The Agilent Intuvo GC – A New Way to Perform Gas Chromatographic Analyses for the Energy, Refining and Petrochemical Lab

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Outline

- Short Overview of Intuvo

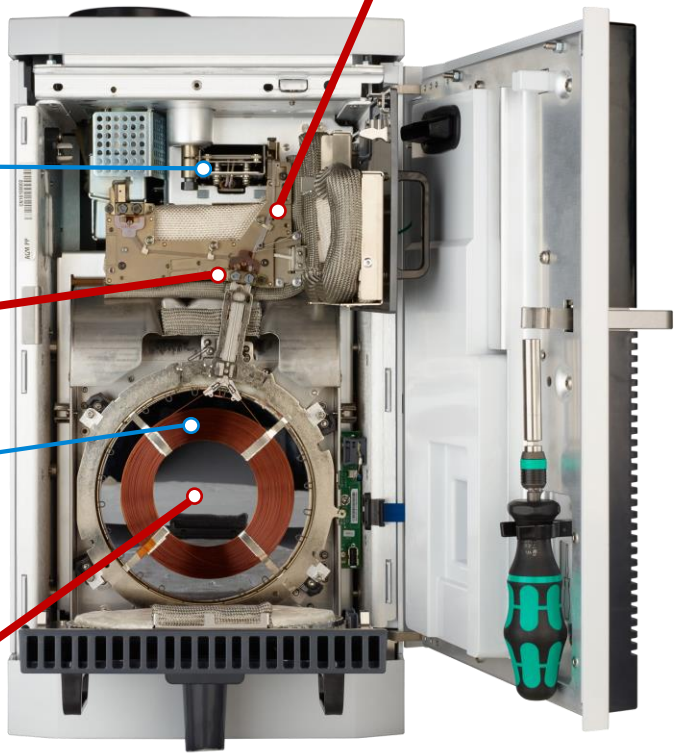
- Applications
  - Detection of Sulfur Compounds in Light Petroleum Liquids
    - Inert Flow Path ensures SCD sensitivity
  - Ultrafast Simulated Distillation
    - Fast column heating combined with 6th Gen EPC provides precise retention time
  - Aromatic Solvent Purity Analysis
    - Small footprint and Graphical User Interface ideal for routine production labs

Innovating a New Path to GC Productivity

- Easier
- Faster
- Smaller
- Smarter
- Greener

Innovating a New Path to GC Productivity

A whole new way to GC



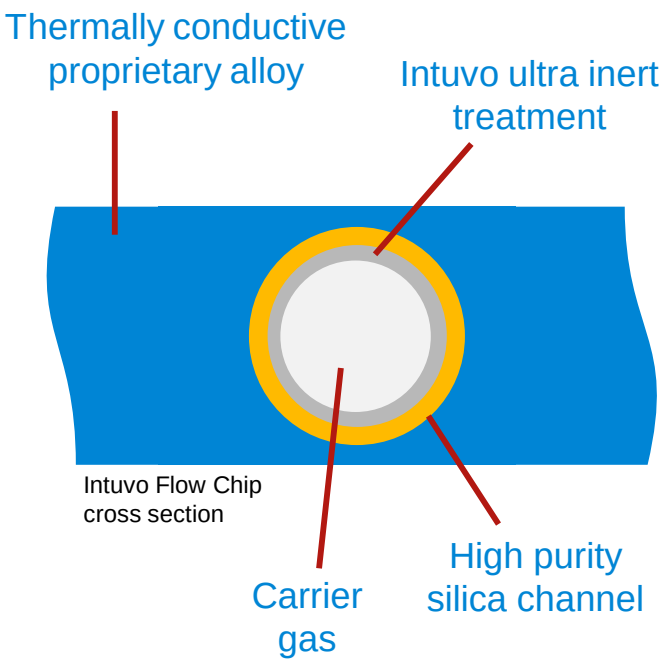
Flexible Compatible Design

Configurable to any application

- SSL, MMI, GSV, LSV inlets
- FID, TCD, ECD, NPD, FPD, NCD, SCD detectors
- SQ and TQ mass spectrometers
- Headspace, thermal desorption, purge and trap samplers
- 16-, 50-, 150-position auto-injectors and trays

Modular Intuvo Flow Chips

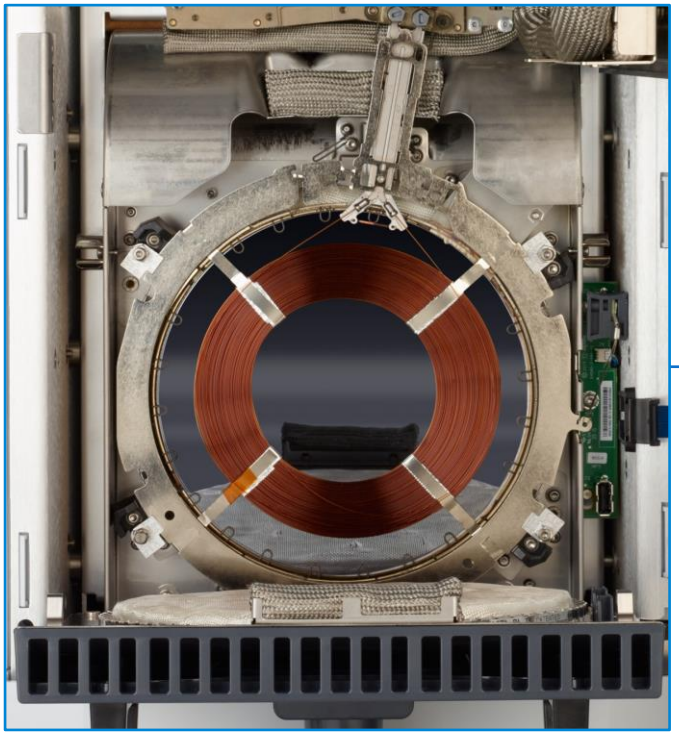
High purity inert silica flow path



- Inert
- Modular, configurable
- Click-and-run connections

Fast Direct Heating and Cooling

Every single minute counts



- Fast, efficient heating
- Ballistic cooling
- Smaller footprint

Optimizing Lab Space

Supporting lean laboratory thinking

Less than half:

- Power consumption
- Heat output into laboratory
- Laboratory bench footprint

- Lower operating costs
- Higher efficiency
- Greener

Intuvo Graphical User Interface

- System status
- Real-time chromatograms
- Step-by-step user maintenance and troubleshooting
- Finding parts fast

Detection of Sulfur Compounds in Light Petroleum Liquids with SCD-ASTM D5623

Rebecca Veeneman

Agilent Technologies

Detection of Sulfur in Light Petroleum Liquids

Why is this important?

