COMPLEX CARBOHYDRATE RESEARCH CENTER

ANALYTICAL SERVICE REPORT

D 04/05/2012

Y

P HPLC OF 7 samples

Sa PEC-NCA,PEC-NCB,PEC-NCC,NCDCA,PHSM,PH12g, NCPropellent

C [redacted]

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S To be invoiced

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The aim of this experiment was to find some nitro compounds in the samples by comparing it with the standard provided (NC Propellant).

Analytical method using HPLC

High-performance liquid chromatography analysis was conducted using C-18 column (250x4.6mm). The mobile phase was water and methanol at a flow rate of 0.8 mL/min. Detection was performed by UV at 240nm. NC Propellant was used as a standard for the analysis of rest of the samples. NC Propellant was weight and diluted in acetone (1mg/ml). It was then evaporated and mixed with methanol and filtered. Only the compounds that was soluble in methanol was used in the analysis since it was only completely soluble in acetone and was precipitating out with eluting solvent.

The crude samples were diluted 5 times with 60% methanol and 10ul was injected in to the C-18 column.

The result below shows the comparison between standard and the samples.

NC Propellant, standard gave a single peak around 32.4 min and peak around the same retention time was found in PEC-NCC, PEC-NCA and NCDCA.
A blank run of acetone was run with same elutant and showed acetone peak at 4.9 min.

PHSM and PM12G gave only one single peak in the entire sample at 3.25 min.
PEC-NCB gave only one single peak at 3.499 min.
PEC-NCA and NCDCA showed many other peaks in the samples which did not correspond to that of NC Propellant.

