

GC-NPD
method

Method Information

NPD.M method by GC-NPD/EPC
by Steven C. Moser

ODAFF Pesticide Lab Residue Section

Method Change History

Operator	Date	Change Information
Steven C. Moser	9/8/2010 7:36:34 AM	This method was created at 9/8/2010 7:36:34 AM and based on method C:\HPCHEM\1\METHODS\DEF_GC.M
Steven C. Moser	9/8/2010 7:36:35 AM	
Steven C. Moser	9/8/2010 7:52:21 AM	
Steven C. Moser	9/8/2010 7:53:22 AM	
Steven C. Moser	9/8/2010 7:56:02 AM	
Steven C. Moser	9/8/2010 7:59:35 AM	
Steven C. Moser	9/8/2010 8:13:48 AM	
Steven C. Moser	9/8/2010 8:14:28 AM	
Steven C. Moser	9/8/2010 10:01:39 AM	added calibration curve
Steven C. Moser	9/8/2010 10:53:19 AM	Updated RT
Steven C. Moser	9/8/2010 10:56:31 AM	This method was created at 9/8/2010 10:56:31 AM and based on method C:\HPCHEM\1\METHODS\NPDSCM2.M
Steven C. Moser	9/8/2010 10:56:32 AM	
Keith Keese	9/21/2010 10:32:02 AM	Updated Sterilant RTs.
Keith Keese	9/21/2010 1:08:14 PM	Adj. RTs.
Keith Keese	9/21/2010 2:24:02 PM	
Keith Keese	9/24/2010 12:30:34 PM	Adj. slope sensitivity.
Keith Keese	10/20/2010 2:50:54 PM	Adj. slope sensitivity from 30 to 10.
Keith Keese	10/20/2010 3:40:28 PM	Disabled adv. baseline int.
Keith Keese	10/20/2010 4:06:14 PM	adjusted makeup flow+column from 10 to 15
Keith Keese	10/20/2010 4:10:32 PM	change back to 10.00 change bead
Keith Keese	10/28/2010 7:16:04 AM	
Keith Keese	10/29/2010 10:15:11 AM	Adj. RT labels.
Keith Keese	1/26/2012 9:23:20 AM	This method was created at 1/26/2012 9:23:20 AM and based on method C:\HPCHEM\1\DATA\2011\03-2011\031011N1\ACETONE.D\RUN.M
Keith Keese	1/26/2012 9:23:28 AM	
Keith Keese	2/8/2012 9:02:50 AM	Adj. RTs
Keith Keese	2/13/2012 8:15:31 AM	
Keith Keese	4/17/2012 3:19:54 PM	This method was created at 4/17/2012 3:19:54 PM and based on method C:\HPCHEM\1\METHODS\NPDSCM.M
Keith Keese	4/17/2012 3:19:56 PM	
Keith Keese	4/18/2012 12:22:52 PM	
Keith Keese	4/18/2012 12:23:02 PM	This method was created at 4/18/2012 12:23:02 PM and based on method