- Sulfur-containing compounds are odorous and corrosive
- These compounds are present in petroleum feed stocks are monitored closely for process control
- There are also increasing regulations that require a reduction in sulfur emissions in order to reduce acid rain and green house gases
  - Refineries are tasked with meeting these regulations so detection of sulfur in gasoline at low levels is imperative

SCD is used since it is sensitive to sulfur species and avoids hydrocarbon quenching and minimizes matrix interferences

Why Intuvo?

- Refineries have limited bench space and at only 27cm, Intuvo is half the size of conventional GCs
- Analysts are typically refinery operators being asked to work outside of their comfort zone. Intuvo's chip-based flow path and click and run connections make the instrument easy to use
- Sulfur analysis can be difficult but when Intuvo is combined with the SCD the chromatographic art is built into the system

GC Methods

GC (7890 & Intuvo)

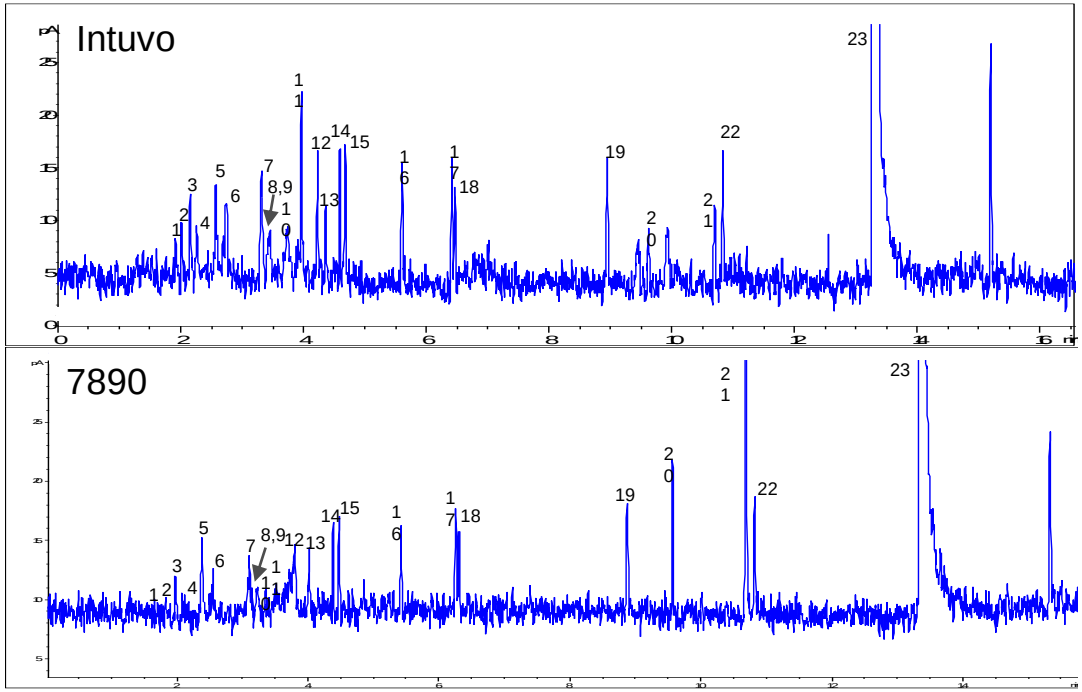
Injection volume	1 µL
Inlet	Split/Splitless 300 °C Split 10:1 Septum purge flow 3mL/min
Liner	UI Splitless liner with a single taper and glass wool at the bottom
Guard chip (*Intuvo only*)	300 °C
Column	Agilent DB-1 30 m x 0.32 mm x 1 µm
Flow	2 mL/min constant flow
Column temperature	40 °C for 0.71 min, 14.1 °C/min to 250 °C hold 1 min

SCD (8355)

Burner	800 °C
Base	280 °C (7890: 250 °C)
Oxidizer	50 mL/min (7890: 60 mL/min)
Upper H_2	38 mL/min (7890: 40 mL/min)
Lower H_2	8 mL/min
Ozone	36 mL/min
Range (stand alone)	6

Light Petroleum Sulfur Analysis

A 20 ppb standard containing 23 compounds was evaluated with a 10:1 split. Most of the analytes can be differentiated from the baseline at 20 ppb making the practical limit of detection 2 ppb.



#	Analyte	SNR (20ppb)
1	ethanethiol	1.76
2	dimethyl sulfide	2.43
3	carbon disulfide	3.35
4	2-propanethiol	2.04
5	2-methyl-2-propanethiol	3.35
6	1-propanethiol	1.48
7	ethylmethyl sulfide	2.47
8	2-butanethiol	4.02
9	thiophene	coleution
10	2-methyl-1-propanethiol	2.00
11	diethyl sulfide	1.34
12	1-butanethiol	6.45
13	dimethyl disulfide	4.79
14	2-methylthiophene	4.83
15	3-methylthiophene	5.04
16	3-chlorothiophene	4.44
17	2-bromothiophene	4.83
18	diethyl disulfide	3.81
19	di-tert-butyl disulfide	4.44
20	thianaphthene	2.40
21	2-methylbenzothiophene	3.31
22	3-methylbenzothiophene	4.79
23	diphenyl sulfide	ISTD

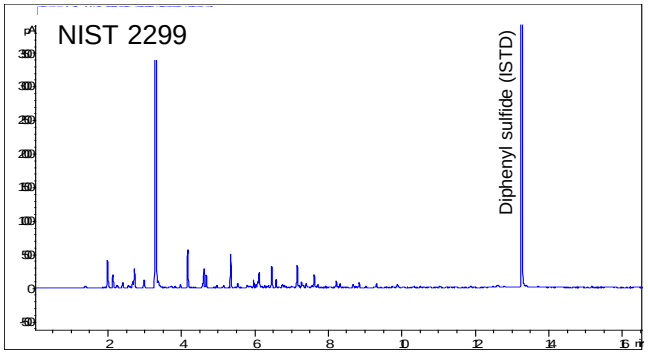
Intuvo GC-SCD Calibration Linearity is Excellent