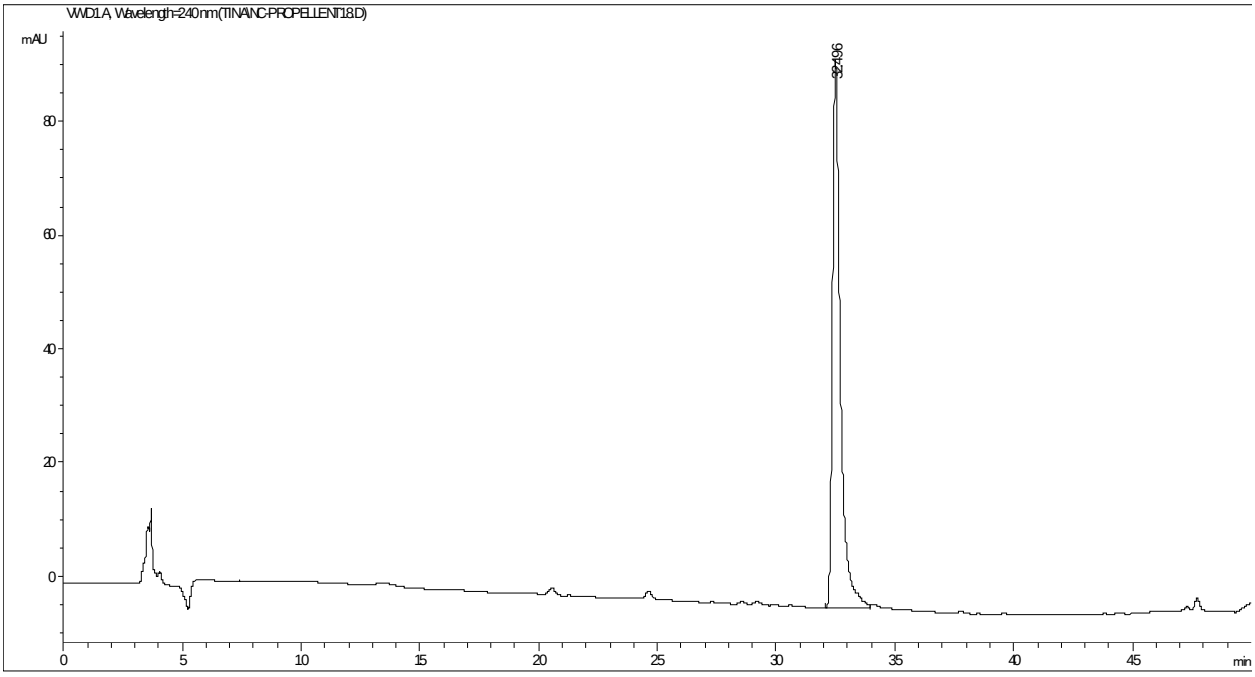

Fig 1. HPLC Chromatogram for NC Propellant

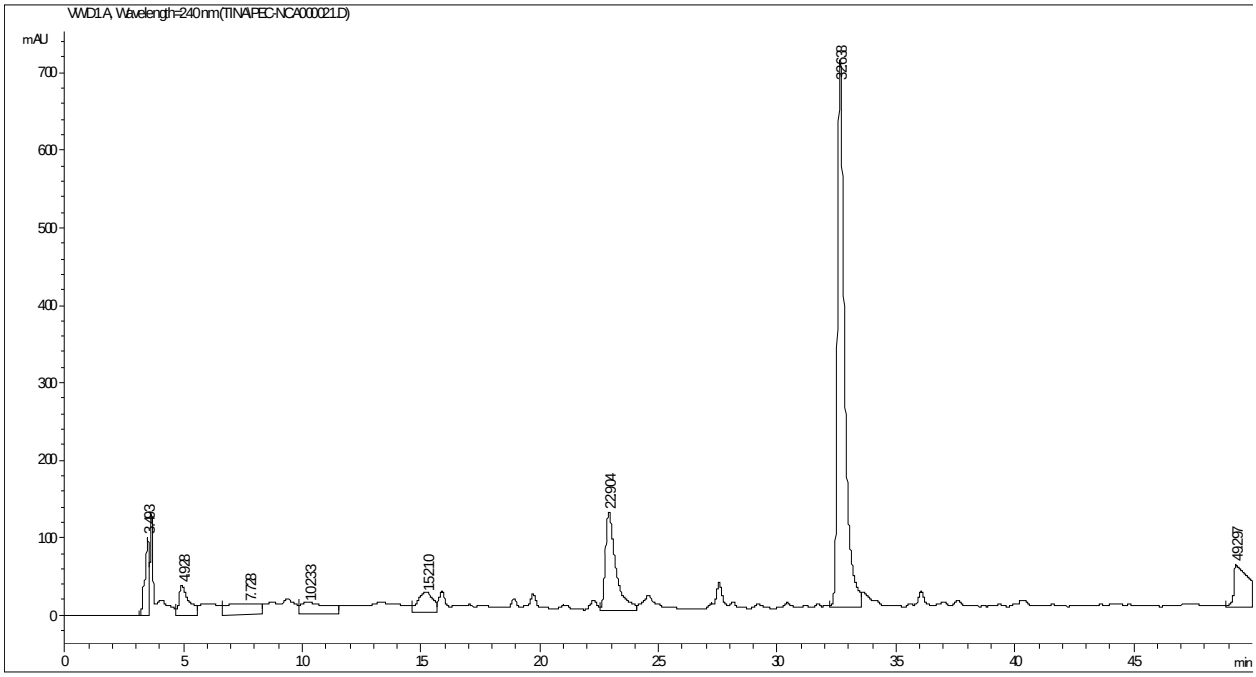