Method Change History

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Keith Keesee	4/18/2012 12:23:22 PM	C:\HPCHEM\1\METHODS\NPD-H2.M Changed Gases to elim Helium
Steven C. Moser	4/19/2012 3:44:48 PM	
Steven C. Moser	4/20/2012 9:55:17 AM	
Steven C. Moser	4/20/2012 10:16:24 AM	adjust offset
Steven C. Moser	4/20/2012 10:22:16 AM	
Steven C. Moser	4/20/2012 11:07:18 AM	
Steven C. Moser	4/20/2012 12:00:06 PM	
Steven C. Moser	4/20/2012 12:46:31 PM	
Steven C. Moser	4/20/2012 2:19:34 PM	
Steven C. Moser	4/20/2012 2:20:27 PM	
Steven C. Moser	4/20/2012 3:13:13 PM	
Steven C. Moser	4/24/2012 11:31:57 AM	
Keith Keesee	4/25/2012 1:29:50 PM	
Keith Keesee	5/2/2012 9:35:57 AM	
Keith Keesee	5/10/2012 10:45:07 AM	Adj. RTs
Keith Keesee	5/10/2012 10:46:25 AM	
Jennifer Bledsoe	6/7/2013 8:41:24 AM	This method was created at 6/7/2013 8:41:24 AM and based on method C:\HPCHEM\1\DATA\011713N1\13-7 04.D\RUN.M
Jennifer Bledsoe	6/7/2013 8:41:59 AM	Pulled old copy of method to check for corruption
Jennifer Bledsoe	6/7/2013 8:50:25 AM	This method was created at 6/7/2013 8:50:25 AM and based on method C:\HPCHEM\1\METHODS\NPD.M
Jennifer Bledsoe	6/7/2013 8:51:03 AM	Put adjust offset back to 45 from 40, did constant column+ flow instead of just strait makeup flow.
Jennifer Bledsoe	6/19/2013 3:34:05 PM	
Jennifer Bledsoe	6/20/2013 8:02:41 AM	Update RT after column clip
Jennifer Bledsoe	6/20/2013 8:05:27 AM	
Jennifer Bledsoe	6/20/2013 8:06:08 AM	This method was created at 6/20/2013 8:06:08 AM and based on method C:\HPCHEM\1\METHODS\NPD-JB.M
Jennifer Bledsoe	6/20/2013 8:06:12 AM	
Jennifer Bledsoe	6/21/2013 8:41:59 AM	Updated RTs
Jennifer Bledsoe	6/26/2013 2:31:15 PM	Added Cyprodinil to Cal
Jennifer Bledsoe	6/26/2013 2:50:09 PM	
Jennifer Bledsoe	6/28/2013 9:07:29 AM	
Jennifer Bledsoe	7/9/2013 8:15:26 AM	Updated RT
Jennifer Bledsoe	7/31/2013 7:13:17 AM	
Jennifer Bledsoe	8/12/2013 8:45:22 AM	
Jennifer Bledsoe	8/23/2013 1:56:22 PM	
Kyle Baker	10/21/2013 9:49:37 AM	
Kyle Baker	11/5/2013 10:24:02 AM	
Kyle Baker	12/2/2013 12:40:40 PM	
Kyle Baker	3/21/2014 2:19:46 PM	
Kyle Baker	4/21/2014 2:30:32 PM	
Kyle Baker	4/22/2014 12:04:08 PM	
Kyle Baker	5/7/2014 5:17:31 PM	
Laura Esquivel	5/9/2014 3:44:31 PM	
Kyle Baker	5/16/2014 12:40:24 PM	

Method Change History

Operator	Date	Change Information
Kyle Baker	6/6/2014 8:36:31 AM	
Jennifer Busey	6/27/2014 12:49:06 PM	
Jennifer Busey	7/2/2014 2:36:57 PM	
Jennifer Busey	8/19/2014 8:18:53 AM	
Jennifer Busey	8/20/2014 8:23:20 AM	
Jennifer Busey	8/21/2014 8:44:54 AM	
Jennifer Busey	8/25/2014 9:54:22 AM	Added S-Metolachlor
Jennifer Busey	8/25/2014 10:20:44 AM	Entered Diazinon
Jennifer Busey	8/26/2014 7:41:30 AM	
Yvette Turner	9/18/2014 9:30:39 AM	
Yvette Turner	11/5/2014 9:02:49 AM	Updating Retention times
Yvette Turner	12/15/2014 4:09:03 PM	updated retention times
Yvette Turner	12/15/2014 4:09:59 PM	
Yvette Turner	12/15/2014 4:20:24 PM	
Yvette Turner	12/15/2014 4:40:08 PM	
Yvette Turner	12/15/2014 4:41:48 PM	
Yvette Turner	2/26/2015 7:47:55 AM	Updating retention times
Yvette Turner	2/26/2015 7:55:02 AM	
Yvette Turner	3/10/2015 12:06:54 PM	
Yvette Turner	3/11/2015 6:49:36 AM	
Yvette Turner	5/7/2015 8:37:52 AM	Updated retention times
Yvette Turner	5/7/2015 9:39:59 AM	Updating Retention times
Yvette Turner	5/11/2015 8:06:29 AM	Updated retention times
Yvette Turner	5/11/2015 3:59:07 PM	updated retention times

Run Time Checklist

Pre-Run Cmd/Macro: off

Data Acquisition: on

Standard Data Analysis: on

Customized Data Analysis: off

Save GLP Data: off

Post-Run Cmd/Macro: off

Save Method with Data: skipped - no ACQ running

Injection Source and Location

Injection Source: GC Injector

Injection Location: Back

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6890 GC METHOD
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OVEN

Initial temp: 60 'C (On) Maximum temp: 350 'C
Initial time: 0.00 min Equilibration time: 0.10 min
Ramps:
Rate Final temp Final time
1 20.00 300 5.00
2 0.0(Off)
Post temp: 60 'C
Post time: 0.00 min
Run time: 17.00 min

