Enhanced Quantitation and Reporting Features for ASTM D5769 Using ChromaTOF®

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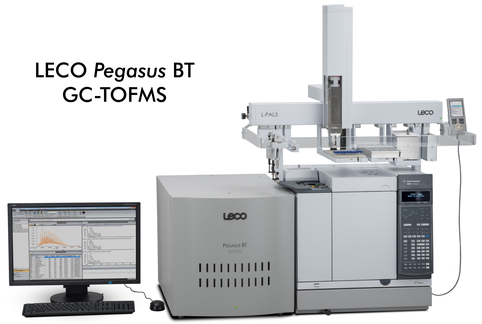
Abstract

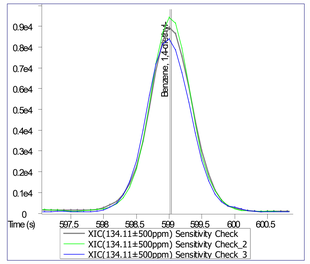
Creating fully customizable reports that meet the variety of needs outlined in the ASTM D5769 Standard Test Method for Determination of Benzene, Toluene, and Total Aromatics in Finished Gasolines by Gas Chromatography/Mass Spectrometry can be a challenge. With LECO's *ChromaTOF* brand software for the Pegasus® BT GC-TOFMS, fully integrated hardware control, data processing, and reporting features are combined with new quantitation types to allow a seamless experience that exceeds method criteria. From collecting wide linear dynamic range and full-mass-range time-of-flight mass spectral data to reporting deconvoluted peaks, ion ratio results, and using multiple internal standards, the *Pegasus* BT system operating on ChromaTOF provides an excellent solution for a variety of established methods, including ASTM D5769.

LECO *Pegasus* BT
GC-TOFMS



Gas Chromatograph	Agilent 7890 with Agilent 7693 Liquid Autosampler
Injection	0.1 µL injection, split 1200:1 @ 260 °C
Carrier Gas	He @ 1.0 ml/min, Constant Flow
Column	Rxi-1ms, 30 m x 0.25 mm i.d. x 1.00 µm coating (Restek, Bellefonte, PA, USA)
Oven Program	55 °C (1 min), to 70 °C @ 20 °C/min (4 min), to 220 °C @ 30 °C/min (5 min)
Transfer Line	280 °C
Mass Spectrometer	**LECO *Pegasus* BT**
Ion Source Temperature	250 °C
Mass Range	35-550 m/z
Acquisition Rate	10 spectra/s

Instrument Checks

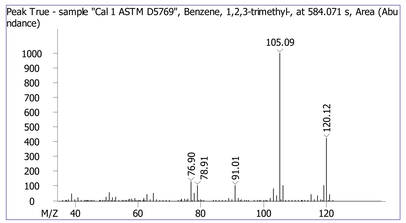


Name	Quant S/N
1,4-Diethylbenzene	141
1,4-Diethylbenzene	151
1,4-Diethylbenzene	132

Figure 1 (left): Triplicate injections of 0.01 mass % 1,4-diethylbenzene are shown. According to Section 6.2.3 of the method, the signal-to-noise (S/N) ratio of 0.01 mass % 1,4-diethylbenzene at mass 134 must be consistently greater than 5. The table above shows an average S/N greater than 100, easily surpassing method requirements.

Figure 2 (right): The deconvoluted Peak True spectrum of 1,2,3-Trimethylbenzene is shown along with a table of relevant ion ratio intensity values as stipulated in Section 9.2.5 of the method.

Ion (m/z)	Required Intensity	Observed Intensity
120	30-60	42
105	100	100
91	7-15	11



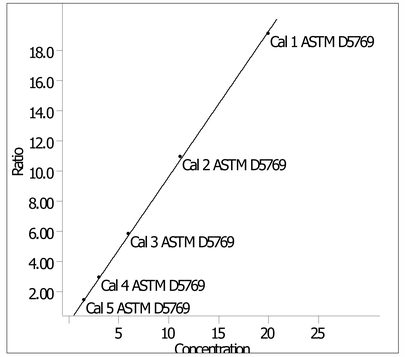


Figure 3 (above): The calibration curve for toluene illustrates the excellent linearity that is shown in the table of R^2 values for each calibrated analyte.

Analyte	R^2
Benzene	0.99994
Toluene	0.99980
Ethylbenzene	0.99998
Benzene, 1,3-dimethyl- + 1,4-dimethyl	0.99998
1,2-Dimethylbenzene	0.99998
Benzene, (1-methylethyl)-	1.00000
Benzene, propyl-	1.00000
Benzene, 1-ethyl-3-methyl-	0.99950
Benzene, 1-ethyl-4-methyl-	0.99980
1,3,5-Trimethylbenzene	0.99996
Benzene, 1-ethyl-2-methyl-	0.99998
Benzene, 1,2,4-trimethyl-	1.00000
Benzene, 1,2,3-trimethyl-	0.99996
Indane	0.99988
Benzene, 1,4-diethyl- + butylbenzene	0.99994
Benzene, 1,2-diethyl-	0.99992
Benzene, 1,2,4,5-tetramethyl-	0.99992
Benzene, 1,2,3,5-tetramethyl-	0.99986
Naphthalene	0.99992
Naphthalene, 2-methyl-	0.99950
Naphthalene, 1-methyl-	0.99954
Average Value	**0.99987**

