LIST of SUPPORTING INFORMATION

Transition-Metal-Free, A Highly Efficient Catalytic Aerobic Alcohol Oxidation Process

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SUPPORTING INFORMATION

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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. ^1H NMR spectrum was recorded on a Varian DRX 400 using CDCl_3 as the solvent with TMS as an internal reference. GC analysis of determination of conversions and selectivities was performed on an Agilent 6890N. GC-MS was performed on Agilent 6890N GC / 5973 MS detector. Benzyl alcohol, cyclohexanol and 3-methyl-2-buten-1-ol were domestic reagent and redistilled before using. TEMPO, 2-chloro-benzyl alcohol, 3-chloro-benzyl alcohol, 4-chloro-benzyl alcohol, 2-methyl-benzyl alcohol, 3-methyl-benzyl alcohol, 4-methyl-benzyl alcohol, α -methyl-benzyl alcohol, 3-pyridinyl methanol, 1-octanol, 2-octanol, 3-octanol, are purchased from Acros, and 2-thionyl methanol from Fluka, directly used for oxidation without further purification. Pyridine hydrobromide perbromide (PyHBr_3) was prepared according to the literature procedure (1).

General procedure of preparation of TEMPO-catalyzed aerobic oxidation: Method A: To a Teflon-lined 316L stainless steel autoclave (300 mL), added 5.00 mL of CH_2Cl_2 contained 64 mg of Br_2 (0.4 mmol) (3.2 g of Br_2 (1.00 mL) was diluted with CH_2Cl_2 in a 250 mL volumetric flask in advance), 27.6 mg of NaNO_2 (0.4 mmol), 15.6 mg of TEMPO (0.1 mmol) and 10.0 mmol of alcohol substrate, diluted the mixture with another 5 mL of clean CH_2Cl_2 . Then closed the autoclave and charged air to 0.4MPa. **Method B:** To a Teflon-lined 316L stainless steel autoclave (300 mL), added 5.00 mL of CH_2Cl_2 contained 64 mg of Br_2 (0.4 mmol), 55.2 mg of NaNO_2 (0.8 mmol), 15.6 mg of TEMPO (0.1 mmol) and 10.0 mmol of alcohol substrate, diluted the mixture with another 5 mL of clean CH_2Cl_2 . Then closed the autoclave and charged air to 0.8 MPa.

General procedure of TEMPO-catalyzed aerobic oxidation: Put the autoclave into the oil bath, which was preheated to 80 °C. A heating period of autoclave to desired temperature was excluded. After the reaction complete, cooled to room temperature and carefully depressurized

the autoclave. Diluted the sample with CH_2Cl_2 and detected the conversion and selectivity by GC without any purification. If the alcohol did not completely converted by GC, then performed another reaction through adjusting the reaction time by primary conversion of one-hour reaction. When GC showed the reaction complete, the liquid in the autoclave was transferred into a separation funnel, carefully washed the autoclave with CH_2Cl_2 , combined all organic solutions. The organic mixture was washed with aqueous $\text{Na}_2\text{S}_2\text{O}_3$ to remove the residual oxidants and TEMPO (2). The organic layer was dried over anhydrous Na_2SO_4 , concentrated to dryness. The yield was calculated based on 10.0 mmol of substrate. Because the selectivities in most reactions were more than 99%, we checked ^1H NMR using isolated 4-chloro-benzaldehyde and acetophenone as examples. See the attached proton NMR spectra.

General GC conditions: 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-chloro-benzyl alcohol, 3-chloro-benzyl alcohol, 4-chloro-benzyl alcohol, 2-methyl-benzyl alcohol, 3-methyl-benzyl alcohol, 4-methyl-benzyl alcohol, α -methyl-benzyl alcohol, 3-pyridine methanol and their corresponding carbonyl compounds were detected under a condition as: column temperature: 100 °C for 20 minutes, raising to 250 °C in a rate of 15 °C / min. TEMPO was also detected under this condition, and its corresponding peak areas were deleted in the GC diagrams.

1-Octanol, 2-octanol, 3-octanol, 2-thionyl methanol and their corresponding carbonyl compounds were performed under a condition as: 80 °C for 20 minutes, raising to 250 °C in a rate of 15 °C / min. TEMPO was also detected under this condition, and its corresponding peak areas were deleted in the GC diagrams.

Cyclohexanol, 3-methyl-2-buten-1-ol and their corresponding carbonyl compounds were performed under a condition as: 50 °C for 5 minutes, raising to 250 °C in a rate of 10 °C / min. TEMPO was also detected under this condition, and its corresponding peak areas were deleted in the GC diagrams.