Fig 2. Chromatogram for PEC-NCA

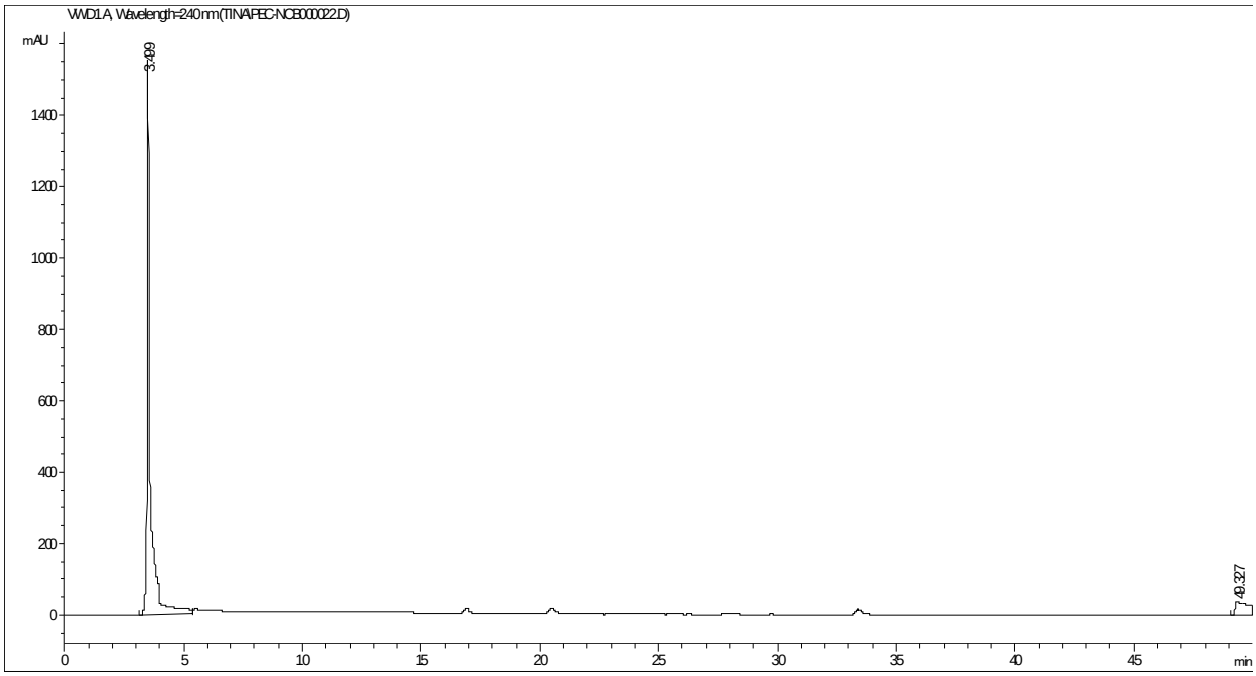

Fig 3. Chromatogram for PEC-NCB