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QC Check

Sample Report

Peak Table - QC Check

Peak #	Name	R.T. (s)	QC Wt %	QC Expected	QC Pass	Quant Masses
6	Benzene	230.514	1.03	0.95 - 1.05	Pass	XIC(78.05±0.3)
14	Toluene	377.725	9.06	8.55 - 9.45	Pass	XIC(92.06±0.3)
21	Ethylbenzene	479.134	3.10	2.85 - 3.15	Pass	XIC(106.08±0.3)
22	m-Xylene + p-Xylene	485.834	3.05	2.85 - 3.15	Pass	XIC(106.08±0.3)
24	o-Xylene	504.236	3.07	2.85 - 3.15	Pass	XIC(106.08±0.3)
38	1,2,4-trimethylbenzene	568.041	2.95	2.85 - 3.15	Pass	XIC(120.09±0.3)
83	1,2,3,5-tetramethylbenzene	625.545	1.94	1.80 - 2.20	Pass	XIC(134.11±0.3)
104	Naphthalene	654.048	1.08	0.90 - 1.10	Pass	XIC(128.06±0.3)
Total*	Total		25.29			

Figure 4 (left): Built-in software checks allow for quick review of quality control (QC) samples and system readiness. Fully-customizable workspaces and automated data processing allow streamlined data display for easier interpretation of QC results.

User-defined text boxes automatically populate with desired information on sample acquisition, sample properties, and data processing methods.

Report Generated Date/Time: 1/5/2018
Sample: Gasoline QC Demo
Sampled Acquired Date: 5/8/2017
Operator: Christina Kelly

Quantitation Acquired Date: 8/25/2017
Quantitation Method: D5769 5.2.36

Sample Weight: 1.0754 g Sample Density: 0.7389 Internal Standard Weight: 0.049 g

Customizable image fields assist in reflecting company branding

Peak #	Name	R.T. (s)	Quant Masses	Area	[Conc. units]
11	Benzene-D6	228.613	XIC(84.08±0.3) XIC(83.08±0.3)	240739833	9.60 mg/100gm
27	Ethylbenzene-D10	474.933	XIC(116.14±0.3) XIC(115.13±0.3)	72721605	19.76 mg/100gm
103	Naphthalene-D8	652.847	XIC(136.11±0.3) XIC(135.11±0.3)	146542156	10.24 mg/100gm

Specially-filtered peak table provides information on internal standard analytes. User-specified concentrations can be edited for each sample and are reflected in all automated calculations

Additional user-defined filters simplify interpretation of analyte results, including rows that aggregate results for uncalibrated components

Peak #	Name	R.T. (s)	Quant Masses	Area	[Wt %]	[Vol %]
1	Uncalibrated C10	64.2003	BPI(134.11±0.3)	67247927	2.43	2.03
3	Uncalibrated C11	104.404	BPI(148.13±0.3)	16028408	0.58	0.48
12	Benzene	230.914	XIC(78.05±0.3)	65161743	0.26	0.22
19	Toluene	378.325	XIC(92.06±0.3)	270264059	3.51	2.93
28	Ethylbenzene	479.434	XIC(106.08±0.3)	30706525	0.79	0.67
31	m-Xylene + p-Xylene	486.234	XIC(106.08±0.3)	176755335	3.39	2.88
35	o-Xylene	504.436	XIC(106.08±0.3)	58538447	1.19	0.99
36	Isopropylbenzene	526.637	XIC(120.09±0.3)	937015	0.03	0.03
38	n-Propylbenzene	545.339	XIC(120.09±0.3)	8241830	0.26	0.22
39	1-methyl-3-ethylbenzene	549.139	XIC(120.09±0.3)	41331484	1.13	0.96
40	1-methyl-4-ethylbenzene	550.539	XIC(120.09±0.3)	14504009	0.41	0.35
41	1,3,5-Trimethylbenzene	553.539	XIC(120.09±0.3)	29101444	0.54	0.46
42	1-methyl-2-ethylbenzene	560.14	XIC(120.09±0.3)	12245194	0.36	0.30
43	1,2,4-trimethylbenzene	568.141	XIC(120.09±0.3)	90278501	1.83	1.53
49	1,2,3-trimethylbenzene	583.342	XIC(120.09±0.3)	19456491	0.46	0.38
54	Indane	590.342	XIC(117.07±0.3)	27757062	0.28	0.21
55	Alkyl Indanes	594.743	BPI(117.07±0.3)	112515283	1.12	0.85
60	1,4-diethylbenzene + n-Butylbenzene	598.343	XIC(134.11±0.3)	18139726	0.65	0.56
63	1,2-diethylbenzene	601.343	XIC(134.11±0.3)	199759	0.01	0.01
80	1,2,4,5-tetramethylbenzene	625.645	XIC(134.11±0.3)	9648137	0.19	0.16
83	1,2,3,5-tetramethylbenzene	627.145	XIC(134.11±0.3)	13494148	0.30	0.24
100	Uncalibrated C12	651.447	BPI(162.14±0.3)	776482	0.03	0.02
104	Naphthalene	654.048	XIC(128.06±0.3)	45506512	0.32	0.24
146	2-methylnaphthalene	697.351	XIC(142.08±0.3)	7302511	0.08	0.06
152	1-methylnaphthalene	704.552	XIC(142.08±0.3)	2054690	0.03	0.02
	Total			1138192722	20.16	16.81

Auto-calculated Total row shows the final result for total aromatics

Figure 5 (above): Sample report with fully user-customizable, automatically populated elements display the results for a gasoline sample, showing values for both calibrated and uncalibrated analytes, as well as total aromatics.