LIST of SUPPORTING INFORMATION

Highly Efficient Catalyzed Aerobic Oxidations of Benzylic Alcohols in Water

Renhua Liu, Chunyan Dong, Xinmiao Liang, Xiujuan Wang and Xinquan Hu *

Dalian Institute of Chemical Physics, the Chinese Academy of Sciences, Dalian, 116023,
People's Republic of China

*To whom correspondence should be address.

E-mail: xinquan@ms.dicp.ac.cn

1. Experimental Section-General (2 page).
2. Figure List (3 pages)
3. GC diagrams and ^1H NMR spectra (57 pages)

SUPPORTING INFORMATION

Highly Efficient Catalyzed Aerobic Oxidations of Benzylic Alcohols in Water

Renhua Liu, Chunyan Dong, Xinmiao Liang, Xiujuan Wang and Xinquan Hu *

Dalian Institute of Chemical Physics, the Chinese Academy of Sciences, Dalian, 116023, People's Republic of China

*To whom correspondence should be address.

E-mail: xinquan@ms.dicp.ac.cn

Experimental Section

General. Equipment and Material All experiments were carried out in a closed Teflon-lined 316L stainless steel autoclave (300 mL), the initial atmospheric air in the autoclave did not exchange for all oxidations. All products in this research are known compounds. ^1H NMR spectrum (400 MHz) was recorded using CDCl_3 as the solvent with TMS as an internal reference. Conversions and selectivities were determined by gas chromatography (GC) analysis. GC-MS was performed on GC with a mass detector. TEMPO, N-Bromosuccinimide (NBS) and 1,3-dibromo-5,5-dimethylhydantoin (DBDMH), all alcohol substrates were commercially available, directly used for oxidation without further purification.

^1H NMR spectra were recorded using isolated products in this method, except that 2-thiophene carboxaldehyde was purified via micro distillation apparatus.

General GC conditions for primary alcohols: HP-5 column, 30m x 0.32mm (id); FID detector, 250 °C; injection: 250 °C; carrier gas: nitrogen; carrier gas rate: 1.2 mL / min, area normalization.

Benzyl alcohol, 2-methyl-benzyl alcohol, 3-methyl-benzyl alcohol, 4-methyl-benzyl alcohol, 2-chloro-benzyl alcohol, 3-chloro-benzyl alcohol, 4-chloro-benzyl alcohol, 3-pyridine methanol and their corresponding aldehydes were detected under a condition as: column temperature: 100 °C for 20 minutes, programmed to 250 °C in a rate of 15 °C / min. 1,3-dibromo-5,5-dimethylhydantoin and its de-brominated 5,5-dimethylhydantoin were also detected under this condition, and their corresponding peak areas were deleted in the GC diagrams.

2-Thionyl methanol and its corresponding aldehyde were performed under a condition as: 80 °C for 20 minutes, programmed to 250 °C in a rate of 15 °C / min..

General GC conditions for secondly alcohols: FFAP column, 30m x 0.32mm (id); FID detector, 250 °C; injection: 250 °C; carrier gas: nitrogen; carrier gas rate: 1.2 mL / min; area normalization.

α -Methyl-benzyl alcohol, 2-octanol, cyclohexanol and their corresponding ketones were detected under a condition as: column temperature: 100 °C for 5 minutes, programmed to 250 °C in a rate of 10 °C / min.

Mixed alcohols and their corresponding carbonyl compounds were detected under a condition as: column temperature: 100 °C for 5 minutes, programmed to 250 °C in a rate of 10 °C / min.

Figure List

- Figure 1. GC diagram of benzyl alcohol.
- Figure 2. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of NBS and 4 mol% of NaNO₂ in 10 mL of CH₂Cl₂.
- Figure 3. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH and 4 mol% of NaNO₂ in 10 mL of CH₂Cl₂.
- Figure 4. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of CH₂Cl₂ and 1.0 mL of H₂O
- Figure 5. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of CH₂Cl₂ and 2.0 mL of H₂O
- Figure 6. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of CH₂Cl₂ and 0.5 mL of H₂O
- Figure 7. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of CH₂ClCH₂Cl and 0.5 mL of H₂O
- Figure 8. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of PhCl and 0.5 mL of H₂O
- Figure 9. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of PhF and 0.5 mL of H₂O
- Figure 10. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of toluene and 0.5 mL of H₂O
- Figure 11. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of α,α,α -trifluorotoluene and 0.5 mL of H₂O
- Figure 12. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of MeCN and 0.5 mL of H₂O
- Figure 13. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of ^tBuOH and 0.5 mL of H₂O
- Figure 14. GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of AcOH and 0.5 mL of H₂O
- Figure 15. GC diagram of oxidation of 10 mmol of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of

- H₂O
- Figure 16. GC diagram of oxidation of 10 mmol of benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 17. GC diagram of oxidation of 5 mmol benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 18. GC diagram of oxidation of 20 mmol benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 19. GC diagram of oxidation of 30 mmol benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 20. GC diagram of oxidation of 40 mmol benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 21. GC diagram of oxidation of 30 mmol benzyl alcohol with 0.9 MPa of air at 80 °C for 1.5 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 22. ¹H NMR spectrum of benzaldehyde
- Figure 23. GC diagram of 4-methyl benzyl alcohol
- Figure 24. GC diagram of oxidation of 30 mmol of 4-methyl benzyl alcohol with 0.9 MPa of air at 80 °C for 2 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 25. ¹H NMR spectrum of 4-tolualdehyde
- Figure 26. GC diagram of 3-methyl benzyl alcohol
- Figure 27. GC diagram of oxidation of 30 mmol of 3-methyl benzyl alcohol with 0.9 MPa of air at 80 °C for 2 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 28. ¹H NMR spectrum of 3-tolualdehyde
- Figure 29. GC diagram of 2-methyl benzyl alcohol
- Figure 30. GC diagram of oxidation of 30 mmol of 2-methyl benzyl alcohol with 0.9 MPa of air at 80 °C for 2.5 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 31. ¹H NMR spectrum of 2-tolualdehyde
- Figure 32. GC diagram of 4-chloro benzyl alcohol
- Figure 33. GC diagram of oxidation of 30 mmol of 4-chloro benzyl alcohol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 34. ¹H NMR spectrum of 4-chloro-benzaldehyde
- Figure 35. GC diagram of 3-chloro benzyl alcohol
- Figure 36. GC diagram of oxidation of 30 mmol of 3-chlorobenzyl alcohol with 0.9 MPa of air

