Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.

Supplemental information

Fully reproducible, low-temperature synthesis of high-quality, few-layer graphene on nickel via preheating of gas precursors using atmospheric pressure chemical vapor deposition

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Raman data (I_D/I_G) from fifteen graphene growth experiments without preheating and with preheating

The corresponding distribution plot of I_D/I_G can be found in the text

Exp. #	No Preheating	stdv	stdv%	With Preheating	stdv	stdv%
1	0.45	0.035	7.8%	0.14	0.031	22.8%
2	1.70	0.196	11.6%	0.15	0.037	24.8%
3	1.05	0.028	2.7%	0.14	0.028	20.4%
4	0.76	0.044	5.8%	0.17	0.021	12.1%
5	1.03	0.061	5.9%	0.14	0.041	29.5%
6	0.62	0.047	7.6%	0.13	0.038	30.1%
7	0.63	0.171	27.0%	0.12	0.055	45.6%
8	0.71	0.114	16.0%	0.11	0.024	21.5%
9	0.67	0.070	10.3%	0.11	0.040	35.7%
10	0.82	0.068	8.3%	0.29	0.072	24.4%
11	1.75	0.095	5.4%	0.02	0.014	67.6%
12	0.88	0.141	16.1%	0.03	0.022	74.0%
13	0.88	0.047	5.3%	0.17	0.044	25.6%
14	0.90	0.047	5.2%	0.37	0.098	26.3%
15	0.41	0.107	26.3%	0.29	0.087	30.1%
Average D/G	0.88			0.16		
Standard deviation	0.39			0.09		

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GC-MS Analysis of Aromatic Materials Collected from Graphene Synthesis Furnace (BIUMS140507b).

Experimental.

GC–MS Analysis. GC−MS analyses were performed on an Agilent 6890/5977A GC-MS system equipped with Agilent 30 m × 0.25 mm i.d. HP-5MS column (5% Phenyl/Methylpolysiloxane, 0.25 µm film thickness). The carrier gas was helium (99.999%) at constant flow rate of 1.2 mL/min. The GC conditions were as follows: injection volume 1.0 µL (Agilent auto-sampler G4513A); injector temperature 250 °C; splitless time 1.0 min; initial oven temperature 70 °C increased to 300 °C at a rate of 10 °C/min with 7 min hold time. MS was performed in the EI positive ion mode, using the electron energy of 70 eV. Transfer line temperature and ion source temperature were maintained at 280 and 300 °C, respectively. MS data were collected in full-scan mode (m/z 50−500) and analyzed with Agilent Chemstation software. Molecular identification was done with NIST MS Search v2.0 and verified by TAMI v3.9 (AVIV Analitical) software.[1]

[1] Tal Alon and Aviv Amirav; "Isotope Abundance Analysis Method and Software for Improved Sample Identification with the Supersonic GC-MS." *Rapid Commun. Mass Spectrom*. 20, 2579-2588 (**2006**).

Quantitative data:

TIC – Total Ion Count

RSIM - Reconstructed Single Ion Monitoring. (Used for specific molecular mass ion and can be used for quantity evaluation in case of high background nose or overlapping peaks of several materials).

Benzene and toluene wasn't detected due to solvent delay of 2 min.

RT – retention time

According to internal markers dioctyl phthalate (RT 20.3 min) and heptadecane (RT (12,4 min), the "NO" sample was at least 3 times concentrated than "WITH" sample. It should be mentioned that due to preconcentration of "NO" sample its background and S/N ratio are higher than in "WITH" sample.

RT	Internal marker	"WITH" TIC Area	"NO" TIC Area	Ratio
20.3	dioctyl phtalate	91953311	312082392	3.39
12.4	heptadecane C17H36	3937190	11906319	3.02

Quantitative data for samples (not normalized):

RT (min)	Mw (m/z)	Mol. formula	Substance	"WITH" TIC Area	"NO" TIC Area	"WITH" RSIM Area	"NO" RSIM Area
2.4-2.7	106	C8H10	Dimethyl benzenes	119772048	363129902	40085727	107863368
3.2-4.1	120	C9H12	Tri methyl benzenes	77235938	223994686	15015470	53287831
4.30	116	C9H8	Indene	8058838	NA*	2877108	60359
6.14	128	C10H8	Naphthalene	93956887	6229997	63310896	1289037
9.36	154	C12H10	Biphenyl/ 2-Vinylnaphthalene	2155889	NA*	722644	16311
9.64	152	C12H8	Acenaphthylene	9669133	2802267	5250484	88221
10.09	154	C12H10	Acenaphthene/ 2-Vinylnaphthalene	4839053	NA**	600417	93278
11.26	166	C13H10	Fluorene/Phenalene	13408541	NA***	3118470	258975
12.03	166	C13H10	Fluorene/Phenalene	3299802	NA*	533883	NA
13.49	178	C14H10	Phenanthrene	41308201	19487185	18022644	2100983
13.60	178	C14H10	Anthracene	20489781	15999355	7033541	290317
14.92	190	C15H10	4,5-Methylenephenanthrene	8771197	6852270	2123110	321432
16.31	202	C16H10	Fluoranthene	8881744	7226444	2968597	672615
16.82	202	C16H10	Pyrene	14178519	13759915	5124378	1349595

NA- no peak was found due to high S/N*

*NA** - Integration not available due to overlapping with stearic acid ester (fatty acid)*

*NA*** - Integration not available due to overlapping with phthalate derivatives (plasticizers)*

You can normalize upper mentioned data with comparison to various factors. In my opinion, you can divide the "NO" data by 3, because it was concentrated during the handling process and according to internal markers. And then normalize all the values by Anthracene "with" area (20489781) or any other choice. I got the following results. The latter demonstrate larger presence of high aromatics in the "with" sample in comparison to "no" sample (especially naphthalene, ahenanthrene and anthracene).

