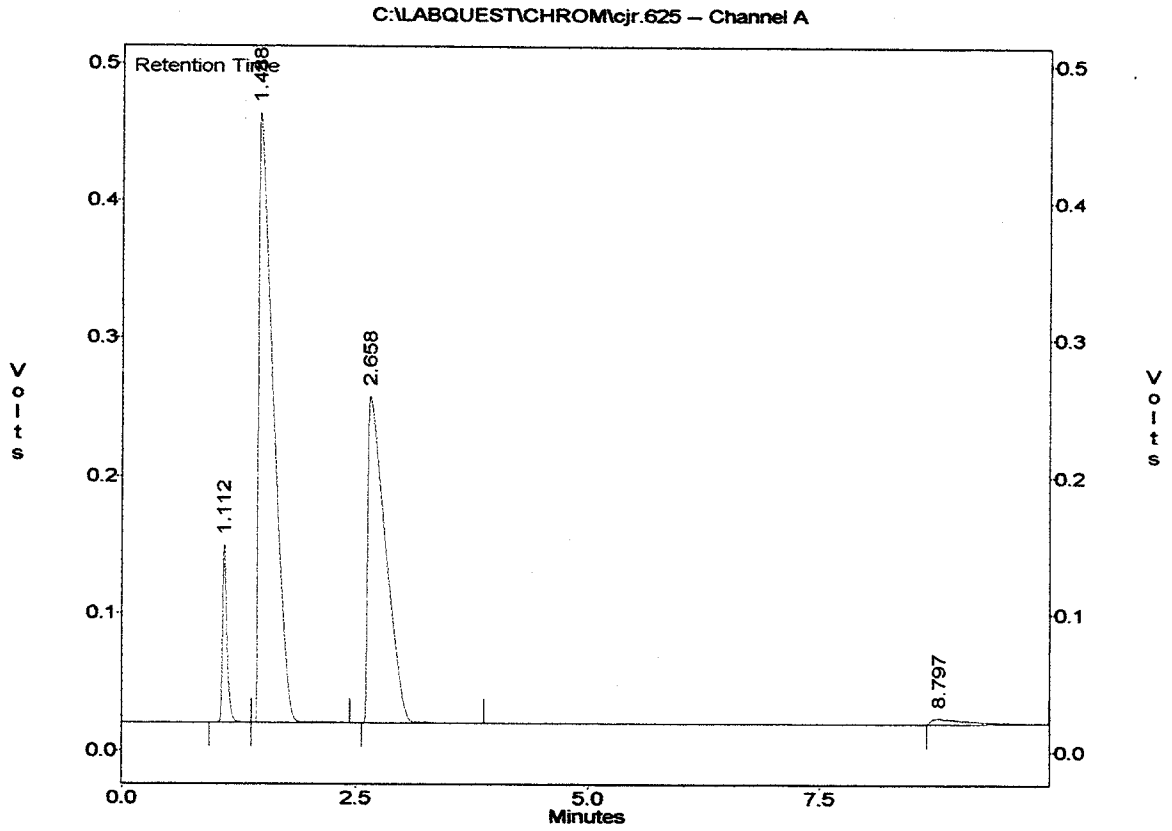
Characterization and Anaerobic Digestion Analysis of Ethanol Process Samples

By

Pinnacle Biotechnologies Inc.

Biogas Analysis

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #1
Printed : Jun 25, 1998 15:00:02



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.488	4691432.5	67.69
3	Carbon Dioxide	2.658	3116158.0	32.31
Totals :			7807590.5	100.00

Gas Analysis
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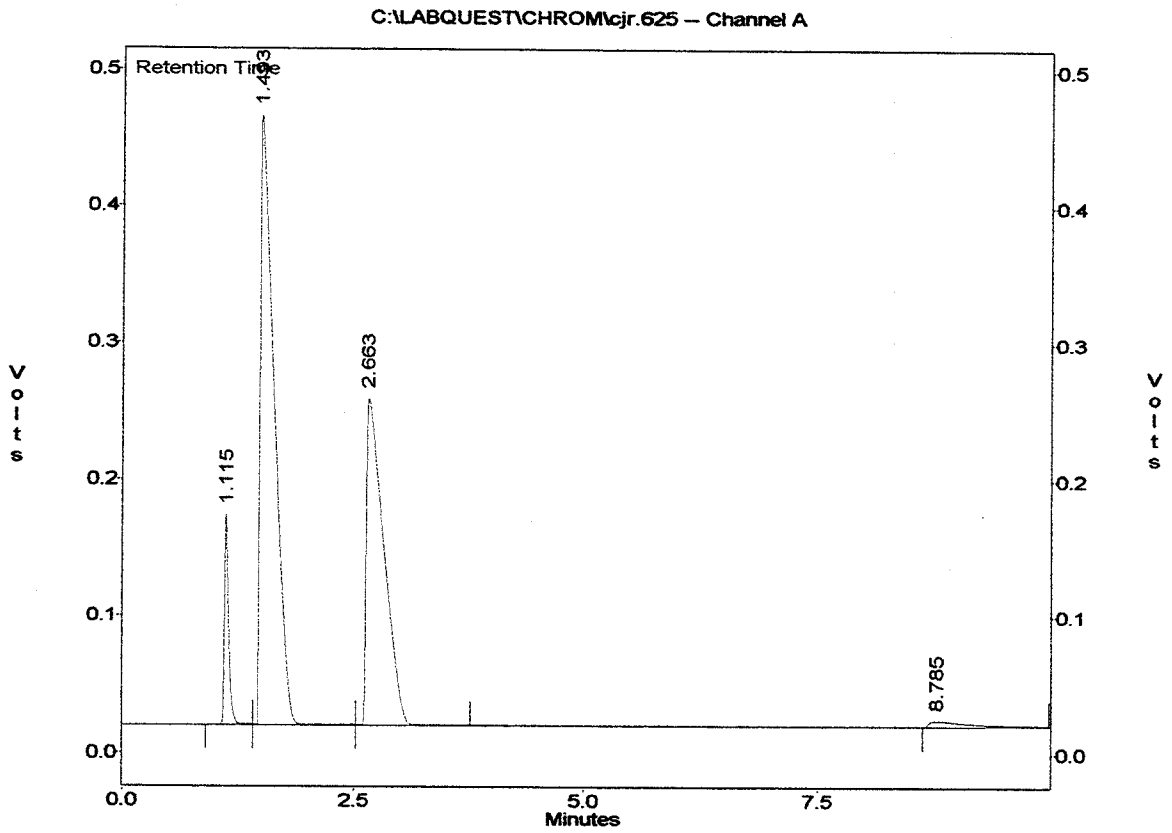
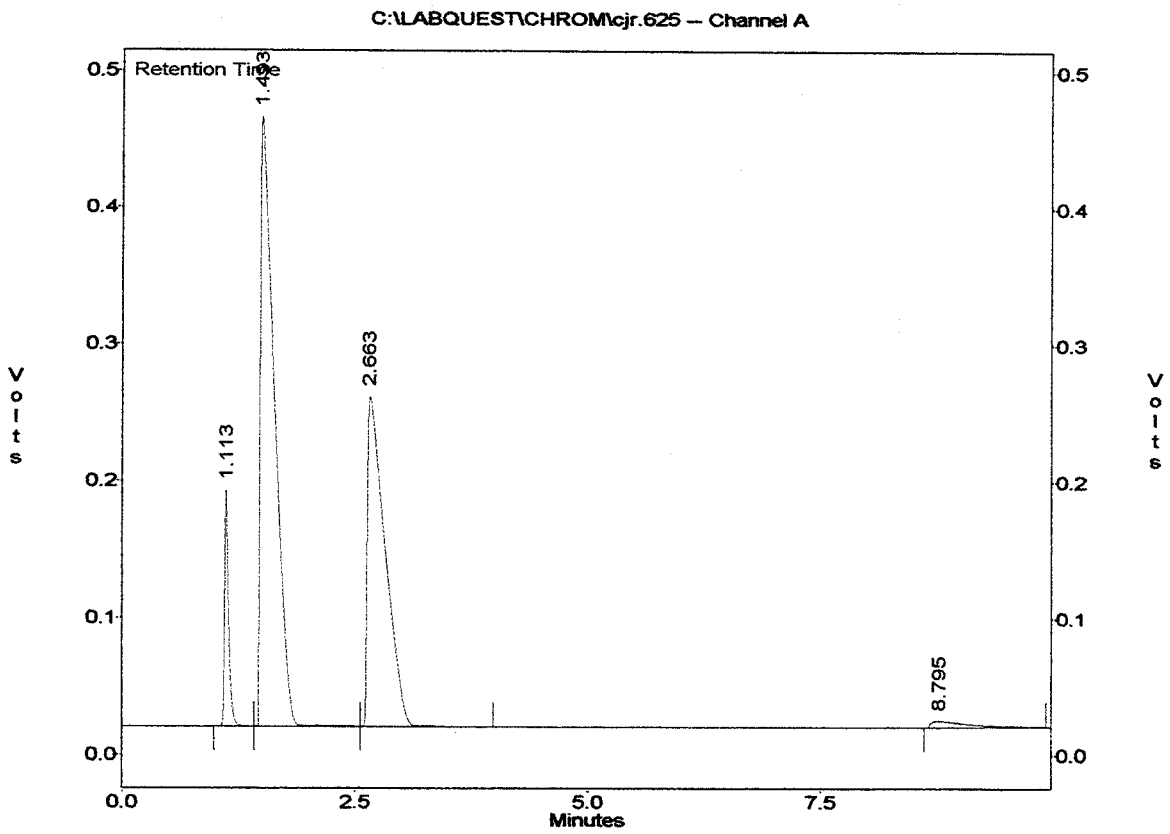
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #2
Printed : Jun 25, 1998 15:28:47



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.493	4724336.0	67.71
3	Carbon Dioxide	2.663	3135141.0	32.29
Totals :			7859477.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #3
Printed : Jun 25, 1998 15:51:57



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.493	4714953.0	67.39
3	Carbon Dioxide	2.663	3175722.0	32.61
Totals :			7890675.0	100.00

Gas Analysis
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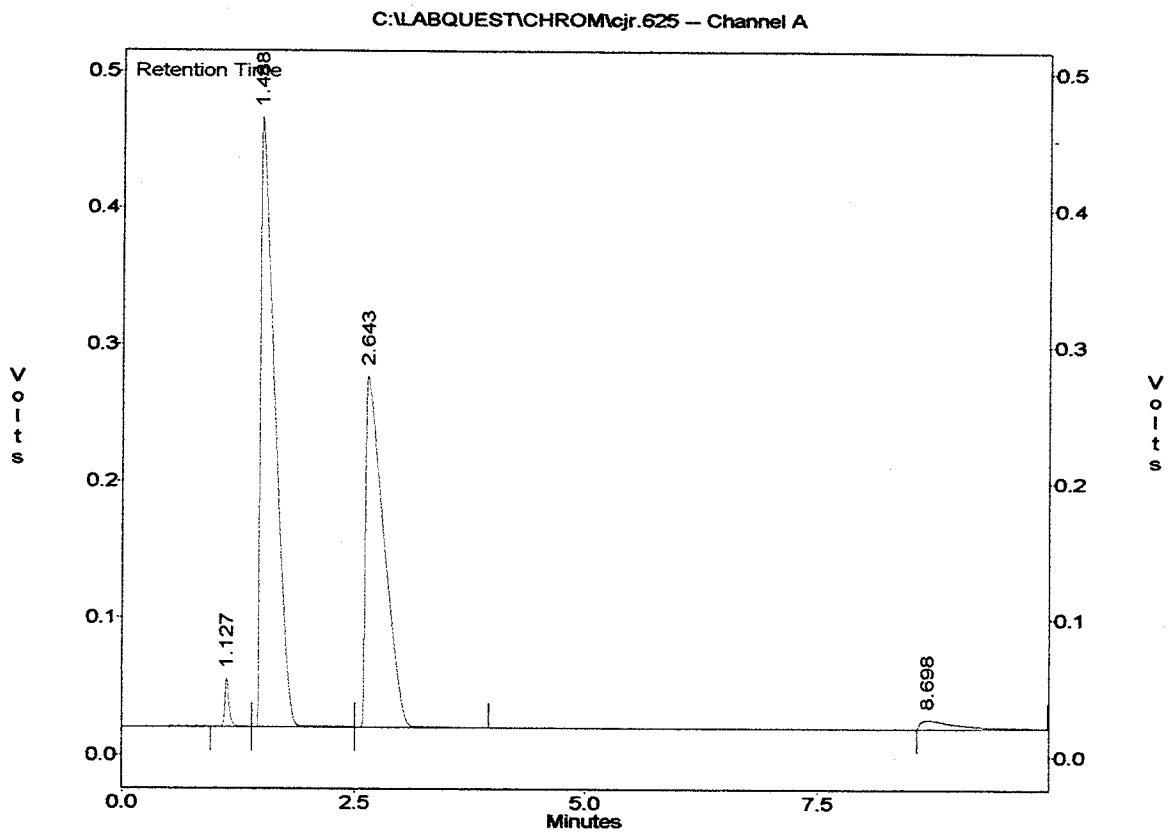
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #4
Printed : Jun 25, 1998 16:25:30



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.488	4693482.5	65.00
3	Carbon Dioxide	2.643	3517557.0	35.00

Totals :

	8211039.5	100.00

Gas Analysis
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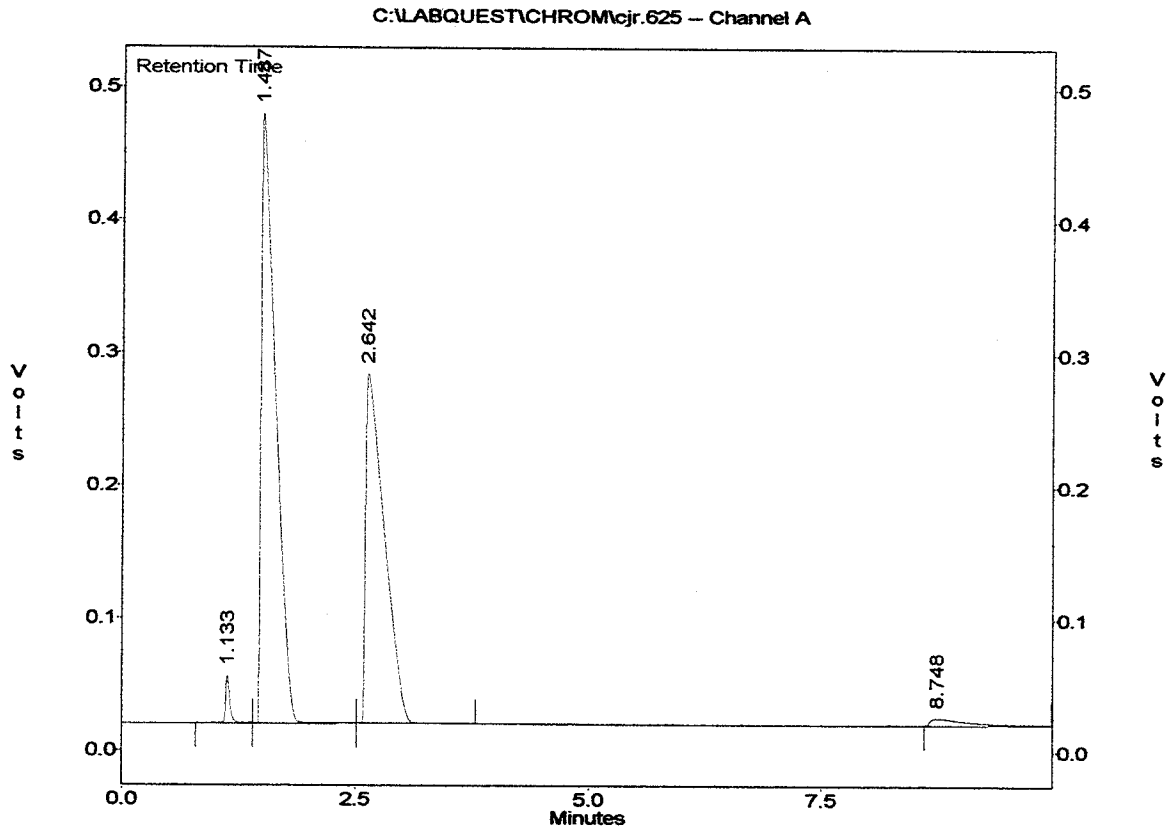
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #5
Printed : Jun 25, 1998 17:33:06



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.487	4919626.5	65.17
3	Carbon Dioxide	2.642	3659567.8	34.83
Totals :			8579194.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #6
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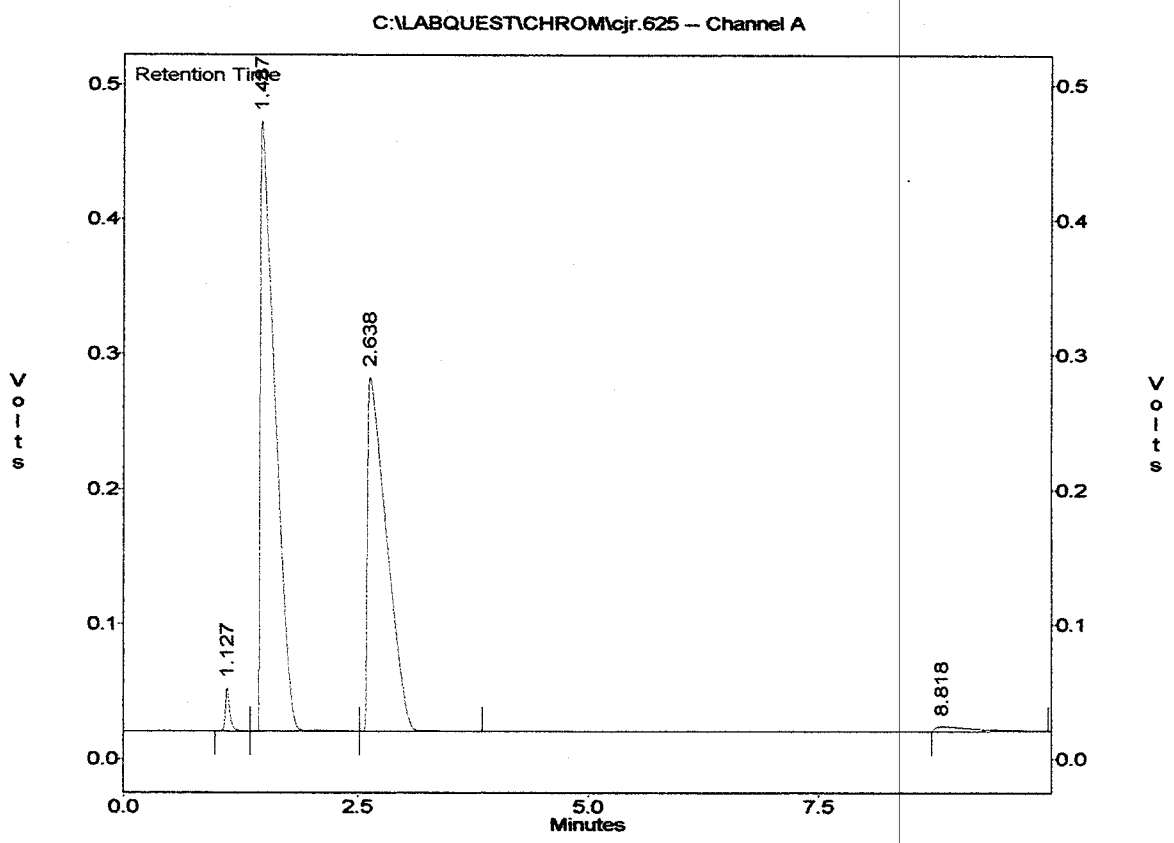