Reference:

1. Djerassi, C.; Scholz, C. R. *J. Am. Chem. Soc.* **1948**, 70, 417.
2. de Nooy, A. E. J.; Besemer, A. C.; van Bekkum, H. *Synthesis*, **1996**, 1153.

Optimization procedure of aerobic oxidation: For our initial experiments of aerobic alcohol oxidation, we chose benzyl alcohol as a test substrate to optimize the reaction conditions (Table 1). Primary results in Table 1 showed that TEMPO catalyzed aerobic benzyl alcohol oxidation with only Br₂ as the co-catalyst, about 8.36% of benzyl alcohol converted to benzaldehyde with a little benzyl bromide under a condition of 0.5 MPa of oxygen at 100 °C for 1 hour (entry 1). Prolonging the reaction time to 5 hours showed some helpful for the conversion (entry 2). Using NaNO₂ only as the co-catalyst showed no oxidation (entry 3). We then used Br₂ (4 mol%) together with NaNO₂ (8 mol%) as co-catalyst, a complete conversion of benzyl alcohol with 100% of selectivity was obtained under 100 °C for 1 hour (entry 4). Lowering the oxygen pressure from 0.5 MPa to 0.2 MPa, the same result was obtained (entry 5). Then we tried to decrease the reaction temperature under 0.2 MPa of oxygen for 1 hour, total conversions in high selectivities were observed at 90 °C (entry 6) and 80 °C (entry 7); trace benzyl alcohol did not convert to product at 70 °C (entry 8) and 60 °C (entry 9); and 85.6% of benzyl alcohol was oxidized at 50 °C in 100% selectivity (entry 10). Then we chose 80 °C as our further reaction temperature for other condition screening. When keeping 4 mol% of Br₂ and reducing the amount of NaNO₂ from 8 mol% to 4 mol% (entry 11), no lowering the conversion and selectivity was observed. Further reducing the amount of NaNO₂ from 4 mol% to 2 mol% with 4 mol% of Br₂ (entry 12), a significant decreasing of conversion was observed, while tried to reduce the amount of Br₂ from 4 mol% to 2 mol% with 4 mol% of NaNO₂ (entry 13), it also resulted in the decreasing of conversion. Thus, we use a catalyst composed of 1 mol% of TEMPO, 4 mol% of Br₂ and 4 mol% of NaNO₂ for the aerobic oxidation to test air as terminal oxidant. It was found that 97.2% of benzyl alcohol was converted to benzaldehyde in 100% selectivity under a condition of 0.2 MPa of air at 80 °C for 1 hour (entry 14). Slightly raising the air pressure to 0.4 MPa can complete the reaction (entry 15). Therefore, an optimal reaction condition for benzyl alcohol was as follows: 1 mol % of TEMPO / 4 mol % of Br₂ / 4 mol % of NaNO₂ as catalyst with 0.4 MPa of air as the terminal oxidant at 80 °C. While, for the less active alcohols, the reaction condition was tuned by adding more NaNO₂ to improve the reaction rate.

Table 1. Optimization of TEMPO-Catalyzed Oxidation of Benzyl Alcohol ^a

Entry	Br ₂ (mmol)	NaNO ₂ (mmol)	Temperature (°C)	P _{O2} (MPa)	P _{air} (MPa)	Time (h)	Conversion (%)	Selectivity of aldehyde (%)
1	0.4	-	100	0.5	-	1	8.36	91.3 ^b
2	0.4	-	100	0.5		5	20.4	95.4 ^b
3	-	0.8	100	0.5	-	1	1.1	100
4	0.4	0.8	100	0.5		1	100	100
5	0.4	0.8	100	0.2		1	100	100
6	0.4	0.8	90	0.2		1	100	100
7	0.4	0.8	80	0.2		1	100	100
8	0.4	0.8	70	0.2		1	>99.9	100
9	0.4	0.8	60	0.2		1	99.7	100
10	0.4	0.8	50	0.2		1	85.6	100
11	0.4	0.4	80	0.2		1	100	100
12	0.4	0.2	80	0.2		1	81.2	100
13	0.2	0.4	80	0.2		1	77.8	100
14	0.4	0.4	80	-	0.2	1	97.2	100
15	0.4	0.4	80	-	0.4	1	100	100

a: the reaction was carried out with 10 mmol of benzyl alcohol, 1 mol % of TEMPO in 10 mL of dichloromethane and 1 atm of air in an autoclave, charge oxygen to the desired pressure, the autoclave then put in the oil bath at desired temperature. The conversions and selectivities were performed on GC (area normalization).

b: The impurity was benzyl bromide by GC-MS.