RT (min)	Mw (m/z)	Mol. formula	Substance	WITH normalized amount	NO normalized amount
2.4-2.7	106	C8H10	dimethyl benzenes	5.8	5.9
3.2-4.1	120	C9H12	trimetyl benzenes	3.8	3.6
4.30	116	C9H8	Indene	0.4	NA*
6.14	128	C10H8	Naphthalene	4.6	0.1
9.36	154	C12H10	Biphenyl/2-Vinylnaphthalene	0.1	NA*
9.64	152	C12H8	Acenaphthylene	0.5	0.0
10.09	154	C12H10	Acenaphthene/2-Vinylnaphthalene	0.2	NA**
11.26	166	C13H10	Fluorene/Phenalene	0.7	NA***
12.03	166	C13H10	Fluorene/Phenalene	0.2	NA*

13.49	178	C14H10	Phenanthrene	2.0	0.3
13.60	178	C14H10	Anthracene	1.0	0.3
14.92	190	C15H10	4,5-Methylenephenanthrene	0.4	0.1
16.31	202	C16H10	Fluoranthene	0.4	0.1
16.82	202	C16H10	Pyrene	0.7	0.2

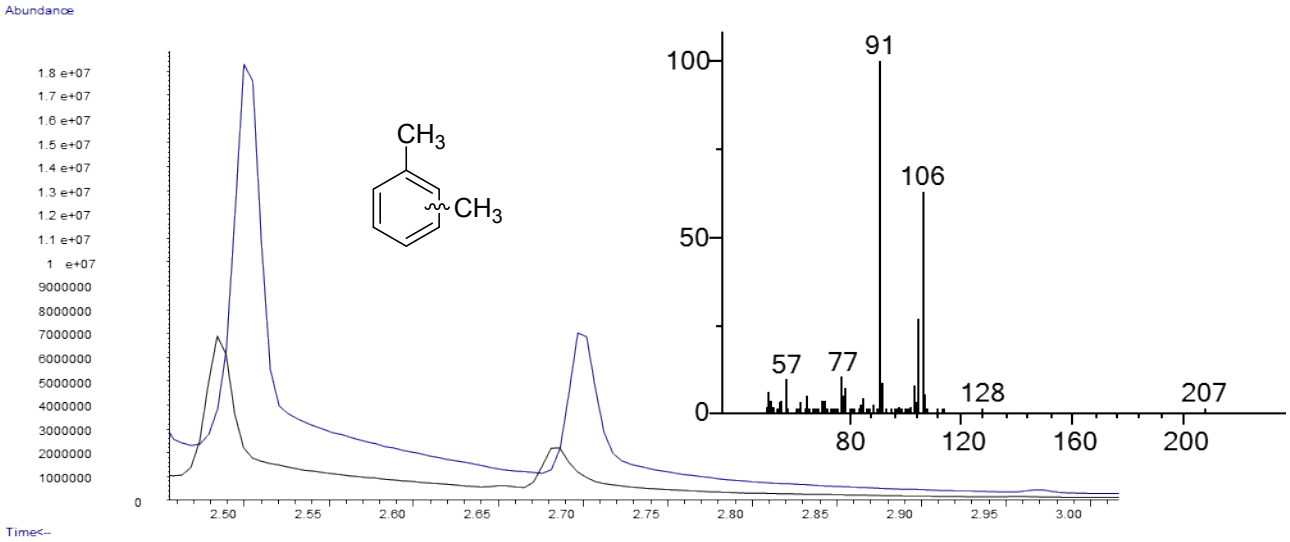
Alternatively, you can use RSIM data and compare "with" and "no" values (with/out 3 dilution factor) because it would allow you more selective comparison. However, you should not use normalization factors across different materials due to variability in molecular ion abundance. This results show that benzene derivatives found in both samples in same amounts, while higher aromatics are prevailing in "with" sample.

RT (min)	Mw (m/z)	Mol. formula	Substance	RSIM ratio with/no
2.4-2.7	106	C8H10	dimethyl benzenes	1.1
3.2-4.1	120	C9H12	trimetyl benzenes	0.8
4.3	116	C9H8	Indene	143.0
6.14	128	C10H8	Naphthalene	147.3
9.36	154	C12H10	Biphenyl/2-Vinylnaphthalene	132.9
9.64	152	C12H8	Acenaphthylene	178.5
10.09	154	C12H10	Acenaphthene/2-Vinylnaphthalene	19.3
11.26	166	C13H10	Fluorene/Phenalene	36.1
12.03	166	C13H10	Fluorene/Phenalene	>150
13.49	178	C14H10	Phenanthrene	25.7
13.6	178	C14H10	Anthracene	72.7
14.92	190	C15H10	4,5-Methylenephenanthrene	19.8
16.31	202	C16H10	Fluoranthene	13.2
16.82	202	C16H10	Pyrene	11.4