- at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 37. ¹H NMR spectrum of 3-chlorobenzaldehyde
- Figure 38. GC diagram of 2-chloro benzyl alcohol
- Figure 39. GC diagram of oxidation of 30 mmol of 2-chlorobenzyl alcohol with 0.9 MPa of air at 80 °C for 7 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 40. ¹H NMR spectrum of 2-chloro benzaldehyde
- Figure 41. GC diagram of α-methyl benzyl alcohol
- Figure 42. GC diagram of oxidation of 30 mmol of α-methyl benzyl alcohol with 0.9 MPa of air at 80 °C for 4 h, 1 mol% of TEMPO, 4 mol% of DBDMH and 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 43. ¹H NMR spectrum of acetophenone
- Figure 44. GC diagram of 3-pyridine methanol
- Figure 45. GC diagram of oxidation of 30 mmol of 3-pyridine methanol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O and 2 mL of AcOH
- Figure 46. ¹H NMR spectrum of 3-pyridine carboxaldehyde
- Figure 47. GC diagram of 2-thiophene methanol
- Figure 48. GC diagram of oxidation of 30 mmol of 2-thiophene methanol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 2 mol% of PyHBr₃, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 49. ¹H NMR spectrum of 2-thiophene carboxaldehyde
- Figure 50. GC diagram of cyclohexanol
- Figure 51. GC diagram of oxidation of 30 mmol of cyclohexanol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 52. GC diagram of 2-octanol
- Figure 53. GC diagram of oxidation of 30 mmol of 2-octanol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH and 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 54. GC diagram of oxidation of 30 mmol of 2-octanol with 0.9 MPa of air at 80 °C for 6 h, 5 mol% of TEMPO, 20 mol% of DBDMH and 20 mol% of NaNO₂ in 10 mL of H₂O
- Figure 55. ¹H NMR spectrum of 2-octanone
- Figure 56. GC diagram of oxidation of mixed alcohols (15 mmol benzyl alcohol and 15 mmol of 2-octanol) with 0.9 MPa of air at 80 °C for 1.5 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O
- Figure 57. GC diagram of oxidation of mixed alcohols (15 mmol benzyl alcohol and 15 mmol of cyclohexanol) with 0.9 MPa of air at 80 °C for 1.5 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112407.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min

```
=====
Injection Date : 11/24/03 12:17:37 PM
Sample Name    : C6H5CH2OH
Acq. Operator  : C
Location       : Vial 2
Inj           : 1
Inj Volume     : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/24/03 12:44:34 PM by C
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 5/20/04 2:51:43 PM
                (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	5.139	BB	0.0454	1151.89136	409.01382	1.000e2

Totals : 1151.89136 409.01382

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 5/20/04 2:51:47 PM

Page 1 of 1

Figure 1.
GC diagram of benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0041902.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, FLOW: 1.2mL/min
1TEMP0.4MBS, 4MNaNO2, 10ml/CH2Cl2,
4atm/air, 80C, 1h

Injection Date : 4/19/04 11:20:03 AM
Sample Name : Benzyl Alcohol
Acq. Operator :
Location : Vial 2
In1 : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 4/19/04 9:35:01 AM
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 4/20/04 1:56:36 PM
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.119	BB	0.0335	214.23058	106.26511	11.10309
2	5.167	BB	0.0453	1693.24841	559.08563	87.75725
3	6.736	BB	0.0536	4.39240	1.32787	0.22765
4	36.532	BB	0.0733	17.59697	3.67837	0.91201

Totals : 1929.46835 670.35698

Results obtained with enhanced integrator!

Instrument 1 4/20/04 2:01:51 PM

Page 1 of 1

Figure 2.

GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of NBS and 4 mol% of NaNO₂

Data File C:\HPCHEM\1\DATA\1\0041901.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
 100C(20min)--15C/min--250C, FLOW: 1.2 mL/min
 1TEMP0, 4DBDMH, 4NaNO₂, 10 mL/CH₂Cl₂,
 4atm/air, 80C, 1h

Injection Date : 4/19/04 10:22:22 AM
 Sample Name : Benzyl Alcohol
 Acq. Operator :
 Location : Vial 2
 Inj : 1
 Inj Volume : Manually
 Acq. Method : C:\HPCHEM\1\METHODS\205.M
 Last changed : 4/19/04 9:35:01 AM
 (modified after loading)
 Analysis Method : C:\HPCHEM\1\METHODS\205.M
 Last changed : 4/20/04 1:56:36 PM
 (modified after loading)

Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.122	BB	0.0310	273.28845	135.63338	10.03281
2	5.180	BB	0.0482	2344.65649	715.68958	86.07568
3	6.738	BB	0.0507	94.89918	28.97915	3.48388
4	28.173	BB	0.0390	4.98862	2.00648	0.18314
5	36.540	BB	0.0729	6.11492	1.26744	0.22449

Totals : 2723.94766 883.59603

Results obtained with enhanced integrator!

Instrument 1 4/20/04 1:56:52 PM

Page 1 of 2

Figure 3.

GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH and 4 mol% of NaNO₂

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00041904.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, FLOW: 1.2 mL/min
ITEMPO, 4 DBDMH, 4 NaNO₂, 10 mL/CH₂Cl₂, 1.0 mL/H₂O,
4 atm/air, 80C, 1h

Injection Date : 4/19/04 1:45:56 PM
Sample Name : Benzyl Alcohol Location : Vial 2
Acq. Operator : In1 : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 4/19/04 9:35:01 AM
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 4/20/04 1:56:36 PM
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.132	BB	0.0348	1422.97729	668.89923	1.000e2

Totals : 1422.97729 668.89923

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 4/20/04 2:04:19 PM

Page 1 of 1

Figure 4.

GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ and 1.0 mL of H₂O

Data File C:\HPCHEM\1\DATA\1\0041905.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, FLOW: 1.2 mL/min
1TEMP0, 4DBDMH, 4NaNO2, 10 mL/CH2Cl2, 2.0 mL/H2O,
4atm/air, 80C, 1h

=====

Injection Date	: 4/19/04 2:38:33 PM	Location	: Vial 2
Sample Name	: Benzyl Alcohol	Inj	: 1
Acq. Operator	:	Inj Volume	: Manually
Acq. Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 4/19/04 9:35:01 AM		
	(modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 4/20/04 1:56:36 PM		
	(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.121	EV	0.0354	1228.40417	563.85400	72.53431
2	5.136	BB	0.0403	465.14499	178.98608	27.46569

Totals : 1693.54916 742.84009

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 4/20/04 2:09:19 PM

Page 1 of 1

Figure 5.

GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ and 2.0 mL of H₂O

Data File C:\HPCHEM\1\DATA\1\0041906.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, FLOW: 1.2 mL/min
1TEMPO, 4DBDMH, 4NaNO₂, 10 mL/CH₂Cl₂, 0.5 mL/H₂O,
4atm/air, 80C, 1h

=====

Injection Date	: 4/19/04 3:39:57 PM	Location	: Vial 2
Sample Name	: Benzyl Alcohol	Inj	: 1
Acq. Operator	:	Inj Volume	: Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 4/19/04 9:35:01 AM
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 4/20/04 1:56:36 PM
(modified after loading)

=====
Area Percent Report
=====

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000
Sample Amount	:	1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.130	BB	0.0357	2246.34741	1020.78741	1.000e2

Totals : 2246.34741 1020.78741

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 4/20/04 2:10:27 PM

Page 1 of 1

Figure 6.

GC diagram of oxidation of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ and 0.5 mL of H₂O

Supporting Information

Data File C:\HPCHEM\1\DATA\DONG\00061601.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMP0, 40SDMH, 4NaNO2, 0.5mL/H2O, 10mL/C2H4Cl2,
4atm/air, 80C, 1h

=====

Injection Date	: 6/16/04 8:13:32 PM	Location	: Vial 2
Sample Name	: Benzyl Alcohol	Inj	: 1
Acq. Operator	:	Inj Volume	: Manually
Acq. Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 6/16/04 1:17:09 PM		
Analysis Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 7/19/04 8:46:36 AM		

(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.188	ES	0.0403	5050.73779	1942.50098	1.000e2

Totals : 5050.73779 1942.50098

Results obtained with enhanced integrator!

*** End of Report ***

Figure 7.
GC diagram of oxidation of benzyl alcohol in $\text{CH}_2\text{ClCH}_2\text{Cl}$

Supporting Information

Data File C:\HPCHEM\1\DATA\DOMG\00061608.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMP0, 4DBDMH, 4NaNO2, 0.5mL/H2O, 10mL/C6H5Cl,
4atm/air, 80C, 1h

Injection Date : 6/16/04 4:15:00 PM
Sample Name : Benzyl Alcohol Location : Vial 2
Acq. Operator : Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 6/16/04 1:17:09 PM
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/19/04 8:50:19 AM
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.163	BB	0.0315	1509.07312	733.00867	98.40331
2	4.921	BB	0.0370	24.48611	10.58067	1.59669

Totals : 1533.55923 743.58934

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 8:50:24 AM

Page 1 of 1

Figure 8.
GC diagram of oxidation of benzyl alcohol in PhCl

Supporting Information

Data File C:\HPCHEM\1\DATA\DMG\00062703.D

Sample Name: Benzyl alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMP0, 4DBDMH, 4NANO2, 10mL/C6H5F, 0.5mL/H2O
9atm/air, 80C, 1h