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.487	4841500.0	64.78
3	Carbon Dioxide	2.638	3663040.0	35.22
Totals :			8504540.0	100.00

Gas Analysis
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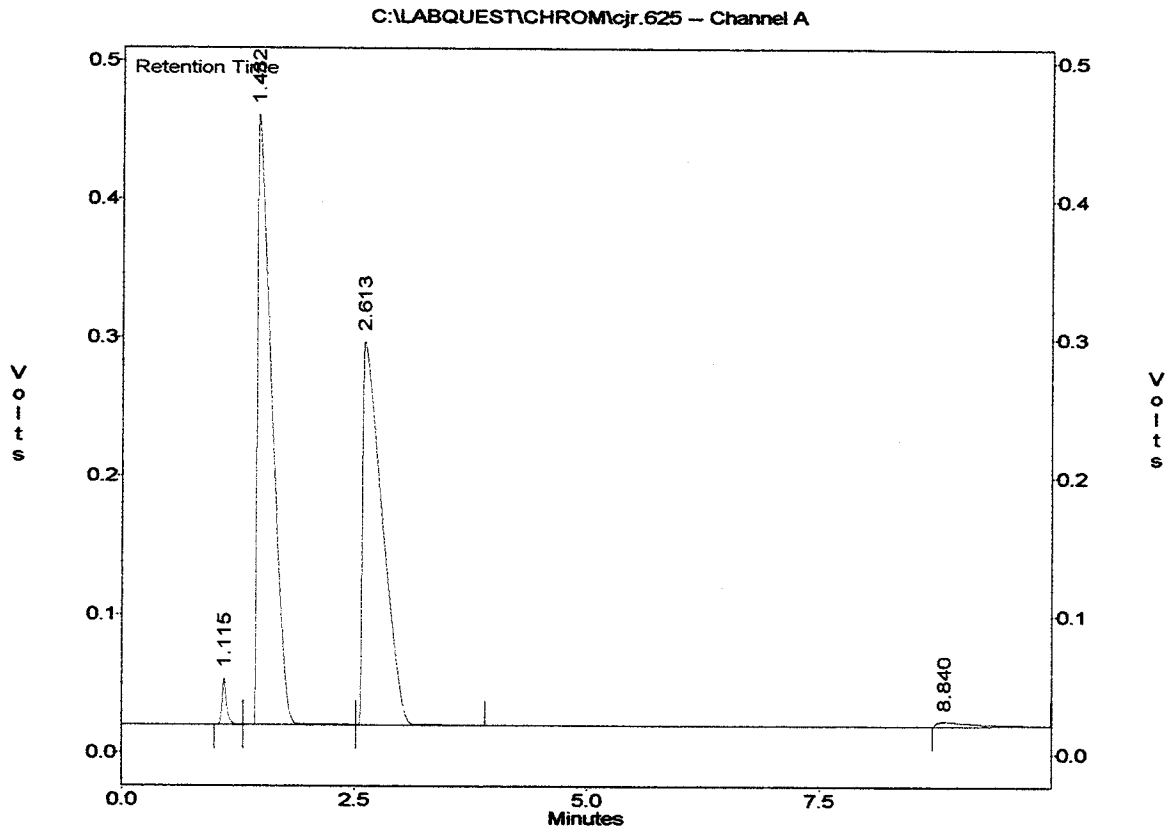
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #7
Printed : Jun 25, 1998 19:07:05



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.482	4612954.0	61.70
3	Carbon Dioxide	2.613	3985246.5	38.30
Totals :			8598200.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #8
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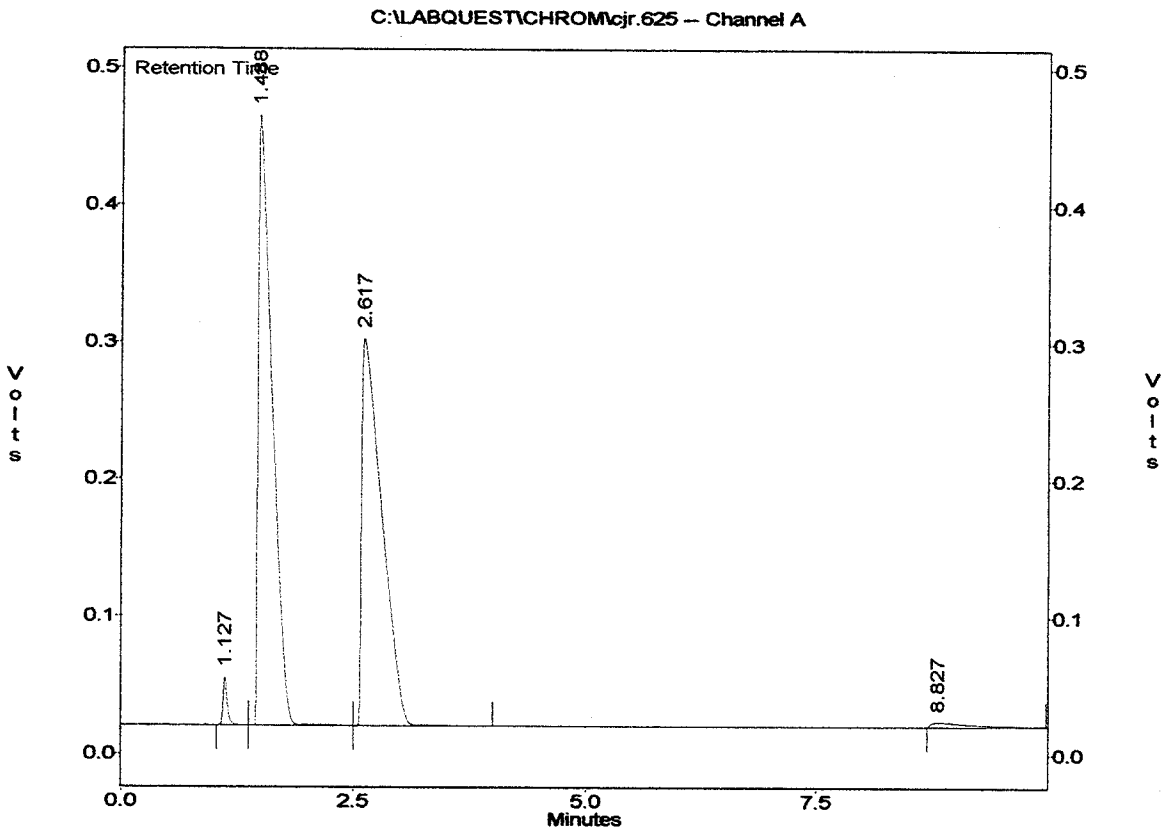

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.488	4666704.0	61.28
3	Carbon Dioxide	2.617	4103121.5	38.72
Totals :			8769826.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #9
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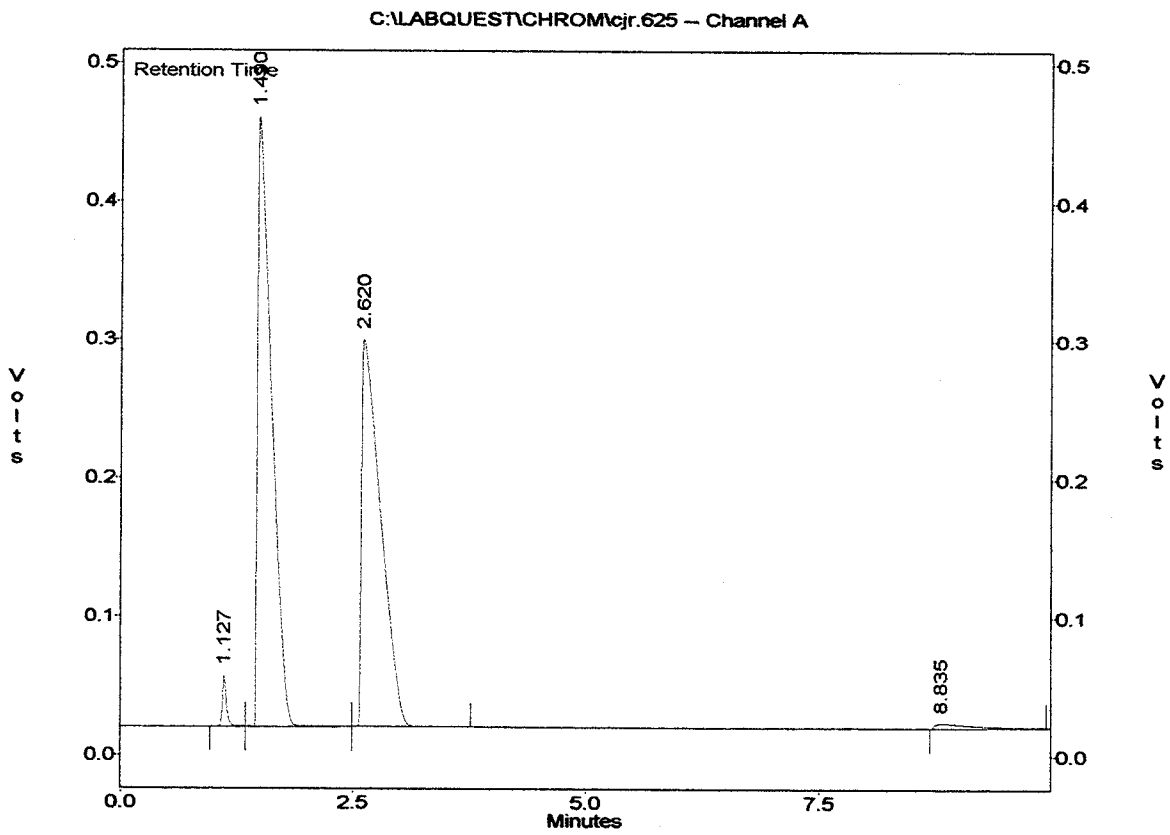

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.490	4582528.5	61.30
3	Carbon Dioxide	2.620	4026208.8	38.70
Totals :			8608737.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #10
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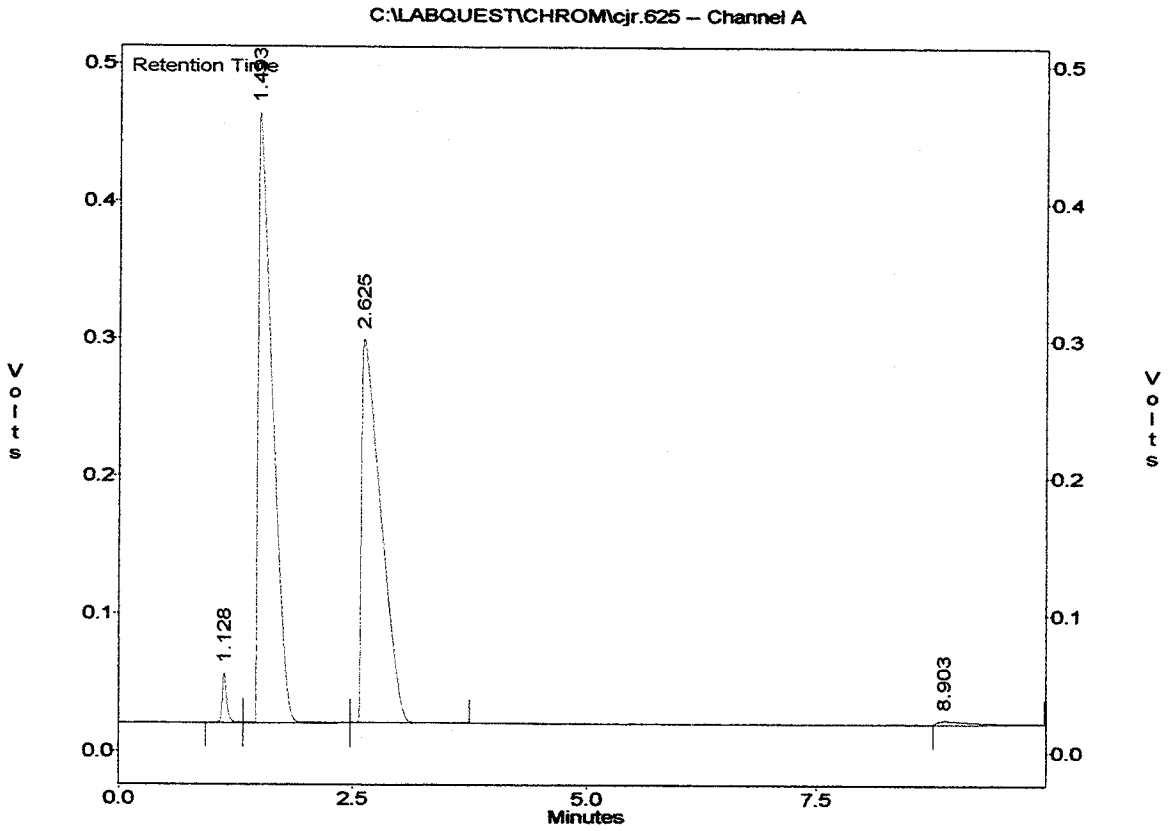

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.493	4664040.5	61.66
3	Carbon Dioxide	2.625	4036362.8	38.34
Totals :			8700403.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #11
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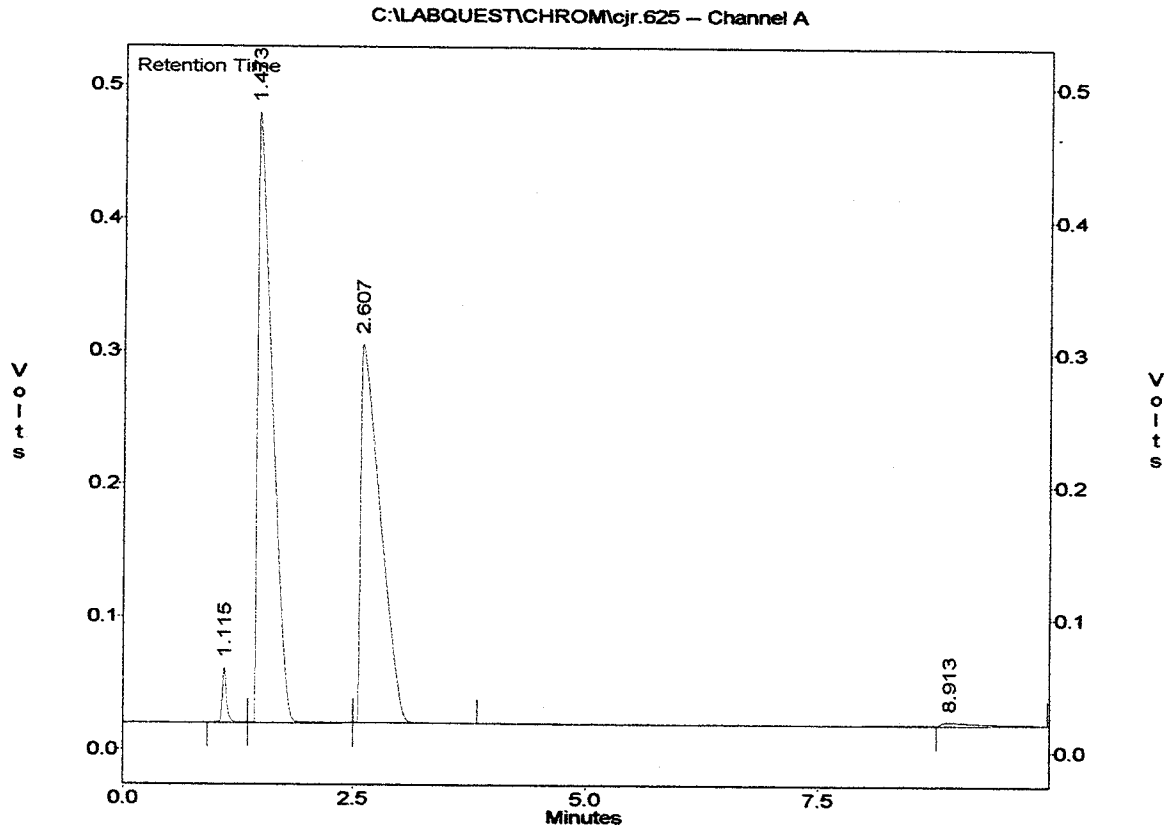