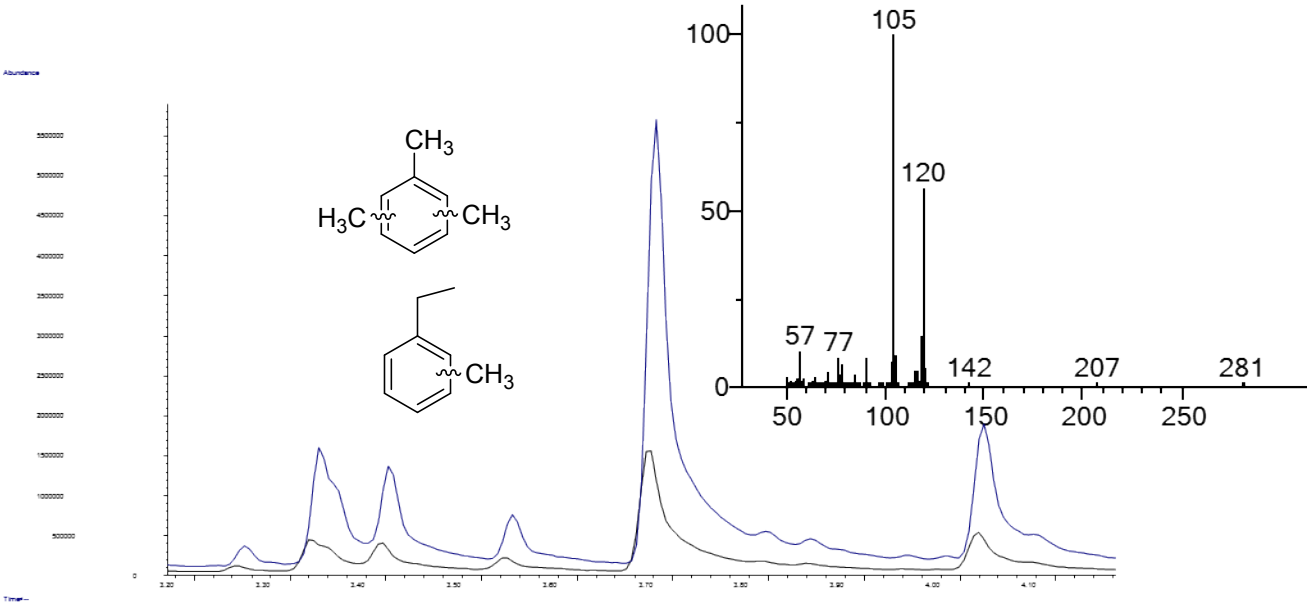
Figures:

All figures include TIC or RSIM of relevant substance and mass spectra used for molecular identification. In all figures blue lines - "NO" / black – "WITH".

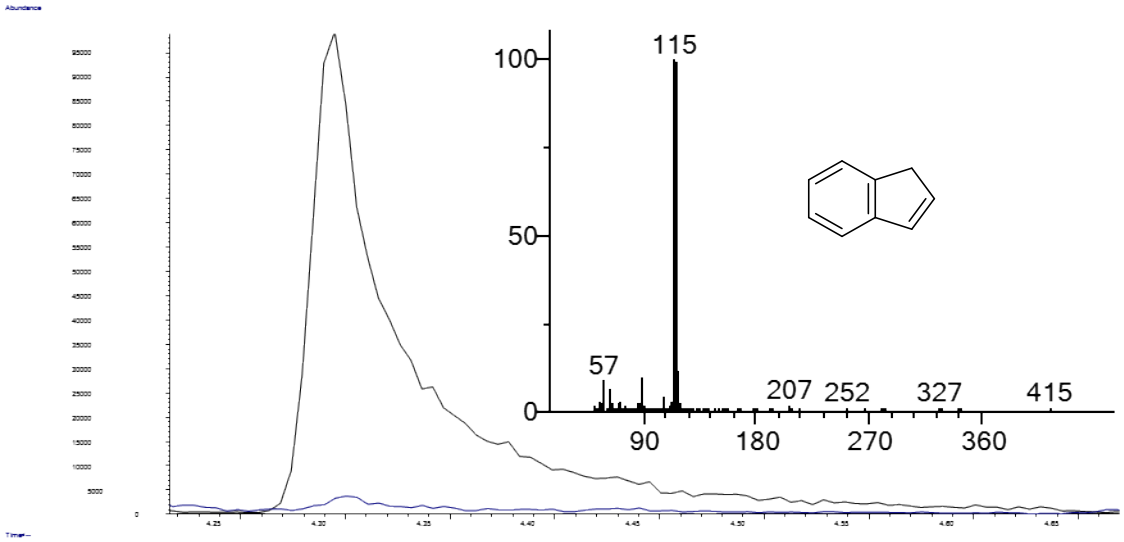
Please notice that some materials are TIC and some RSIM. Let me know if you need additional variations.



