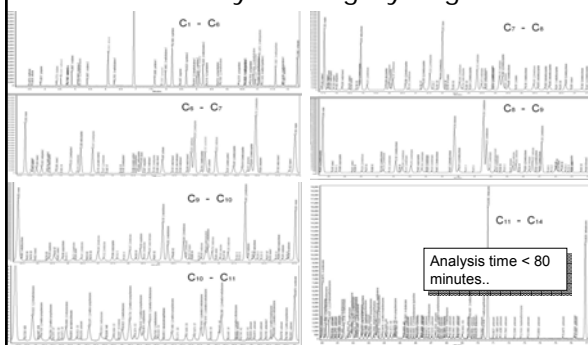
- Using hydrogen we can operate at near 2 times the linear velocity as used for helium
 - This results in significant shorter run time
- To get the same chromatogram/separation, we need to adjust the oven temperature program

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Fast DHA Analysis using Hydrogen...



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Fast DHA using Hydrogen

- Same GC
- Same method
- Same Injection/detection techniques
- Same column
- No issues with overloading, peak shifts, tailing, high inlet pressures, discrimination and reduced life time..



Neil Johansen

All retention data and identification is generated by Neil Johansen, developer of the D6730 DHA method.

Available on the Restek website
: http://www.restek.com/promo_gc_pona.asp#004

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Hydrogen : Safety Issues

Need to make sure there is no accumulation of H₂ possible in the oven..

Hydrogen is combustible over a concentration range of 4% to 74% by volume;
Diffusion VERY fast;

- Use of Hydrogen generators: limited amount of hydrogen
- Hydrogen Flow restriction to set with GC inlet (electronic flow setting)
- Hydrogen detection systems (sniffer)

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Rtx-DHA Columns for Other ASTM Methods

For ASTM D6733 : Rtx-DHA-50 50m x 0.25mm, df = 0.5 μ m

For ASTM D6729 : Rtx-DHA-100 100m x 0.25mm, df = 0.5 μ m

For ASTM D5501 : Rtx-DHA-150 150m x 0.25mm, df = 1.0 μ m

NOTE:

This column was called earlier Rtx-1 PONA, which is a WRONG name as the application is DHA, not PONA.

Competing products are still called: HP-PONA, DB-PONA, CP-Sil PONA CB, BP-1PONA and Petrocol

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Have good peak shape for alcohols.....



Have sufficient theoretical plates



Coupling of "tuning" capillary



Analysis time too long, wish for faster methods ..



Reproducibility of column quality parameters



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Customer referral

I just wanted to relay to you how pleased I am with the performance of the Restek DHA PONA bonded to the 5 meter RTX-5 pre-column. We've found that the optimal tuning length is 2.05 meters \pm 0.5 meters, which means less time finding the optimal length as compared to the prior Supelco columns.

The peaks look great – far superior to the prior supplier. I am especially pleased with the critical separation of t-butanol and 2-methylbutene-2. The t-butanol peak only tails minimally with the temperature programming, and it keeps us from having to scrap failed columns. This improvement process of switching columns has improved our production rate by nearly half and cut our waste costs as well.

Terry Osenbach
PerkinElmer Health Science
GC Application and Engineered Solutions
Chemist
710 Bridgeport Ave, Shelton, CT 06614

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Product information Rtx DHA columns

- Rtx-DHA sales companion + presentation
- Restek Refined brochure PCFL1195A: <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
Brochure Rtx-DHA 100 PCFL1007A: <http://www.restek.com/restek/images/external/PCFL1007A.pdf>
- Recorded WEBINAR
- Applications see: <http://www.restek.com/restek/class/5004088.asp>; and http://www.restek.com/info_chromatograms.asp
- Technical Literature: http://www.restek.com/aci_lit_main.asp
- Article: **Faster DHA Analyses Using Helium or Hydrogen:** http://www.restek.com/aci_petro_A025.asp
- Article: **How Good is Your PONA Column?:** http://www.restek.com/aci_petro_A006.asp



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What does every refinery do?