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.473	4918913.0	62.21
3	Carbon Dioxide	2.607	4158397.5	37.79
Totals :			9077310.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #12
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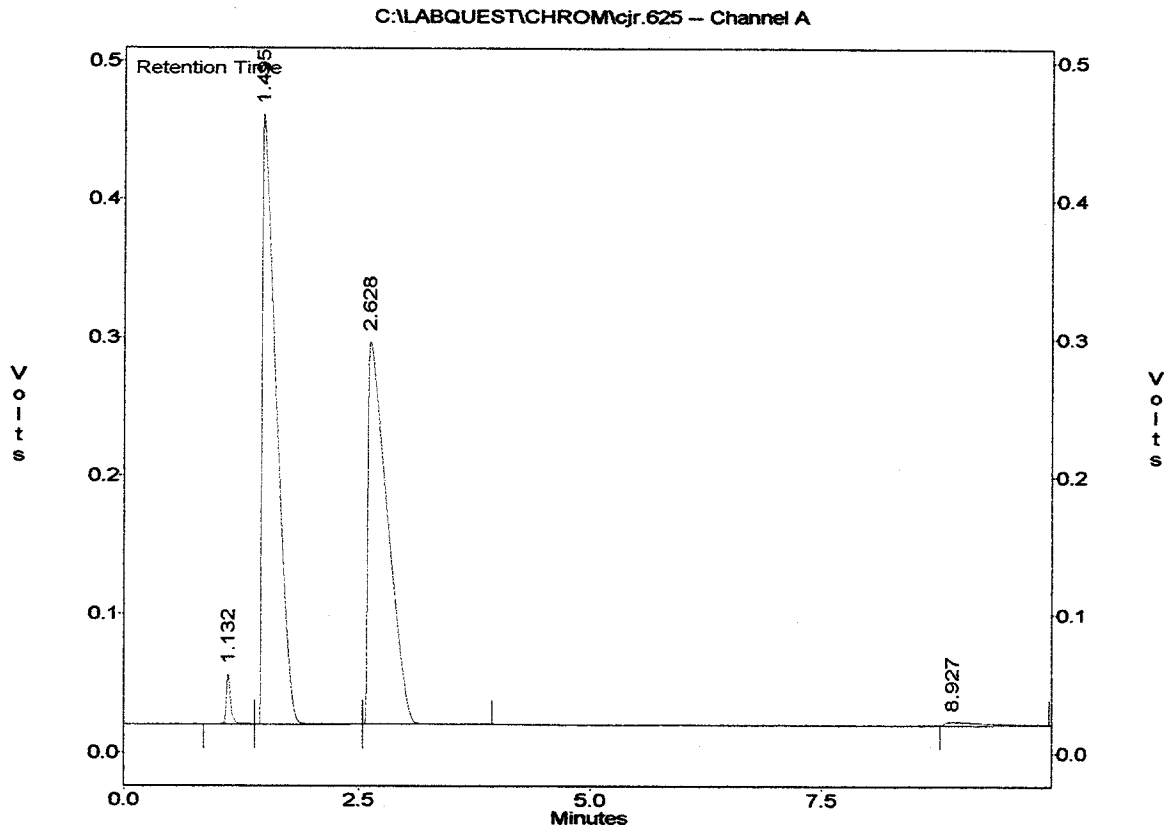

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.495	4582253.0	61.72
3	Carbon Dioxide	2.628	3955167.8	38.28
Totals :			8537421.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #13
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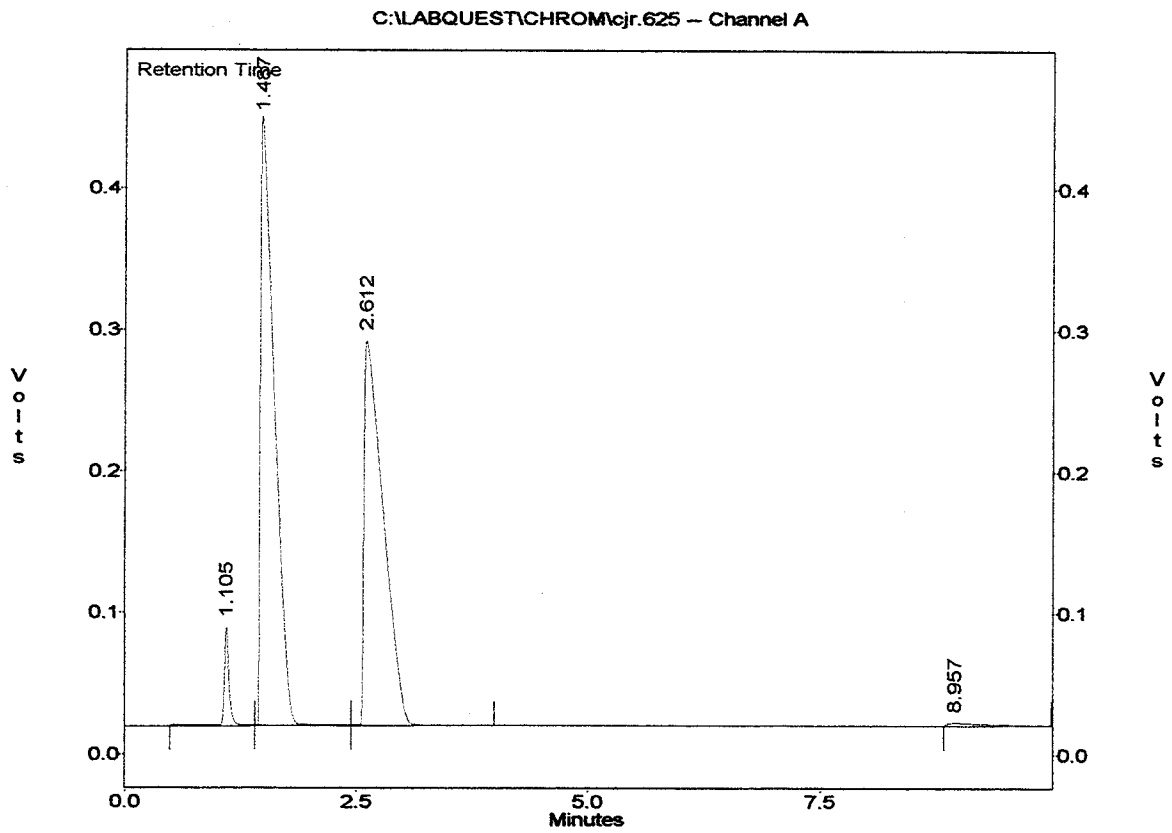

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.487	4431616.0	61.49
3	Carbon Dioxide	2.612	3862540.3	38.51
Totals :			8294156.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #14
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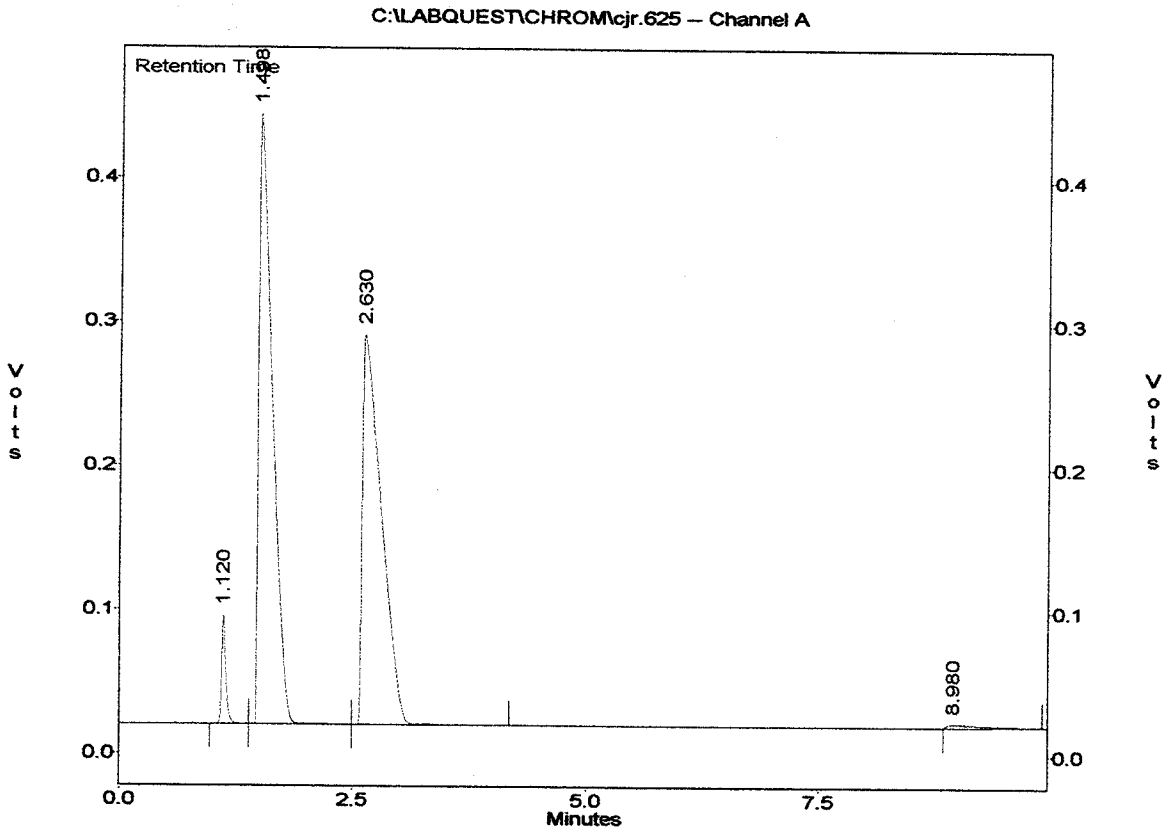

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.498	4286963.0	61.03
3	Carbon Dioxide	2.630	3810111.0	38.97
Totals :			8097074.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #15
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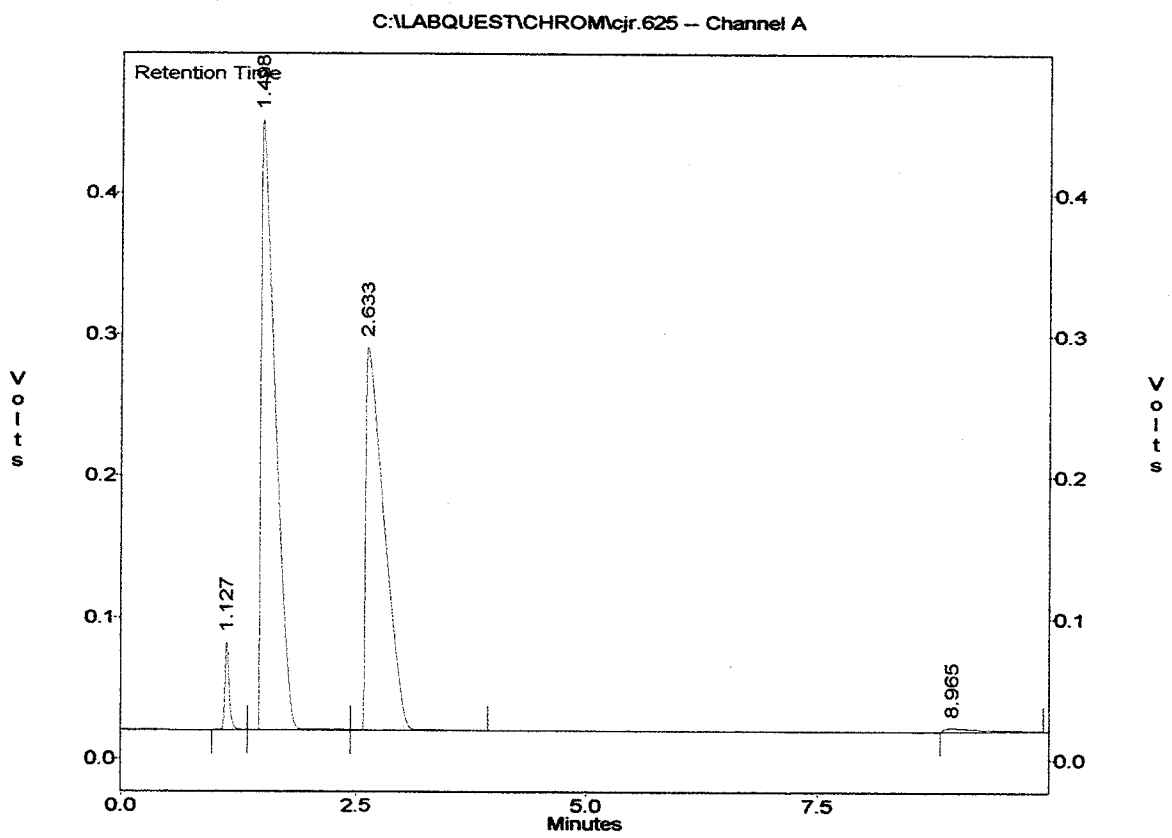

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.498	4404101.5	61.68
3	Carbon Dioxide	2.633	3807966.8	38.32
Totals :			8212068.0	100.00

Gas Analysis
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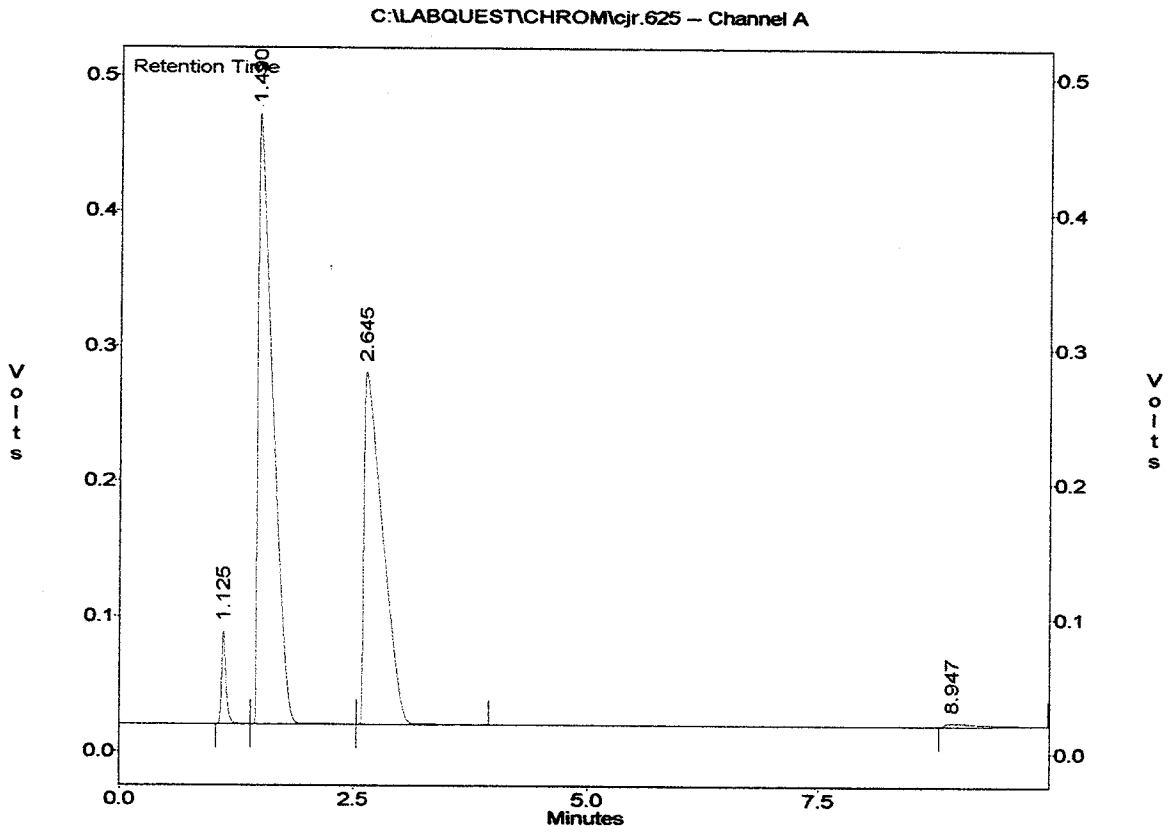
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #16
Printed : Jun 26, 1998 15:00:53



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.490	4794559.5	64.89
3	Carbon Dioxide	2.645	3610497.5	35.11

Totals :

	8405057.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #17
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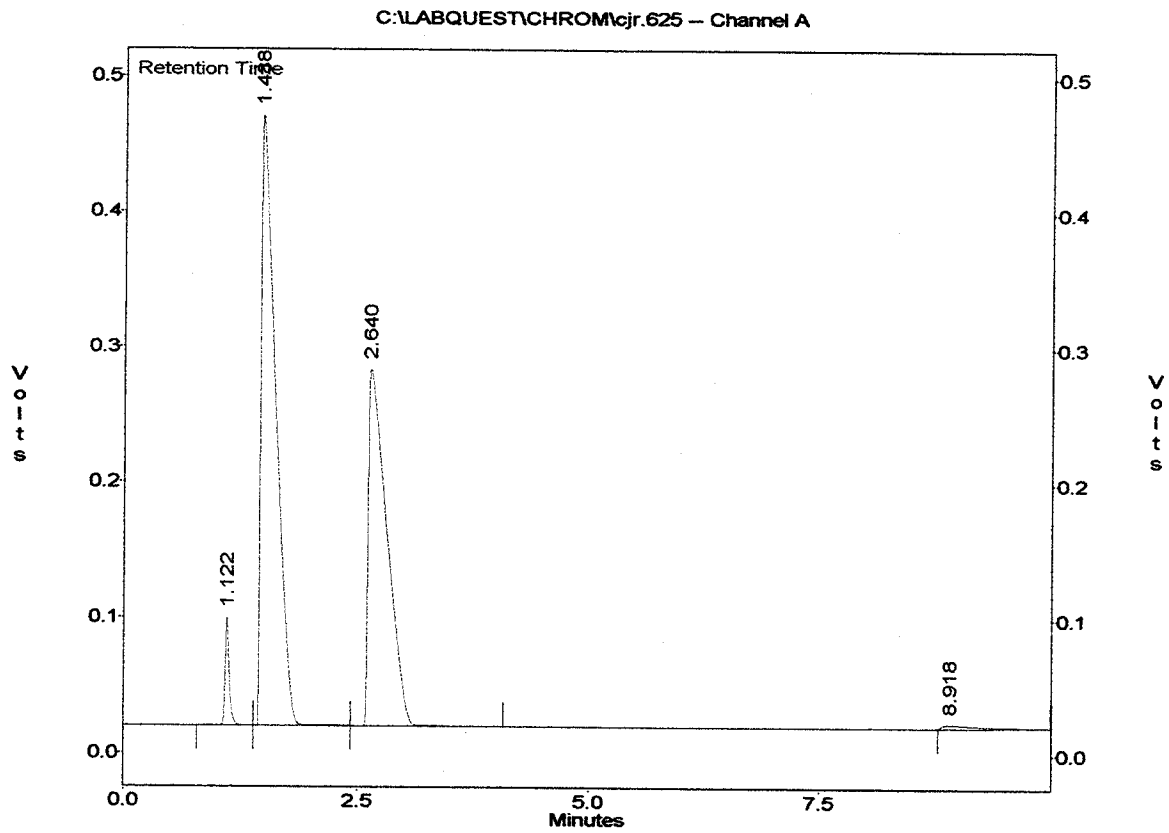