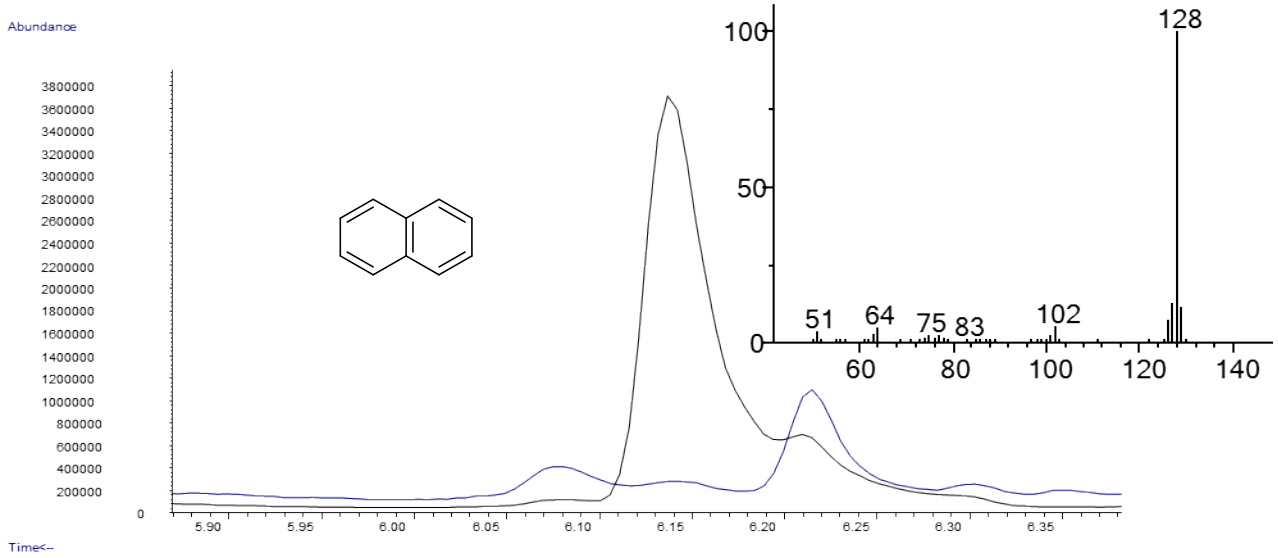
TIC RT: 2.4-2.7 min. C8H10 - dimethyl benzenes



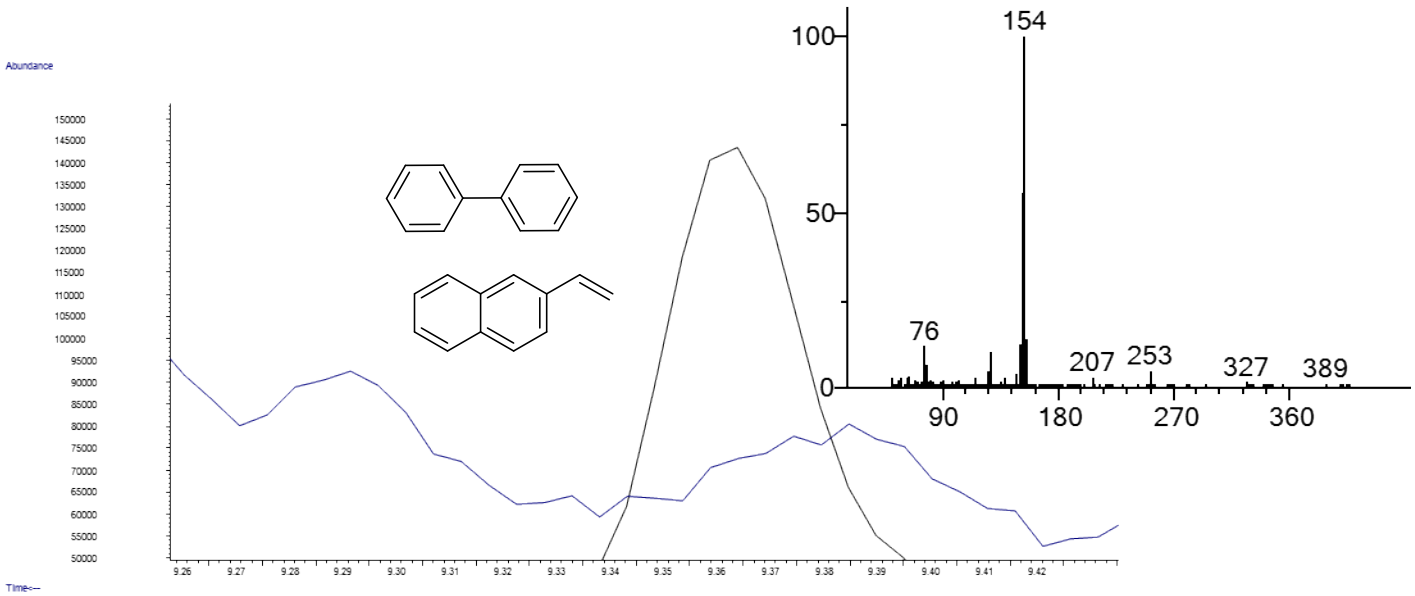
TIC RT: 3.2-4.1 min. C9H12 - trimethyl benzenes / Ethyl methyl benzenes