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- Figure 50: GC diagram of TEMPO at 80 °C (initial column temperature).
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Supporting Information

Data File C:\HPCHEM\1\DATA\1\00111001.D

COLUMN: HP-5
100 C (20 min) -- 15 C/min -- 250 C, Flow 1.2 mL/min
TEMPO + CH₂CL₂

=====

Injection Date	: 11/10/03 10:07:08 AM	Location	: Vial 2
Sample Name	: C6H5CH2OH	Inj	: 1
Acq. Operator	: BCM	Inj Volume	: Manually

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Analysis Method : C:\HPCHEM\1\METHODS\205.M
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(modified after loading)

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Area Percent Report

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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	7.799	BB	0.0658	250.28362	60.45930	1.000e2

Totals : 250.28362 60.45930

Results obtained with enhanced integrator!

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*** End of Report ***

Instrument 1 11/11/03 3:00:58 PM BCM

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Figure 1.
GC diagram of TEMPO at 100 °C (initial column temperature).

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

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Acq. Operator  : C
Location       : Vial 2
Int            : 1
Inj Volume     : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
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Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/24/03 1:19:28 PM by C
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Area Percent Report

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Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
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Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	5.139	FB	0.0454	1153.27295	409.08969	1.000e2

Totals : 1153.27295 409.08969

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/24/03 1:19:35 PM C

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Figure 2.
GC diagram of benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00110702.D

Sample Name: mixture sample

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
C6H5CH2OH / C6H5CHO / C6H5COOH

```
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Injection Date : 11/7/03 10:17:51 AM
Sample Name    : mixture sample          Location : Vial 2
Acq. Operator  : BCM                     Int : 1
                                           Inj Volume : Manually

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Analysis Method : C:\HPCHEM\1\METHODS\205.M
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Dilution       : 1.0000
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Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.145	BV	0.0595	1.41215e4	3927.04712	34.20482
2	5.260	VB	0.0910	1.47335e4	2189.13232	35.68728
3	12.017	MM	1.8544	1.23651e4	111.13181	29.95065
4	40.282	BB	0.0716	64.91951	13.99303	0.15725

Totals : 4.12850e4 6241.30429

Results obtained with enhanced integrator!

Instrument 1 11/11/03 2:57:29 PM BCM

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Figure 3.
GC diagram of mixed sample of benzaldehyde, benzyl alcohol and benzoic acid.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0112501.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br2, 100C, 5 atm/O2, 1 hours

Injection Date : 11/25/03 10:15:54 AM
Sample Name : C6H5CH2OH Location : Vial 2
Acq. Operator : C Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
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Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/25/03 11:12:58 AM by C
(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	4.108	BB	0.0330	91.09718	41.61977	7.63458
2	5.138	BP	0.0425	1093.51184	392.34210	91.64396
3	6.717	MM	0.0562	8.60857	2.55434	0.72146

Totals : 1193.21759 436.51621

Results obtained with enhanced integrator!

Instrument 1 11/25/03 11:13:10 AM C

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Figure 4.

GC diagram of oxidation of benzyl alcohol with 0.5 MPa of O₂ at 100 °C for 1 hour, 4 mol% of Br₂ as co-catalyst only.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112412.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br2, 100C, 5 atm/O2, 5 hours

Injection Date : 11/24/03 4:20:19 PM
Sample Name : C6H5CH2OH Location : Vial 2
Acq. Operator : C Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
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Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/24/03 5:31:18 PM by C
(modified after loading)

Area Percent Report

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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	4.113	BB	0.0355	257.08191	117.59270	19.49053
2	5.148	BB	0.0423	1049.63733	378.58865	79.57772
3	6.735	MM	0.0550	12.28986	3.72104	0.93175

Totals : 1319.00910 499.90240

Results obtained with enhanced integrator!

Instrument 1 11/24/03 5:31:25 PM C

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Figure 5.

GC diagram of oxidation of benzyl alcohol with 0.5 MPa of O₂ at 100 °C for 5 hours, 4 mol% of Br₂ as co-catalyst only.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112413.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
TEMPO + NaNO₂ 0.8, 100C, 5 atm/O₂, 1 hours

=====
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Sample Name : C6H5CH2OH Location : Vial 2
Acq. Operator : C Inj : 1
Inj Volume : Manually
Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/24/03 5:31:18 PM by C
(modified after loading)

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

Sorted By : Signal
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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	4.114	BV	0.0335	19.85177	8.91231	1.09568
2	5.159	BB	0.0463	1791.97168	575.96368	98.90432

Totals : 1811.82345 584.87600

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/24/03 5:38:44 PM C

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Figure 6.

GC diagram of oxidation of benzyl alcohol with 0.5 MPa of O₂ at 100 °C for 1 hour, 8 mol% of NaNO₂ as co-catalyst only.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00110707.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min) -- 15 C/min -- 250 C, Flow 1.2 mL/min
TEMPO + NaNO₂ + Br₂, 100 C, 5 atm O₂,
1 hour

```

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Acq. Operator  : BCM
Location       : Vial 2
Inj           : 1
Inj Volume     : Manually

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Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/11/03 2:45:37 PM by BCM
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Area Percent Report

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Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
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Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.127	VB	0.0480	1.01013e4	3100.62744	1.000e2

Totals : 1.01013e4 3100.62744

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/11/03 2:50:41 PM BCM

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Figure 7.