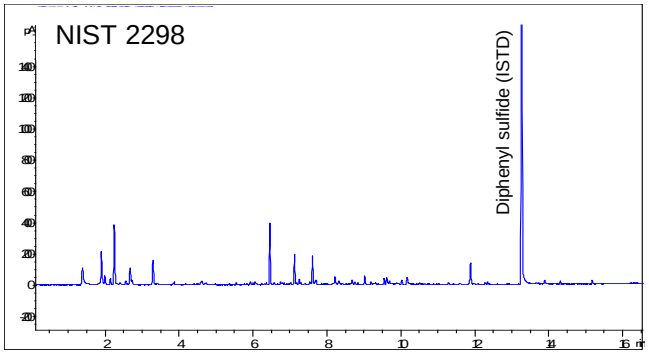
- Linearity was acceptable for all analytes evaluated
  - Average linearity was 0.999
- Area RSDs were all below 10% with the majority of analytes below 5%
  - Average area repeatability was 3.1%
  - Earlier eluting and lower concentration components showed higher variability (typical of this standard)

Analyte	0.1ppm	1ppm	10ppm	100ppm	R^2
Ethanethiol	8.5%	4.6%	5.4%	3.9%	0.998
Dimethyl sulfide	5.5%	5.9%	6.9%	4.1%	0.9971
Carbon disulfide	3.9%	6.8%	4.0%	7.3%	0.9972
2-propanethiol	3.1%	3.6%	3.9%	3.7%	0.9985
2-methyl-2-propanethiol	4.3%	2.6%	1.6%	1.0%	0.9994
1-propanethiol	6.7%	2.2%	5.3%	2.9%	0.9994
Ethylmethyl sulfide	3.8%	5.3%	5.1%	3.0%	0.9986
2-butanethiol	3.1%	4.2%	2.4%	3.8%	0.9992
Thiophene	3.9%	3.6%	4.5%	4.0%	0.9989
2-methyl-1-propanethiol	3.0%	3.2%	1.4%	1.1%	0.9997
Diethyl sulfide	6.9%	2.6%	3.7%	1.9%	0.9991
n-butanethiol	4.0%	3.7%	3.7%	1.9%	0.9944
Dimethyl disulfide	3.3%	3.5%	2.2%	4.0%	0.9993
2-methylthiophene	2.8%	3.5%	2.7%	1.7%	0.9998
3-methylthiophene	4.4%	3.3%	1.5%	0.94%	0.9998
3-chlorothiophene	4.7%	3.2%	1.2%	0.76%	0.9998
2-bromothiophene	2.8%	0.92%	2.3%	0.52%	0.9998
Diethyl disulfide	3.1%	1.2%	1.6%	0.56%	0.9999
Di-tert-butyl disulfide	2.5%	1.9%	0.66%	0.74%	0.9994
Thianaphthene	4.7%	0.66%	1.2%	0.54%	0.9995
2-methylbenzothiophene	2.7%	1.4%	1.2%	0.64%	0.9979
3-methylbenzothiophene	1.5%	2.4%	0.59%	0.18%	0.999

NIST Gasoline Reference Standards with the Intuvo GC-SCD are Within Specification

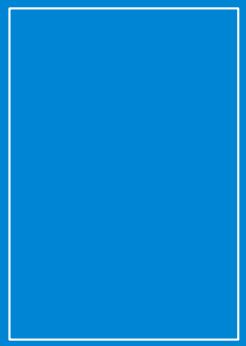
- An analysis of NIST 2299 (reformulated) was found to be within specification. Total sulfur was determined to be 14.3 µg/g (spec is 13.6±1.5 µg/g)
- An analysis of NIST 2298 (high octane) was found to be within specification. Total sulfur was 4.0 µg/g (spec is 4.7±1.3 µg/g).





Ultrafast Simulated Distillation

Agilent Intuvo 9000 Performance for ASTM Method D7798



Jim McCurry

Agilent Technologies

ASTM D7798 – A New Ultrafast SimDis Method for Middle Distillates

- UFGC method for the analysis of middle distillates
  - diesel, kerosene, heating oil, jet fuel
- Uses short, narrow columns with high flow rates and fast oven programming
- Run times reduced 10-fold compared to ASTM D2887

- Challenges for UFGC when running SimDis
  - retention time precision
    - small variation in RT can lead to large errors in BP determination
  - system discrimination
    - incomplete transfer of entire sample from inlet to detector causes failure final results of BP cuts

Why Intuvo for UFGC SimDis

- Extremely high retention time precision for UFGC
  - New Column Heating Technology
    - unique approach to direct column heating
      - up to 250 ^{o}C/ min over entire oven programming range
    - fewer column elements to fail
      - no complex direct heating/sensing elements
    - no complex in-oven connections
  - 6th Generation EPC
    - Works with New Column Oven to delivers precise carrier flow during rapid column heating
    - assures retention time and detector response stability
- Extremely good recovery for low to high boiling compounds
  - MMI combined with Intuvo Flow Technology assure quantitative transfer of all compound from inlet to column