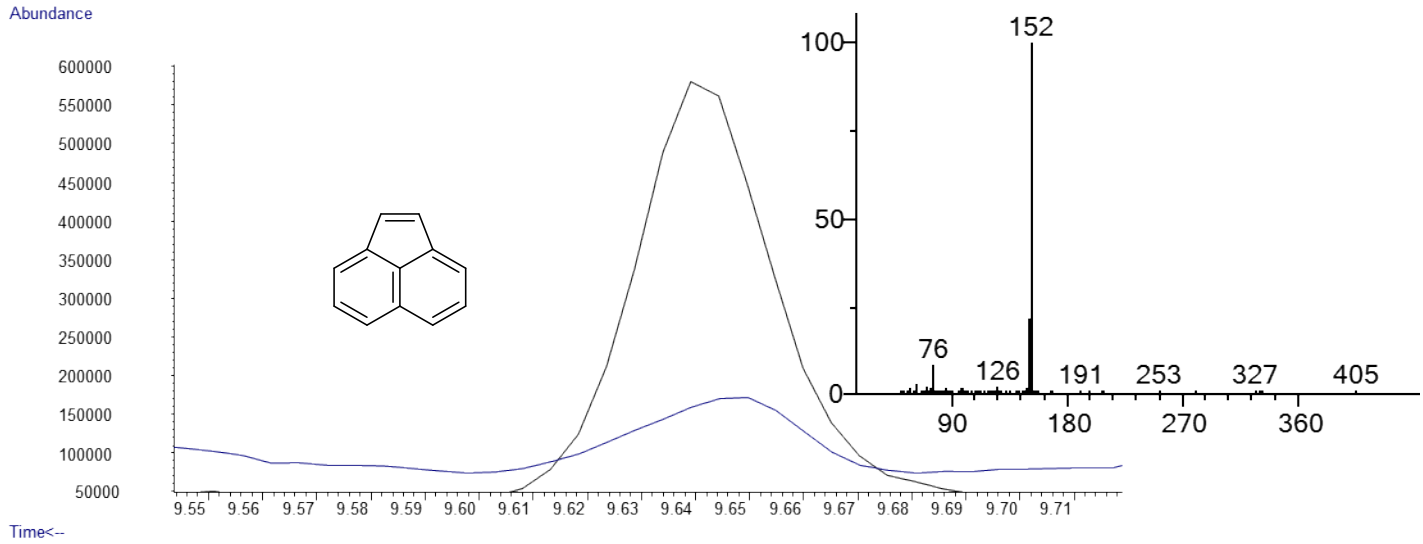
RSIM RT: 4.30 min. C9H8 - Indene



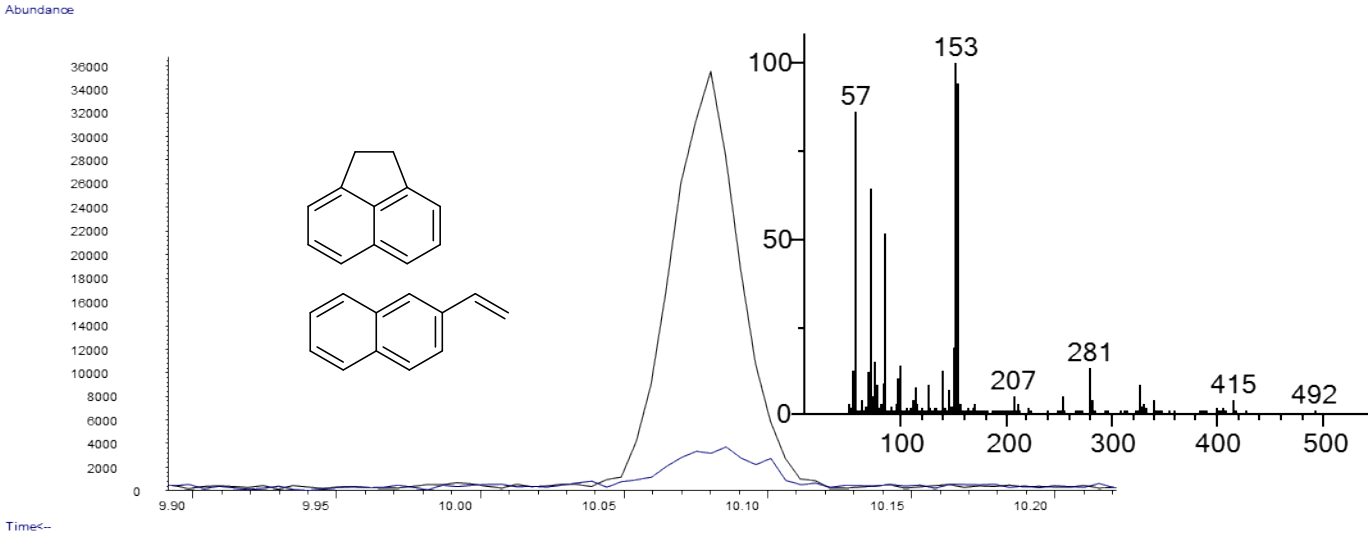
TIC RT: RT: 6.14 min. C10H8 – Naphthalene