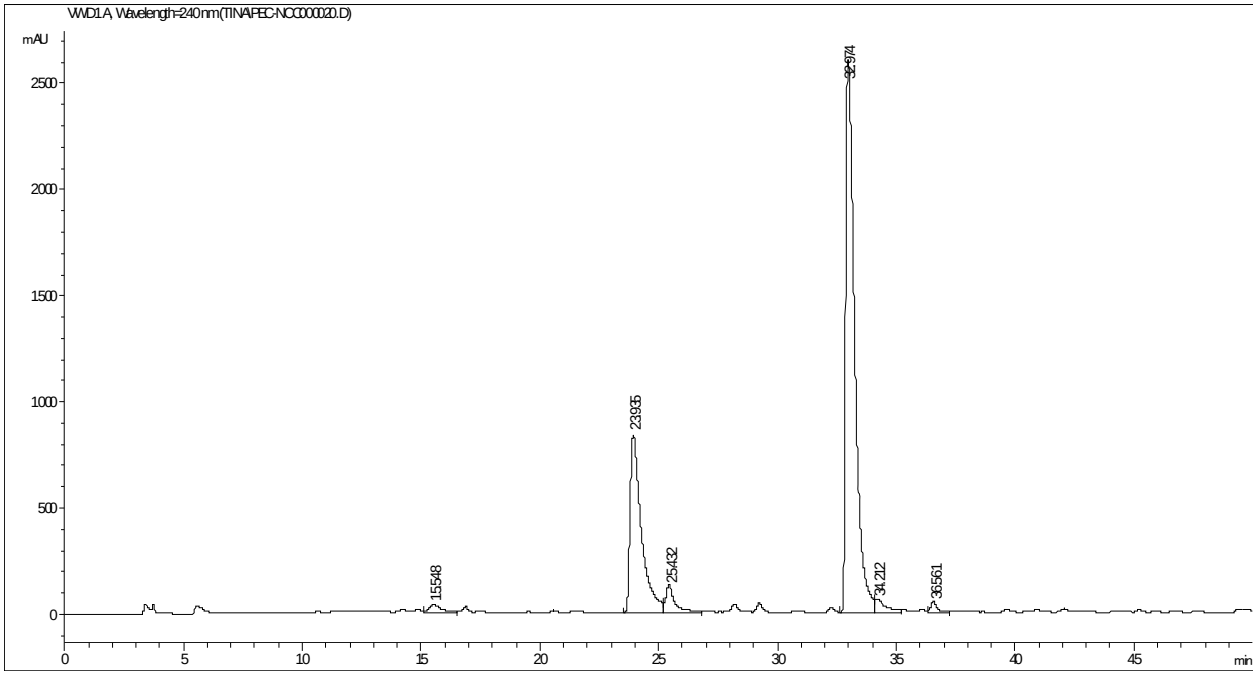

Fig 4. Chromatogram of PEC-NCC

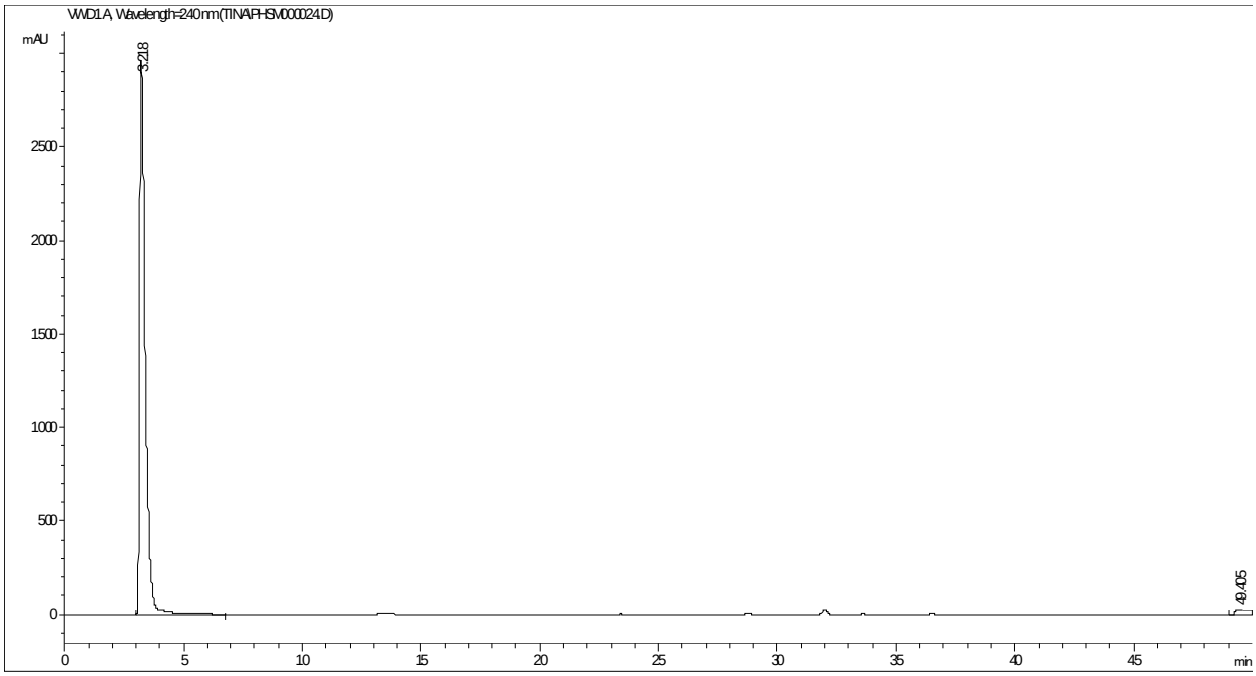

Fig 5. Chromatogram of PHSM.