GC diagram of oxidation of benzyl alcohol with 0.5 MPa of O₂ at 100 °C for 1 hour, 4 mol% of Br₂ and 8 mol% of NaNO₂ as co-catalysts.

Data File C:\HPCHEM\1\DATA\1\00111004.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMPO + Br₂ + NaNO₂, 100 C, 2atm / O₂
,1 hour

=====

Injection Date	: 11/10/03 2:32:04 PM	Location	: Vial 2
Sample Name	: C6H5CH2OH	Inj	: 1
Acq. Operator	: BCM	Inj Volume	: Manually

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(modified after loading)

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Area Percent Report

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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	4.201	VV	0.0710	3.17867e4	6337.87598	1.000e2

Totals : 3.17867e4 6337.87598

Results obtained with enhanced integrator!

=====

*** End of Report ***

Instrument 1 11/11/03 2:49:12 PM BCM

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Figure 8.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 100 °C for 1 hour, 4 mol% of Br₂ and 8 mol% of NaNO₂ as co-catalysts.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00111005.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMPO + Br₂ + NaNO₂, 90 C, 2atm / O₂,
1 hour

```
=====
Injection Date : 11/10/03 3:43:40 PM
Sample Name    : C6H5CH2OH                Location : Vial 2
Acq. Operator  : BCM                      Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/10/03 1:35:25 PM by BCM
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/11/03 2:45:37 PM by BCM
                (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	4.142	VB	0.0482	1.12120e4	3426.23535	1.000e2

Totals : 1.12120e4 3426.23535

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/11/03 2:53:47 PM BCM

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Figure 9.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 90 °C for 1 hour, 4 mol% of Br₂ and 8 mol% of NaNO₂ as co-catalysts.

Data File C:\HPCHEM\1\DATA\1\00111006.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMPO + Br₂ + NaNO₂, 80 C, 2atm / O₂,
1 hour

=====

Injection Date	: 11/10/03 4:49:03 PM	
Sample Name	: C6H5CH2OH	Location : Vial 2
Acq. Operator	: BCM	Inj : 1
		Inj Volume : Manually
Acq. Method	: C:\HPCHEM\1\METHODS\205.M	
Last changed	: 11/10/03 1:35:25 PM by BCM	
	(modified after loading)	
Analysis Method	: C:\HPCHEM\1\METHODS\205.M	
Last changed	: 11/11/03 2:45:37 PM by BCM	
	(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	4.166	VB	0.0599	1.68794e4	4385.36182	1.000e2

Totals : 1.68794e4 4385.36182

Results obtained with enhanced integrator!

=====

*** End of Report ***

Instrument 1 11/11/03 2:54:26 PM BCM

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Figure 10.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 80 °C for 1 hour, 4 mol% of Br₂ and 8 mol% of NaNO₂ as co-catalysts.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00111104.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br2 + NaNO2, 70 C, 2atm / O2,
1 hour

Injection Date : 11/11/03 3:43:07 PM
Sample Name : C6H5CH2OH Location : Vial 2
Acq. Operator : BCM Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/11/03 3:02:11 PM by BCM
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/11/03 4:36:44 PM by BCM
(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	4.157	VB	0.0627	1.56198e4	4033.81763	99.95241
2	5.102	MM	0.0425	7.43692	2.91660	0.04759

Totals : 1.56272e4 4036.73422

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/11/03 4:36:48 PM BCM

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Figure 11.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 70 °C for 1 hour, 4 mol% of Br₂ and 8 mol% of NaNO₂ as co-catalysts.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00111102.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br₂ + NaNO₂, 60 C, 2atm / O₂,
1 hour

```

=====
Injection Date   : 11/11/03 12:57:37 PM
Sample Name     : C6H5CH2OH                Location : Vial 2
Acq. Operator   : BCM                      Inj : 1
                                           Inj Volume : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 11/11/03 10:56:27 AM by BCM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 11/11/03 2:45:37 PM by BCM
                  (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	4.148	VV	0.0599	1.42024e4	3909.05518	99.67902
2	5.096	BB	0.0399	45.73327	17.87122	0.32098