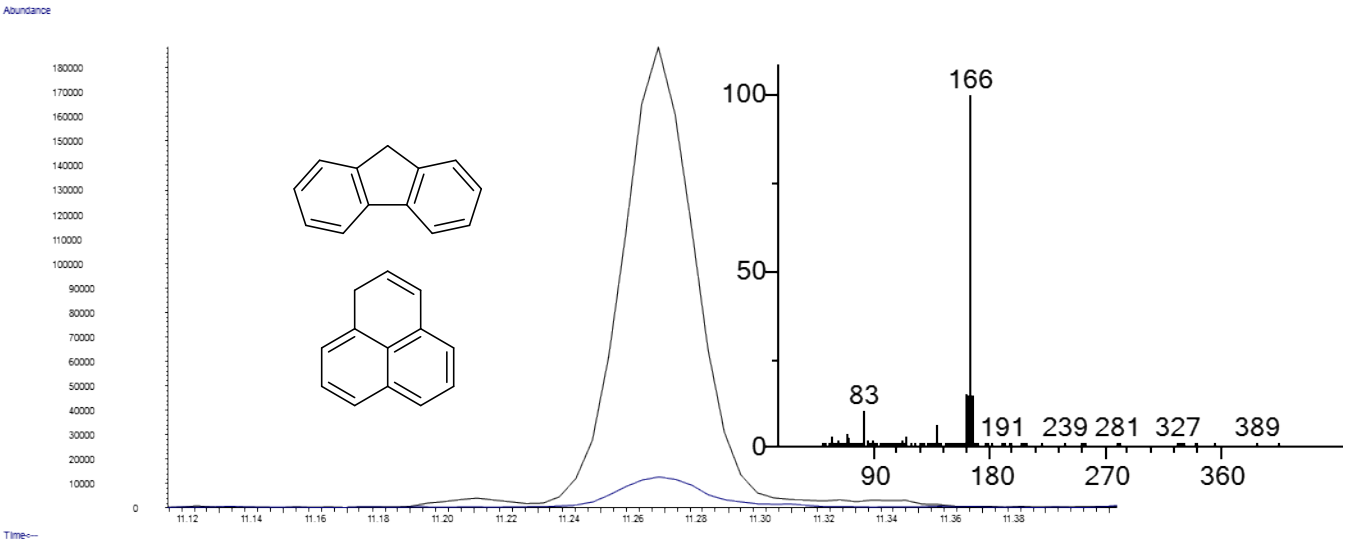
TIC RT: RT: 9.36 min. C12H10 – Biphenyl/ 2-Vinylnaphthalene



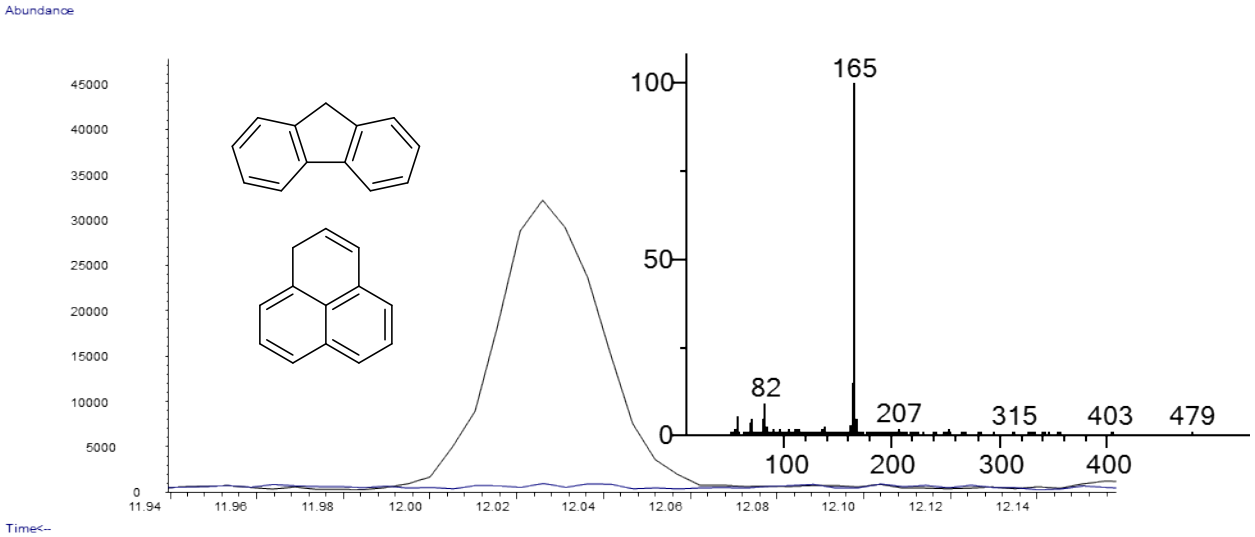
TIC RT: 9.64 min. C12H8 – Acenaphthylene