```
=====
Injection Date   : 6/27/04 2:08:06 PM
Sample Name      : Benzyl alcohol
Acq. Operator    :
Location         : Vial 2
Inj              : 1
Inj Volume       : Manually

Acq. Method      : C:\HPCHEM\1\METHODS\205.M
Last changed     : 6/27/04 10:37:41 AM
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\205.M
Last changed     : 7/19/04 8:56:48 AM
                  (modified after loading)
=====
```

```
=====
Area Percent Report
=====
Sorted By       : Signal
Multiplier      : 1.0000
Dilution        : 1.0000
Sample Amount   : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak RetTime Type Width Area Height Area
# [min] [min] [nA*s] [nA] %
-----|-----|-----|-----|-----|
1 4.153 BB 0.0341 2263.64526 993.46759 1.000e2

Totals : 2263.64526 993.46759

Results obtained with enhanced integrator!
=====
*** End of Report ***
=====
```

Instrument 1 7/19/04 8:56:53 AM

Page 1 of 1

Figure 9.
GC diagram of oxidation of benzyl alcohol inPhF

Supporting Information

Data File C:\HPCHEM\1\DATA\DOMG\00061606.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMP0, 4DBDMH, 4NaNO2, 0.5mL/H2O, 10mL/C6H5CH3,
4atm/air, 80C, 1h

```

=====
Injection Date : 6/16/04 2:29:06 PM
Sample Name    : Benzyl Alcohol
Acq. Operator  :                               Location : Vial 2
                                           In1 : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 6/16/04 1:17:09 PM
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 7/19/04 8:50:19 AM
                (modified after loading)
  
```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.164	BB	0.0315	1344.67053	653.87061	1.000e2

Totals : 1344.67053 653.87061

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 8:51:31 AM

Page 1 of 1

Figure 10.
GC diagram of oxidation of benzyl alcohol in toluene

Supporting Information

Data File C:\HPCHEM\1\DATA\DONG\DO062702.D

Sample Name: Benzyl alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2ml/min
1TEMP0, 40SDMH, 40Na02, 10ml/C6H5CF3, 0.5ml/H2O
9atm/air, 80C, 1h

```
=====
Injection Date : 6/27/04 11:37:07 AM
Sample Name    : Benzyl alcohol
Acq. Operator  :                               Location : Vial 2
                                           Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 6/27/04 10:37:41 AM
               (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 7/19/04 8:55:46 AM
               (modified after loading)
=====
```

```
=====
                        Area Percent Report
=====
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Sample Amount  :      1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak RetTime Type Width Area Height Area
# [min] [min] [min] [nA*s] [nA] %
----|-----|----|-----|-----|-----|
1 4.158 BB 0.0338 1084.80298 530.78101 1.000e2

Totals : 1084.80298 530.78101

Results obtained with enhanced integrator!
=====
*** End of Report ***
=====
```

Instrument 1 7/19/04 8:55:49 AM

Page 1 of 1

Figure 11.
GC diagram of oxidation of benzyl alcohol in α,α,α -trifluorotoluene

Supporting Information

Data File C:\HPCHEM\1\DATA\DONG\00061604.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMP0, 40SDMH, 4NaNO2, 0.5mL/H2O, 10mL/CH3CN,
4atm/air, 80C, 1h

```
=====
Injection Date   : 6/16/04 12:52:41 PM
Sample Name      : Benzyl Alcohol
Acq. Operator    :                               Location : Vial 2
                                                    Inj : 1
                                                    Inj Volume : Manually
Acq. Method      : C:\HPCHEM\1\METHODS\205.M
Last changed     : 6/16/04 1:17:09 PM
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\205.M
Last changed     : 7/19/04 8:50:19 AM
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.156	BB	0.0314	1201.34497	586.05902	1.000e2

Totals : 1201.34497 586.05902

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 8:52:22 AM

Page 1 of 1

Figure 12.
GC diagram of oxidation of benzyl alcohol in MeCN

Supporting Information

Data File C:\HPCHEM\1\DATA\BONG\B0061602.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMP0, 40SDMH, 4NaNO2, 0.5mL/H2O, 10mL/BuOH,
4atm/air, 80C, 1h

=====

Injection Date	: 6/16/04 8:59:34 PM	Location	: Vial 2
Sample Name	: Benzyl Alcohol	Inj	: 1
Acq. Operator	:	Inj Volume	: Manually
Acq. Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 6/16/04 1:17:09 PM		
Analysis Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 7/19/04 8:53:14 AM		

(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.179	BB	0.0370	3179.26538	1371.90210	85.96873
2	5.189	BB	0.0392	518.89954	207.31200	14.03127

Totals : 3698.16492 1579.21410

Results obtained with enhanced integrator!