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.488	4767781.5	64.45
3	Carbon Dioxide	2.640	3659716.5	35.55

Totals :

	8427498.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\cjr.625
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL Sample Bottle #18
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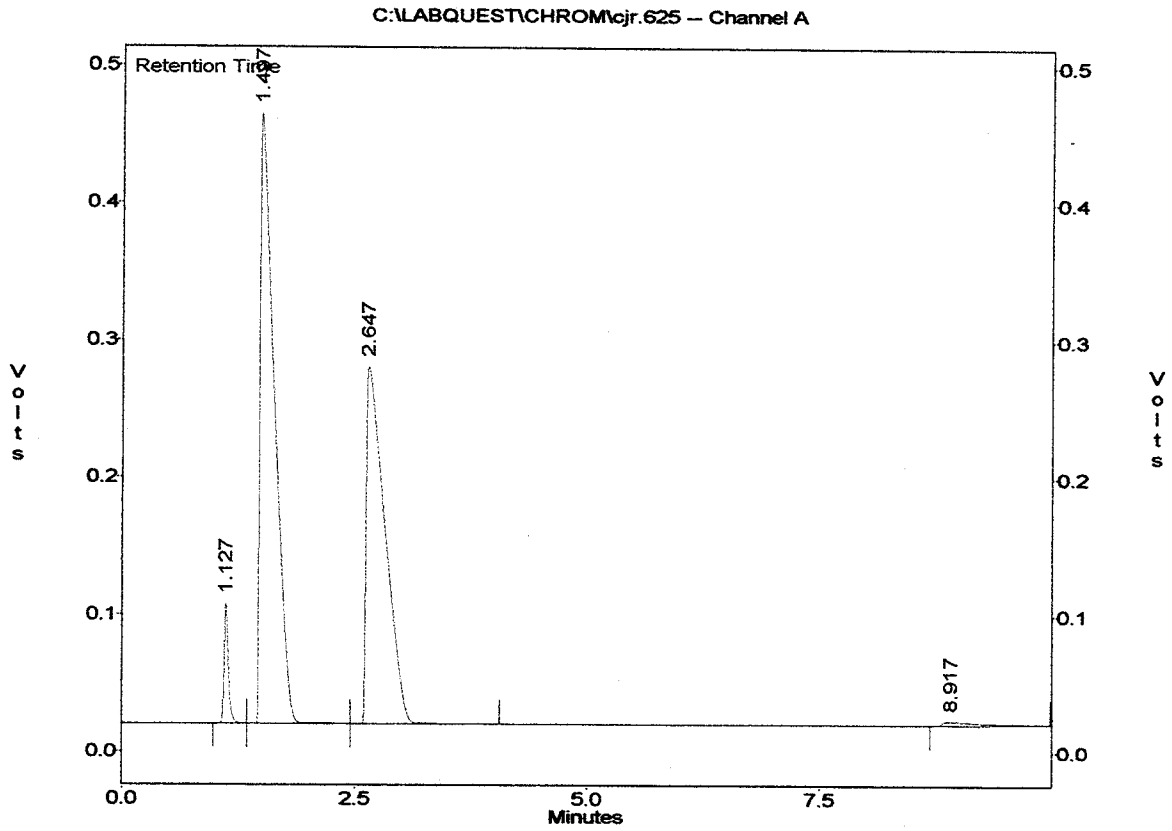

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.497	4672798.0	64.33
3	Carbon Dioxide	2.647	3605458.5	35.67
Totals :			8278256.5	100.00

Gas Analysis
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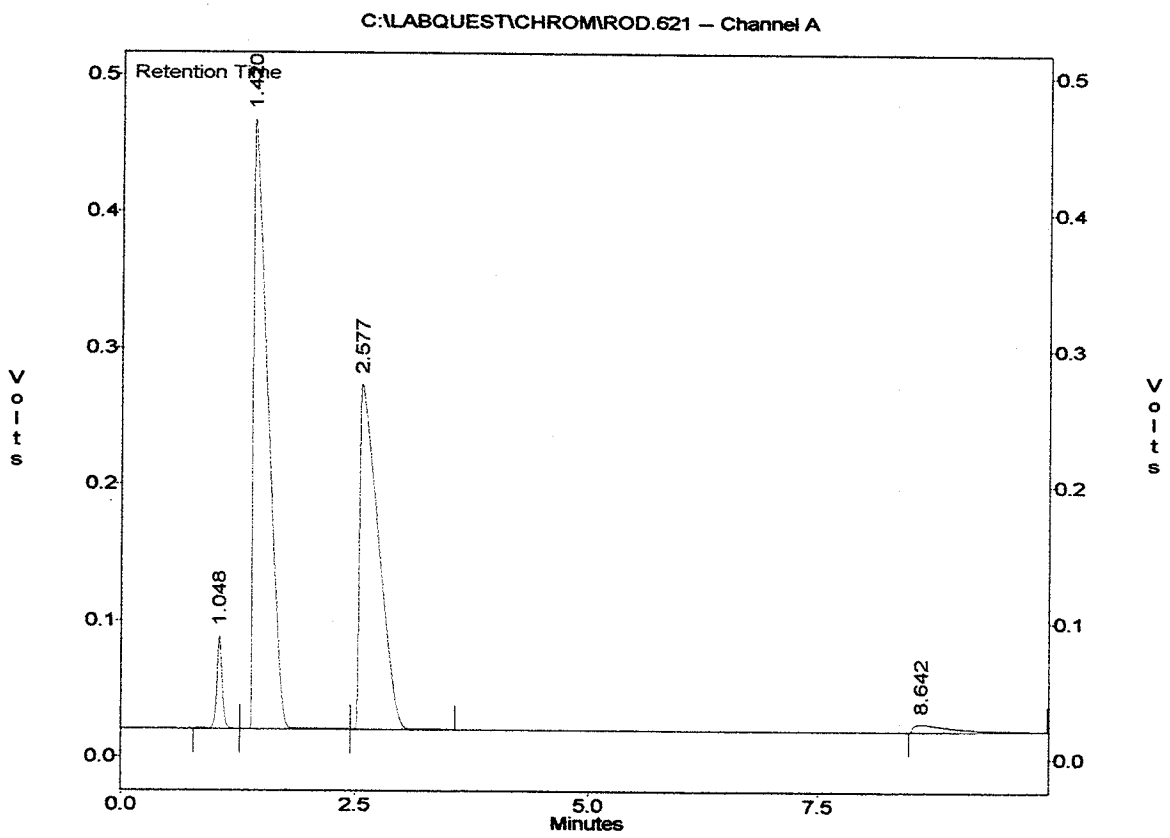
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NERL /16
Printed : Jun 22, 1998 00:57:12



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.420	4664980.0	65.53
3	Carbon Dioxide	2.577	3415075.5	34.47
Totals :			8080055.5	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NERL /13
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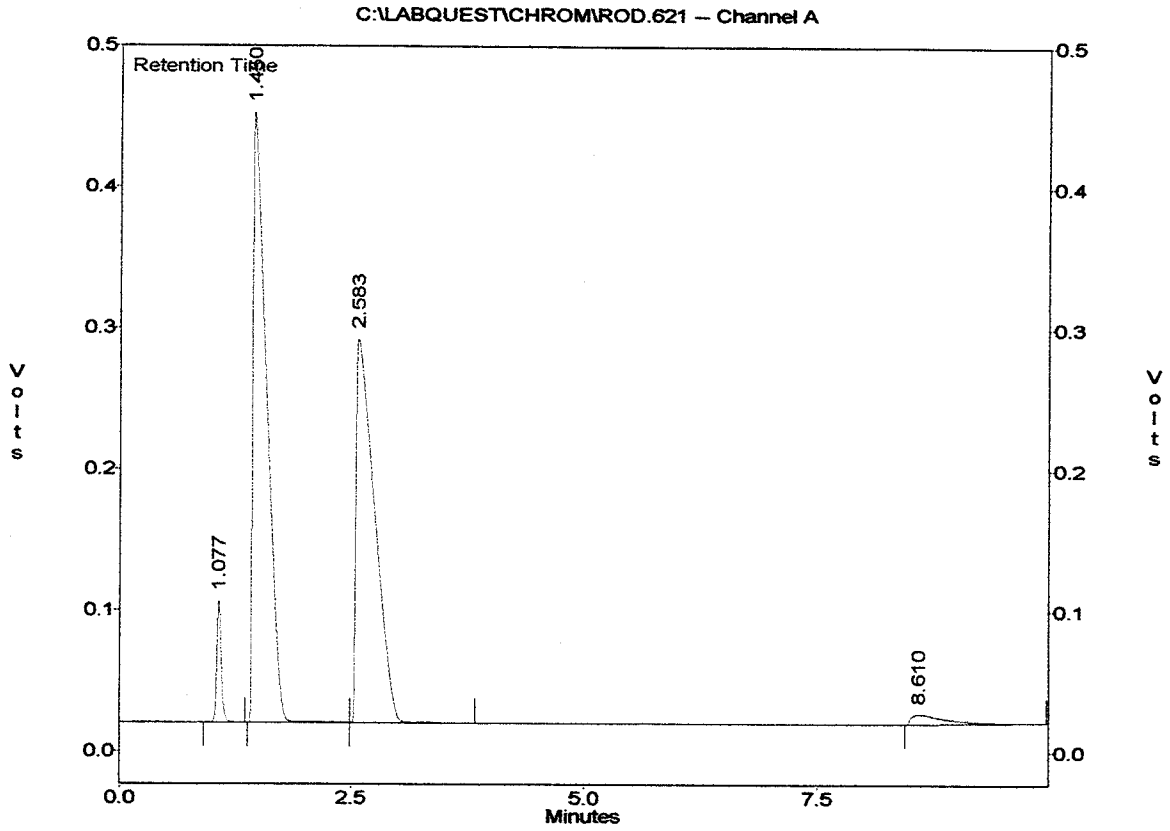

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.450	4351107.0	61.66
3	Carbon Dioxide	2.583	3764813.3	38.34
Totals :			8115920.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NERL /10
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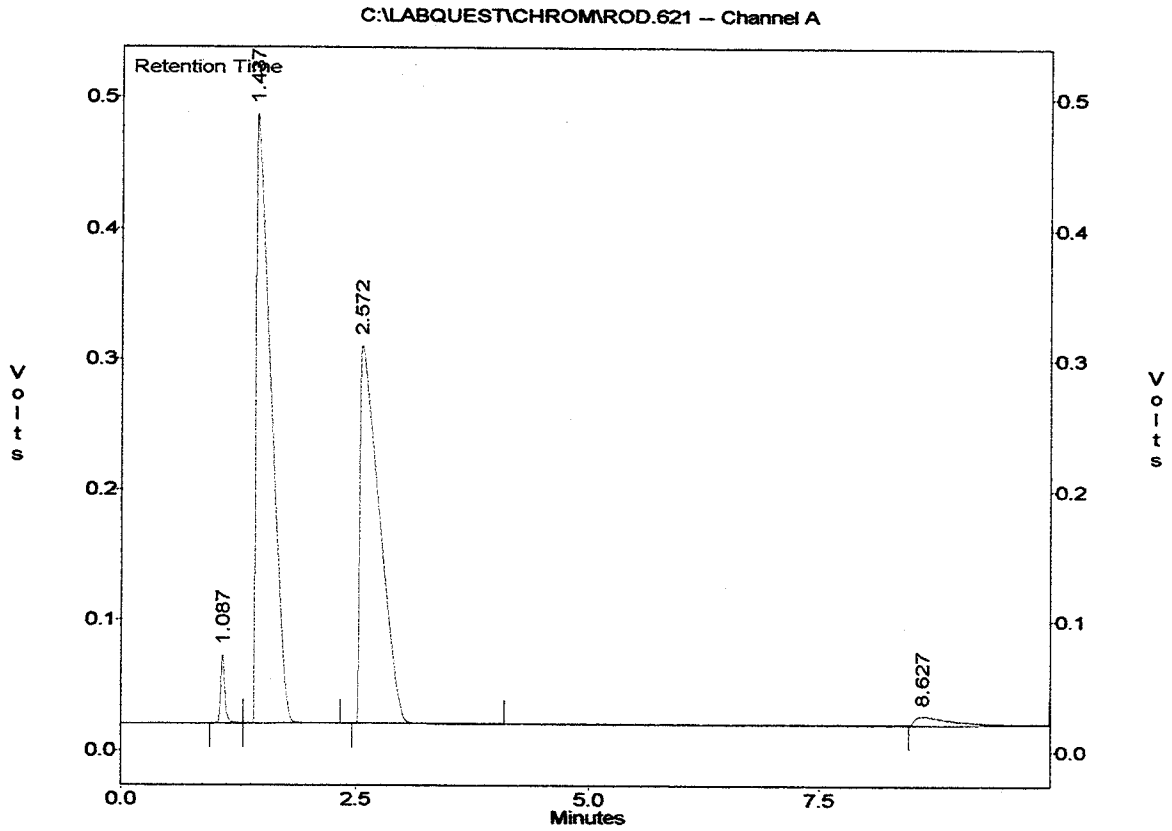

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.437	4935281.5	62.19
3	Carbon Dioxide	2.572	4175114.3	37.81
Totals :			9110396.0	100.00

Gas Analysis
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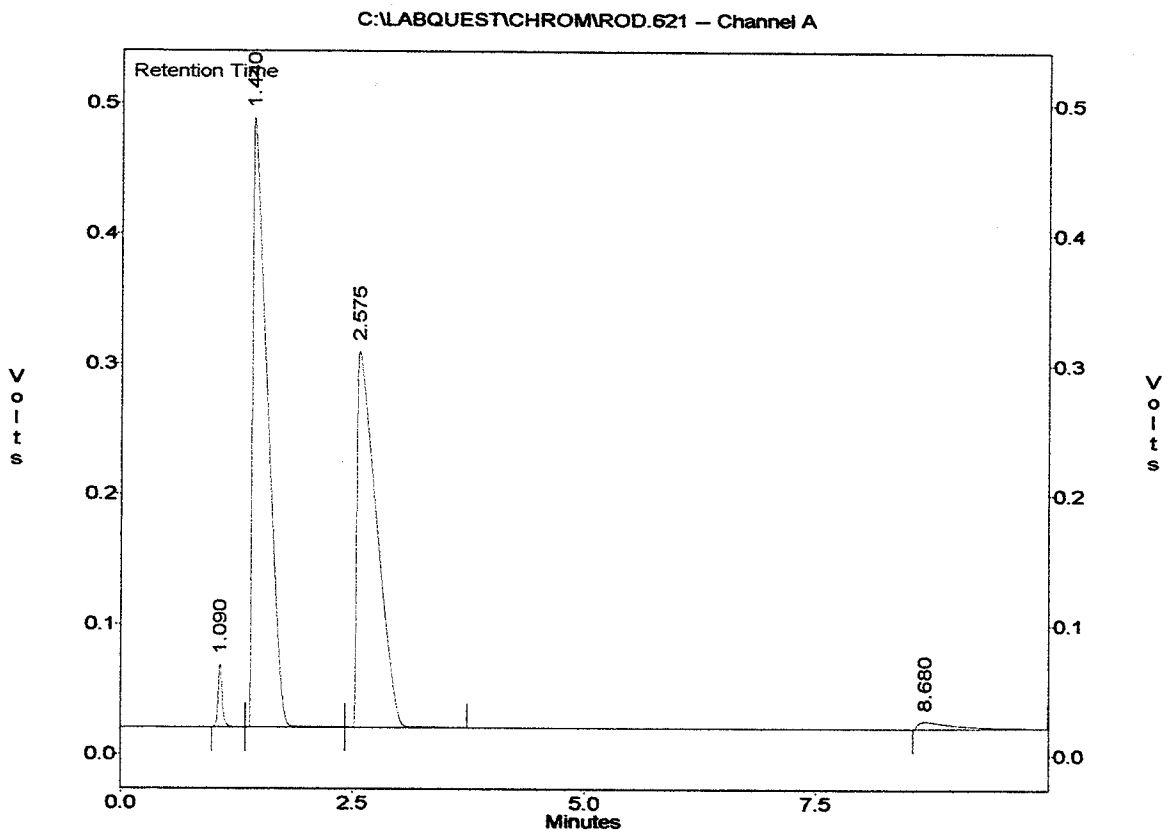