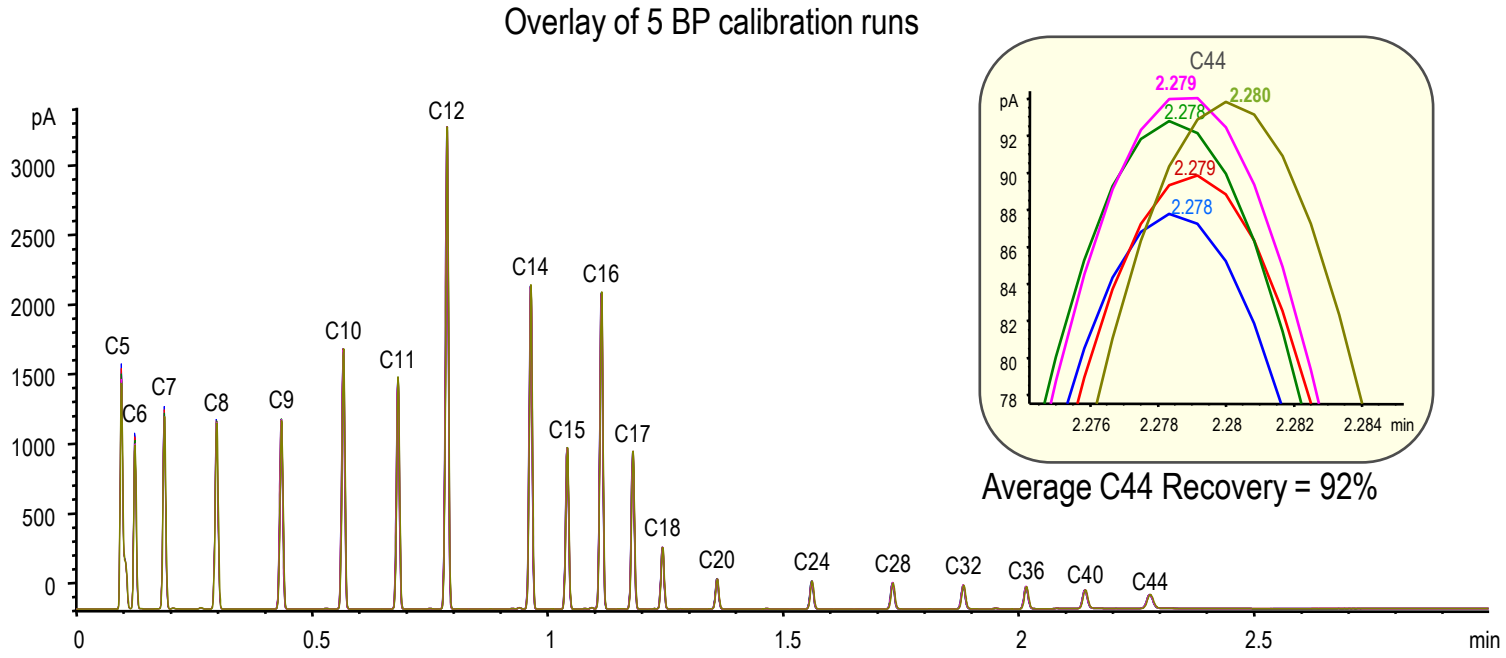
Typical D7798 GC Conditions

Conditions for Intuvo 9000

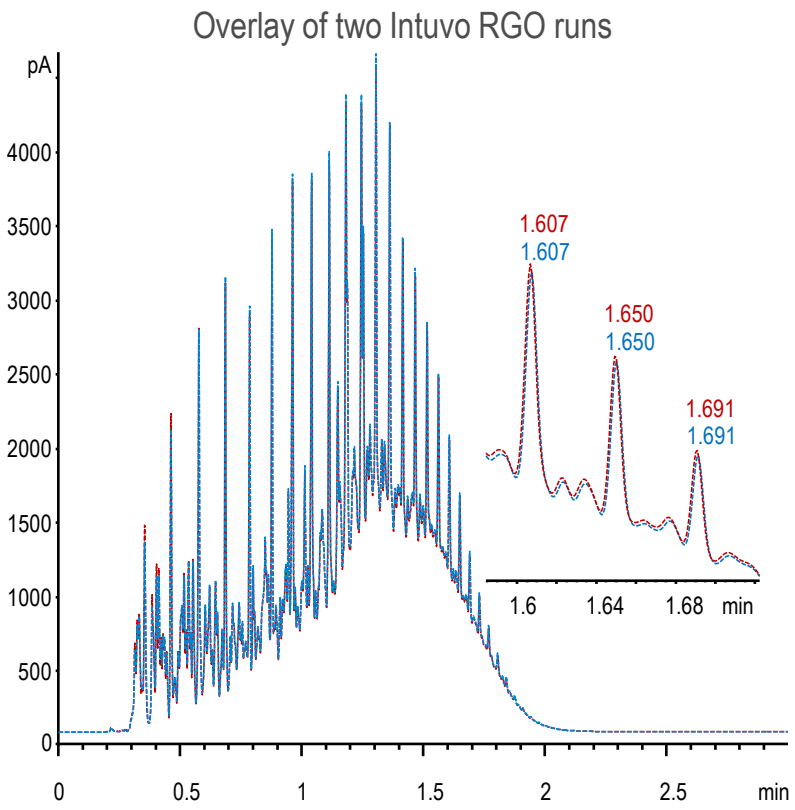
- ALS with 10 μL syringe, 0.1 μL injection
- MMI Inlet, split 30:1, 5190-2293 UI liner, 350 °C, Merlin Microseal
- Guard Chip Jumper, 350 °C
- DB-1 column, 4 m x 0.25 mm ID x 0.5 μm
  - helium carrier gas @ 8 mL/min constant flow
  - oven program, 40 °C for 0 min, 160 °C/min to 350 °C, hold 1 min
- FID, 350 °C

Challenges for UFGC When Running SimDis

- retention time precision
  - small variations in RTs can lead to imprecise BP determination
- system discrimination
  - incomplete transfer of entire sample from inlet to detector causes failed BP cut determination



SimDis QC Sample Runs - Reference Gas Oil (RGO)



	Ref	Allowed	RGO Start		RGO End	
	Temp (°C)	Diff (°C)	Temp (°C)	Diff (°C)	Temp (°C)	Diff (°C)
IBP	115	7.6	115	0	115	0
5	151	3.8	151	0	151	0
10	176	4.1	177	1	177	1
15	201	4.5	203	2	203	2
20	224	4.9	227	3	227	3
25	243		246		246	
30	259	4.7	262	3	262	3
35	275		277		277	
40	289	4.3	291	2	291	2
45	302		303		303	
50	312	4.3	314	2	314	2
55	321	4.3	323	2	323	2
60	332	4.3	333	1	333	1
65	343	4.3	345	2	345	2
70	354	4.3	355	1	355	1
75	365	4.3	367	2	367	2
80	378	4.3	380	2	380	2
86	391	4.3	393	2	393	2
90	407	4.3	409	2	409	2
95	428	5	430	2	430	2
FBP	475	11.8	473	2	473	2