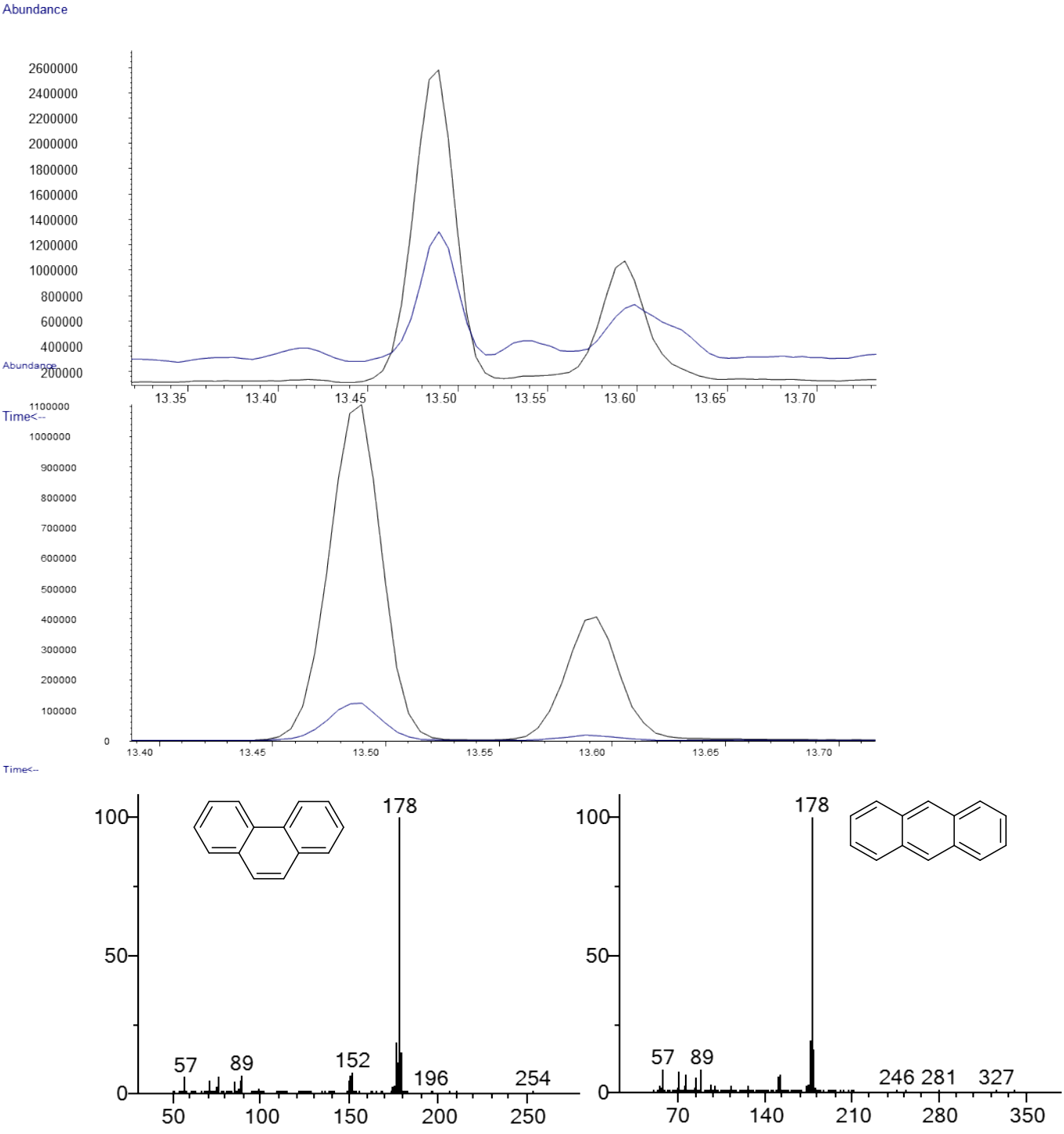
RSIM RT: 10.09 min. C12H10 – Acenaphthene / 2-Vinylnaphthalene



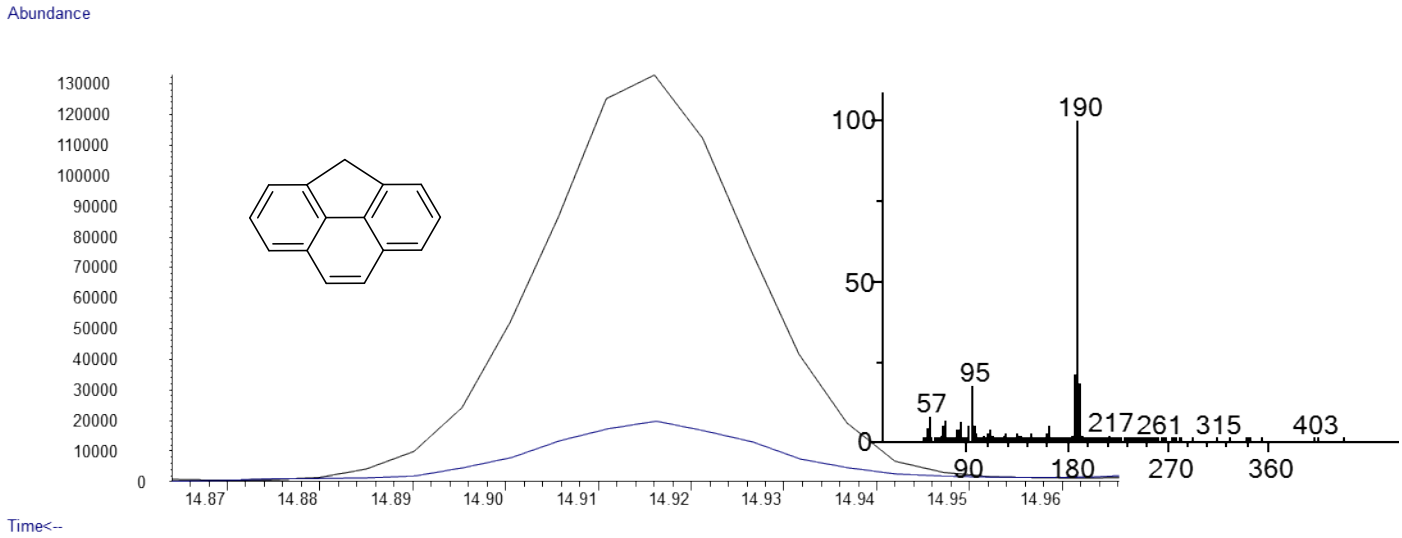
RSIM RT: 11.26 min. C13H10 - Fluorene/Phenalene