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- Detailed Hydrocarbon Analysis
- Benzene / Toluene/ ethanol in gasoline
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions
does Restek offer?

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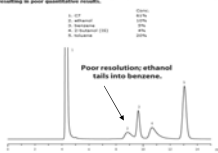
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Benzene / Toluene/ Ethanol in Gasoline

ASTM Method D3606

Figure 1. HPLC columns often fail to adequately resolve benzene from ethanol, resulting in poor identification results.



Present solution is problematic:

Big issues with separations and peak shapes

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Modified ASTM D3606 Method using the D3606 Column Set

Uses a SET of packed columns:

Material	Siltek deactivated SS
ID / OD	2mm / 1/8"
Lengths	6' and 16' long
Phases	Rtx-1 and proprietary

- First part of eluting peaks is sent to the second, highly selective column, where the alcohols and aromatics are separated
- Higher boiling compounds are back-flushed
- Unique column set, only from Restek

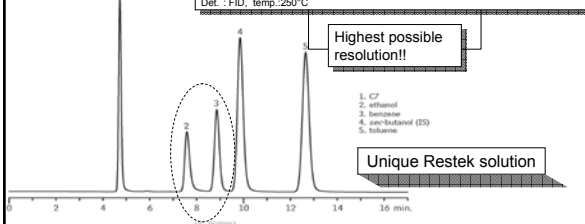
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New D3606 Method

Columns : Rt™-D3606 Column Set (cat.# 83606)
 column 1 : Rt™-D3606-1: 6'x 1/8"OD (1.8m x 2mm ID), nonpolar RtxB-1
 column 2 : Rt™-D3606-2: 16'x 1/8"OD (4.9m x 2mm ID), proprietary packing
 Injection : 0.05µL, direct injection; Inj. temp. :200°C
 Car. Gas : He, constant flow; Flow rate:20mL/min.
 Oven : 135°C
 Det. : FID, temp: 250°C



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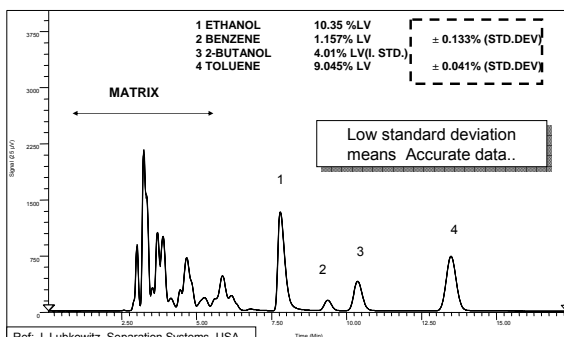


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Repeatability of 4 Injections of Walmart Gasoline



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Product Information for D3606

- Restek Refined brochure: [PCFL1195A: http://www.restek.com/restek/images/external/PCFL1195A.pdf](http://www.restek.com/restek/images/external/PCFL1195A.pdf)
- Applications see : <http://www.restek.com/restek/class/5004088.asp>
- Technical Literature: http://www.restek.com/aoi_lit_main.asp
- Article: **New D3606 Column Set Outperforms TCEP Columns for Benzene Analysis:**
http://www.restek.com/aoi_petro_A020.asp



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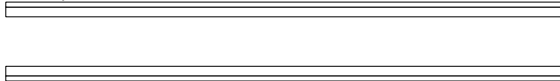
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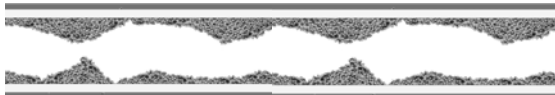
PLOT Layer is often deposited as non homogeneous film

FLOW Restriction

Ideal "open Path: Flow-restriction factor = 1.0



PLOT column with Flow-Restrictions: Flow restriction factor "F" = 0.2-0.8



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This was one of the reasons why we could replace Varian and Agilent PLOT columns for:

- Siemens
- Agilent micro GC
- C2V (Thermo)
- Schlumberger
- Many companies that supply Analyzer solutions (PAC, Interscience,...)

