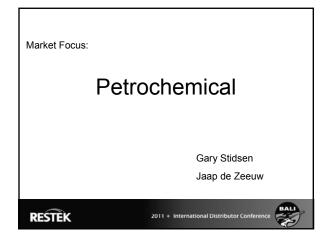
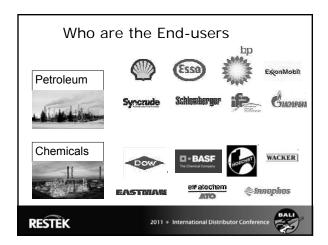
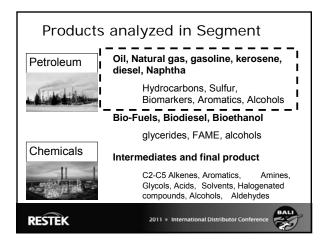
Petrochemical APPLICATIONS

- 2011 Restek Overview









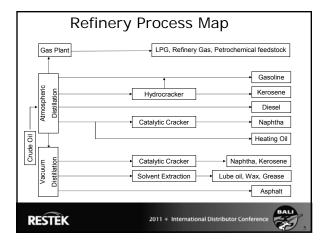


- · MXT-1HT Sim Dist
- MXT-2887
- Rtx-DHA
- · D3606 packed column set
- · Rt-XL Sulfur packed column
- And Siltek deactivation for sulfurs
- · PLOT columns
- Appendix
 - Products for biodiesel and chemical analysis



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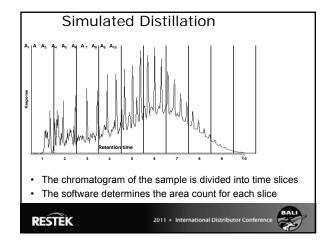


What does every refinery do?					
Simulated Distillation	 				
Detailed Hydrocarbon Analysis					
Benzene /Toluene					
Refinery gases					
Sulfur analysis	What Solutions				
Oxygenates	does Restek offer?				

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Challenges

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

POLY MINISTRACTOR CONFERENCE 2011 * International Distributor Conference

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!











Simulated Distillation of Crude oil

Analysis: Simulated Distillation

C5-C110 D6352 High Temp

C5-C44 D2887

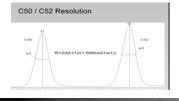
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ASTM Spec for High Temperature Sim Dist

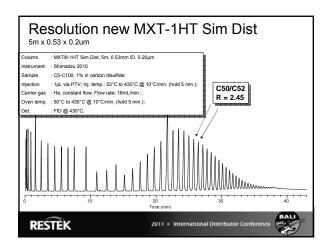
- In order to be approved for ASTM D86 and 6352, 7169 and 7500:
 - Resolution between C50 and C52 > 2.0, running under ASTM conditions

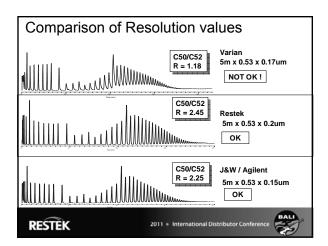


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Resolution values

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









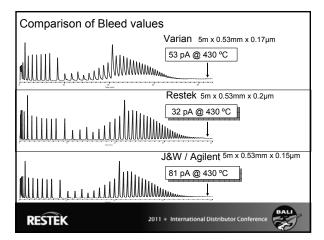
Column Stability / Lifetime

- 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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Performance-ASTM D7169

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

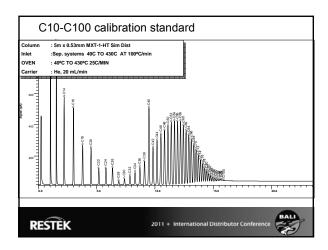


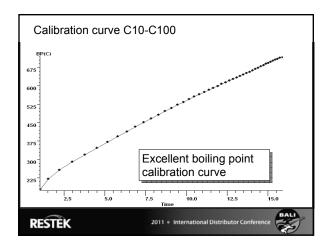
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Column Cutting For installation in a PTV or on-column injector, the column inlet must be well-cut..