Totals : 1.42481e4 3926.92639

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/11/03 2:45:43 PM BCM

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Figure 12.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 60 °C for 1 hour, 4 mol% of Br₂ and 8 mol% of NaNO₂ as co-catalysts.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00111103.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMPO + Br₂ + NaNO₂, 50 C, 2atm / O₂,
1 hour

```

=====
Injection Date : 11/11/03 2:37:14 PM
Sample Name    : C6H5CH2OH                Location : Vial 2
Acq. Operator  : BCM                      Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/11/03 3:02:11 PM by BCM
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/11/03 4:38:09 PM by BCM
                (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	4.151	VP	0.0634	1.64340e4	4176.35254	85.71117
2	5.141	BB	0.0523	2739.69409	802.20697	14.28883

Totals : 1.91737e4 4980.55951

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/11/03 4:38:12 PM BCM

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Figure 13.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 50 °C for 1 hour, 4 mol% of Br₂ and 8 mol% of NaNO₂ as co-catalysts.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112503.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br₂, NaNO₂ 0.4, 80C, 2 atm/O₂, 1 hours

```
=====
Injection Date : 11/25/03 2:15:38 PM
Sample Name    : C6H5CH2OH
Acq. Operator  : C
Location       : Vial 2
Inj           : 1
Inj Volume     : Manually

Method         : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/25/03 11:12:58 AM by C
                (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] [pA*s] [pA] %
----|-----|-----|-----|-----|-----|
1 4.106 BB 0.0354 986.66779 453.19391 1.000e2

Totals : 986.66779 453.19391

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

Instrument 1 11/25/03 3:49:37 PM C

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Figure 14.
GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 80 °C for 1 hour, 4 mol% of Br₂ and 4 mol% of NaNO₂ as co-catalysts.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112505.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br₂, NaNO₂ 0.2, 80C, 2 atm/O₂, 1 hours

=====

Injection Date	: 11/25/03 5:00:05 PM	Location	: Vial 2
Sample Name	: C6H5CH2OH	Inj	: 1
Acq. Operator	: C	Inj Volume	: Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/25/03 11:12:58 AM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/25/03 5:41:44 PM by C
(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	4.106	BV	0.0360	771.52960	346.26770	81.82892
2	5.115	BB	0.0408	171.32732	64.81120	18.17108

Totals : 942.85692 411.07890

Results obtained with enhanced integrator!

Instrument 1 11/25/03 5:41:58 PM C

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Figure 15.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 80 °C for 1 hour, 4 mol% of Br₂ and 2 mol% of NaNO₂ as co-catalysts.

Data File C:\HPCHEM\1\DATA\1\0111201.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
 100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
 TEMPO + Br2/0.2 + NaNO2/0.4, 80C, 2at
 m/02, 1hour

=====

Injection Date	: 11/12/03 10:32:17 AM	Location	: Vial 2
Sample Name	: C6H5CH2OH	Inj	: 1
Acq. Operator	: BCM	Inj Volume	: Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
 Last changed : 11/12/03 10:32:01 AM by BCM
 (modified after loading)
 Analysis Method : C:\HPCHEM\1\METHODS\205.M
 Last changed : 11/12/03 12:59:00 PM by BCM
 (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	4.148	PV	0.0528	1.16607e4	3379.51636	77.76216
2	5.161	PB	0.0526	3334.63940	914.09930	22.23784

Totals : 1.49953e4 4293.61566

Results obtained with enhanced integrator!

=====

*** End of Report ***

Instrument 1 11/12/03 12:59:35 PM BCM

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Figure 16.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of O₂ at 80 °C for 1 hour, 2 mol% of Br₂ and 4 mol% of NaNO₂ as co-catalysts.

Data File C:\HPCHEM\1\DATA\1\DO111401.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
 100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
 TEMPO + Br₂ + NaNO₂ 0.4, 80C, 2atm /
 air, 1hour

=====

Injection Date	: 11/14/03 10:26:42 AM	Location	: Vial 2
Sample Name	: C6H5CH2OH	Inj	: 1
Acq. Operator	: BCM	Inj Volume	: Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
 Last changed : 11/14/03 9:27:42 AM by BCM
 (modified after loading)
 Analysis Method : C:\HPCHEM\1\METHODS\205.M
 Last changed : 11/14/03 3:09:14 PM by BCM
 (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	4.218	BV	0.0760	3.14545e4	6017.78711	97.16351
2	5.146	BB	0.0410	918.24963	345.71567	2.83649

Totals : 3.23728e4 6363.50278

Results obtained with enhanced integrator!

=====

*** End of Report ***

Instrument 1 11/14/03 3:12:48 PM BCM

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Figure 17.