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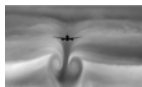
Transportation



Transportation of PLOT columns

During transport columns are subjected to uncontrolled:

- Vibrations
- Pressure changes
- Temperature changes
- Shocks



Which all can destabilize the adsorption layer..

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HROM or Zymol Flow 40/60 100/20 150/30
ECH Flow 40/60 100/20 150/30
Website: www.restek.com.au Email: info@restek.com.au Tel: 61 6762 2016 ... @ AUSTRALIA

Australian Distributors
Suppliers of Gas Chromatography
www.restek.com.au

12/13

Dow Chemical, J . Luong

One of the concerns involves the use of PLOT column technology is adsorbent particle shedding. The shedding of adsorbent particles has the potential of plugging the flow channel of the capillary flow Deans switch device. This in turn can cause retention time shift.

Prior to connecting the columns to the capillary flow Deans switch device, we highly recommend the columns be purged with carrier gas at a flow of 50 mL/min for 15 min to remove any particles that might have been dislodged during shipment of the columns from the manufacturers.



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Refinery Gas Analysis

- C1-C5
 - Rt AluminaBOND KCl or Na2SO4
- Permanent gases, C1-C2 and CO2:
 - Rt Msieve 5A / Rt-Q BOND
 - ShinCarbon



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C1-C5 Hydrocarbons Rt-Alumina BOND

KCl : Non polar Al2O3 surface

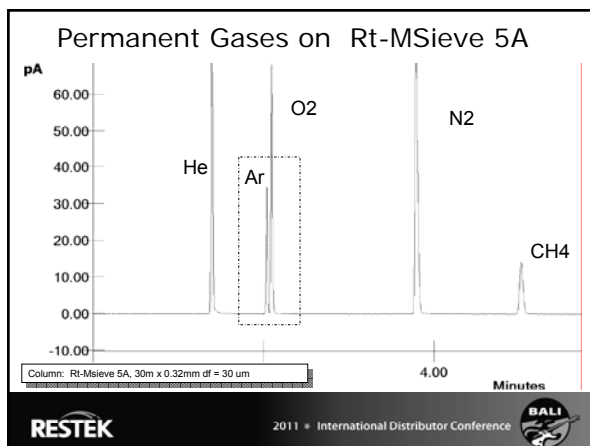
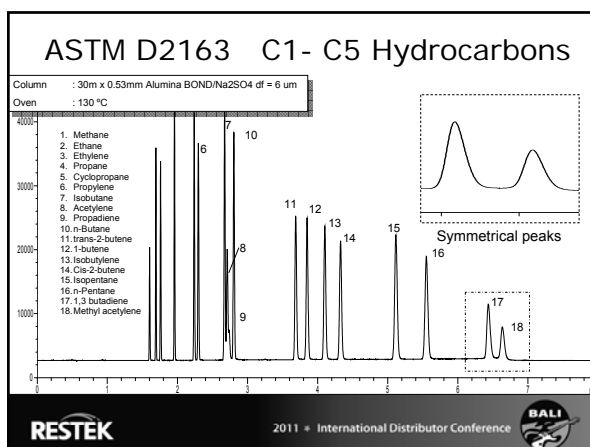
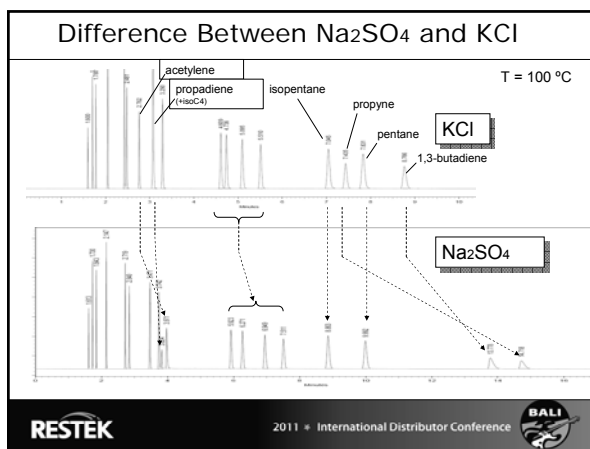
Na2SO4 : Polar Al2O3 surface