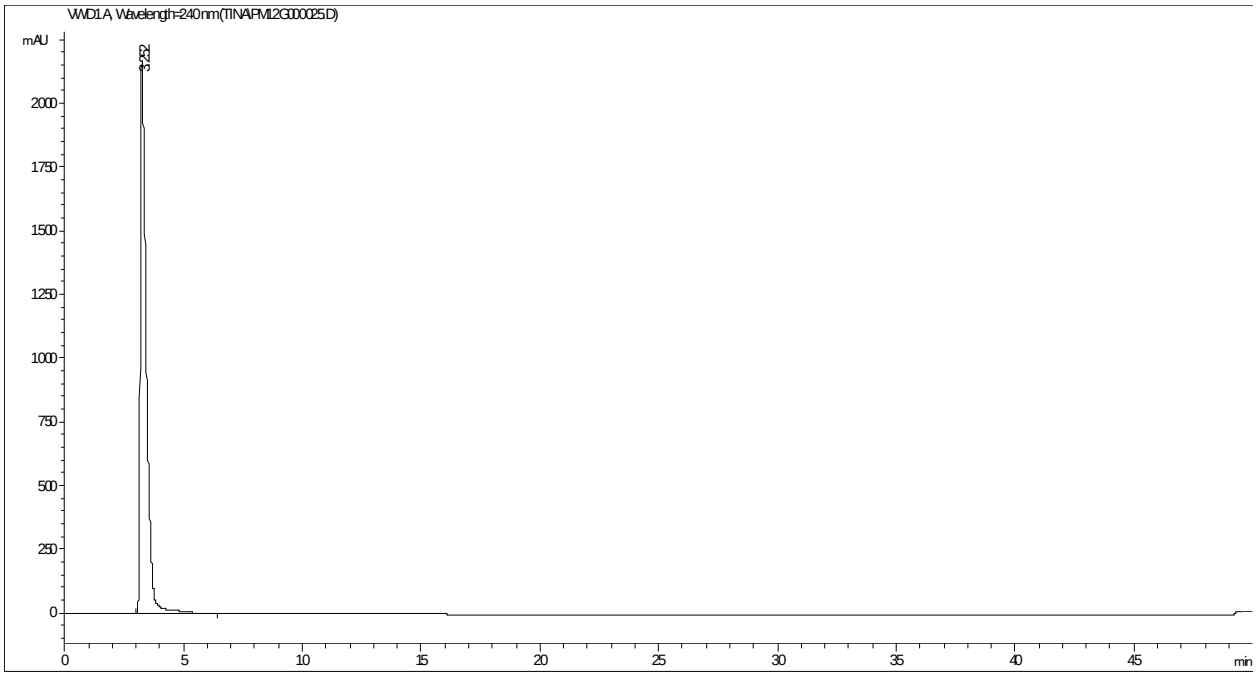

Fig 6. Chromatogram of PM12G

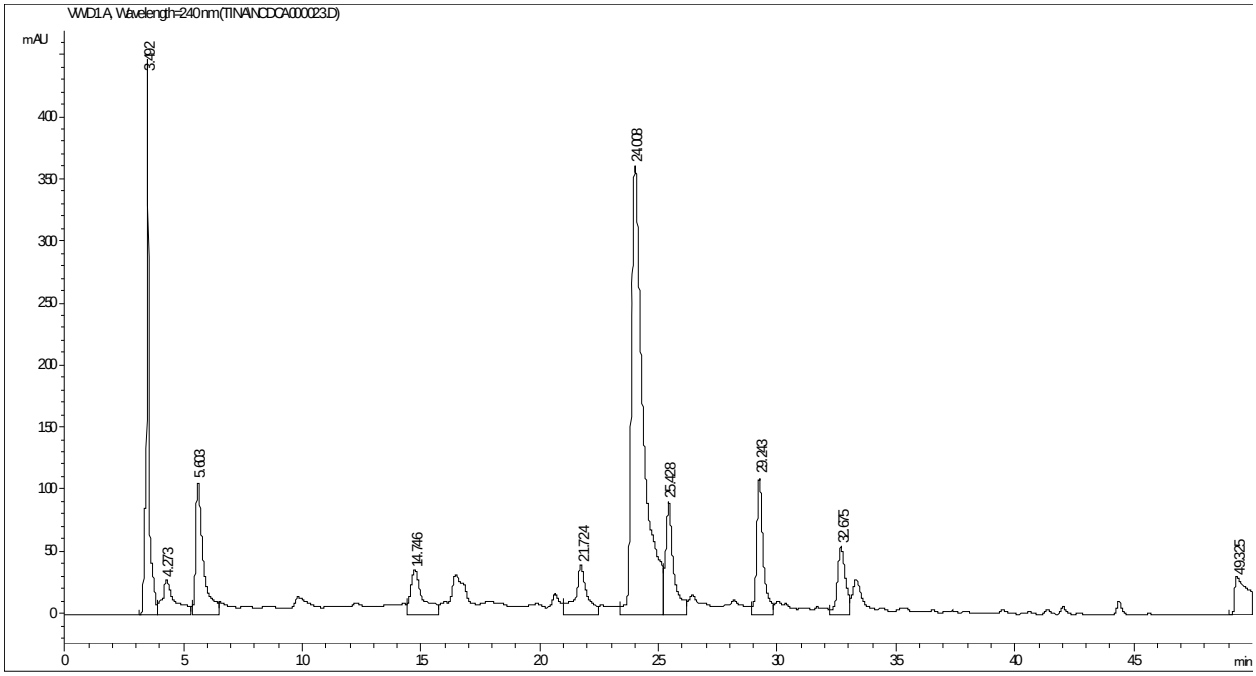

Fig 7. Chromatogram of NCDCA

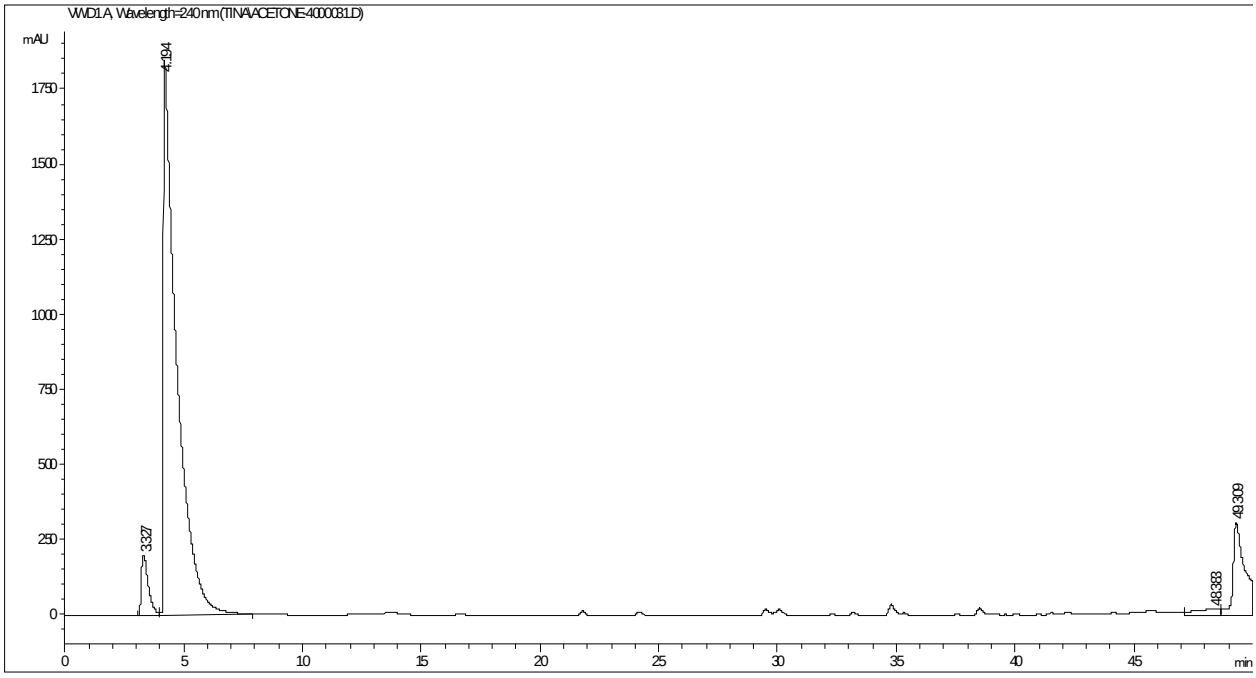

Figure 8. Chromatogram of Acetone

NCDCA

#	Time	Area	Height	Width	Area%	Symmetry
1	3.492	3208.8	449.6	0.0978	11.092	0.895
2	4.273	1084.9	28.2	0.5257	3.75	0.434
3	5.603	2393.2	106.5	0.3233	8.273	0.365
4	14.746	1321.8	35.7	0.5278	4.569	0.506
5	21.724	1383.9	40.8	0.4526	4.784	0.952
6	24.008	12758.3	361.4	0.5051	44.103	0.393
7	25.428	2170.2	90.5	0.335	7.502	0.541
8	29.243	2175.3	110.4	0.2896	7.52	0.637
9	32.675	1313.3	54.6	0.3548	4.54	0.741
10	49.325	1118.6	31.2	0.4717	3.867	0.166

NC PROPELLANT

#	Time	Area	Height	Width	Area%	Symmetry
1	32.496	2240.7	96.6	0.342	100	0.555