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.440	4952168.0	62.38
3	Carbon Dioxide	2.575	4156688.5	37.62
Totals :			9108856.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NERL / 4
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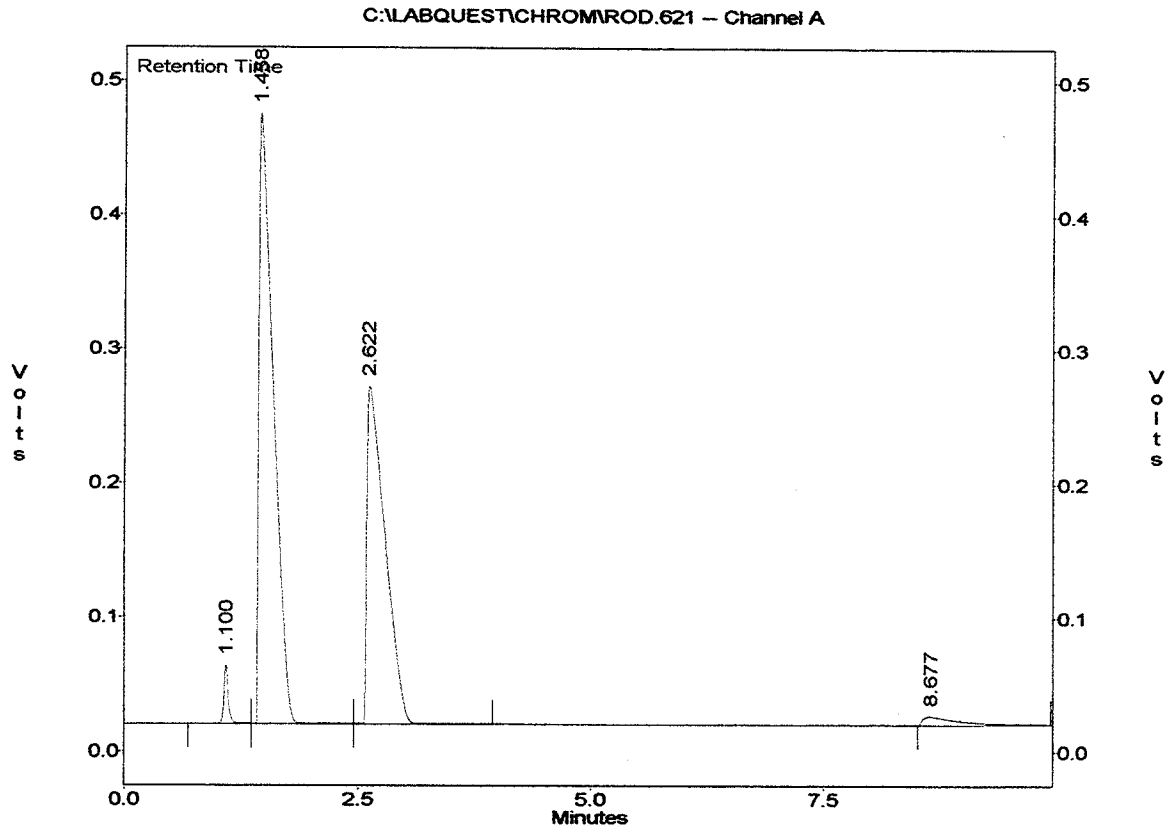

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.458	4751608.0	66.55
3	Carbon Dioxide	2.622	3323181.8	33.45
Totals :			8074790.0	100.00

Gas Analysis
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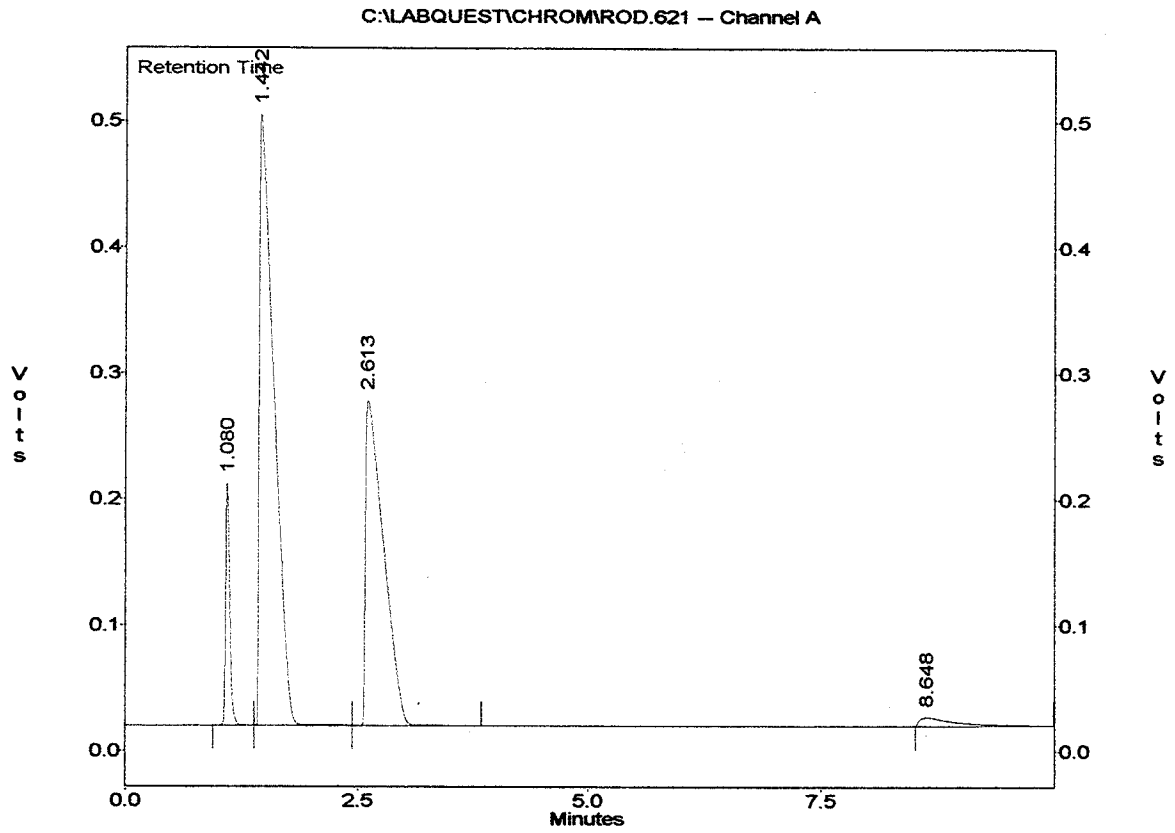

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.442	5291346.0	68.01
3	Carbon Dioxide	2.613	3463263.0	31.99
Totals :			8754609.0	100.00

Gas Analysis
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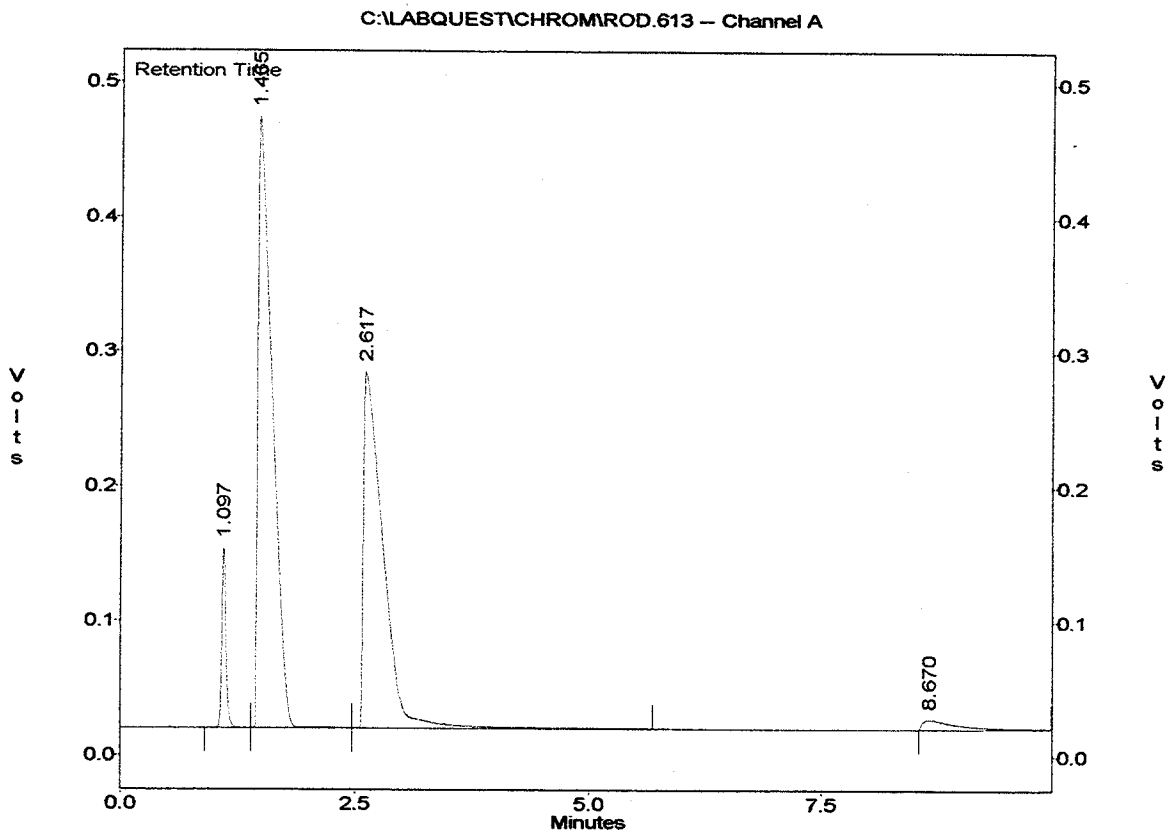
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 16
Printed : Jun 13, 1998 22:36:09



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.465	4714369.0	63.07
3	Carbon Dioxide	2.617	3841557.8	36.93
Totals :			8555927.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 13
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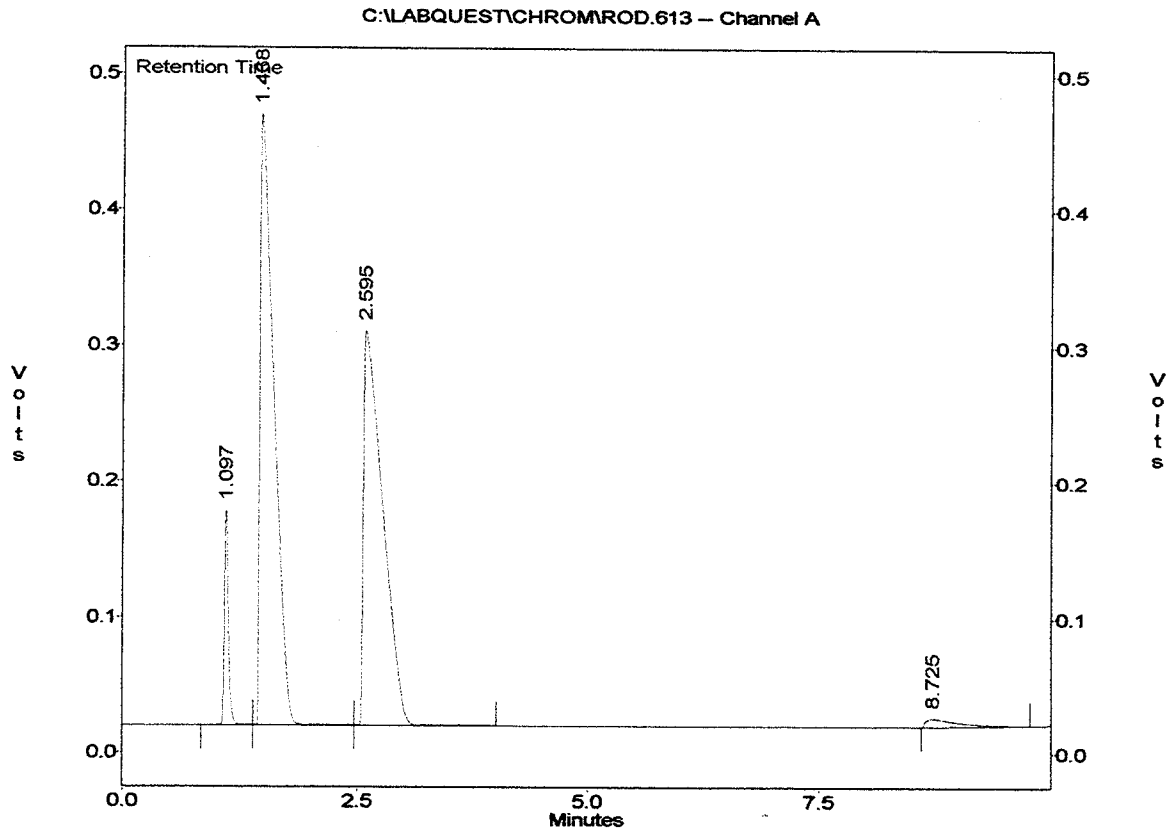

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.468	4625750.5	60.57
3	Carbon Dioxide	2.595	4191349.0	39.43
Totals :			8817100.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 10
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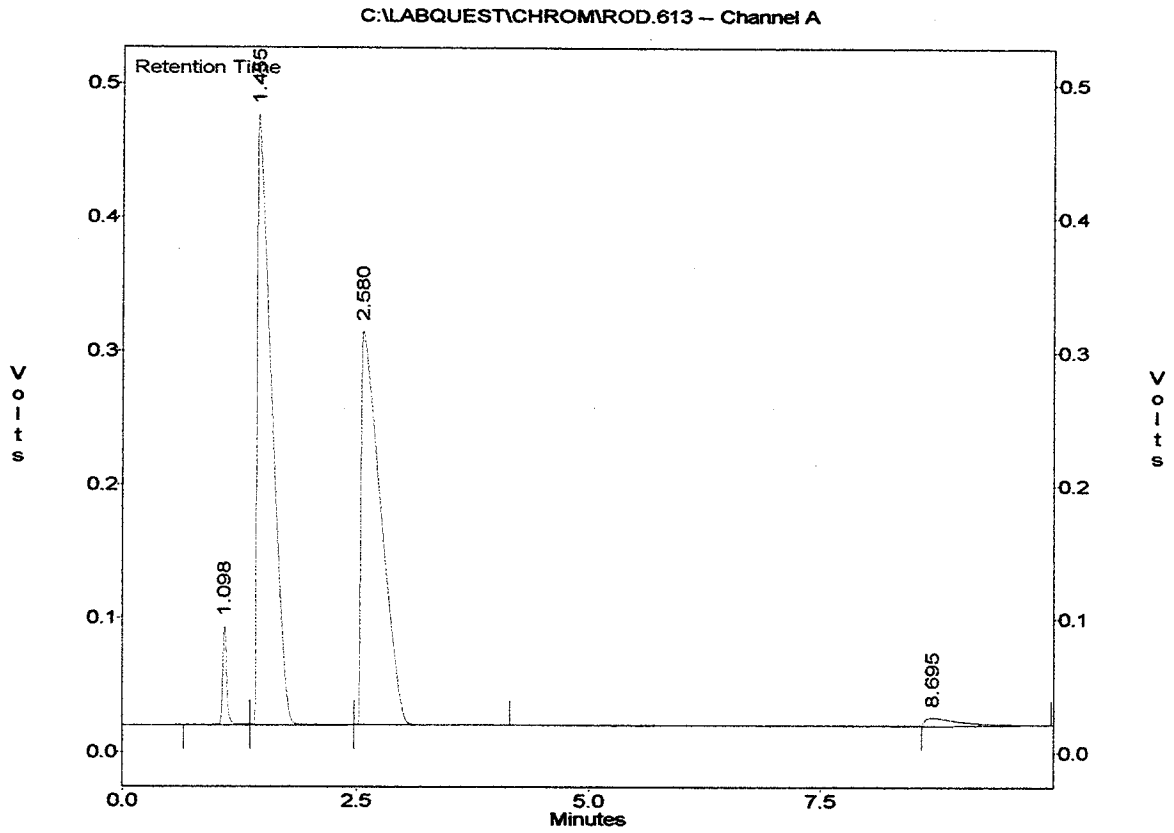

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.455	4755520.0	60.62
3	Carbon Dioxide	2.580	4299400.0	39.38
Totals :			9054920.0	100.00

Gas Analysis
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Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 7
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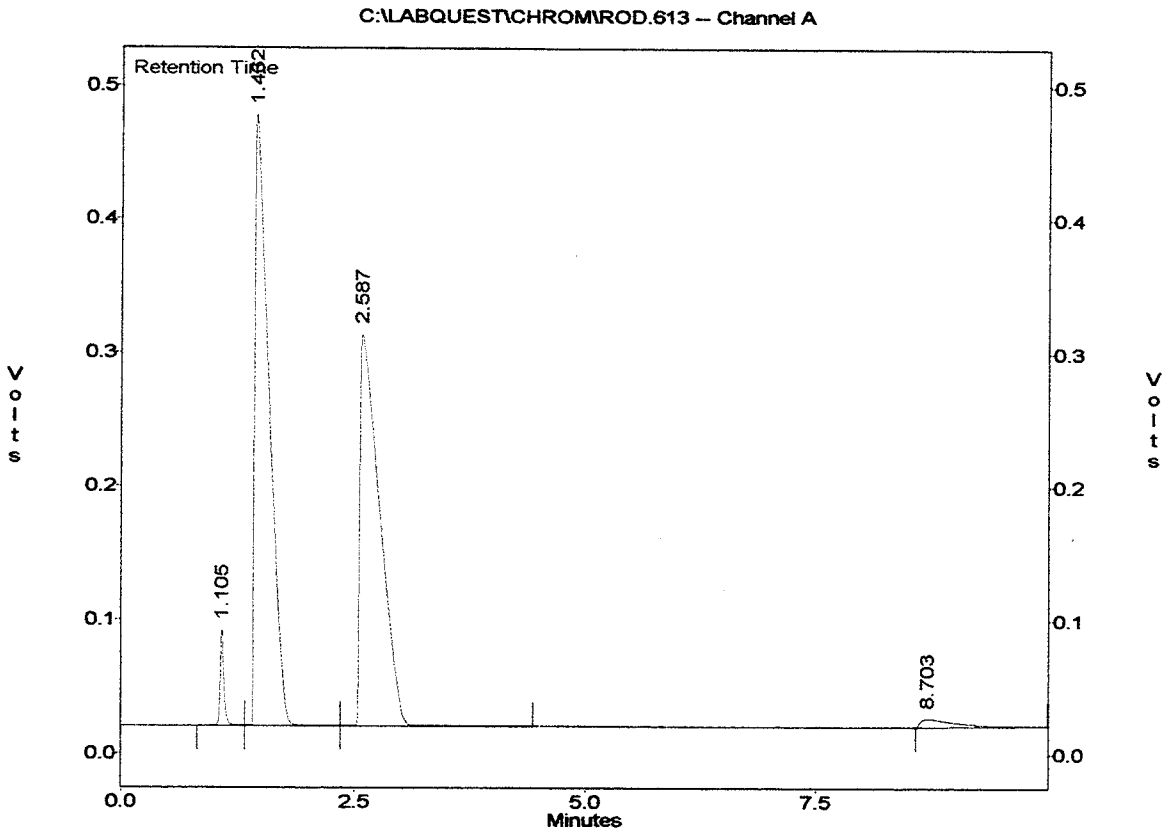