*** End of Report ***

Fig 13
GC diagram of oxidation of benzyl alcohol in ^tBuOH

Supporting Information

Data File C:\HPCHEM\1\DATA\BONG\B0061609.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMP0, 4DBDMH, 4NaNO2, 0.5mL/H2O, 10mL/AcOH,
4atm/air, 80C, 1h

```

=====
Injection Date   : 6/16/04 5:01:30 PM
Sample Name     : Benzyl Alcohol
Acq. Operator   :
Location        : Vial 2
In1             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 6/16/04 1:17:09 PM
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 7/19/04 8:54:24 AM
                  (modified after loading)
  
```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.173	BB	0.0323	1994.53967	936.71472	1.000e2

Totals : 1994.53967 936.71472

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 8:54:28 AM

Page 1 of 1

Fig 14
GC diagram of oxidation of benzyl alcohol in AcOH

Supporting Information

Data File C:\HPCHEM\1\DATA\DOMG\00061612.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMP0, 4DBDMH, 4NaNO2, 10mL/H2O,
4atm/air, 80C, 1h

=====

Injection Date	: 6/16/04 7:21:43 PM	Location	: Vial 2
Sample Name	: Benzyl Alcohol	Inj	: 1
Acq. Operator	:	Inj Volume	: Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 6/16/04 1:17:09 PM
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/19/04 8:58:24 AM
(modified after loading)

=====

Area Percent Report

=====

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000
Sample Amount	:	1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.186	EV	0.0460	6227.39697	2171.51392	22.63254
2	5.376	BB	0.1058	2.06355e4	2512.07349	74.99657
3	36.585	BB	0.0723	652.35785	138.95197	2.37090

Totals : 2.75152e4 4822.53937

Results obtained with enhanced integrator!

=====

*** End of Report ***

Instrument 1 7/19/04 8:58:31 AM

Page 1 of 1

Fig 15

GC diagram of oxidation of 10 mmol of benzyl alcohol with 0.4 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Supporting Information

Data File C:\HPCHEM\1\DATA\DOMG\00062102.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
ITEMPO, 4DBDMH, 4NaNO₂, 10mL/H₂O,
9atm/air, 80C, 1h

```
=====
Injection Date : 6/21/04 4:12:21 PM
Sample Name    : Benzyl Alcohol
Acq. Operator  :                               Location : Vial 2
                                           Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 6/21/04 3:55:46 PM
               (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 7/19/04 9:00:37 AM
               (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.146	BB	0.0340	555.12402	270.40610	58.76452
2	5.165	BB	0.0392	389.53445	155.51433	41.23548

Totals : 944.65846 425.92043

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:00:40 AM

Page 1 of 1

Fig 16

**GC diagram of oxidation of 10 mmol of benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h,
1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O**

Supporting Information

Data File C:\HPCHEM\1\DATA\DOMG\00062205.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
ITEMPO, 4DBDMH, 4NaNO₂, 10mL/H₂O, 5mmol/C₆H₅CH₂OH,
9atm/air, 80C, 1h

```

=====
Injection Date : 6/22/04 4:36:58 PM
Sample Name    : Benzyl Alcohol
Acq. Operator  :                               Location : Vial 2
                                           Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 6/22/04 4:51:38 PM
               (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 7/19/04 9:00:37 AM
               (modified after loading)
  
```

```

=====
                        Area Percent Report
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.149	EB	0.0307	99.56452	49.98580	14.70574
2	5.175	EB	0.0420	577.48071	228.74501	85.29426

Totals : 677.04523 278.73081

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:01:40 AM

Page 1 of 1

Fig 17

GC diagram of oxidation of 5 mmol of benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Supporting Information

Data File C:\HPCHEM\1\DATA\BONG\B0062202.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
1TEMPO, 4DBDMH, 4NaNO₂, 10mL/H₂O, 20mmol/C₆H₅C₂H₂OH,
9atm/air, 80C, 1h

```
=====
Injection Date   : 6/22/04 1:47:16 PM
Sample Name      : Benzyl Alcohol
Acq. Operator    :
Location         : Vial 2
Inj              : 1
Inj Volume       : Manually

Acq. Method      : C:\HPCHEM\1\METHODS\205.M
Last changed     : 6/22/04 11:12:30 AM
                  (modified after loading)
Analysis Method   : C:\HPCHEM\1\METHODS\205.M
Last changed     : 7/19/04 9:02:25 AM
                  (modified after loading)
=====
```

```
=====
Area Percent Report
=====
Sorted By       : Signal
Multiplier      : 1.0000
Dilution        : 1.0000
Sample Amount   : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak RetTime Type Width Area Height Area
# [min] [min] [min] [nA*s] [nA] %
-----|-----|-----|-----|-----|-----
1 4.171 BP 0.0368 2783.87573 1209.99451 82.78877
2 5.181 BB 0.0406 578.74915 220.87321 17.21123

Totals : 3362.62488 1430.86671

Results obtained with enhanced integrator!
=====
*** End of Report ***
```

Instrument 1 7/19/04 9:02:31 AM

Page 1 of 1

Fig 18

**GC diagram of oxidation of 20 mmol of benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h,
1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O**

Supporting Information

Data File C:\HPCHEM\1\DATA\DONG\DO062207.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/C6H5CH2OH,
1TEMP0, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 1h

```
=====
Injection Date   : 6/22/04 10:05:46 PM          Location : Vial 2
Sample Name      : Benzyl Alcohol              Inj       : 1
Acq. Operator    :                               Inj Volume: Manually

Acq. Method      : C:\HPCHEM\1\METHODS\205.M
Last changed     : 6/22/04 4:51:38 PM
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\205.M
Last changed     : 7/19/04 9:03:27 AM
                  (modified after loading)
=====
```

```
=====
Area Percent Report
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [nA*s]	Height [nA]	Area %
1	4.153	BV	0.0402	3602.61445	1389.55249	96.30730
2	5.151	BB	0.0421	138.14232	50.21368	3.69270

Totals : 3740.95677 1439.76617

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:03:30 AM

Page 1 of 1

Fig 19

**GC diagram of oxidation of 30 mmol of benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h,
1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O**

Supporting Information

Data File C:\HPCHEM\1\DATA\DOMG\00062301.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
40mmol/C6H5CH2OH,
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 1h