Carrier gas considerations

Avoid introduction of oxidizers

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Impact of Oxygen/Moisture

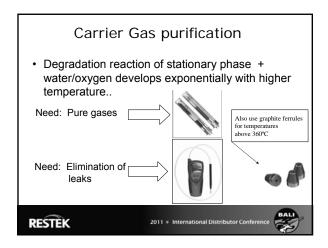
- · Increase of bleed
- · Retention times drop fast
- · Peaks will broaden
- · Peaks will tail

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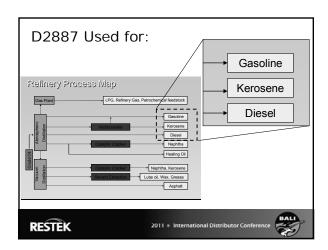








Simulated Distillation of Gasoline Range Hydrocarbons Analysis: Simulated Distillation C5-C110 D6352 High Temp C5-C44 D2887 Also Called "gasoline-range" Sim Dist







Gasoline Range Sim Dist

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

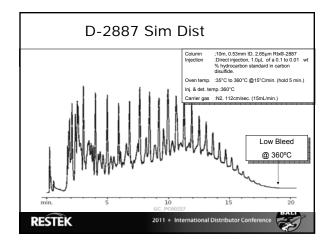
Uses a THICK film because the sample is injected on the column with no dilution;

Need LOADABILITY!!

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Restek New Generation MXT Sim Dist Columns

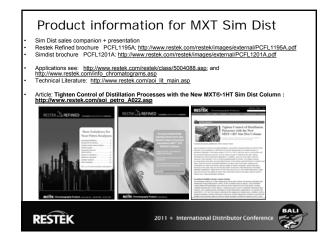
- Features: Low bleeding, Temperature Stable, Better Efficiency
- Benefit: Longer Column Lifetime, Less Replacement, less calibrations, Saving \$\$\$\$\$

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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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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

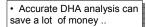
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Why do the Refiners Run DHA Analysis?

- 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











Important Quality Parameter for DHA Analysis

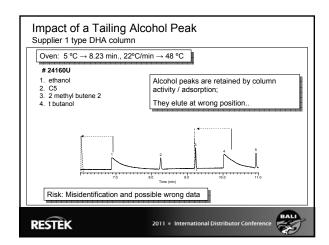
- · The column must not only elute hydrocarbons
- Also need to elute alcohols

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

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ASTM D 6730 Specifications

Specifications DHA column: for AstmD6730

fusedsilica Material 100m Length Internaldiameter 0.25mm methylsilicone Liquidphase Filmthickness 0.50 µm

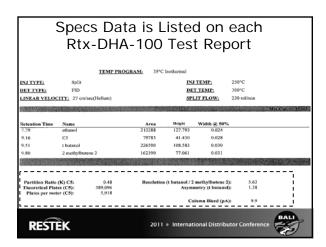
Theoretical plates, n, pentane at $35^{\circ}C$; 400000 to 500000 0.45 to 0.50 Retention factor, k, pentane at 35°C Resolution, R,t-butanoland2-methylbutene-2at 35°C3.25 to 5.25

Peak symmetry, t-butanolat 35°C >1.0 to <5.0

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Standard ASTM-Specs Test for all Restek Rtx-DHA 100 Columns ethanol t-butanol 2-methyl-butene-2 pentane RESTEK 2011 * International Distributor Co







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

Set by ASTM

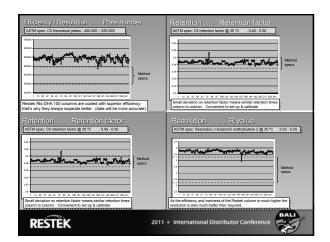
· Inertness

• 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..

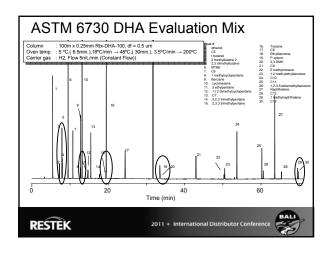
Tuning pre-column: Rtx-5, 2-5 meters Length to be determined empirically

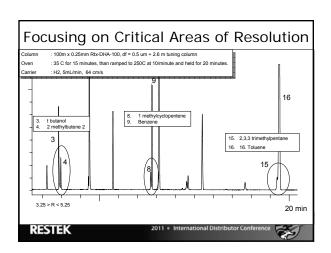
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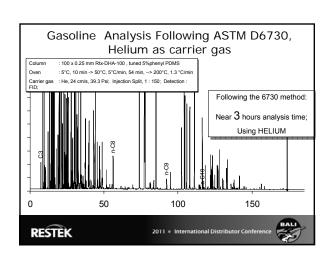
