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.462	4768752.0	60.80
3	Carbon Dioxide	2.587	4278701.0	39.20
Totals :			9047453.0	100.00

Gas Analysis
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Sample ID : NREL / 4
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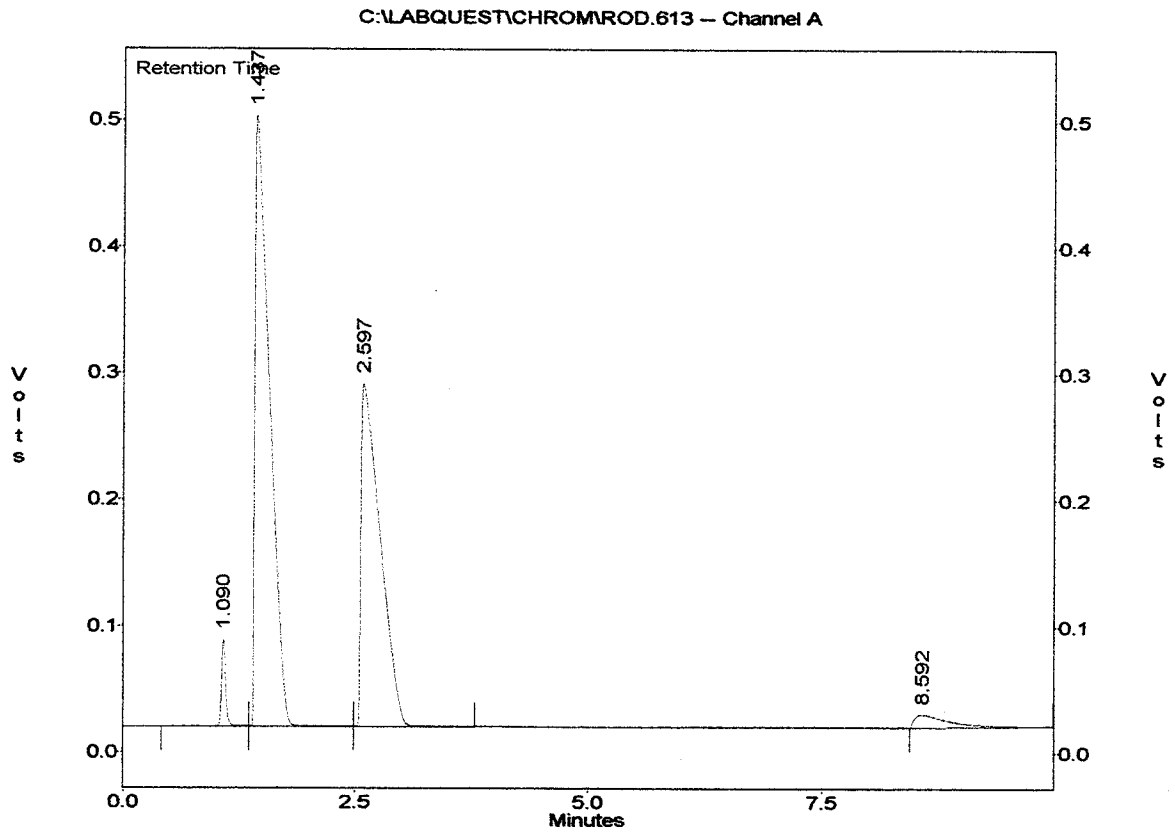

Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.437	5242003.0	65.99
3	Carbon Dioxide	2.597	3759144.8	34.01
Totals :			9001148.0	100.00

Gas Analysis
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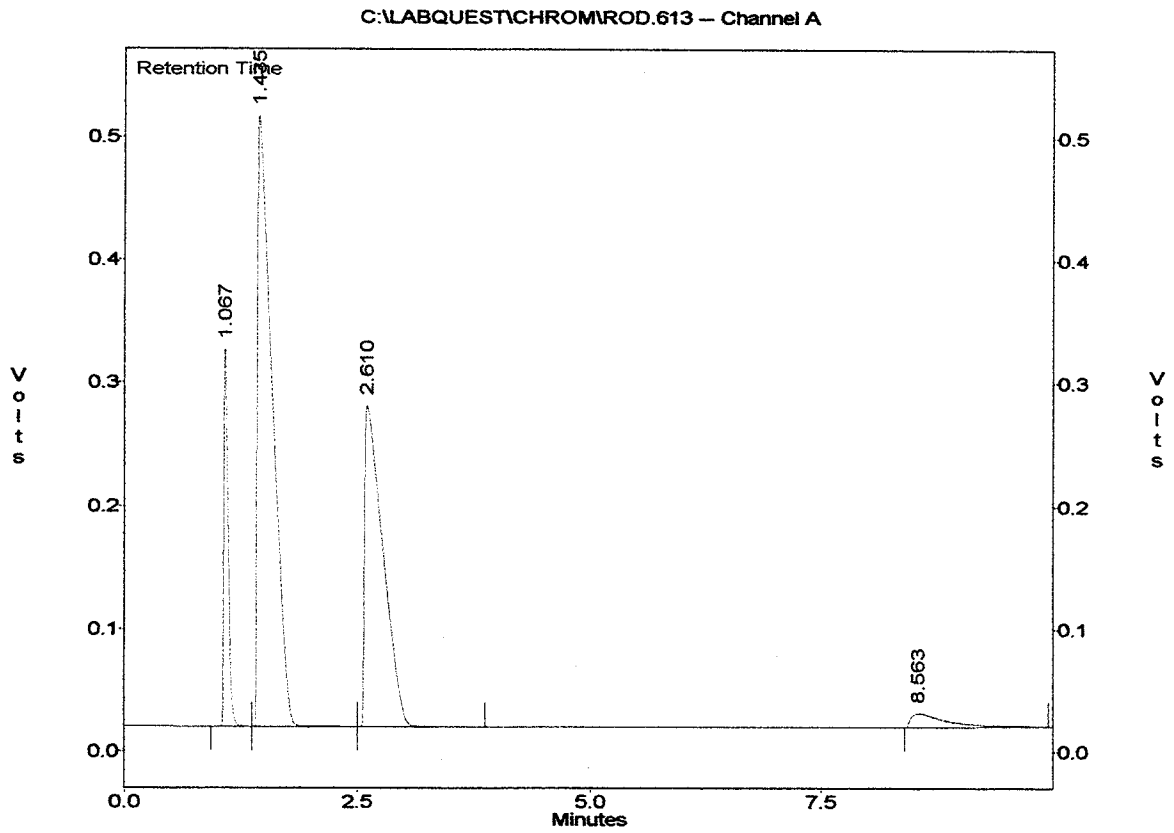
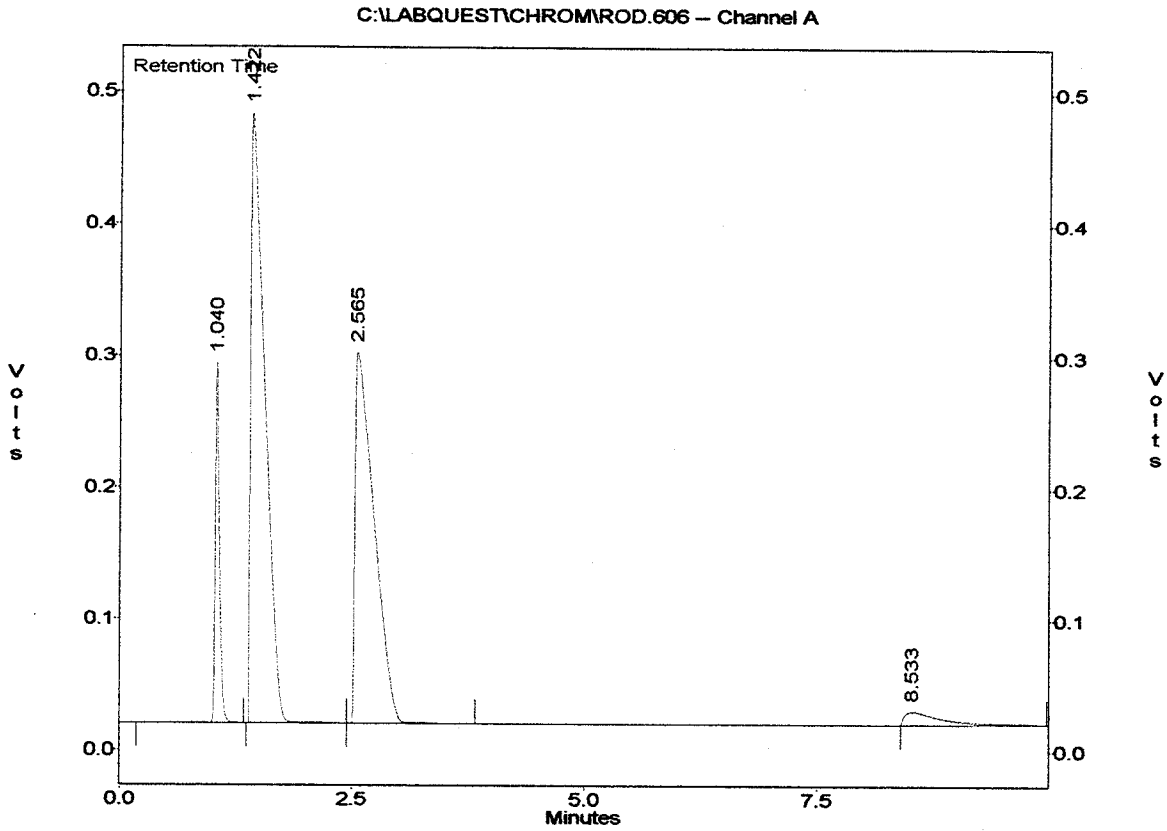
Sample ID : NREL / 1
Printed : Jun 13, 1998 19:41:23



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.435	5506645.0	68.42
3	Carbon Dioxide	2.610	3537088.0	31.58
Totals :			9043733.0	100.00

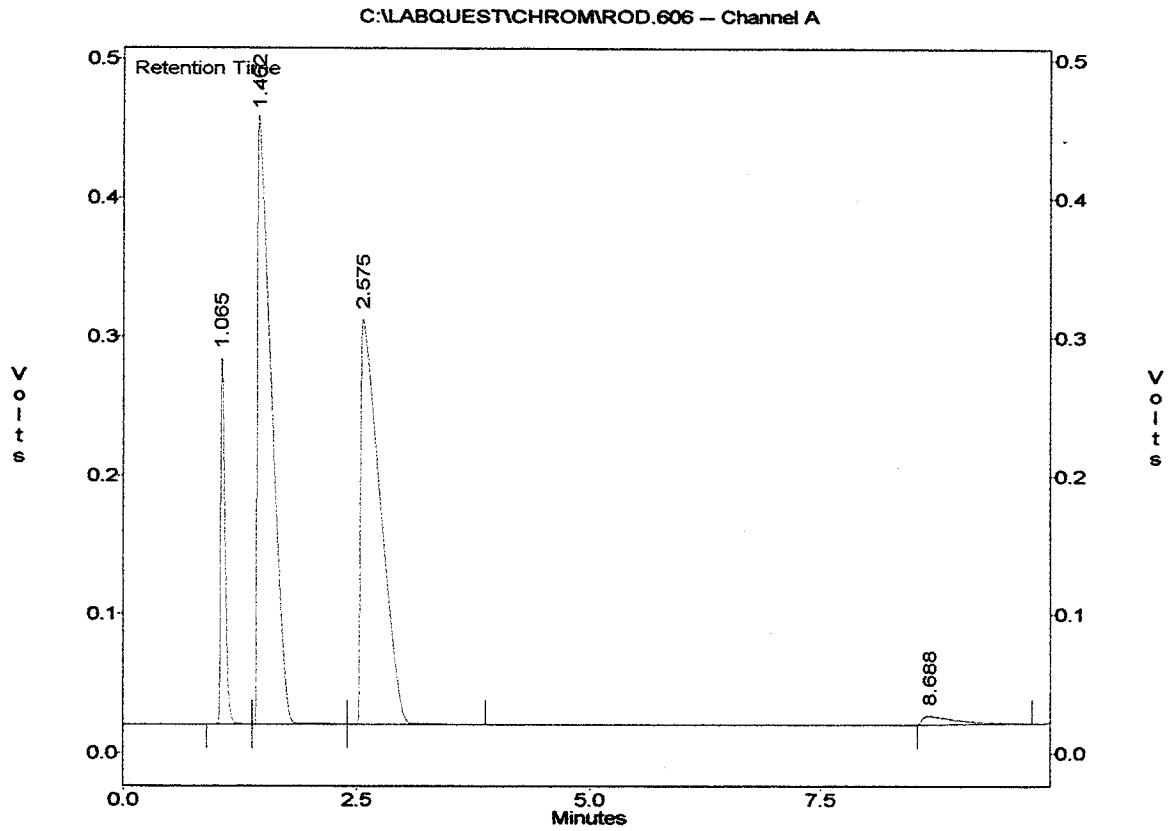
Gas Analysis
File : C:\LABQUEST\CHROM\ROD.606
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 17
Printed : Jun 06, 1998 23:25:15



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.422	4875957.0	63.00
3	Carbon Dioxide	2.565	3986138.3	37.00
Totals :			8862095.0	100.00

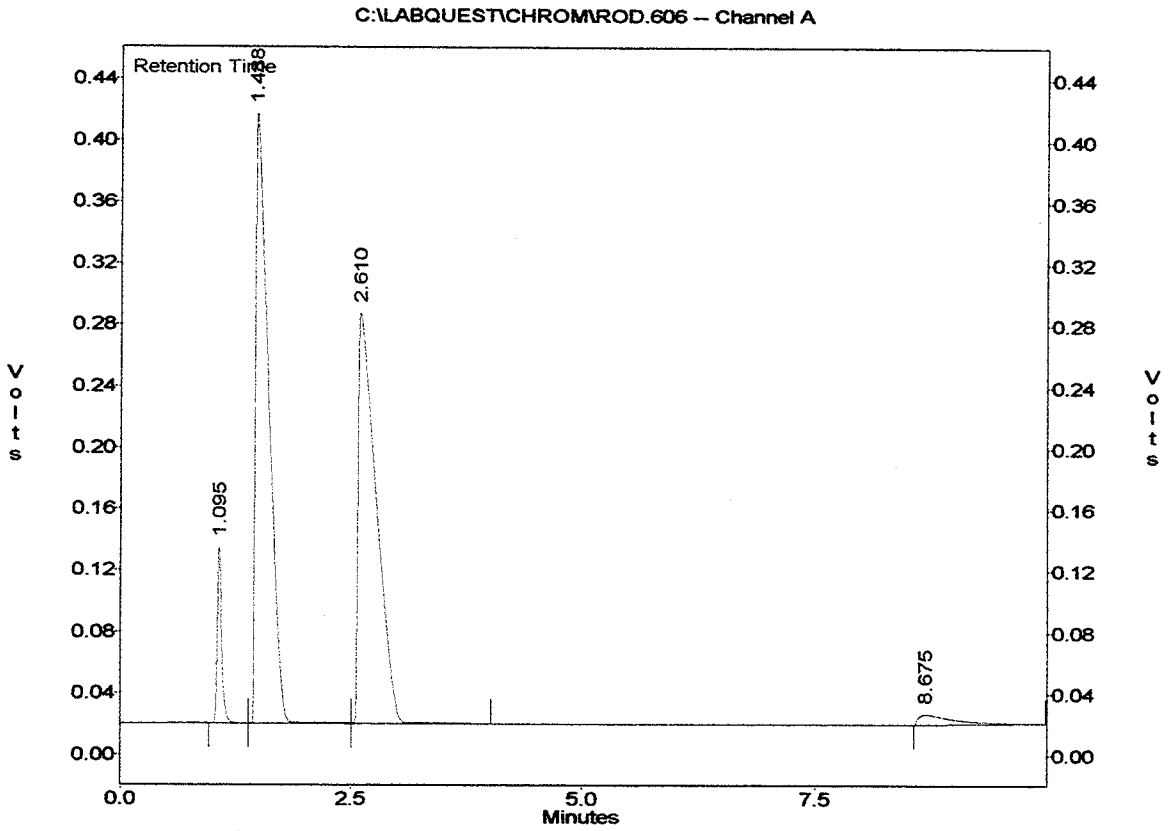
Gas Analysis
File : C:\LABQUEST\CHROM\ROD.606
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 14
Printed : Jun 06, 1998 13:58:58



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.462	4437186.0	59.35
3	Carbon Dioxide	2.575	4230029.0	40.65
Totals :			8667215.0	100.00

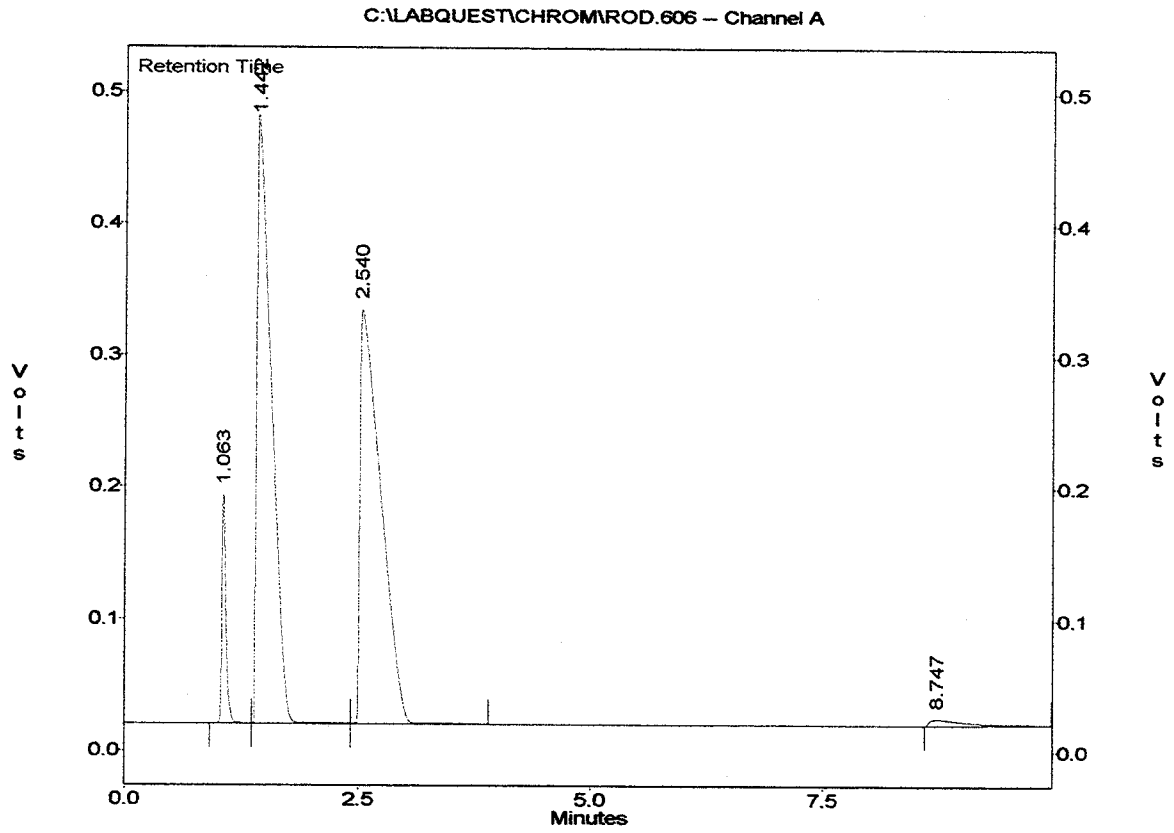
Gas Analysis
File : C:\LABQUEST\CHROM\ROD.606
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 11
Printed : Jun 06, 1998 13:42:28