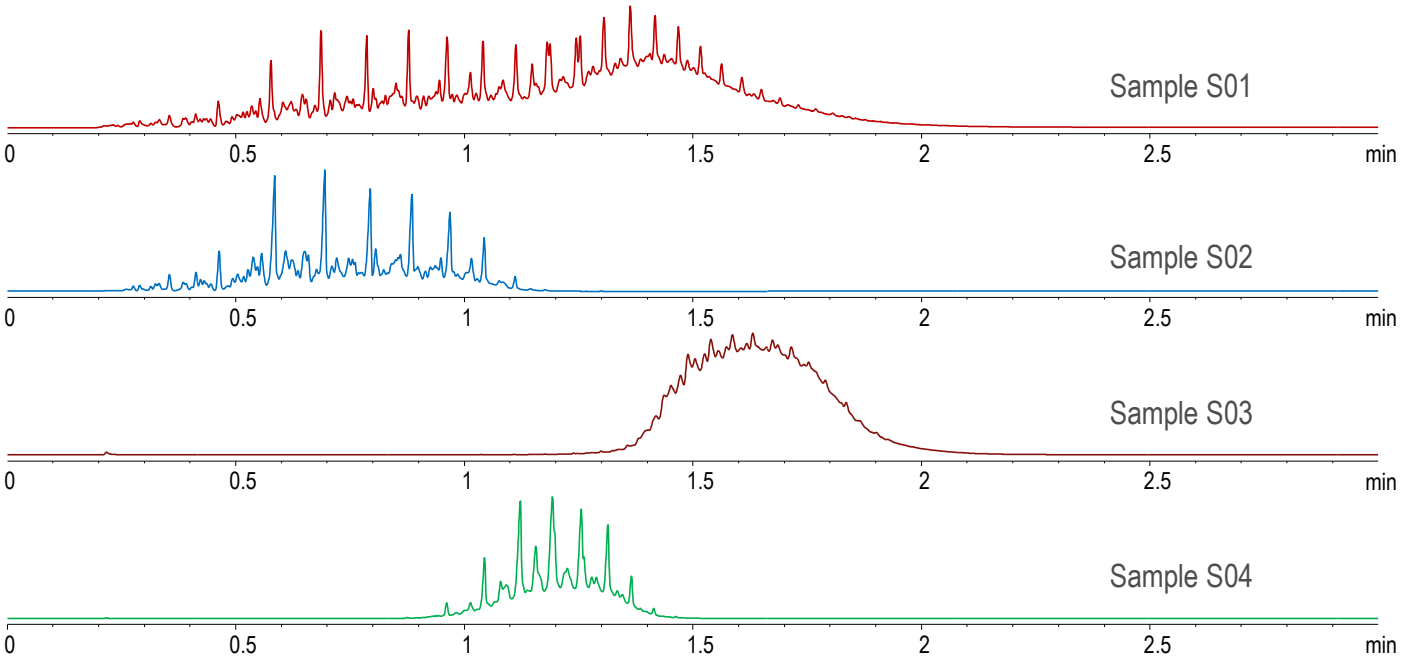
Comparing UFGC SimDis (D7798) and Conventional SimDis (D2887)

Experimental Details

- 24 middle distillate duplicates run on 7890 series GC using ASTM D2887 conditions
- Same samples run on Intuvo GC using ASTM D7798 conditions
- Instrument blanks run before and after sample set to assure consistent baseline and no carry-over
- QC sample (reference gas oil) run before and after sample set to assure system performance

% Off	Temperature (deg. C)							
	Sample 01		Sample 02		Sample 03		Sample 04	
	D2887	D7798	D2887	D7798	D2887	D7798	D2887	D7798
IBP	119	117	107	104	331	336	246	247
5	174	174	146	145	360	362	268	269
10	198	198	162	163	368	370	275	276
15	217	218	169	170	374	376	282	282
20	235	236	175	176	379	381	288	288
25	252	253	180	181	385	387	290	290
30	268	270	186	188	389	391	294	295
35	284	286	193	195	394	396	298	298
40	298	301	198	198	398	400	302	303
45	310	313	202	204	403	405	305	305
50	321	324	209	211	407	409	307	307
55	331	334	217	217	411	414	310	311
60	341	344	219	221	416	419	313	314
65	349	353	227	229	420	423	317	319
70	358	361	233	235	425	428	320	320
75	367	370	237	239	430	433	323	325
80	376	380	246	247	435	439	327	329
86	389	392	253	254	441	445	332	333
90	404	408	259	262	449	452	335	338
95	428	431	271	272	461	464	344	346
FBP	481	481	290	290	490	490	363	365