GC diagram of oxidation of benzyl alcohol with 0.2 MPa of air at 80 °C for 1 hour, 4 mol% of Br₂ and 4 mol% of NaNO₂ as co-catalysts.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0111402.D

Sample Name: C6H5CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMPO + Br2 + NaNO2 0.4, 80C, 4atm /
air, 1hour

=====

Injection Date	: 11/14/03 1:38:27 PM	Location	: Vial 2
Sample Name	: C6H5CH2OH	Inj	: 1
Acq. Operator	: BCM	Inj Volume	: Manually
Acq. Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 11/14/03 1:36:05 PM by BCM		
	(modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\205.M		
Last changed	: 11/14/03 3:09:14 PM by BCM		
	(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	4.169	VB	0.0558	1.35119e4	3643.07104	1.000e2

Totals : 1.35119e4 3643.07104

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/14/03 3:09:22 PM BCM

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Figure 18.
GC diagram of oxidation of benzyl alcohol with Method A for 1 hour.

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	Location	: Vial 2
Sample Name	: 2-ClC6H4CH2OH	Inj	: 1
Acq. Operator	: C	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	: 11/20/03 3:46:03 PM by C		
	(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	11.434	BB	0.1247	2673.94067	284.44644	1.000e2

Totals : 2673.94067 284.44644

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/20/03 3:46:08 PM C

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Figure 19.
GC diagram of 2-chloro-benzyl alcohol

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00112004.D

Sample Name: 2-ClC6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMPO + Br2 + NaNO2 0.4, 80C, 4ata /
air, 1hour

=====

Injection Date	: 11/20/03 12:11:19 PM	Location	: Vial 2
Sample Name	: 2-ClC6H4CH2OH	Inj	: 1
Acq. Operator	: C	Inj Volume	: Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/20/03 11:27:19 AM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/20/03 1:12:48 PM by C
(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	7.419	PP	0.0676	2457.77808	519.93915	1.000e2

Totals : 2457.77808 519.93915

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/20/03 1:15:30 PM C

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Figure 20.

GC diagram of oxidation of 2-chloro-benzyl alcohol with Method A for 2 hours.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\0112403.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
Acq. Operator  : C
Location       : Vial 2
Int            : 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   : 11/24/03 1:20:41 PM by C
                (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] [pA*s] [pA] %
-----|-----|-----|-----|-----|-----|
1 13.223 VB 0.1569 2958.84326 243.25612 1.000e2

Totals : 2958.84326 243.25612

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

Instrument 1 11/24/03 1:20:48 PM C

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Figure 21.
GC diagram of 3-chloro-benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112003.D

Sample Name: 3-ClC6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br2 + NaNO2 0.4, 80C, 4atm /
air, 1hour

Injection Date : 11/20/03 11:21:53 AM
Sample Name : 3-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 11:27:19 AM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/20/03 1:12:48 PM by C
(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	7.520	BV	0.0783	2894.64844	555.07715	99.32022
2	7.626	VV	0.0523	19.81181	5.80799	0.67978

Totals : 2914.46025 560.88514

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/20/03 1:13:00 PM C

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Figure 22.
GC diagram of oxidation of 3-chloro-benzyl alcohol with Method A for 2 hours.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00112008.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
Acq. Operator  : C
Location       : Vial 2
Int            : 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   : 11/21/03 4:24:31 PM by C
                (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	12.885	FV	0.1320	1765.75488	183.95264	1.000e2

Totals : 1765.75488 183.95264

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/21/03 4:24:36 PM C

Page 1 of 1

Figure 23.
GC diagram of 4-chloro-benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00111907.D

Sample Name: 4-ClC6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMPO + Br2 + NaNO2 0.4, 80C, 4atm /
air, 1hour

=====

Injection Date	: 11/19/03 4:29:02 PM	Location	: Vial 2
Sample Name	: 4-ClC6H4CH2OH	Inj	: 1
Acq. Operator	: C	Inj Volume	: Manually

Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/19/03 9:56:40 AM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/20/03 11:27:19 AM by C
(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	7.717	VV	0.0736	2483.95557	494.22797	1.000e2

Totals : 2483.95557 494.22797

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/20/03 11:31:36 AM C

Page 1 of 1

Figure 24
GC diagram of oxidation of 4-chloro-benzyl alcohol with Method A for 2 hours.

Figure 25.
¹H NMR spectrum of 4-chloro-benzaldehyde (isolated)

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112102.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                      Int : 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     : 11/21/03 3:28:49 PM by C
                  (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	2349.66064	400.62854	1.000e2

Totals : 2349.66064 400.62854

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/21/03 3:28:54 PM C

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Figure 26.
GC diagram of 2-methyl-benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112106.D

Sample Name: 2-CH3C6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMP0 + Br2 + NaNO2 0.4, 80C, 4atm/air, 1.5 hour

=====
Injection Date : 11/21/03 2:54:49 PM
Sample Name : 2-CH3C6H4CH2OH Location : Vial 2
Acq. Operator : C Inj : 1
Inj Volume : Manually
Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/21/03 3:28:49 PM by C
(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	5.972	BB	0.0526	2286.17334	664.62964	98.97127
2	6.267	MM	0.0500	11.70890	3.90369	0.50689
3	10.780	MM	0.0958	12.05414	2.09792	0.52184

Totals : 2309.93637 670.63126

Results obtained with enhanced integrator!