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.488	3759369.8	59.01
3	Carbon Dioxide	2.610	3633971.0	40.99
Totals :			7393341.0	100.00

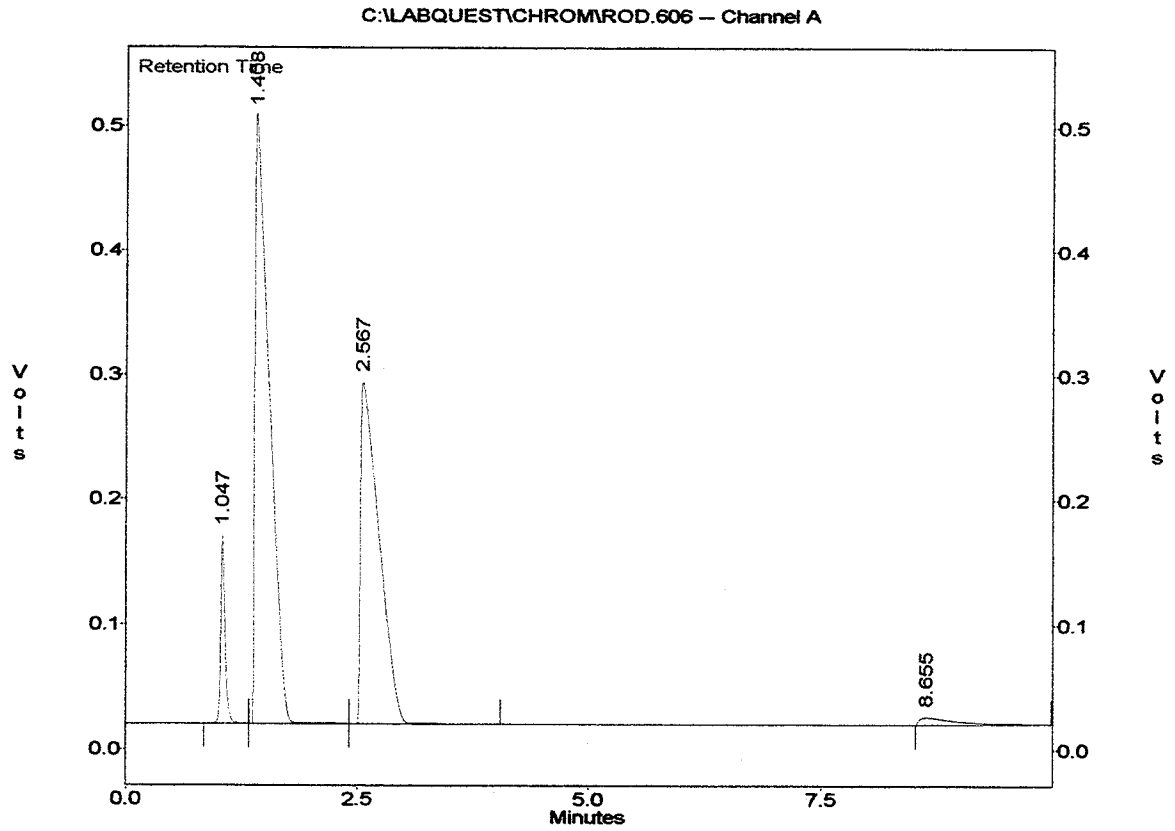
Gas Analysis
File : C:\LABQUEST\CHROM\ROD.606
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 8
Printed : Jun 06, 1998 13:21:33



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.442	4820590.5	58.50
3	Carbon Dioxide	2.540	4760156.5	41.50
Totals :			9580747.0	100.00

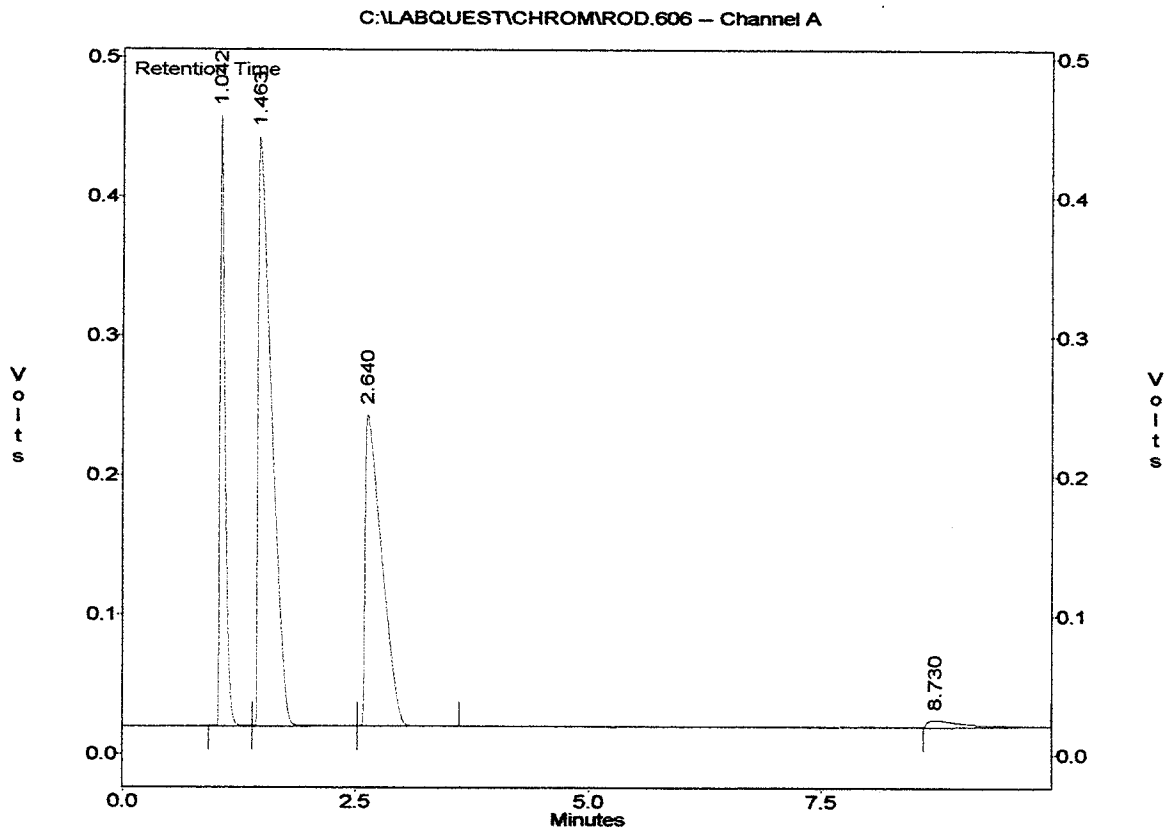
Gas Analysis
File : C:\LABQUEST\CHROM\ROD.606
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 5
Printed : Jun 06, 1998 13:05:53



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.408	5293552.5	66.13
3	Carbon Dioxide	2.567	3772505.0	33.87
Totals :			9066058.0	100.00

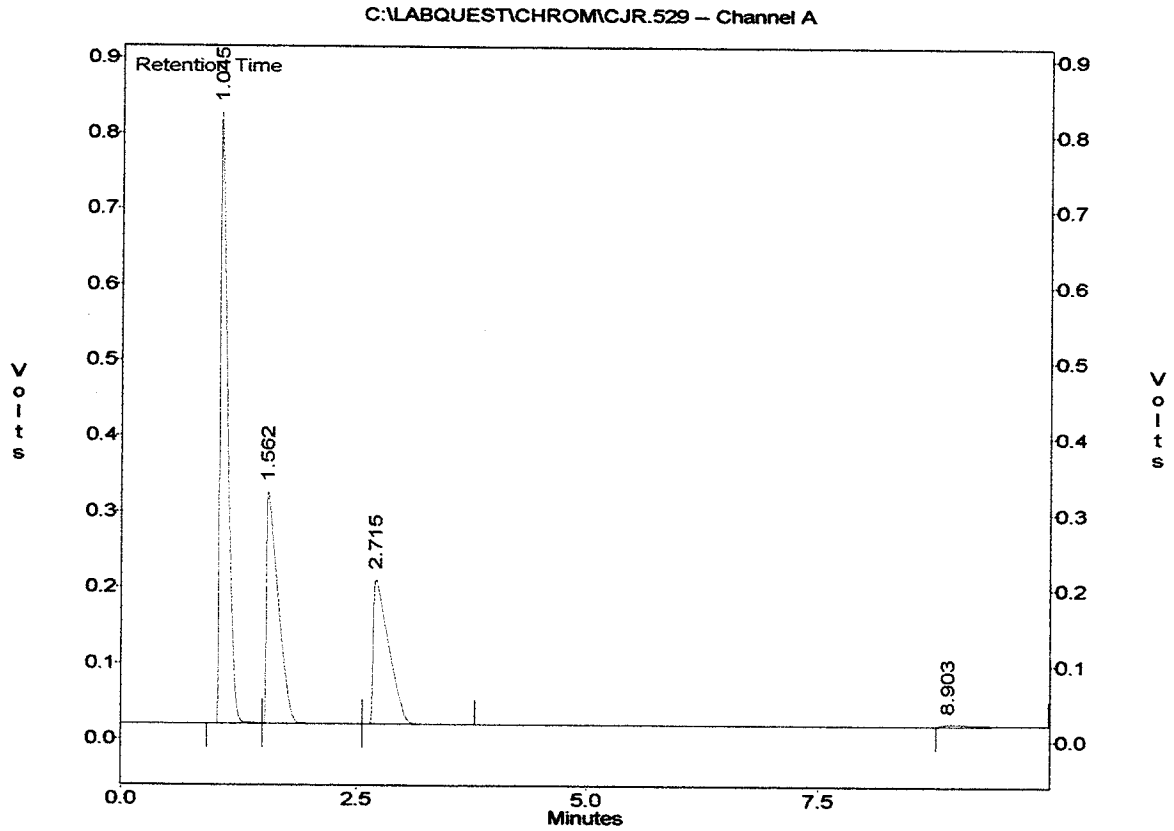
Gas Analysis
File : C:\LABQUEST\CHROM\ROD.606
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL / 2
Printed : Jun 06, 1998 12:47:11



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.463	4211825.0	68.16
3	Carbon Dioxide	2.640	2738296.5	31.84
Totals :			6950121.5	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\CJR.529
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL #1
Printed : May 29, 1998 19:03:47



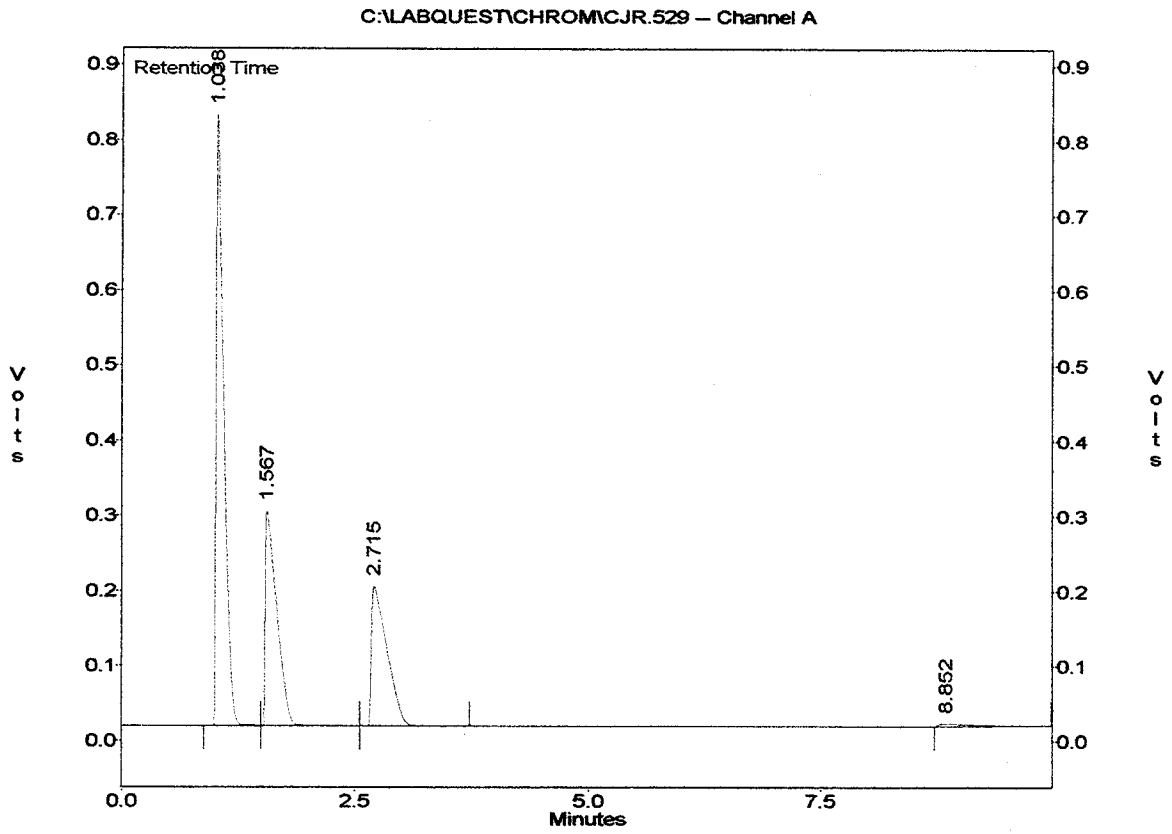
Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.562	2638135.8	62.41
3	Carbon Dioxide	2.715	2211822.8	37.59

Totals :

	4849958.5	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\CJR.529
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL #4
Printed : May 29, 1998 19:31:11



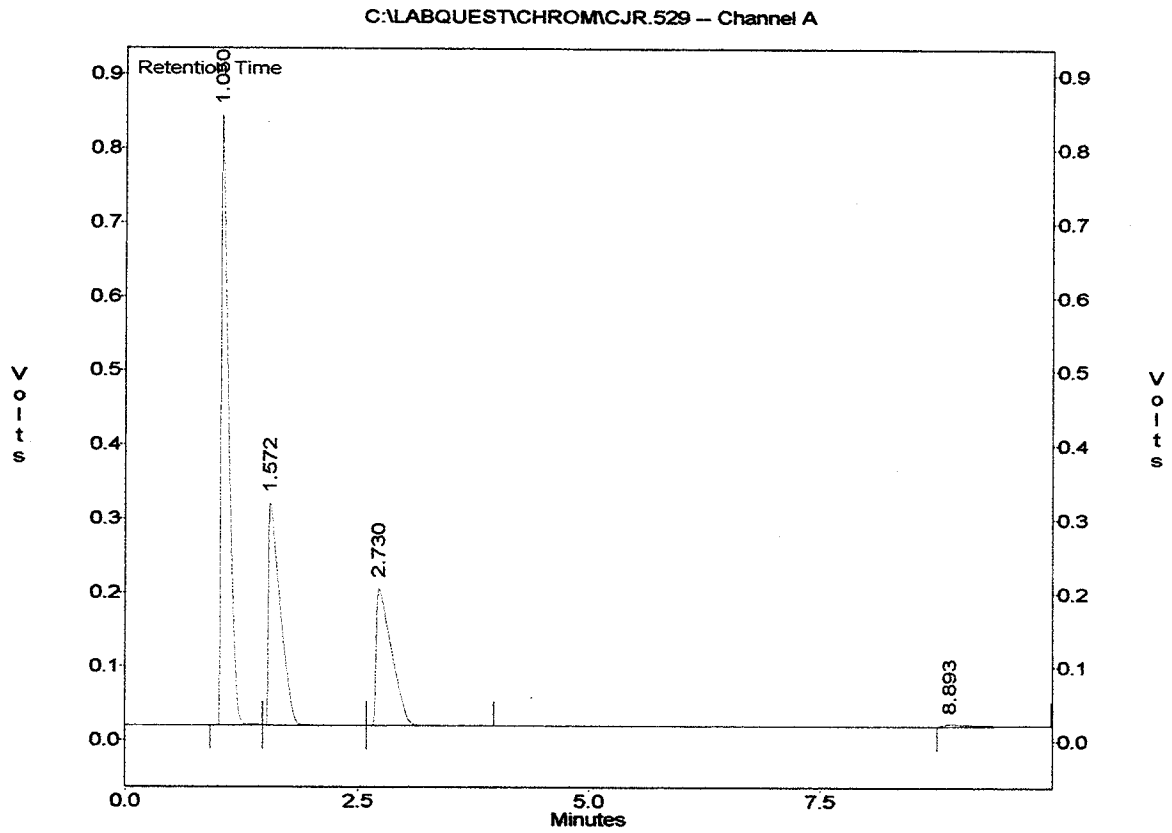
Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.567	2387867.3	60.99
3	Carbon Dioxide	2.715	2125146.0	39.01

Totals :