```

=====
Injection Date : 6/23/04 10:31:19 AM
Sample Name    : Benzyl Alcohol
Acq. Operator  :                               Location : Vial 2
                                           Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 6/23/04 10:15:00 AM
               (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 7/19/04 9:03:27 AM
               (modified after loading)
  
```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.174	EV	0.0348	3505.10791	1497.10352	95.35863
2	5.169	BB	0.0385	170.60329	69.77390	4.64137

Totals : 3675.71120 1566.87742

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:04:24 AM

Page 1 of 1

Fig 20

**GC diagram of oxidation of 40 mmol of benzyl alcohol with 0.9 MPa of air at 80 °C for 1 h,
1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O**

Supporting Information

Data File C:\HPCHEM\1\DATA\DOMG\DOMG62304.D

Sample Name: Benzyl Alcohol

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/C6H5CH2OH,
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 1.5h

```

=====
Injection Date   : 6/23/04 4:03:02 PM
Sample Name     : Benzyl Alcohol
Acq. Operator   :
Location        : Vial 2
Inj             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 6/23/04 10:15:00 AM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 7/19/04 9:03:27 AM
                  (modified after loading)
  
```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [nA*s]	Height [nA]	Area %
1	4.168	BB	0.0336	2759.08691	1231.34070	99.77457
2	5.169	FB	0.0393	6.23397	2.48269	0.22543

Totals : 2765.32086 1233.82339

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:05:04 AM

Page 1 of 1

Fig 21

GC diagram of oxidation of 30 mmol of benzyl alcohol with 0.9 MPa of air at 80 °C for 1.5 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Figure 22.
¹H NMR spectrum of benzaldehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0112009.D

Sample Name: 4-CH3C6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min

```

=====
Injection Date : 11/20/03 3:39:02 PM
Sample Name    : 4-CH3C6H4CH2OH          Location : Vial 2
Acq. Operator  : C                      Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/20/03 3:46:03 PM by C
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 5/20/04 2:50:42 PM
                (modified after loading)
=====
  
```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Tvbe	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.804	BB	0.0870	3266.80127	529.03265	1.000e2

Totals : 3266.80127 529.03265

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 5/20/04 2:50:45 PM

Page 1 of 1

Figure 23.
GC diagram of 4-methyl benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\DONG\DO062504.D

Sample Name: 4-Me-C6H4CH2OH

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/4-Me-C6H4CH2OH,
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 2h

```
=====
Injection Date : 6/25/04 5:06:10 PM
Sample Name    : 4-Me-C6H4CH2OH
Acq. Operator  :                               Location : Vial 2
                                           Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 6/25/04 8:32:41 AM
               (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 7/19/04 9:05:55 AM
               (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.398	BB	0.0571	2413.12915	631.67383	1.000e2

Totals : 2413.12915 631.67383

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:05:59 AM

Page 1 of 1

Figure 24.

**GC diagram of oxidation of 30 mmol of benzyl alcohol with 0.9 MPa of air at 80 °C for 2 h,
1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O**

Figure 25.
¹H NMR spectrum of 4-tolualdehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0112016.D

Sample Name: 3-CH3C6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min

```
=====
Injection Date : 11/21/03 10:32:40 AM
Sample Name    : 3-CH3C6H4CH2OH          Location : Vial 2
Acq. Operator  : C                      Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/21/03 8:37:15 AM by C
                                           (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 5/20/04 2:46:03 PM
                                           (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.841	BV	0.0934	4234.14453	610.79565	1.000e2

Totals : 4234.14453 610.79565

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 5/20/04 2:46:07 PM

Page 1 of 1

Figure 26.
GC diagram of 3-methyl benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\DONG\DO062501.D

Sample Name: 3-Me-C6H4CH2OH

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/3-Me-C6H4CH2OH,
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 2h

```
=====
Injection Date   : 6/25/04 8:36:07 AM
Sample Name      : 3-Me-C6H4CH2OH
Acq. Operator    :
Location         : Vial 2
Inj              : 1
Inj Volume       : Manually

Acq. Method      : C:\HPCHEM\1\METHODS\205.M
Last changed     : 6/25/04 8:32:41 AM
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\205.M
Last changed     : 7/19/04 9:05:55 AM
                  (modified after loading)
=====
```

Area Percent Report

```
-----
Sorted By       : Signal
Multiplier      : 1.0000
Dilution        : 1.0000
Sample Amount   : 1.00000 [ng/ul] (not used in calc.)
-----
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.059	EB	0.0549	2666.38623	734.25342	99.71030
2	7.775	FP	0.0477	7.74689	2.76029	0.28970

Totals : 2674.13312 737.01370

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:06:57 AM

Page 1 of 1

Figure 27.

GC diagram of oxidation of 30 mmol of 3-methyl benzyl alcohol with 0.9 MPa of air at 80 °C for 2 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Figure 28.
¹H NMR spectrum of 3-tolualdehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0112102.D

Sample Name: 2-CH3C6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min

```
=====
Injection Date : 11/21/03 11:58:28 AM
Sample Name    : 2-CH3C6H4CH2OH          Location : Vial 2
Acq. Operator  : C                      Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/21/03 8:37:15 AM by C
                                           (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 5/20/04 2:41:41 PM
                                           (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	8.115	BB	0.0884	2348.73486	400.59509	1.000e2

Totals : 2348.73486 400.59509

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 5/20/04 2:41:47 PM

Page 1 of 1

Figure 29.
GC diagram of 2-methyl benzyl alcohol

Data File C:\HPCHEM\1\DATA\DOMG\00063002.D

Sample Name: 2-Me-C6H4CH2OH

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/2-Me-C6H4CH2OH
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 2.5h

=====

Injection Date	: 6/30/04 2:43:40 PM	Location	: Vial 2
Sample Name	: 2-Me-C6H4CH2OH	In1	: 1
Acc. Operator	:	Inj Volume	: Manually

Acc. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 6/30/04 1:53:54 PM
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/19/04 9:08:00 AM
(modified after loading)

=====
Area Percent Report
=====

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000
Sample Amount	:	1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.066	BB	0.0649	4249.20605	1046.46777	1.000e2

Totals : 4249.20605 1046.46777

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:08:04 AM

Page 1 of 1

Figure 30.

GC diagram of oxidation of 30 mmol of 2-methyl benzyl alcohol with 0.9 MPa of air at 80 °C for 2.5 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Figure 31.
¹H NMR spectrum of 2-tolualdehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\12008.D

Sample Name: 4-ClC6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min