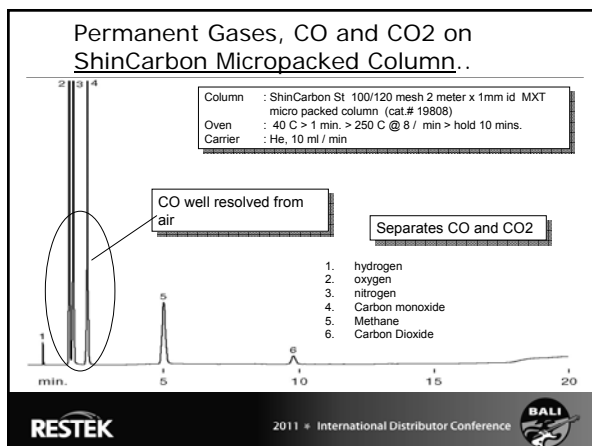
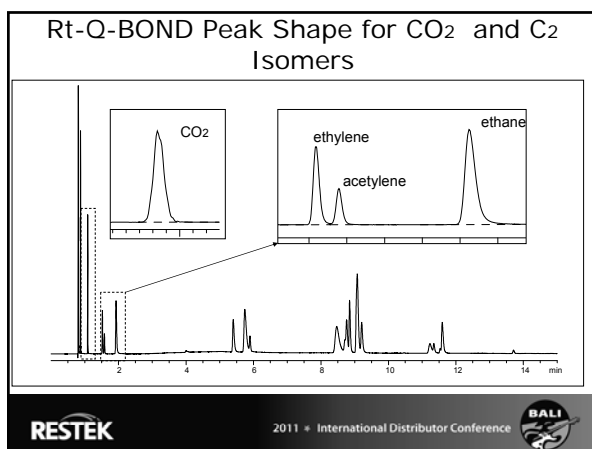
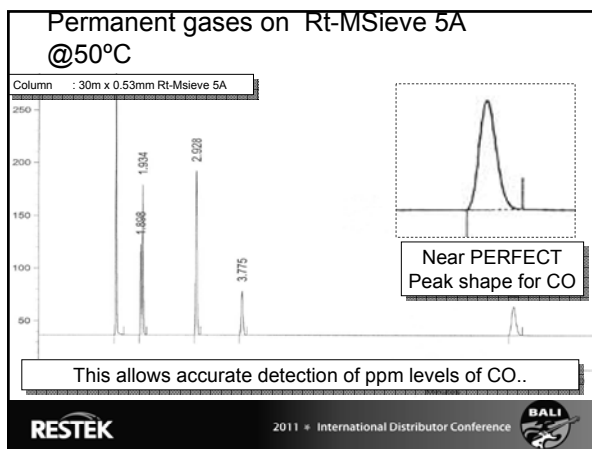
Deactivation is necessary to reduce the activity of the alumina..



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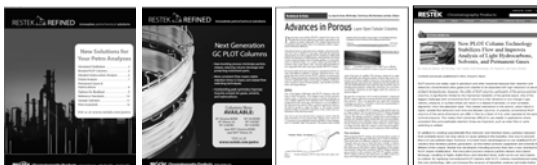






Product Information for PLOT

- Restek Refined brochure PCFL1195A: <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Next generation PLOT columns PCFL1163A: <http://www.restek.com/restek/images/external/PCFL1163A.pdf>
- Applications see: <http://www.restek.com/restek/class/5004088.asp>
- Technical Literature: http://www.restek.com/aol_lit_main.asp
- Article: New PLOT Column Technology Stabilizes Flow and Improves Analysis of Light Hydrocarbons, Solvents, and Permanent Gases: http://www.restek.com/aol_petro_A023.asp
- Article: Advances in Porous Layer Open Tubular Columns: http://www.icpubs.com/Media/PublishingTitles/dec/eeuw%20layout_0.pdf



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Additional UNIQUE Restek Development

Making PLOT and LIQUID PHASE columns available in MXT..