There will be always a wish from customers to SPEED UP the analysis

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

_

 $\begin{array}{ll} \hbox{Column} & : 100 \hbox{m x 0.25mm Rtx-DHA-100, df = 0.5 } \mu \hbox{m coupled with} \\ 2.6 \hbox{m x 0.25mm Rtx-5DHA tuning column, df = 1.0 } \mu \hbox{m} \\ \end{array}$

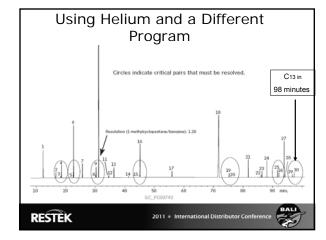
• Carrier gas : He, Flow 2.3 mL/min (Constant Flow), 28 cm/s

• Oven temp. : $5 \, ^{\circ}\text{C} \rightarrow 15 \text{min.}, \ 5 \, ^{\circ}\text{C/min} \rightarrow 50 \, ^{\circ}\text{C}, \ 50 \text{min.}$

 $8^{\circ}\text{C/min} \rightarrow 200^{\circ}\text{C}, 10 \text{ min}$

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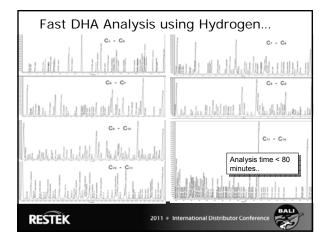
Speeding up the Application using HYDROGEN Carrier Gas

- 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 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..

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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Neil Johansen





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 μm

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

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

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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Biggest Challenges for customers running ASTM D6730

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









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 funing length is 2.05 meters ± 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. 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

- Restek Refined brochure Brochure Rtx-DHA 100

- - 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/aoi_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

What Solutions does Restek offer?

· Oxygenates

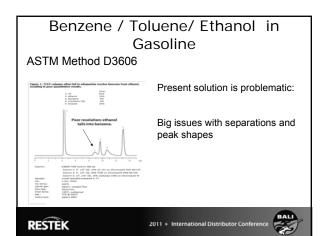
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Modified ASTM D3606 Method using the D3606 Column Set

Uses a SET of packed columns:

Material Siltek deactivated SS

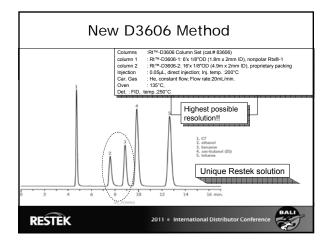
ID / OD 2mm / 1/8"

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

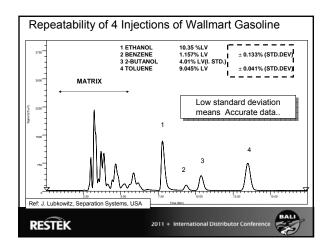
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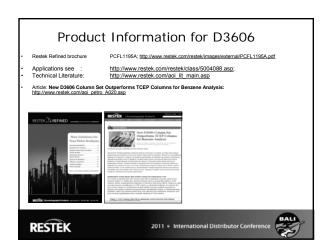












What does every refinery do? Simulated Distillation Detailed Hydrocarbon Analysis Benzene / Toluene/ ethanol in gasoline Refinery gases Sulfur analysis Oxygenates What Solutions does Restek offer?





Refinery Gases

- · Refinery Gas, what is it?
- · Gas produced in petroleum refineries by cracking, reforming, and other processes;
- · Mainly Light hydrocarbons and permanent gases

Separation done with PLOT columns

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Restek PLOT Columns Versus Competitor Columns

The PLOT columns supplied by competition have issues with:

- Particle stabilization
- Reproducibility of retention
- · Reproducibility of Flow
- All Restek PLOT columns have unique bonding chemistry to eliminate particle generation and are ALL tested for Flow-Restriction.
- The Flow Restriction Factor (F) is printed on each test report