FRONT INLET (SPLIT/SPLITLESS)

Mode: Splitless
Initial temp: 230 'C (On)
Pressure: 3.78 psi (On)
Purge flow: 0.0 mL/min
Purge time: 0.00 min
Total flow: 7.8 mL/min
Gas saver: Off
Gas type: Hydrogen

BACK INLET (SPLIT/SPLITLESS)

Mode: Splitless
Initial temp: 210 'C (On)
Pressure: 4.98 psi (On)
Purge flow: 15.0 mL/min
Purge time: 0.50 min
Total flow: 24.4 mL/min
Gas saver: Off
Gas type: Hydrogen

COLUMN 1

Capillary Column
Model Number: Restek 13324
Rxi-MS
Max temperature: 350 'C
Nominal length: 30.0 m
Nominal diameter: 320.00 um
Nominal film thickness: 0.25 um
Mode: constant flow
Initial flow: 1.4 mL/min
Nominal init pressure: 3.80 psi
Average velocity: 29 cm/sec
Inlet: Front Inlet
Outlet: Front Detector
Outlet pressure: ambient

COLUMN 2

Capillary Column
Model Number: Restek 13324
Rxi-1MS, 30m x 0.32mm x 0.25um
Max temperature: 350 'C
Nominal length: 30.0 m
Nominal diameter: 320.00 um
Nominal film thickness: 0.25 um
Mode: constant flow
Initial flow: 2.0 mL/min
Nominal init pressure: 5.12 psi
Average velocity: 39 cm/sec
Inlet: Back Inlet
Outlet: Back Detector
Outlet pressure: ambient

FRONT DETECTOR (µECD)

Temperature: 325 'C (On)
Mode: Constant makeup flow
Makeup flow: 60.0 mL/min (On)
Makeup Gas Type: Nitrogen
Electrometer: On

BACK DETECTOR (NPD)

Temperature: 325 'C (On)
Hydrogen flow: 1.5 mL/min (On)
Air flow: 60.0 mL/min (On)
Mode: Constant column+makeup flow
Combined flow: 8.5 mL/min
Makeup flow: On
Makeup Gas Type: Nitrogen
Adjust offset: 45.00
Electrometer: On
Bead: On
Equilibration time: 20.00

SIGNAL 1

Data rate: 20 Hz
Type: front detector
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

SIGNAL 2

Data rate: 50 Hz
Type: back detector
Save Data: On
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1

Derive from front detector

COLUMN COMP 2

Derive from back detector

POST RUN
Post Time: 0.00 min

TIME TABLE	Specifier	Parameter & Setpoint
Time		

GC Injector

Front Injector:
No parameters specified

Back Injector:

Sample Washes	1
Sample Pumps	2
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
PostInj Solvent A Washes	3
PostInj Solvent B Washes	1
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

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 Integration Events
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Results will be produced with the enhanced integrator.

 Default Integration Event Table "Event"

Event	Value	Time
Initial Slope Sensitivity	1.000	Initial
Initial Peak Width	0.040	Initial
Initial Area Reject	1.000	Initial
Initial Height Reject	1.700	Initial
Initial Shoulders	OFF	Initial

 Detector Default Integration Event Table "Event_TCD"

Event	Value	Time
Initial Slope Sensitivity	100.000	Initial
Initial Peak Width	0.040	Initial
Initial Area Reject	1.000	Initial
Initial Height Reject	1.000	Initial
Initial Shoulders	OFF	Initial

 Detector Default Integration Event Table "Event_ADC"

Event	Value	Time
Initial Slope Sensitivity	20.000	Initial
Initial Peak Width	0.040	Initial
Initial Area Reject	1.000	Initial
Initial Height Reject	1.000	Initial
Initial Shoulders	OFF	Initial

 Detector Default Integration Event Table "Event_FID"

Event	Value	Time
Initial Slope Sensitivity	50.000	Initial
Initial Peak Width	0.040	Initial
Initial Area Reject	1.000	Initial
Initial Height Reject	1.000	Initial
Initial Shoulders	OFF	Initial

 Detector Default Integration Event Table "Event_ECD"

Event	Value	Time
Initial Slope Sensitivity	100.000	Initial
Initial Peak Width	0.080	Initial
Initial Area Reject	1.000	Initial
Initial Height Reject	1.000	Initial
Initial Shoulders	OFF	Initial

 Detector Default Integration Event Table "Event_FPD"