In PROCESS type applications, customers look for:

RELIABILITY..

MXT columns will offer extra security and value..

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MXT-MSieve 5A

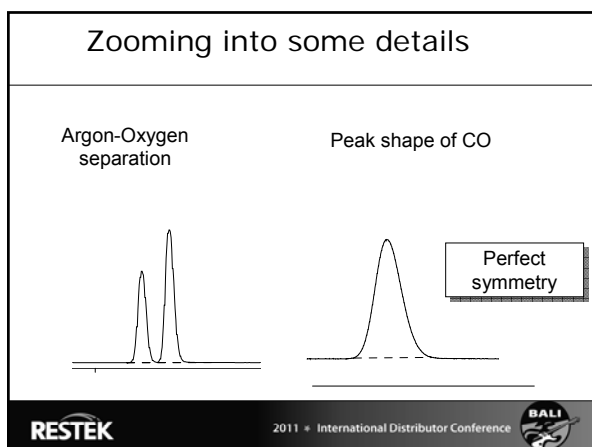
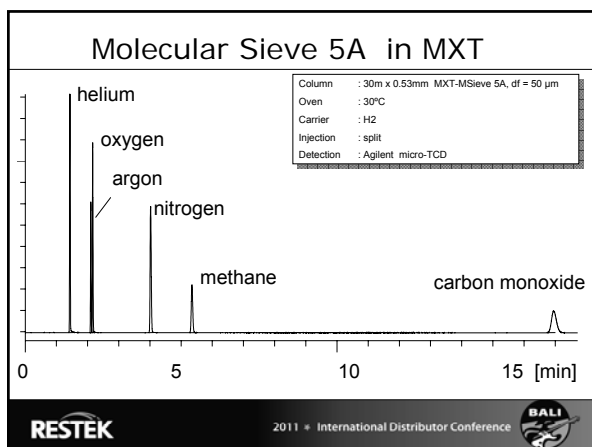
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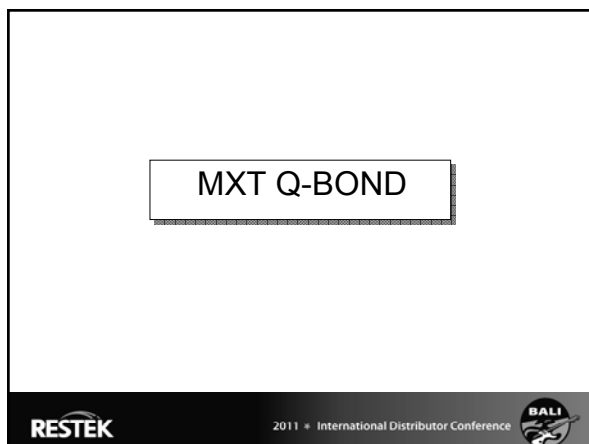
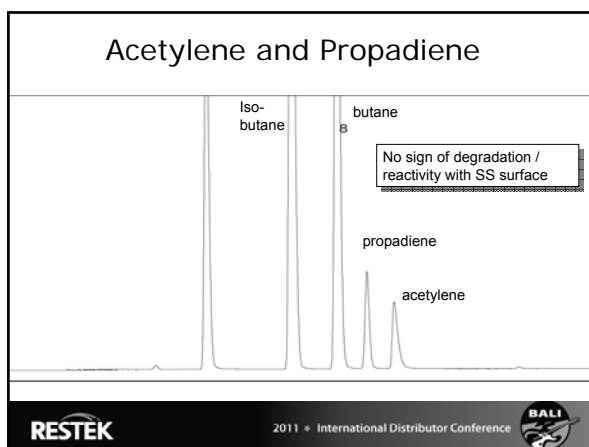
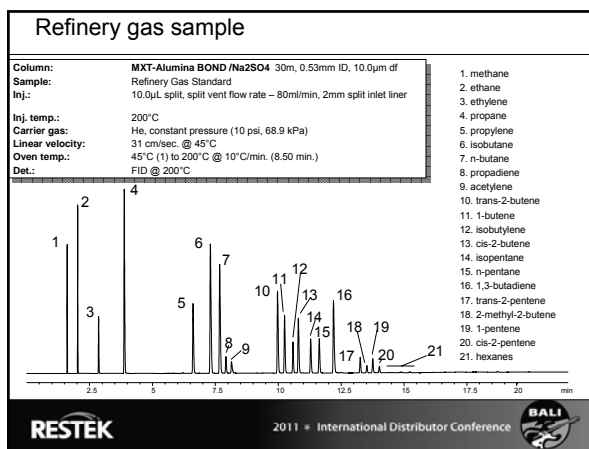
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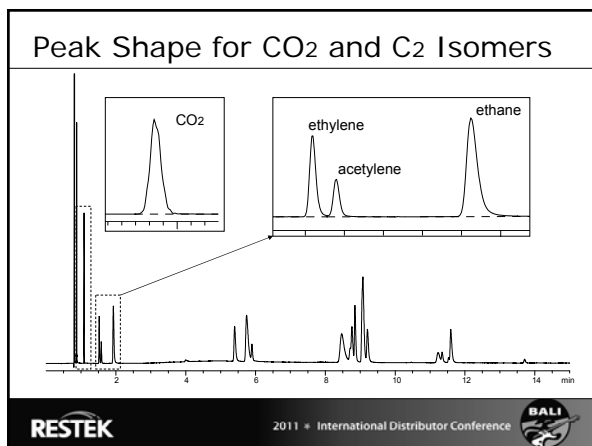
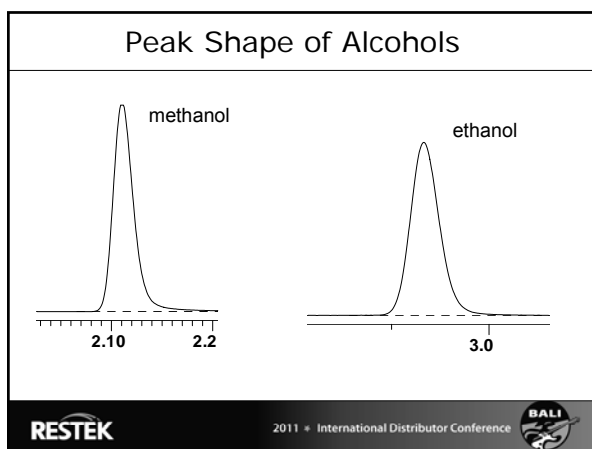
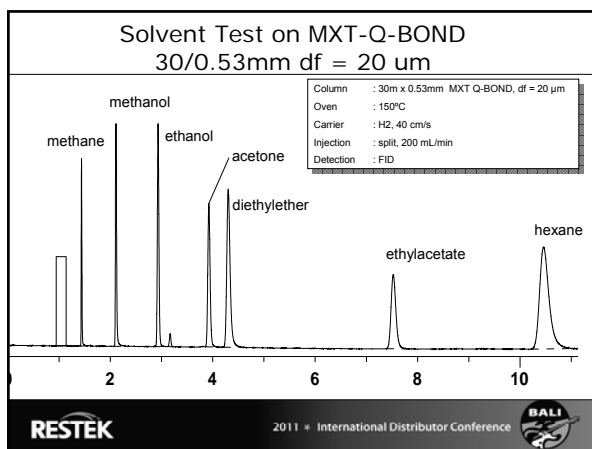
HROM or Zymolam 40/60 50/60 50/80 50/100 50/120 50/150 50/200 50/250 50/300 50/350 50/400 50/450 50/500 50/550 50/600 50/650 50/700 50/750 50/800 50/850 50/900 50/950 50/1000 50/1100 50/1200 50/1300 50/1400 50/1500 50/1600 50/1700 50/1800 50/1900 50/2000 50/2100 50/2200 50/2300 50/2400 50/2500 50/2600 50/2700 50/2800 50/2900 50/3000 50/3100 50/3200 50/3300 50/3400 50/3500 50/3600 50/3700 50/3800 50/3900 50/4000 50/4100 50/4200 50/4300 50/4400 50/4500 50/4600 50/4700 50/4800 50/4900 50/5000 50/5100 50/5200 50/5300 50/5400 50/5500 50/5600 50/5700 50/5800 50/5900 50/6000 50/6100 50/6200 50/6300 50/6400 50/6500 50/6600 50/6700 50/6800 50/6900 50/7000 50/7100 50/7200 50/7300 50/7400 50/7500 50/7600 50/7700 50/7800 50/7900 50/8000 50/8100 50/8200 50/8300 50/8400 50/8500 50/8600 50/8700 50/8800 50/8900 50/9000 50/9100 50/9200 50/9300 50/9400 50/9500 50/9600 50/9700 50/9800 50/9900 50/10000 50/10100 50/10200 50/10300 50/10400 50/10500 50/10600 50/10700 50/10800 50/10900 50/11000 50/11100 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MXT-Alumina BOND (Na₂SO₄ deactivation)