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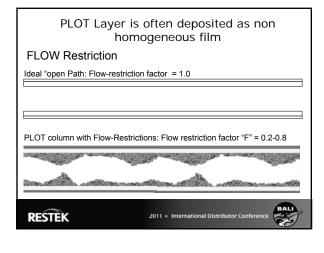
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RESTEK Quality Assurance Report								
110 Senner Circle + Detertoria, PA 10823 + 614-303-1300 + 800-395-1088 + fox 614-303-1309 + www.restek.com								
CATALOG No. 86172 SEQ SOVE He v S. Hom v tom MESSAA, No. 971944								
	150°C Inothermal Salik				1900			
	FID			EMP:	250°C			
LINEAR VELOCITE:				LELOW:	55 militain			
- HOCHE			M.L.			-		
					THE R			
Retartion(min) Nan		Arms	Thight	Wide	SRY'S MARKET	Aquenty		
2.67 Mod	Baserii Baserii	11360	10.794	6.13	8,4127	1.066		
		28863 82983	40.760	0.14	5,6179	1.115		
1.36 Eth		83993 161132	29.170	0.14	8.6031	1.339		
1.88 Aug 2.66 Dist	Red Eiler	119073	51.353	0.14	8.6031	1.139		
	Hyl Eiber	19975	51.360	0.78	0.0032	1.334		
	of Acceptance	112738	26.620	0.26	0.0041	1.235		
	I Aconomic	31509	36.862	0.51	0.1189	1.853		
SECTION SECTION	100000000000000000000000000000000000000		entropy of the	0.000	COLUMN TO			
Purities Ratio (Fiftal Australia 6.05 Releasing Index (Edy) Australia 30.5 8								
Plates Meter (Ethyl Au	otate(s	1877	Returnis	on Buden(Are	tom/s	451.9		
Flow-restriction Sector	(F)c	0.00						
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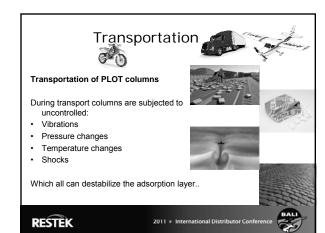


This was one of the reasons why we could replace Varian and Agilent PLOT columns for:

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

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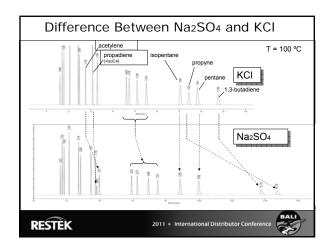


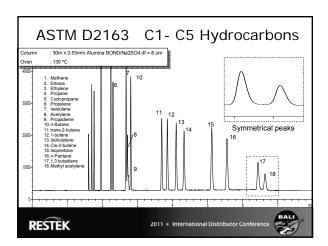


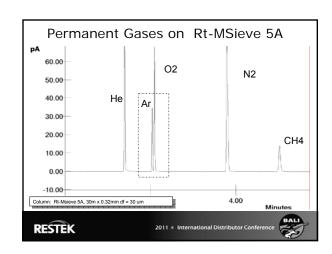
Dow Chemical, J. Luong	
One of the concerns involves the use of PLOT column technology is adsorbent particles hedding. The shedding of adsorbent particles has the potential of plugging the flow channel of the capillary flow Deans switch device. This in turn can cause retention time shift.	
Prior to connecting 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 	
PALL	
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C1-C5 Hydrocarbons Rt-Alumina BOND	
KCI : Non polar Al2O3 surface	
Na2SO4 : Polar Al2O3 surface	
Deactivation is necessary to reduce the activity of	
the alumina	





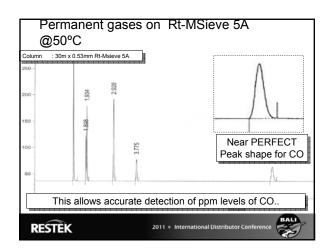


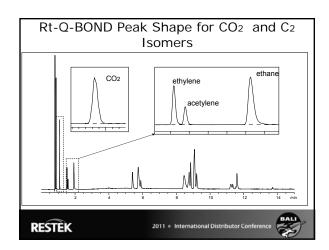


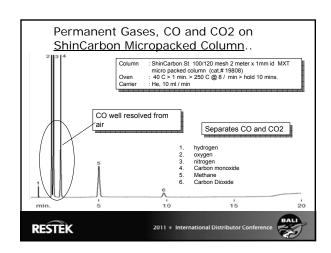






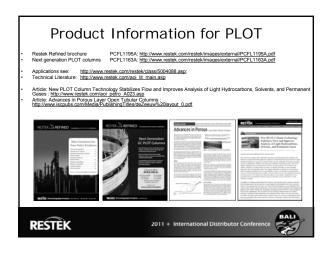












Additional UNIQUE Restek Development

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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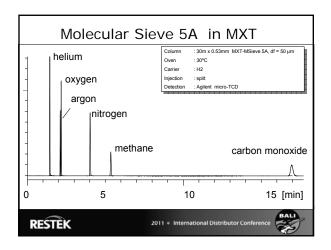
MXT-MSieve 5A