```

=====
Injection Date : 11/20/03 3:04:43 PM
Sample Name    : 4-ClC6H4CH2OH          Location : Vial 2
Acq. Operator  : C                      Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/20/03 1:12:48 PM by C
               : (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 5/20/04 2:49:28 PM
               : (modified after loading)
=====
  
```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Tvbe	Width [min]	Area [pA*s]	Height [pA]	Area %
1	12.885	BB	0.1325	1773.88098	183.91830	1.000e2

Totals : 1773.88098 183.91830

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 5/20/04 2:49:31 PM

Page 1 of 1

Figure 32.
GC diagram of 4-chloro benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\DONG\00070103.D

Sample Name: 4-Cl-C6H4CH2OH

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/4-Cl-C6H4CH2OH
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 7h

```

=====
Injection Date   : 7/1/04 5:21:25 PM
Sample Name     : 4-Cl-C6H4CH2OH
Acq. Operator   :
Location        : Vial 2
Inj             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 7/1/04 1:25:40 PM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 7/19/04 9:09:02 AM
                  (modified after loading)
=====
  
```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
=====
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.711	BB	0.0722	1796.38599	382.93546	1.000e2

Totals : 1796.38599 382.93546

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:09:05 AM

Page 1 of 1

Figure 33.

GC diagram of oxidation of 30 mmol of 4-chlorobenzyl alcohol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Figure 34.
¹H NMR spectrum of 4-chloro-benzaldehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\12403.D

Sample Name: 3-CLC6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min

```

=====
Injection Date   : 11/24/03 10:47:27 AM
Sample Name     : 3-CLC6H4CH2OH           Location : Vial 2
Acq. Operator   : C                      In1 : 1
                                           Inj Volume : Manually
Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 11/24/03 9:44:46 AM by C
                                           (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 5/20/04 2:44:34 PM
                                           (modified after loading)
=====
  
```

```

=====
                        Area Percent Report
=====
Sorted By          :      Signal
Multiplier         :      1.0000
Dilution           :      1.0000
Sample Amount      :      1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak RetTime Type  Width  Area      Height  Area
# [min]           [min] [pA*s]   [pA]     %
-----|-----|-----|-----|-----|
1  13.223 BB      0.1568 2957.27197 243.23935 1.000e2

Totals :                      2957.27197 243.23935

Results obtained with enhanced integrator!
=====
                        *** End of Report ***
=====
  
```

Instrument 1 5/20/04 2:44:41 PM

Page 1 of 1

Figure 35.
GC diagram of 3-chloro benzyl alcohol

Data File C:\HPCHEM\1\DATA\DONG\00062906.D

Sample Name: 3-Cl-C6H4CH2OH

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/3-Cl-C6H4CH2OH
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 7h

=====

Injection Date : 6/29/04 5:10:07 PM
Sample Name : 3-Cl-C6H4CH2OH
Acq. Operator :
Location : Vial 2
Inj : 1
Inj Volume : Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 6/29/04 8:32:07 AM
(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/19/04 9:10:02 AM
(modified after loading)

=====

Area Percent Report

=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.592	BB	0.0700	2432.59595	493.36432	1.000e2

Totals : 2432.59595 493.36432

Results obtained with enhanced integrator!

=====

*** End of Report ***

Instrument 1 7/19/04 9:10:06 AM

Page 1 of 1

Figure 36.

GC diagram of oxidation of 30 mmol of 3-chlorobenzyl alcohol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Figure 37.
¹H NMR spectrum of 3-chlorobenzaldehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\12006.D

Sample Name: 2-ClC6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min

```

=====
Injection Date : 11/20/03 1:47:27 PM
Sample Name    : 2-ClC6H4CH2OH          Location : Vial 2
Acq. Operator  : C                      Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/20/03 1:12:48 PM by C
               : (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 5/20/04 2:39:04 PM
               : (modified after loading)
=====
  
```

Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount   : 1.00000 [ng/ul] (not used in calc.)
=====
  
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Tvbe	Width [min]	Area [pA*s]	Height [pA]	Area %
1	11.434	BB	0.1247	2673.94702	284.44830	1.000e2

Totals : 2673.94702 284.44830

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 5/20/04 2:39:19 PM

Page 1 of 1

Figure 38.
GC diagram of 2-chloro benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\DONO\DOO62802.D

Sample Name: 2-Cl-C₆H₄CH₂OH

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/2-Cl-C₆H₄CH₂OH
1TEMPO, 4DBDMH, 4NaNO₂, 10mL/H₂O, 9atm/air, 80C, 7h

```
=====
Injection Date   : 6/28/04 4:24:16 PM
Sample Name      : 2-Cl-C6H4CH2OH          Location : Vial 2
Acq. Operator    :                          In1   : 1
                                           Inj Volume : Manually

Acq. Method      : C:\HPCHEM\1\METHODS\205.M
Last changed     : 6/28/04 3:34:03 PM
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\205.M
Last changed     : 7/19/04 9:12:00 AM
                  (modified after loading)
=====
```

Area Percent Report

```
-----
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
-----
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.920	EV	0.2064	4.95127e4	2875.92920	98.97064
2	11.397	EB	0.0918	426.01224	71.72533	0.85155
3	31.821	EB	0.0410	88.95460	33.49314	0.17781

Totals : 5.00277e4 2981.14767

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:12:27 AM

Page 1 of 1

Figure 39.

GC diagram of oxidation of 30 mmol of 2-chlorobenzyl alcohol with 0.9 MPa of air at 80 °C for 7 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Figure 40.
¹H NMR spectrum of 2-chloro benzaldehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00041406.D

Sample Name: a-C8H9OH

Column FAAP
100C(5min)-10C/min-250C, flow= 1.2 mL/min
0.2TEMP0.40BDMM,8NaH02,0.5ml/H2O,15ml/CH2Cl2,
4atm/air,80C,2h

```
=====
Injection Date   : 4/14/04 2:42:35 PM
Sample Name     : a-C8H9OH
Acq. Operator   :
Location        : Vial 1
Inj             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 4/14/04 1:54:01 PM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 5/5/04 8:46:21 PM
                  (modified after loading)
=====
```

```
=====
Area Percent Report
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000

Signal 1: FID1 A,

Peak RetTime Type Width Area Height Area
# [min] [min] [pA*s] [pA] %
-----|-----|-----|-----|-----|
1 11.097 PB 0.0588 1.31319e4 3018.60986 1.000e2

Totals : 1.31319e4 3018.60986

Results obtained with enhanced integrator!
=====
*** End of Report ***
```

Instrument 1 5/5/04 8:51:06 PM

Page 1 of 1

Figure 41.
GC diagram of α -methyl benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\H00Q\H0062801.D

Sample Name: a-C8H9OH

Column:FFAp
100C (5min), 10C/min--250C, Flow rate: 1.2mL/min
30mmol a-C8H9OH,
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 4h

=====

Injection Date	: 6/28/04 2:56:44 PM	Location	: Vial 1
Sample Name	: a-C8H9OH	Inj	: 1
Acq. Operator	:	Inj Volume	: Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 6/28/04 2:52:39 PM
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/19/04 9:16:51 AM
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	10.979	BB	0.0756	7063.87354	1417.63501	99.56200
2	12.457	BB	0.0683	31.07602	7.13949	0.43800

Totals : 7094.94956 1424.77450

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:16:56 AM

Page 1 of 1

Figure 42.