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

Instrument 1 11/21/03 3:34:33 PM C

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Figure 27.
GC diagram of oxidation of 2-methyl-benzyl alcohol with Method A for 1.5 hours.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00112016.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 : 11/21/03 3:19:52 PM by C
(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	4234.13037	610.79468	1.000e2

Totals : 4234.13037 610.79468

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/21/03 3:26:37 PM C

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Figure 28.
GC diagram of 3-methyl-benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0112013.D

Sample Name: 3-CH3C6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br2 + NaNO2 0.4, 80C, 4atm/air, 1.5hour

Injection Date : 11/21/03 8:47:30 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 : 11/24/03 8:02:07 AM by C
(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	5.995	VB	0.0513	2473.14478	697.81445	1.000e2

Totals : 2473.14478 697.81445

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/24/03 8:30:57 AM C

Page 1 of 1

Figure 29.

GC diagram of oxidation of 3-methyl-benzyl alcohol with Method A for 1.5 hours.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112009.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   : 11/20/03 4:59:49 PM by C
               (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 7.804 PB 0.0671 3267.46289 529.05701 1.000e2

Totals : 3267.46289 529.05701

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

Instrument 1 11/20/03 4:59:54 PM C

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Figure 30.
GC diagram of 4-methyl-benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112011.D

Sample Name: 4-CH3C6H4CH2OH

COLUMN: HP-5
100 C (20 min)-- 15 C/min--250 C, Flow 1.2 mL/min
TEMPO + Br2 + NaNO2 0.4, 80C, 4atm/air, 1.5hour

=====

Injection Date	: 11/20/03 4:52:23 PM	Location	: Vial 2
Sample Name	: 4-CH3C6H4CH2OH	Inj	: 1
Acq. Operator	: C	Inj Volume	: Manually

Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/20/03 4:59:49 PM by C
(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	6.347	BV	0.0685	4390.13818	957.21063	99.56274
2	7.438	BP	0.0583	19.28061	5.19516	0.43726

Totals : 4409.41880 962.40579

Results obtained with enhanced integrator!

=====

*** End of Report ***

Instrument 1 11/20/03 5:58:28 PM C

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Figure 31.
GC diagram of oxidation of 4-methyl-benzyl alcohol with Method A for 1.5 hours.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112104.D

Sample Name: C6H5C2H5OH

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

```
=====
Injection Date : 11/21/03 1:27:13 PM
Sample Name    : C6H5C2H5OH
Acq. Operator  : C
Location       : Vial 2
Int:           : 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   : 11/21/03 3:19:52 PM by C
                (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	5.718	PV	0.0600	2984.31201	774.34106	1.000e2

Totals : 2984.31201 774.34106

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/21/03 3:19:55 PM C

Page 1 of 1

Figure 32.
GC diagram of α -methyl-benzyl alcohol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112105.D

Sample Name: C6H5C2H5OH

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

TEMP0 + Br2 + NaNO2 0.4, 80C, 4atm/air, 1.5 hour

```

=====
Injection Date : 11/21/03 2:04:04 PM
Sample Name    : C6H5C2H5OH
Acq. Operator  : C
Location       : Vial 2
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   : 11/21/03 3:19:52 PM by C
                (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	5.931	VB	0.0586	3037.89282	812.62866	98.96978
2	8.535	BB	0.0765	31.62262	6.53040	1.03022

Totals : 3069.51544 819.15906

Results obtained with enhanced integrator!

Instrument 1 11/21/03 3:21:22 PM C

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Figure 33.

GC diagram of oxidation of α -methyl-benzyl alcohol with Method A for 1.5 hours.

Figure 34.
¹H NMR spectrum of acetophenone (isolated).