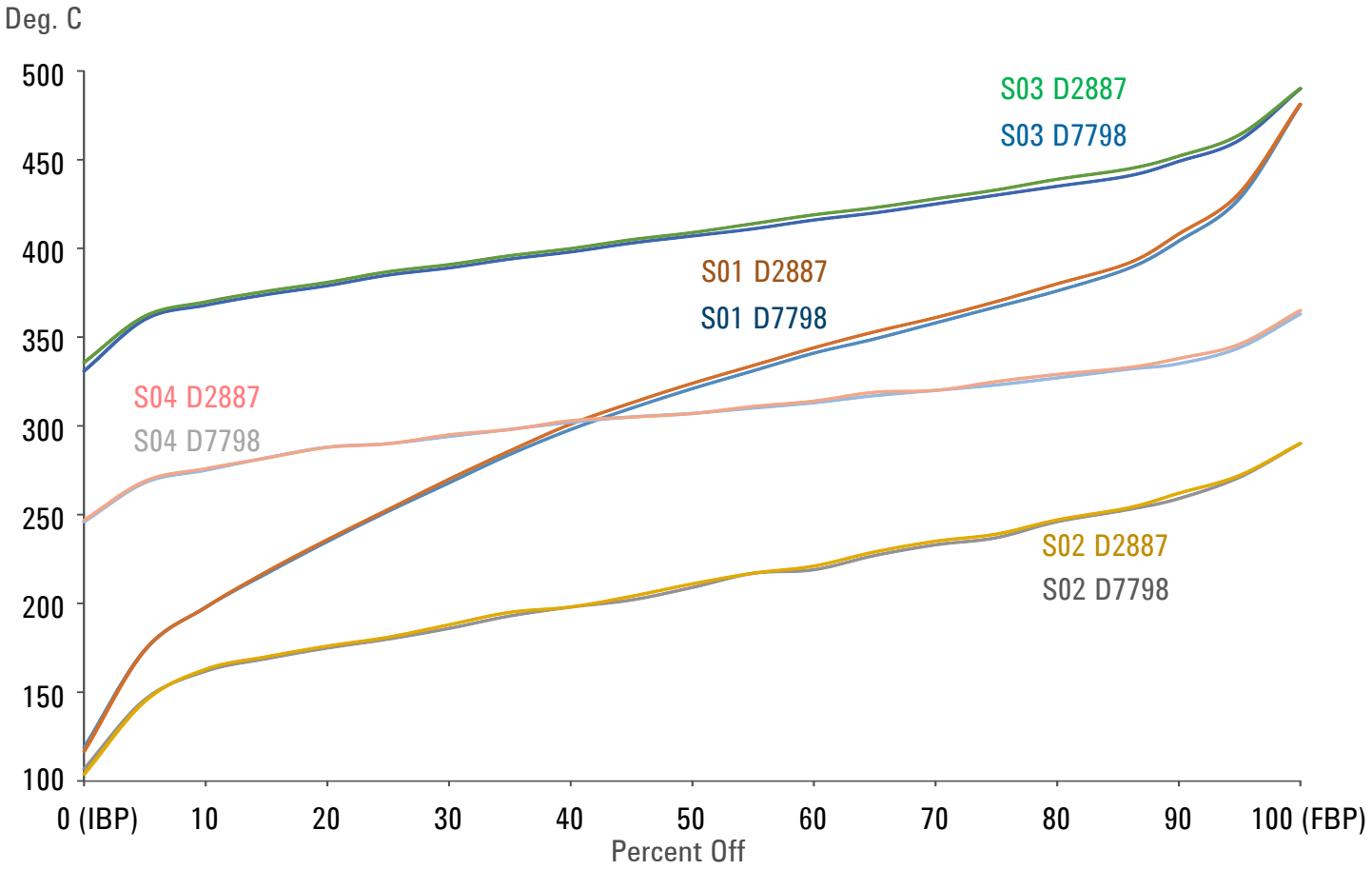
UFGC SimDis Sample Runs



Four different sample types

- S01 – wide boiling range
- S02 – narrow, lower boiling range
- S03 - narrow, high boiling
- S04 – narrow, mid boiling range

Comparing UFGC SimDis (D7798) and Conventional SimDis (D2887)



Conclusions

- A new technology for fast capillary column heating uses a direct heating disk to rapidly transfer heat to a planar capillary GC column.
- This enables fast and precise column heating up to 250 ^{o}C across the a temperature range of 40 ^{o}C to 450 ^{o}C.

- Extremely fast temperature programming is ideal for minimizing GC analysis times when individual peak resolution is not required.
- High retention time precision and low boiling point discrimination assured precise and reliable results.

Aromatic Solvent Purity Analysis – ASTM D7504

Jim McCurry

Agilent Technologies

ASTM D7504 – Aromatic Solvent Purity

Latest ASTM Method Relies on Advanced GC Performance

- Widely used method to measure trace impurities and major compound purity in monomer precursors
- No calibration – uses Effective Carbon Number (ECN) based on FID response
  - FID must be capable of detecting large peaks (% level) and small peaks (ppm level in a single run

Instrument Conditions for Agilent 9000 GC	
Inlet	270 °C, split 100:1, Liner 2295
AQM Flow Path	Jumper to column connector at 270 °C isothermal
Column	HP-Innowax, 60m x 0.32 mm x 0.5 um Flow: helium at 2.1 mL/min constant
Column Oven	Initial: 60 °C for 10 min Ramp: 5 °C/min Final: 150 °C for 10 min
Detector Tail	300 °C
Detector	FID at 300 °C
Injection	0.5 uL

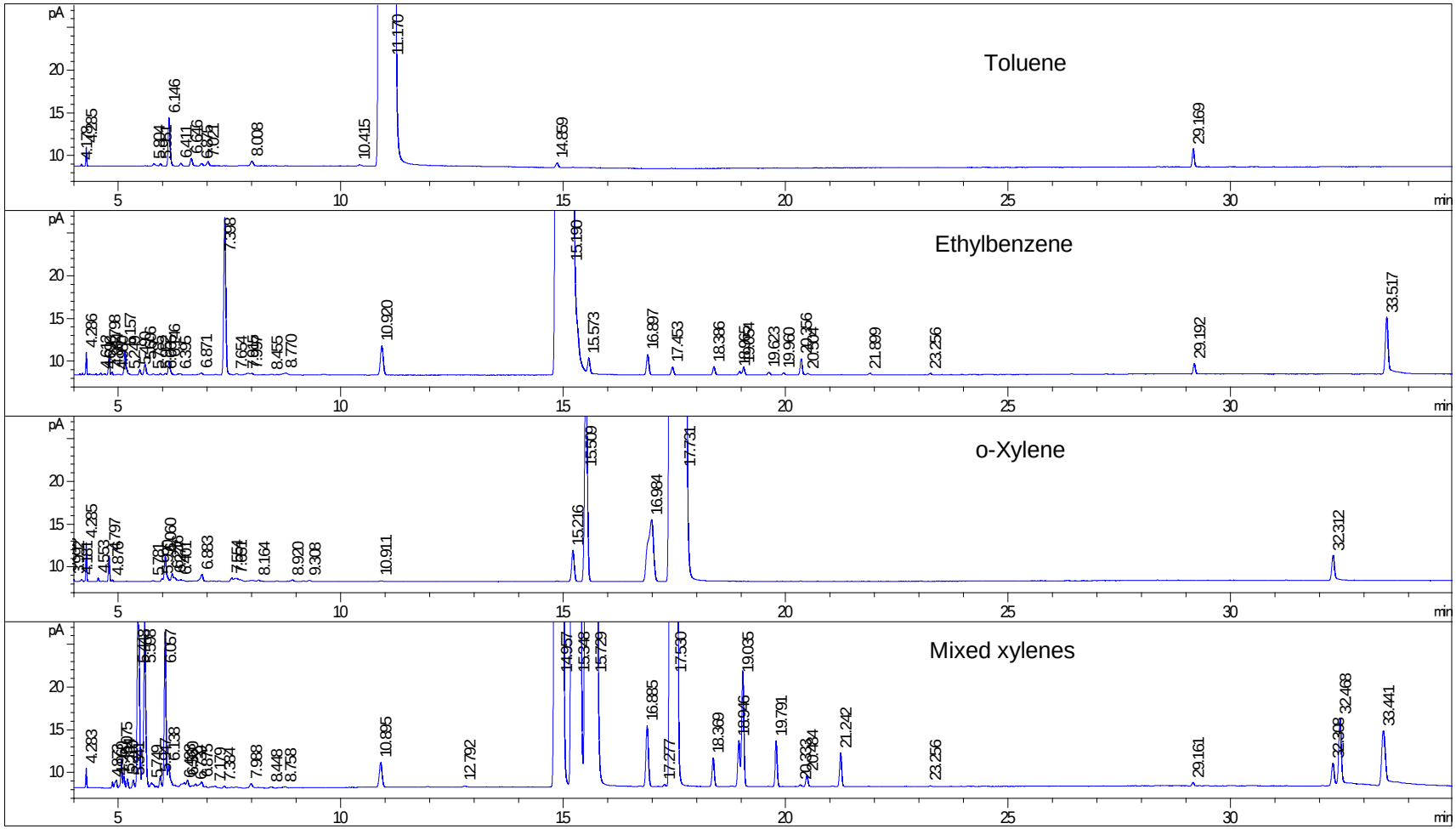
Why Intuvo for D7504 Analysis of Aromatic Solvent Purity