GC diagram of oxidation of 30 mmol of α -methylbenzyl alcohol with 0.9 MPa of air at 80°C for 4 h, 1 mol% of TEMPO, 4 mol% of DBDMH and 4 mol% of NaNO₂ in 10 mL of H₂O

Figure 43.
¹H NMR spectrum of acetophenone

Data File C:\HPCHEM\1\DATA\1\00113008.D

Sample Name: 3-Py-CH2OH

COLUMN: HP-5
100 C [20 min]--15 C/min--250 C, Flow 1.2 mL/min

=====

Injection Date	: 11/30/03 2:22:46 PM	Location	: Vial 2
Sample Name	: 3-Py-CH2OH	Inj	: 1
Acq. Operator	: C	Inj Volume	: Manually
Acq. Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 11/30/03 2:19:38 PM by C		
	(modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 5/20/04 2:48:06 PM		
	(modified after loading)		

=====

=====

Area Percent Report

=====

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000
Sample Amount	:	1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.141	BV	0.2058	4507.47070	283.00287	1.000e2

Totals : 4507.47070 283.00287

Results obtained with enhanced integrator!

=====

*** End of Report ***

Instrument 1 5/20/04 2:48:12 PM

Page 1 of 1

Figure 44.
GC diagram of 3-pyridine methanol

Supporting Information

Data File C:\HPCHEM\1\DATA\DOMG\00071201.D

Sample Name: 3-PyCH2OH

COLUMN: HP-5
100C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/3-PyCH2OH
1TEMPO, 4DBDMH, 4NaNO2, 2mL/AcOH, 10mL/H2O,
9atm/air, 80C, 6h

```
=====
Injection Date : 7/12/04 3:27:04 PM
Sample Name : 3-PyCH2OH
Acq. Operator :
Location : Vial 2
Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/12/04 3:20:18 PM
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/19/04 9:13:59 AM
(modified after loading)
=====
```

```
=====
Area Percent Report
=====
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.649	BB	0.0415	2465.10742	846.81915	98.22759
2	7.885	VB	0.0679	44.48023	9.81040	1.77241

Totals : 2509.58765 856.62955

Results obtained with enhanced integrator!

```
=====
*** End of Report ***
```

Instrument 1 7/19/04 9:14:03 AM

Page 1 of 1

Figure 45.

GC diagram of oxidation of 30 mmol of 3-pyridine methanol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂, 10 mL of H₂O, 2 mL of AcOH

Figure 46.
¹H NMR spectrum of 3-pyridine carboxaldehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\12801.D

Sample Name: 2-C4H3S-CH2OH

COLUMN: HP-5
80 C (20 min)--15 C/min--250 C, Flow 1.2 ml/min

```
=====
Injection Date   : 11/28/03 10:03:16 AM
Sample Name      : 2-C4H3S-CH2OH          Location : Vial 2
Acq. Operator    : C                      Inj      : 1
                                           Inj Volume: Manually
Acq. Method      : C:\HPCHEM\1\METHODS\205.M
Last changed     : 11/28/03 9:57:05 AM by C
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\205.M
Last changed     : 5/20/04 2:43:13 PM
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By       : Signal
Multiplier      : 1.0000
Dilution        : 1.0000
Sample Amount   : 1.00000 [ng/ul] (not used in calc.)
=====
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	9.073	BP	0.1569	6008.19678	485.35428	1.000e2

Totals : 6008.19678 485.35428

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 5/20/04 2:43:17 PM

Page 1 of 1

Figure 47.
GC diagram of 2-thiophene methanol

Data File C:\HPCHEM\1\DATA\DOMG\00070605.D

Sample Name: C4H3S-2-CH2OH

COLUMN: HP-5
80C(20min)--15C/min--250C, Flow rate:1.2mL/min
30mmol/C4H3S-2-CH2OH
1TEMPO, 2PyHBr3, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 6h

=====

Injection Date	: 7/6/04 6:41:45 PM	Location	: Vial 2
Sample Name	: C4H3S-2-CH2OH	Inj	: 1
Acq. Operator	:	Inj Volume	: Manually
Acq. Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 7/6/04 6:35:51 PM		
	(modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 7/19/04 9:15:01 AM		
	(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.729	BB	0.0731	1334.08887	280.05386	98.38175
2	22.883	BB	0.0616	21.94397	5.49041	1.61825

Totals : 1356.03283 285.54428

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:15:05 AM

Page 1 of 1

Figure 48.

GC diagram of oxidation of 30 mmol of 2-thiophene methanol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 2 mol% of PyHBr₃, 4 mol% of NaNO₂, 10 mL of H₂O

Figure 49.
¹H NMR spectrum of 2-thiophene carboxaldehyde

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00041405.D

Sample Name: C-C6H11OH

Column FAAP
100C(5min)-10C/min-250C, flow= 1.2 mL/min

```

=====
Injection Date   : 4/14/04 2:03:24 PM
Sample Name     : C-C6H11OH
Acq. Operator   :
Location        : Vial 1
Inj             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 4/14/04 1:54:01 PM
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\205.M
Last changed    : 5/5/04 8:46:21 PM
                  (modified after loading)
=====
  
```

Area Percent Report

```

=====
Sorted By       : Signal
Multiplier      : 1.0000
Dilution        : 1.0000
  
```

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [nA*s]	Height [nA]	Area %
1	6.558	FB	0.0774	7346.54590	1310.69910	1.000e2

Totals : 7346.54590 1310.69910

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 5/5/04 8:48:49 PM

Page 1 of 1

Figure 50.
GC diagram of cyclohexanol

Data File C:\HPCHEM\1\DATA\H00Q\H0071402.D

Sample Name: Cyclohexanol

Column:FFAO
100C (5min), 10C/min--250C, Flow rate: 1.2mL/min
30mmol/C6H11OH
1TEMPO, 4DBDMH, 4NaNO₂, 10mL/H₂O, 9atm/air, 80C, 6h

=====

Injection Date : 7/14/04 4:15:10 PM
Sample Name : Cyclohexanol
Acq. Operator :
Location : Vial 1
Inj : 1
Inj Volume : Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/14/04 3:02:01 PM
(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/19/04 9:19:10 AM
(modified after loading)

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.756	BB	0.0868	610.09900	110.61793	10.72308
2	6.436	BB	0.1194	5079.49072	627.90985	89.27692

Totals : 5689.58972 738.52778

Results obtained with enhanced integrator!