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Product information MXT PLOT

- Brochure Restek Refined PCFL1195A: <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Brochure Next generation PLOT columns PCFL1163A: <http://www.restek.com/restek/images/external/PCFL1163A.pdf>
- Applications see: <http://www.restek.com/restek/class/5004088.asp> and http://www.restek.com/info_chromatograms.asp
- Technical Literature: http://www.restek.com/aci_lit_main.asp



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What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene / Toluene/ ethanol in gasoline
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions
does Restek offer?

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Reference

Restek is internationally known for the Siltek / and SilcoSteel deactivations;

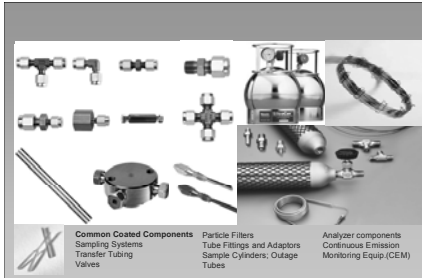
Siltek treated parts are widely used and acknowledged in the petrochemical Industry for sulfur compound stability...

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All parts in the GC system that come in contact with sulfur compounds can be deactivated by the Siltek technology...

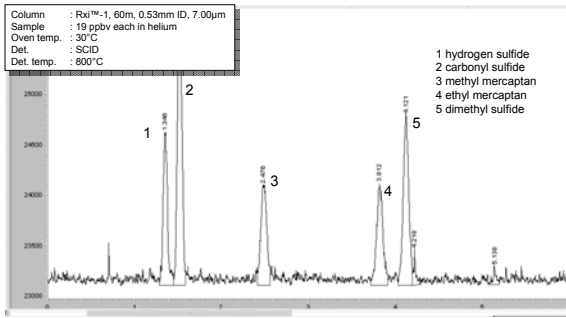


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Sulfur Gases at 19 ppb



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Most Important Issue for Trace Sulfur Analysis

System Inertness

Use a complete Inert system

- Deactivated transfer tubing, Inert Valves / coupling devices, Inert columns, Inert detection liners, Pressure regulators