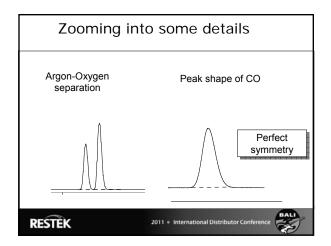
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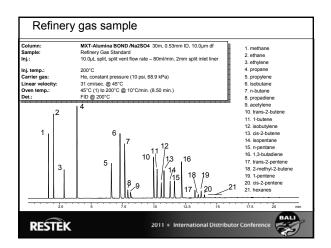
MXT-Alumina BOND
(Na₂SO₄ deactivation)

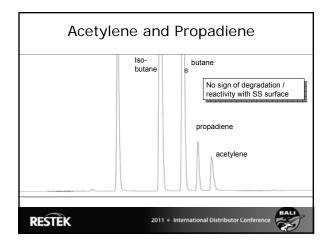
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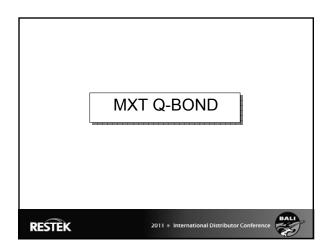
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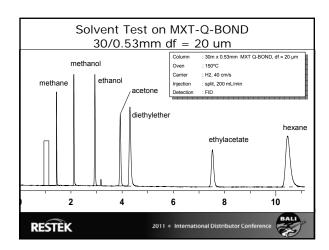


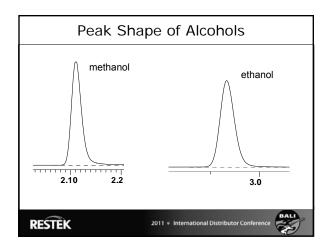


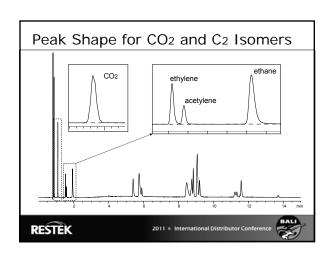
















Product information MXT PLOT PCFL1195A; http://www.restek.com/restek/images/external/PCFL1195A.pdf ration PLOT columns PCFL1163A; http://www.restek.com/restek/images/external/PCFL1163A.pdf Applications see: http://www.restek.com/restek/class/5004088.asp and http://www.restek.com/info chromatograms.asp Technical Literature: http://www.restek.com/aoi lit main.asp RESTEK 2011 # International Distributor Cor

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...



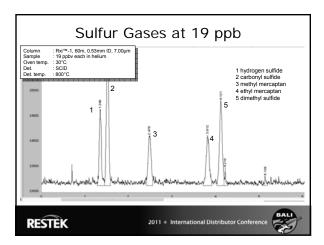




All parts in the GC system that come in contact with sulfur compounds can be deactivated by the Siltek technology...



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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...

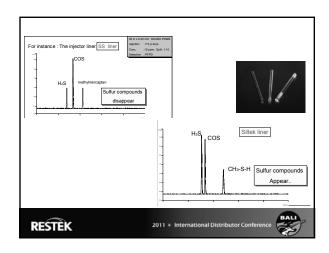
Restek Developed Siltek deactivated tubing..

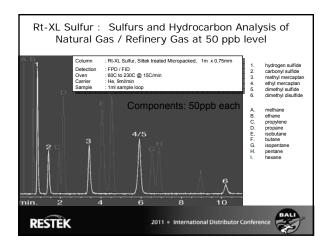
RESTEK

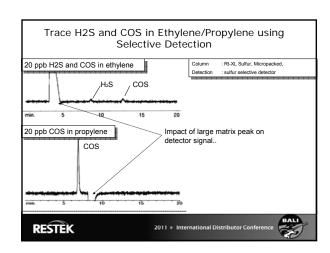
















Product information Sulfur Resease Refined socious PCT-1 1964, 1962 A 1

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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· Sulfur analysis

Oxygenates

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What Solutions

does Restek offer?