=====
*** End of Report ***

Instrument 1 7/19/04 9:19:13 AM

Page 1 of 1

Figure S1.

GC diagram of oxidation of 30 mmol of cyclohexanol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00041501.D

Sample Name: 2-Octanol

Column FAAP
100C(5min)-10C/min-250C, flow= 1.2 mL/min

```

=====
Injection Date   : 4/15/04 11:03:29 AM
Sample Name     : 2-Octanol
Acq. Operator   :
Location        : Vial 1
Inj             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 4/15/04 10:58:49 AM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 5/5/04 8:46:21 PM
                  (modified after loading)
  
```

```

=====
Area Percent Report
=====

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000

Signal 1: FID1 A,

Peak RetTime Type Width Area Height Area
# [min] [min] [pA*s] [pA] %
-----|-----|-----|-----|-----|
1 6.856 BB 0.0910 8787.06836 1297.93274 1.000e2

Totals : 8787.06836 1297.93274

Results obtained with enhanced integrator!
=====
*** End of Report ***
  
```

Instrument 1 5/5/04 8:46:29 PM

Page 1 of 1

Figure S2.
GC diagram of 2-octanol

Supporting Information

Data File C:\HPCHEM\1\DATA\H0071403.D

Sample Name: 2-Octanol

Column: FFAp
100C (5min), 10C/min--250C, Flow rate: 1.2mL/min
30mmol/C8H17OH
1TEMPO, 4DBDMH, 4NaNO2, 10mL/H2O, 9atm/air, 80C, 6h

=====
Injection Date : 7/14/04 4:51:43 PM
Sample Name : 2-Octanol
Acq. Operator :
Location : Vial 1
Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/14/04 3:02:01 PM
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 7/19/04 9:16:51 AM
(modified after loading)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.490	BB	0.0790	1326.37427	262.41113	15.32430
2	6.734	BB	0.1195	7328.99365	861.30615	84.67570

Totals : 8655.36792 1143.71729

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:18:26 AM

Page 1 of 1

Figure 53.

GC diagram of oxidation of 30 mmol of 2-octanol with 0.9 MPa of air at 80 °C for 6 h, 1 mol% of TEMPO, 4 mol% of DBDMH and 4 mol% of NaNO₂ in 10 mL of H₂O

Data File C:\HPCHEM\1\DATA\H0072702.D

Sample Name: 2-Octanol

Column: FFAP
 100C (5min), 10C/min--250C, Flow rate: 1.2mL/min
 30mmol/2-Octanol
 STEMP0, 20DBDMH, 20NaN02, 10mL/H2O
 9atm/air, 80C, 10h

```

=====
Injection Date : 7/27/04 4:47:39 PM
Sample Name    : 2-Octanol
Acq. Operator  :                               Location : Vial 1
                                           Inj : 1
                                           Inj Volume : Manually
Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 7/27/04 4:41:04 PM
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 7/27/04 8:41:58 PM
                (modified after loading)
  
```

```

=====
Area Percent Report
=====
  
```

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
  
```

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.497	BB	0.0776	2302.92969	446.87451	1.000e2

```
Totals :                2302.92969  446.87451
```

Results obtained with enhanced integrator!

```

=====
*** End of Report ***
  
```

Instrument 1 7/27/04 8:42:05 PM

Page 1 of 1

Figure 54.

GC diagram of oxidation of 30 mmol of 2-octanol with 0.9 MPa of air at 80 °C for 6 h, 5 mol% of TEMPO, 20 mol% of DBDMH and 20 mol% of NaNO₂ in 10 mL of H₂O

Figure 55
¹H NMR spectrum of 2-octanone

Data File C:\HPCHEM\1\DATA\H00Q\H0070802.D

Sample Name: mixed alcohols

Column:FFAP
 100C (5min), 10C/min--250C, Flow rate: 1.2mL/min
 15mmol Benzyl alcohol + 15mmol 2-Octanol
 1TEMPO, 4DBDMH, 4NaNO₂, 10mL/H₂O,
 9atm/air, 80C, 1.5h

 Injection Date : 7/8/04 4:06:22 PM
 Sample Name : mixed alcohols Location : Vial 1
 Acc. Operator : Inj : 1
 Inj Volume : Manually
 Acq. Method : C:\HPCHEM\1\METHODS\205.M
 Last changed : 7/8/04 2:23:53 PM
 (modified after loading)
 Analysis Method : C:\HPCHEM\1\METHODS\205.M
 Last changed : 7/19/04 9:20:22 AM
 (modified after loading)

Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.490	BB	0.0789	688.77649	136.43953	9.84505
2	6.706	BB	0.1228	2566.69873	314.24997	36.68718
3	8.794	BB	0.0758	3093.67407	618.84015	44.21951
4	14.049	BB	0.0645	647.02454	152.64374	9.24826

Totals : 6996.17383 1222.17339

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:21:33 AM

Page 1 of 1

Figure 56.

GC diagram of oxidation of mixed alcohols (15 mmol benzyl alcohol and 15 mmol of 2-octanol) with 0.9 MPa of air at 80 °C for 1.5 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O

Supporting Information

Data File C:\HPCHEM\1\DATA\H00Q\H0070801.D

Sample Name: mixed alcohols

Column:FFAo
100C (5min), 10C/min--250C, Flow rate: 1.2ml/min
15mmol Benzyl alcohol + 15mmol Cyclohexanol
1TEMPO, 4DBDMH, 4NaNO₂, 10ml/H₂O,
9atm/air, 80C, 1.5h

```
=====
Injection Date   : 7/8/04 2:28:49 PM
Sample Name      : mixed alcohols
Acq. Operator    :
Location         : Vial 1
Int              : 1
Inj Volume       : Manually

Acq. Method      : C:\HPCHEM\1\METHODS\205.M
Last changed     : 7/8/04 2:23:53 PM
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\205.M
Last changed     : 7/19/04 9:22:21 AM
                  (modified after loading)
=====
```

Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
```

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.748	BB	0.0823	231.30177	43.34938	7.53426
2	6.411	BB	0.1278	1288.03247	157.64651	41.95548
3	8.776	BB	0.0779	812.06171	163.74324	26.45153
4	14.047	BB	0.0673	738.60254	173.07465	24.05873

Totals : 3069.99849 537.81378

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 7/19/04 9:22:32 AM

Page 1 of 1

Figure 57.

GC diagram of oxidation of mixed alcohols (15 mmol benzyl alcohol and 15 mmol of cyclohexanol) with 0.9 MPa of air at 80 °C for 1.5 h, 1 mol% of TEMPO, 4 mol% of DBDMH, 4 mol% of NaNO₂ in 10 mL of H₂O