Event	Value	Time
Initial Slope Sensitivity	50.000	Initial
Initial Peak Width	0.040	Initial
Initial Area Reject	1.000	Initial
Initial Height Reject	1.000	Initial
Initial Shoulders	OFF	Initial

 Detector Default Integration Event Table "Event_uECD"

Event	Value	Time
Initial Slope Sensitivity	500.000	Initial
Initial Peak Width	0.080	Initial
Initial Area Reject	1.000	Initial
Initial Height Reject	1.000	Initial
Initial Shoulders	OFF	Initial

 Detector Default Integration Event Table "Event_NPD"

Event	Value	Time
Initial Slope Sensitivity	5.000	Initial
Initial Peak Width	0.050	Initial
Initial Area Reject	0.010	Initial
Initial Height Reject	0.010	Initial
Initial Shoulders	DROP	Initial
Integration	OFF	0.000
Integration	ON	2.500

Apply Manual Integration Events: No

Advanced Baseline : No

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 Calibration Table
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NPD

Calib. Data Modified : 5/11/2015 3:58:47 PM

Calculate : Height Percent

Rel. Reference Window : 0.500 %

Abs. Reference Window : 0.000 min

Rel. Non-ref. Window : 0.500 %

Abs. Non-ref. Window : 0.000 min

Use Multiplier & Dilution Factor with ISTDs

Uncalibrated Peaks : compound name not specified

Partial Calibration : Yes, identified peaks are recalibrated

Correct All Ret. Times: No, only for identified peaks

Curve Type : Linear

Origin : Included

Weight : Equal

Recalibration Settings:

Average Response : Average all calibrations

Average Retention Time: Floating Average New 75%

Calibration Report Options :

Printout of recalibrations within a sequence:
 Calibration Table after Recalibration
 Normal Report after Recalibration
 If the sequence is done with bracketing:
 Results of first cycle (ending previous bracket)

Signal 1: NPD2 B,

RetTime [min]	Lvl Sig	Amount [ppm]	Height	Amt/Height	Ref Grp	Name
5.030	1 1	1.00000	1.92978	5.18195e-1		Diuron
6.162	1 1	100.00000	77.26389	1.29427		Tebuthiuron
7.334	1 1	1.00000	1.00000	1.00000		Trifluralin
7.383	1 1	5.00000e-1	10.46079	4.77975e-2		Simazine
7.447	1 1	1.00000	2.66649	3.75025e-1		Prometon
7.459	1 1	5.00000e-1	10.72244	4.66312e-2		Atrazine
7.505	1 1	1.00000e-1	23.99158	4.16813e-3		Propazine
8.247	1 1	1.02000	14.51938	7.02509e-2		Acetochlor
8.339	1 1	2.00000	6.73656	2.96887e-1		Alachlor
8.348	1 1	1.00000	0.00000	0.00000		Metalaxyl
8.367	1 1	1.00000	12.56123	7.96100e-2		Bromacil
8.550	1 1	1.00000	1.67348	5.97558e-1		Prodiamine
8.676	1 1	1.00000	0.00000	0.00000		Parathion
8.713	1 1	1.00000e-1	0.00000	0.00000		Chlorpyrifos
8.807	1 1	2.00000	3.73602	5.35328e-1		Metolachlor
9.010	1 1	1.00000	17.48367	5.71962e-2		Pendimethalin
9.051	1 1	1.00000	12.46276	8.02390e-2		Cyprodinil
9.639	1 1	1.00000	1.00000	1.00000		Myclobutanil
9.723	1 1	1.00000	1.73312	5.76995e-1		Oxadiazon
12.408	1 1	1.00000e-1	95.66788	1.04528e-3		Esfenvalerate I
12.531	1 1	1.00000e-1	421.10040	2.37473e-4		EsfenvalerateII

More compound-specific settings:

Compound: Alachlor
 Time Window : From 8.319 min To 8.356 min

Compound: Metalaxyl
 Time Window : From 8.327 min To 8.365 min

Compound: Prodiamine
 Time Window : From 8.537 min To 8.570 min

5 Warnings or Errors :

- Warning : Overlapping peak time windows at 7.447 min, signal 1
- Warning : Overlapping peak time windows at 8.339 min, signal 1
- Warning : Overlapping peak time windows at 8.348 min, signal 1
- Warning : Overlapping peak time windows at 8.676 min, signal 1
- Warning : Overlapping peak time windows at 9.01 min, signal 1

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 Peak Sum Table
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No Entries in table
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