- D7504 is designed as an easy-to-use method
  - no sample prep or calibration standards

Why not have an easy-to use GC for an easy-to-use method?

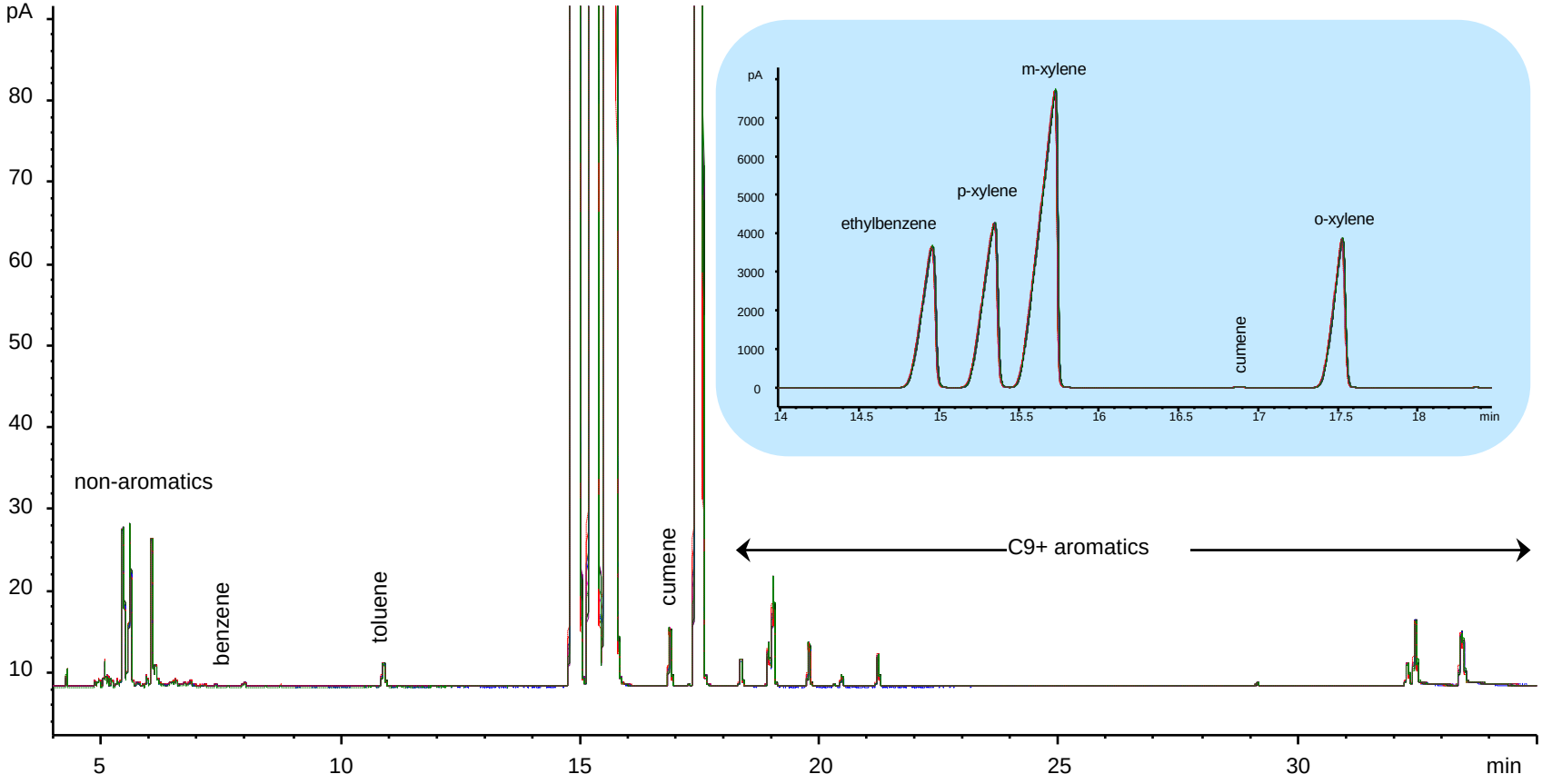
- Intuvo's FID is ideally suited to using Effective Carbon Number response
  - uses the Agilent's FID with autoranging electronics
    - detector response is linear for peaks from ppm to 99+ % in a single run
- Intuvo is an easy to use
  - Intuvo click and run connections for easy column install
  - Intuvo Smart ID Key
  - Intuvo Health Report and Early Maintenance Feedback (EMF)