	4513013.0	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\CJR.529
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL #7
Printed : May 29, 1998 20:07:53



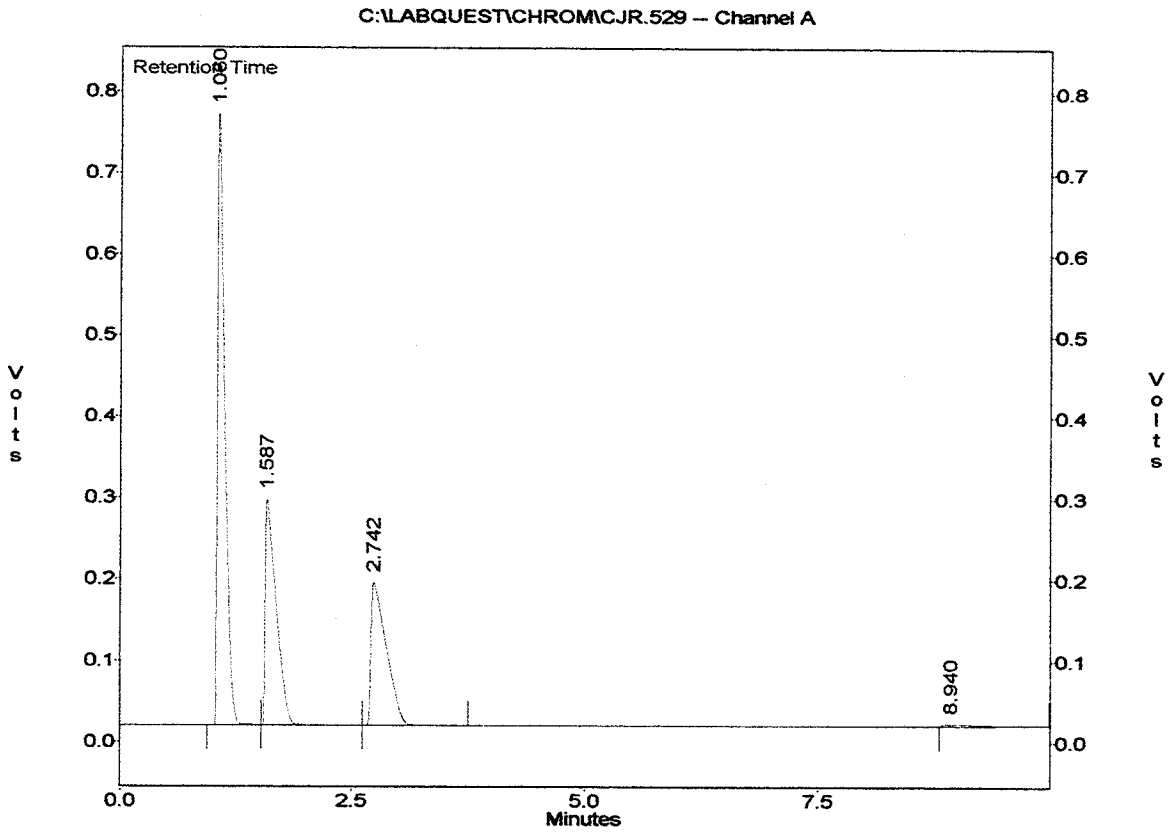
Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.572	2584103.3	62.76
3	Carbon Dioxide	2.730	2133925.0	37.24

Totals :

	4718028.0	100.00

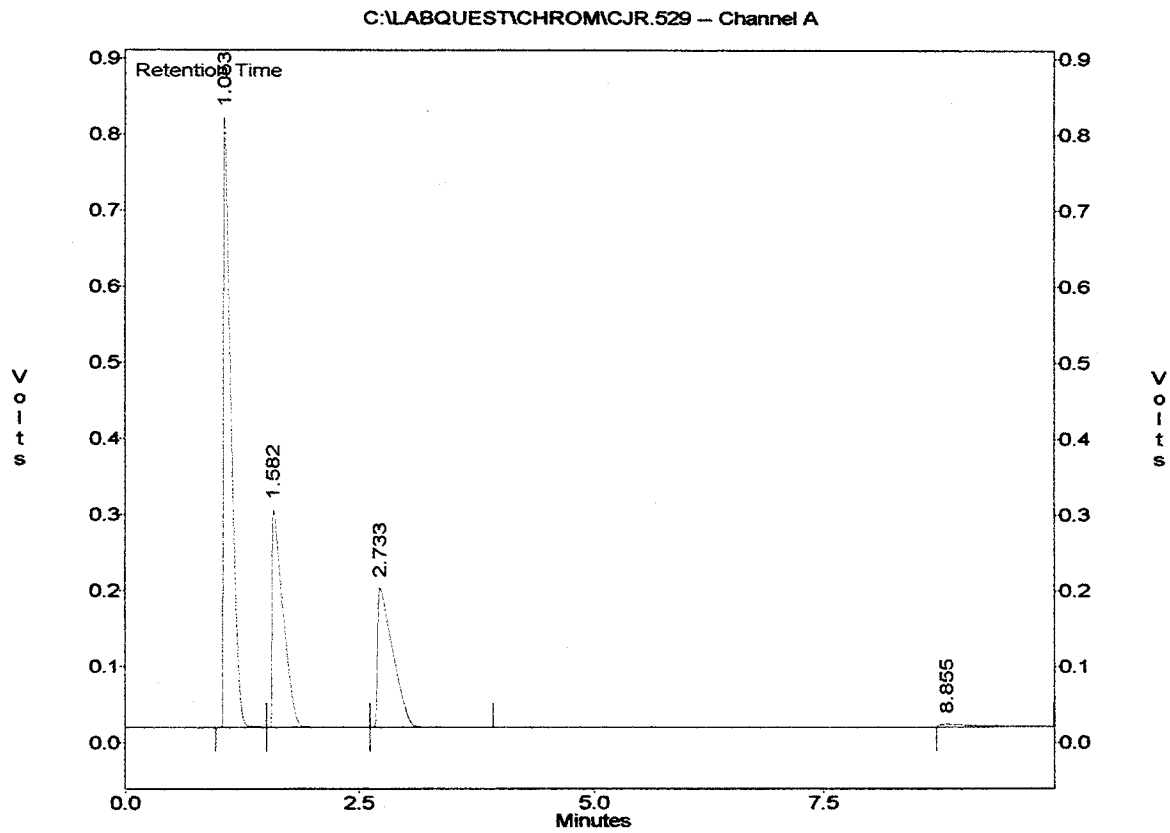
Gas Analysis
File : C:\LABQUEST\CHROM\CJR.529
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL #10
Printed : May 29, 1998 20:30:15



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.587	2262604.5	61.85
3	Carbon Dioxide	2.742	1942272.1	38.15
Totals :			4204876.5	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\CJR.529
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL #13
Printed : May 29, 1998 20:46:19



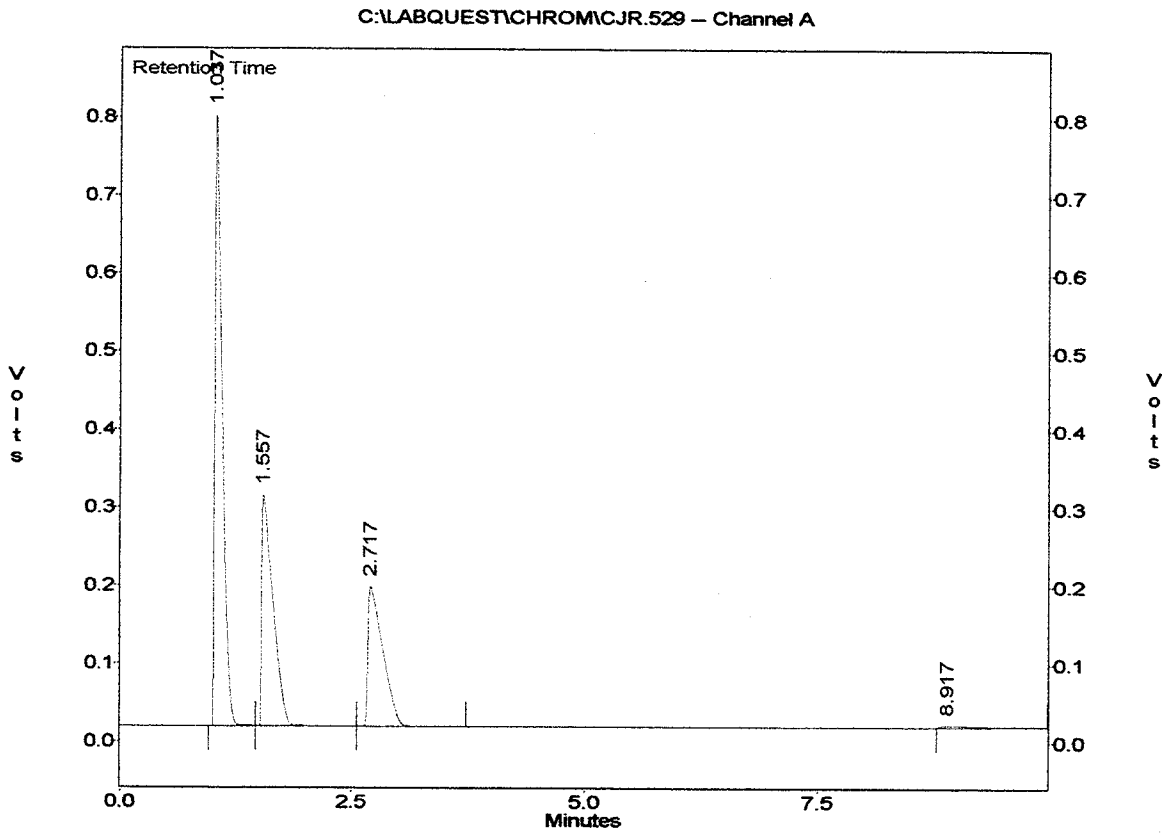
Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.582	2361568.3	61.30
3	Carbon Dioxide	2.733	2075262.1	38.70

Totals :

	4436830.5	100.00

Gas Analysis
File : C:\LABQUEST\CHROM\CJR.529
Method : C:\LABQUEST\METHODS\biogas.MET
Sample ID : NREL #16
Printed : May 29, 1998 21:08:24



Channel A Results

Peak Number	Peak Name	Retention Time	Area	Norm Conc
2	Methane	1.557	2523193.3	63.47
3	Carbon Dioxide	2.717	2021501.1	36.53

Totals :

	4544694.5	100.00

Calculated Data

Other Analysis

LABORATORY SOLIDS WORKSHEET

Time	2355									
Date	5/10/95									
Sample I.D.	MTX 7F BR							BF 172014		
Crucible I.D.			1	2	3		4	5	6	
WEIGHTS	**#**	**Equation**								
Crucible	1		1.096	1.102	1.103		1.105	1.101	1.108	
Wet Sample + Crucible	2									
Wet Sample	3	2 - 1	2.923	2.912	2.882		2.891	2.906	2.909	
Dry Sample + Crucible	4									
Dry Sample	5	4 - 1	.140	.125	.127		.084	.078	.076	
Ash Sample + Crucible	6		1.113	1.113	1.115		1.117	1.110	1.117	
Ash Sample	7	6 - 1	.017	.011	.012		012	.09	.9	
Volatile Solids	8	5 - 7	.123	.114	.115		.072	.069	.063	
ANALYSIS										
% Total Solids		(5 / 3) x 100	4.79	4.29	4.41	4.49	2.91	2.68	2.61	2.73
% Volatile Solids		(8 / 5) x 100	87.85	91.20	90.55	89.86	85.71	88.46	88.15	87.44

LABORATORY SOLIDS WORKSHEET

Time		0320							
Date		5/7/98							
Sample I.D.		T. O_2							
Crucible I.D.			1	2	3				
WEIGHTS	#	Equation							
Crucible	1		1.108	1.097	1.100				
Wet Sample + Crucible	2								
Wet Sample	3	2 - 1	2.922	2.940	2.975				
Dry Sample + Crucible	4								
Dry Sample	5	4 - 1	.169	.153	.170				
Ash Sample + Crucible	6		1.147	1.143	1.142				
Ash Sample	7	6 - 1	.039	.046	.042				
Volatile Solids	8	5 - 7	.130	.107	.128				
ANALYSIS									
% Total Solids		(5 / 3) x 100	5.78	5.20	5.71	5.54			
% Volatile Solids		(8 / 5) x 100	76.92	69.93	75.29	74.04			

LABORATORY SOLIDS WORKSHEET

Time	2350								
Date	5/11/98								
Sample I.D.	HYDROLY 3A								
Crucible I.D.	CONTROL		1	2	3				
WEIGHTS	**#**	**Equation**							
Crucible	1		1.103	1.105	1.116				
Wet Sample + Crucible	2								
Wet Sample	3	2 - 1	2.901	2.901	2.906				
Dry Sample + Crucible	4								
Dry Sample	5	4 - 1	.151	.151	.151				
Ash Sample + Crucible	6		1.129	1.132	1.143				
Ash Sample	7	6 - 1	.026	.027	.027				
Volatile Solids	8	5 - 7	.125	.124	.124				
ANALYSIS									
% Total Solids		(5 / 3) x 100	5.21	5.21	5.20	5.20			
% Volatile Solids		(8 / 5) x 100	82.38	82.11	82.11	82.33			

LABORATORY SOLIDS WORKSHEET

Time			5-30-98						
Date			NOON						
Sample I.D.			Bacto Peptone						
Crucible I.D.			1	2	3				
WEIGHTS	**#**	**Equation**							
Crucible	1		0.991	0.984	1.024				
Wet Sample + Crucible	2								
Wet Sample	3	2 - 1							
Dry Sample + Crucible	4								
Dry Sample	5	4 - 1	0.170	0.120	0.101				
Ash Sample + Crucible	6		0.999	0.988	1.029				
Ash Sample	7	6 - 1	0.008	0.004	0.005				
Volatile Solids	8	5 - 7	0.162	0.116	0.096				
ANALYSIS									
% Total Solids		(5 / 3) x 100	5.81	6.10	5.09				
% Volatile Solids		(8 / 5) x 100	95.29	96.67	95.05				

%TS = 5.67
%VS = 95.67

```
Temp          90 C
Mode         Auto
             100..0%

  0.0M     2.924 g
  5.0M      87.9 %
 10.0M      75.0 %
 15.0M      62.9 %
 20.0M      51.5 %
 25.0M      40.6 %
 30.0M      30.2 %
 35.0M      21.3 %
 40.0M      13.1 %
 45.0M       6.7 %
Time eff.   47.5Min
             5.81 %
            0.170 g
```

```
Temp          90 C
Mode         Auto
             100..0%

  0.0M     1.966 g
  5.0M      88.5 %
 10.0M      76.3 %
 15.0M      64.8 %
 20.0M      54.1 %
 25.0M      43.9 %
 30.0M      34.3 %
 35.0M      25.2 %
 40.0M      17.0 %
 45.0M      10.3 %
 50.0M       6.3 %
Time eff.   50.5Min
             6.10 %
            0.120 g
```

LABORATORY SOLIDS WORKSHEET

Time			5:30pm						
Date			5-30-98						
Sample I.D.			Terminal Island Sewage Sludge						
Crucible I.D.			1	2	3				
WEIGHTS	#	Equation							
Crucible	1		0.983	0.988					
Wet Sample + Crucible	2								
Wet Sample	3	2 - 1							
Dry Sample + Crucible	4								
Dry Sample	5	4 - 1	0.067	0.064					
Ash Sample + Crucible	6		1.006	1.014					
Ash Sample	7	6 - 1	0.023	0.026					
Volatile Solids	8	5 - 7	0.044	0.038					
ANALYSIS									
% Total Solids		(5 / 3) x 100	3.20	3.01					
% Volatile Solids		(8 / 5) x 100	65.67	59.38					

Temp 90 C
Mode ~~Auto~~
100..0%

0.0M	2.094 g
5.0M	89.5 %
10.0M	76.4 %
15.0M	63.7 %
20.0M	51.6 %
25.0M	39.6 %
30.0M	28.3 %
35.0M	18.2 %
40.0M	10.1 %
45.0M	5.1 %

Time eff. 49.0Min
3.20 %
0.067 g

Temp 90 C
Mode Auto
100..0%

0.0M	2.128 g
5.0M	85.3 %
10.0M	69.9 %
15.0M	55.2 %
20.0M	40.7 %
25.0M	27.0 %
30.0M	15.1 %
35.0M	7.7 %
40.0M	4.0 %

Time eff. 43.0Min
3.01 %
0.064 g

%TS = 3.105
% VS = 62.53

5:30pm 0.5 ml into 9.5 ml water

1:20

				Actual Conc mg/L	
Bkg	0	0			
STD 0.5	45	45		450	
STD 1.0	99	97		970	
Pep 1	1650	277		55,400	
Pep 2	1650	291		58,200	57,400
Pep 3	1650	293		58,600	
7F 1	1650	172		34,400	
7F 2	1650	174		34,800	34,800
7F 3	1650	176		35,200	
TiO_2 1	1650	271	TiO_2 1	54,200	
BF 1	1650	282	TiO_2 2	56,400	55,800
3A 1	1650	284	TiO_2 3	56,800	

Terminal Island Anaerobic digester Sludge

pH = 7.43

BF1	140	28,000	
BF2	141	28,200	28,267
BF3	143	28,600	
3A 1	267	53,400	
3A 2	275	55,000	54,400
3A3	274	54,800	

final pH

1	7.25
2	7.28
3	7.27
4	7.36
5	7.37
6	7.36
7	7.23
8	7.24
9	7.25
10	7.22
11	7.22
12	7.23
13	7.23
14	7.23
15	7.24
16	7.23
17	7.23
18	7.25

COD RESULTS

Secondary (X1000)	Primary CR
#1 - Control Hydrolyzate	
1- 63	
2- 53	54,400
3- 51	
AVG - 55.66	
#2 - BF 772014	
1- 27	
2- 26	28,267
3- 26	
AVG - 26.33	
#3 - MTX 7F BROTH	
1- 31	
2- 34	34,800
3- 32	
AVG - 32.33	
#4 - TiO_2 TREATED	
1- 50	
2- 58	55,800
3- 52	
AVG - 53.33	

Graphics