PEC-NCA

#	Time	Area	Height	Width	Area%	Symmetry
1	3.493	1251	100.3	0.1731	4.626	1.913
2	4.928	1134	38.4	0.4187	4.193	0.335
3	7.728	1388.4	14.7	1.1779	5.133	2.135
4	10.233	1221.2	15.8	0.9952	4.515	0.302
5	15.21	1164.1	26.8	0.6575	4.304	1.19
6	22.904	3884.5	126.5	0.4443	14.363	0.449
7	32.638	14867.9	707.6	0.3107	54.973	0.52
8	49.297	2134.9	54.8	0.4892	7.894	0.177

PEC-NCC

#	Time	Area	Height	Width	Area%	Symmetry
1	15.548	1737.2	41.3	0.5915	1.621	0.533
2	23.935	28345.3	844.6	0.4847	26.458	0.365
3	25.432	4092.7	139.4	0.4007	3.82	0.424
4	32.974	69427.1	2612.5	0.4105	64.804	0.344
5	34.212	2268.2	66.9	0.4497	2.117	0.191
6	36.561	1263.2	56.5	0.316	1.179	0.587

PHSM

#	Time	Area	Height	Width	Area%	Symmetry
1	3.218	48974.1	2972.4	0.2257	97.372	0.376
2	49.405	1321.6	32.6	0.5614	2.628	0.321

PM12G

#	Time	Area	Height	Width	Area%	Symmetry
1	3.252	38354.6	2174.8	0.2392	100	0.355

PEC-NCB

#	Time	Area	Height	Width	Area%	Symmetry
1	3.414	118170.9	4524.8	0.3309	96.72	0.958
2	3.883	2122.2	182.6	0.1589	1.737	0.167
3	49.375	1885.3	56.7	0.43	1.543	0.216

NC Propellant peak around 32.4 min and peak around the same retention time in PEC-NCC, PEC-NCA and NCDCA might be same.

I ran PEC-NCB in 5 times dilution and concentrate solution and could not find any peak corresponding to NC Propellant. This might be interesting since PEC-NCA and PEC-NCB samples are supposed to be similar.

The integration results are provided above.

If you have any further questions please contact Dr. Parastoo Azadi at 706-583-0629 or azadi@ccrc.uga.edu.