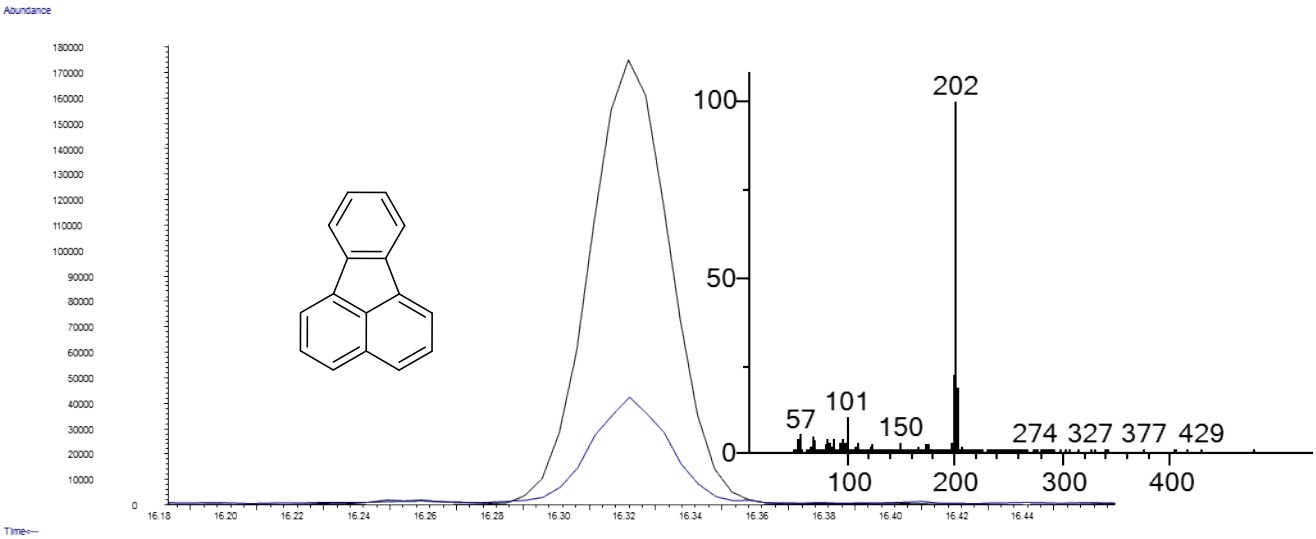
RSIM RT: RT: 12.03 min. C13H10 - Fluorene/Phenalene



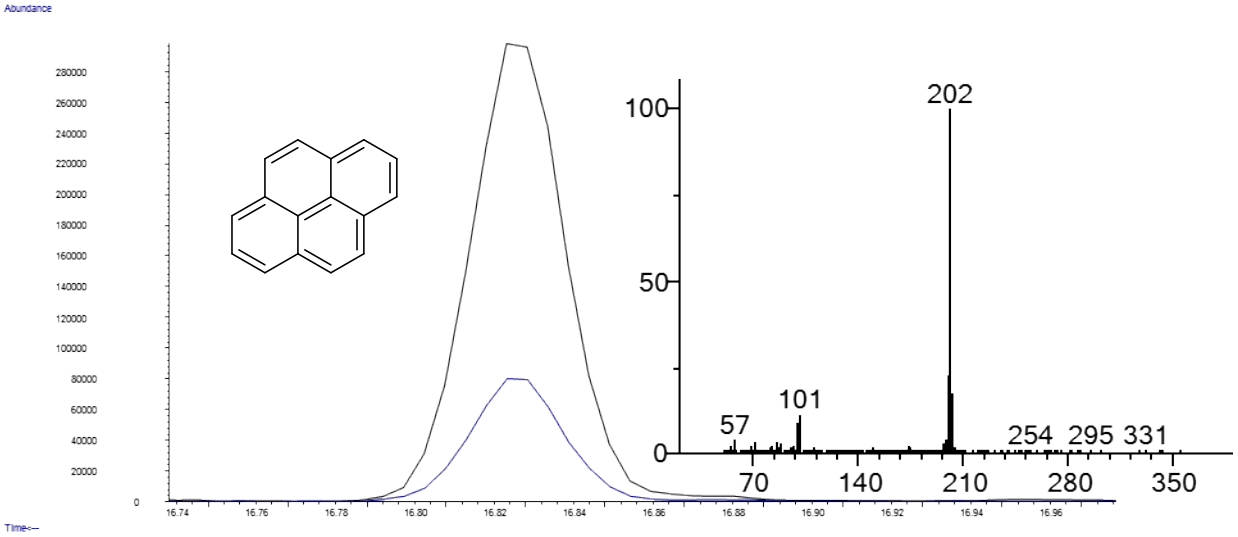
TIC (top) & RSIM (bottom) of C14H10: Phenanthrene (RT 13.49 min) and Anthracene (RT 13.60 min)



RSIM RT: 14.92min. C15H10 - 4,5-Methylenephenanthrene



RSIM RT: 16.31 min. C16H10 – Fluoranthene



RSIM RT: 16.82 min. C16H10 - Pyrene