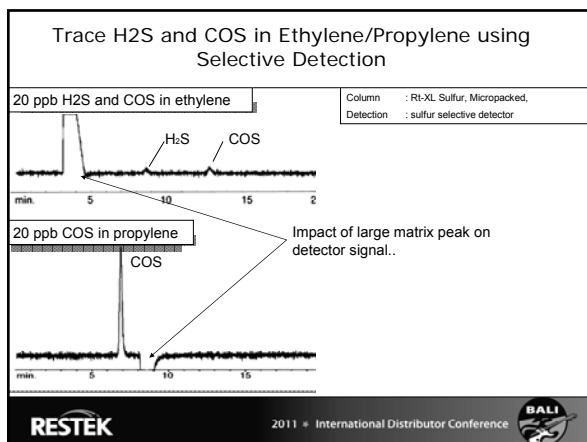
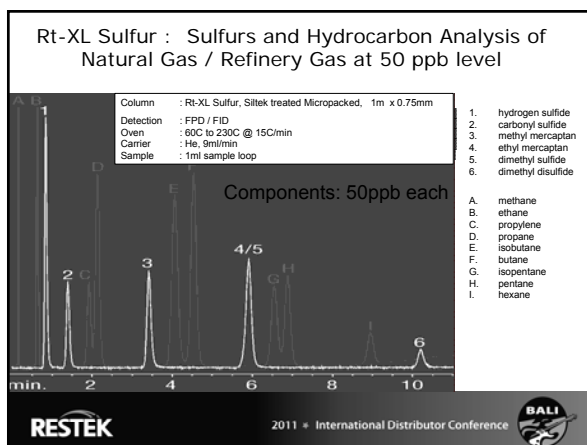
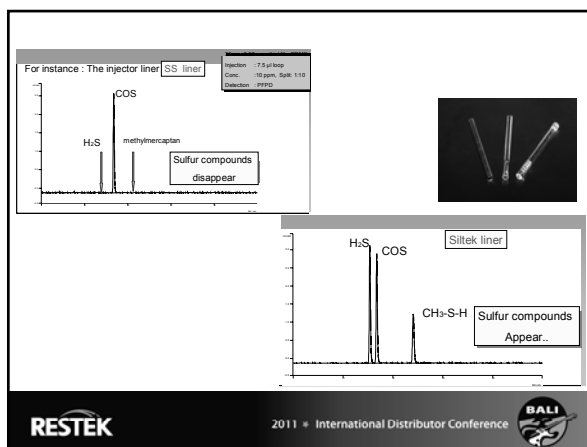
Sulfur compounds will disappear especially at low levels..

Restek Developed Siltek deactivated tubing..

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Product information Sulfur

- Restek Refined brochure : PCFL1195A; <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Applications : [gc_pc00202.pdf](#); [gc_pc00202.pdf](#); [gc_pc00202.pdf](#); [gc_pc00199.pdf](#); [gc_pc00199.pdf](#)
- Powerpoint handout : http://www.restek.com/restek/images/external/PASS6_SF_TraceLevelSulfurs_Restek.pdf
- Applications see : <http://www.restek.com/restek/class/504088.asp>
- Technical Literature : http://www.restek.com/aol_111_main.asp
- Article: Stable Sulfur & Mercury Sampling in Refineries Using Siltek® and Sulfinet® Surface Treated Components: http://www.restek.com/aol_petro_A016.asp



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What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene / Toluene/ ethanol in gasoline
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions
does Restek offer?

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Oxygenates in Gasoline Analyses: ASTM D4815

- Oxygenates in Finished Gasoline (Octane boosters) D4815
- Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C1 to C4 Alcohols in Gasoline by Gas Chromatography
- TCEP column plus Rtx-1 column or Rtx-DHA column

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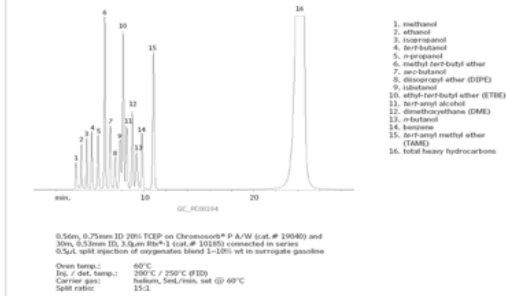
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Alcohols in Gasoline by ASTM D4815



*Silcosteel® treatment is a proprietary surface treatment for passivating steel and stainless steel, U.S. Patent 6,513,760.

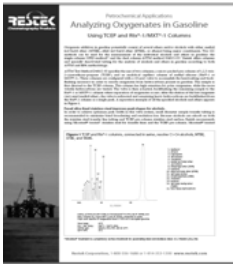
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Product Information for Oxygenates

- Applications : gc_pc00194.pdf
- Application article : Oxygenates in gasoline: 59587A



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