ASTM D7504 – Aromatic Solvent Purity



Ten Overlays of Mixed Xylene Analysis

Autoranging FID Quantitatively Detects Small and Large Peaks in a Single Run



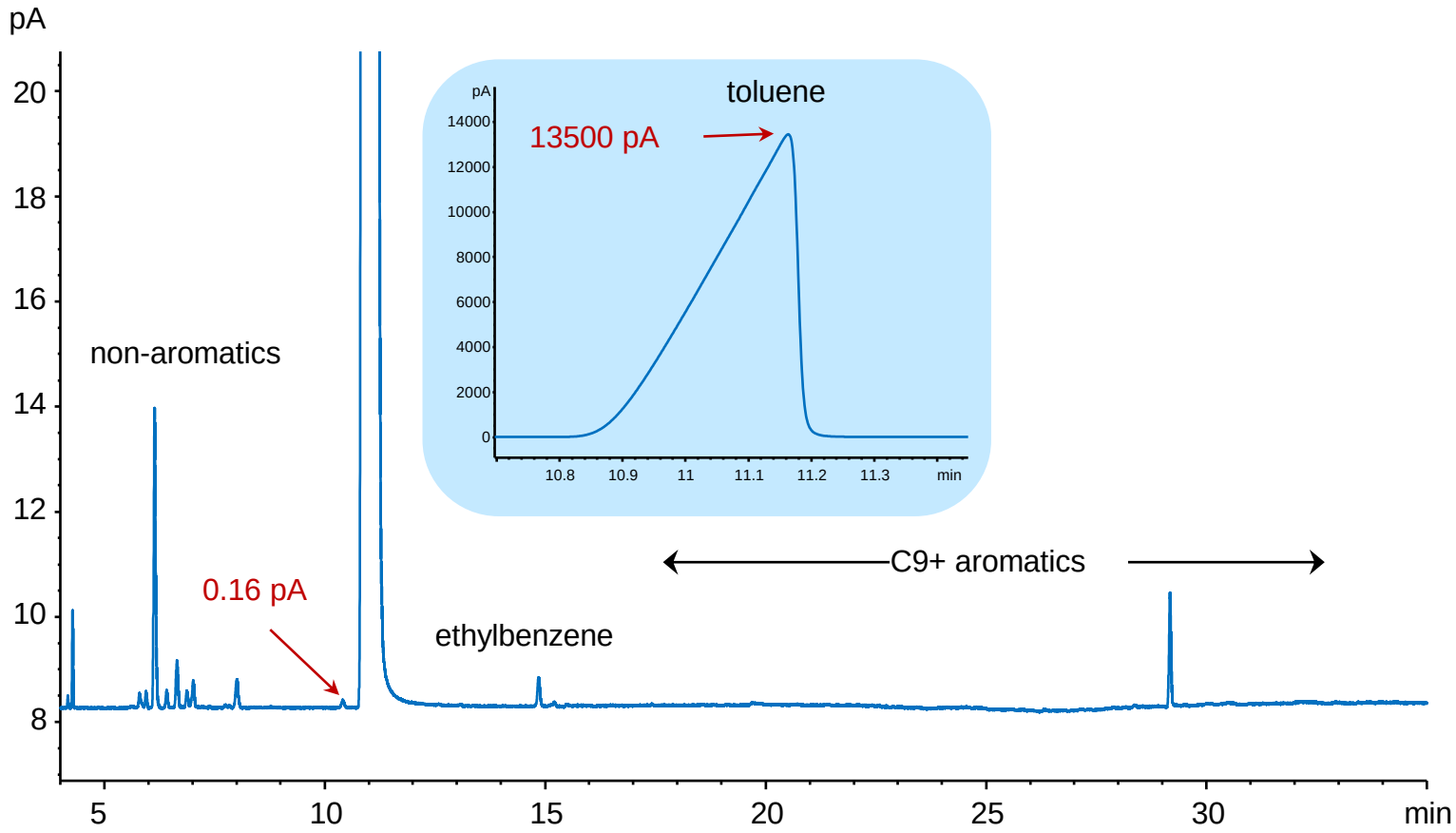
Results for Ten D7504 Analyses of Mixed Xylenes

Run	wt %	wt %	wt %	wt %	wt %	wt %	wt %	wt %
1	0.1982	0.0101	16.84	21.05	46.43	0.0221	15.29	0.1667
2	0.1991	0.0100	16.83	21.05	46.43	0.0219	15.30	0.1662
3	0.1986	0.0100	16.84	21.05	46.43	0.0218	15.29	0.1646
4	0.1994	0.0100	16.83	21.05	46.43	0.0219	15.30	0.1595
5	0.1984	0.0100	16.83	21.04	46.43	0.0219	15.30	0.1623
6	0.1993	0.0101	16.84	21.04	46.43	0.0219	15.31	0.1651
7	0.2008	0.0100	16.83	21.04	46.43	0.0219	15.30	0.1632
8	0.1998	0.0101	16.84	21.04	46.43	0.0219	15.30	0.1566
9	0.2005	0.0100	16.83	21.04	46.43	0.0219	15.30	0.1624
10	0.2005	0.0100	16.83	21.04	46.43	0.0219	15.31	0.1633
Mean	0.1995	0.0100	16.83	21.04	46.43	0.0219	15.30	0.1630
StdDev	0.00092	0.00005	0.004	0.004	0.003	0.00007	0.006	0.00307
ASTM SD	0.00700	0.01400	0.007	0.029	0.021	0.00030	0.010	0.00100

Exceeds ASTM Precision Requirements

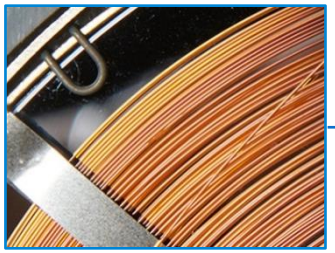
D7504 Analysis of Purified Toluene

Precisely measures very large components (99.97%) and very small components (18 ppm) in a single run



Intuvo 9000 GC System

Appealing to all stakeholders



Operator and Scientist

- Simpler operation, less training, fewer mistakes
- Innovative enabling technology: Future of GC

Lab Manager

- Less unplanned downtime and disruption
- Delivering results with confidence

Business Executive

- A better financial return on innovation
- Improved operations and business continuity


DREAM
BIGGER
GC