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112409.D

Sample Name: 1-OCTANOL

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

```

=====
Injection Date   : 11/24/03 1:38:31 PM
Sample Name     : 1-OCTANOL
Acq. Operator   : C
Location        : Vial 2
Inj             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 11/24/03 1:20:41 PM by C
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 11/24/03 2:19:06 PM by C
                  (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	11.001	BB	0.1349	4013.90747	416.63077	1.000e2

Totals : 4013.90747 416.63077

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/24/03 2:19:18 PM C

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Figure 35
GC diagram of 1-octanol.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0112410.D

Sample Name: 1-OCTANOL

COLUMN: HP-5
80 C (20 min)-- 15 C/min---250 C, Flow 1.2 mL/min
TEMPO + Br2 + NaNO2O.8, 80C, 9atm/air, 3hour

Injection Date : 11/24/03 2:25:06 PM
Sample Name : 1-OCTANOL Location : Vial 2
Acq. Operator : C Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/24/03 2:19:06 PM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/27/03 8:40:47 AM by C
(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	7.598	BP	0.0629	1334.01355	325.21274	86.00132
2	31.261	VB	0.0387	217.14116	88.26449	13.99868

Totals : 1551.15471 413.47723

Results obtained with enhanced integrator!

Instrument 1 11/27/03 8:40:56 AM C

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Figure 36
GC diagram of oxidation of 1-octanol with Method B for 3 hours.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00112709.D

Sample Name: 2-Octanol

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

```
=====
Injection Date : 11/27/03 9:08:59 PM
Sample Name    : 2-Octanol
Acq. Operator  : C
Location       : Vial 2
Int            : 1
Inj Volume     : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/27/03 9:05:09 PM by C
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/27/03 10:21:35 PM by C
                (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	7.135	BV	0.0696	78.03357	17.48225	1.12157
2	7.556	VB	0.1008	6879.47070	935.06750	98.87843

Totals : 6957.50427 952.54976

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/27/03 10:21:40 PM C

Page 1 of 1

Figure 37
GC diagram of 2-octanol.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\000112901.D

Sample Name: 2-Octanol

COLUMN: HP-5
80 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
ITEMP0 + 4Br2 + 6NaNO2, 80C, 9atm/air, 4hours

=====
Injection Date : 11/29/03 2:08:35 PM
Sample Name : 2-Octanol
Acq. Operator : C
Location : Vial 2
Int : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/29/03 2:01:52 PM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/29/03 7:33:22 PM by C
(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	7.177	BB	0.0689	3326.42334	720.38269	99.43791
2	7.493	PB	0.0571	18.80332	4.91722	0.56209

Totals : 3345.22666 725.29991

Results obtained with enhanced integrator!

Instrument 1 11/29/03 7:33:29 PM C

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Figure 38
GC diagram of oxidation of 2-octanol with Method B for 4 hours.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00112711.D

Sample Name: 3-Octanol

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

```
=====
Injection Date : 11/28/03 8:22:42 AM
Sample Name    : 3-Octanol
Acq. Operator  : C
Location       : Vial 2
Int            : 1
Inj Volume     : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/28/03 8:19:02 AM by C
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/28/03 9:07:53 AM by C
                (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	7.394	VV	0.0894	4839.43408	813.86871	1.000e2

Totals : 4839.43408 813.86871

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/28/03 9:07:59 AM C

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Figure 39
GC diagram of 3-octanol.

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112712.D

Sample Name: 3-Octanol

COLUMN: HP-5
80 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
1TEMP0+4Br2+8NaNO2, 9atm/air, 4hours

Injection Date : 11/28/03 9:12:30 AM
Sample Name : 3-Octanol Location : Vial 2
Acq. Operator : C Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/28/03 9:07:53 AM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/28/03 9:57:05 AM by C
(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	7.072	BV	0.0665	3956.20361	854.24457	98.45227
2	7.298	VV	0.0586	35.75182	9.56804	0.88970
3	24.487	VB	0.0500	26.44226	8.22725	0.65803

Totals : 4018.39769 872.03986

Results obtained with enhanced integrator!

Instrument 1 11/28/03 9:57:12 AM C

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Figure 40
GC diagram of oxidation of 3-octanol with Method B for 3 hours.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00112808.D

Sample Name: Cyclohexanol

COLUMN: HP-5
50 C (5 min)--10 C/min--250 C, Flow 1.2 mL/min

```
=====
Injection Date : 11/28/03 8:43:20 PM
Sample Name    : Cyclohexanol
Acq. Operator  : C
Location       : Vial 2
Int:           : 1
Inj Volume     : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/28/03 8:54:11 PM by C
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/28/03 9:28:15 PM by C
                (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] [pA*s] [pA] [%]
-----|-----|-----|-----|-----|-----|
1 8.586 BV 0.0841 6580.84229 1111.13367 99.20068
2 8.785 VB 0.0379 33.51434 12.85804 0.50520
3 12.303 VV 0.0411 19.51161 7.31883 0.29412

Totals : 6633.86824 1131.31053

Results obtained with enhanced integrator!
=====
```

Instrument 1 11/28/03 9:28:20 PM C

Page 1 of 1

Fig. 41
GC diagram of cyclohexanol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0112805.D

Sample Name: Cyclohexanol

COLUMN: HP-5
50 C (5 min)--10 C/min--250 C, Flow 1.2 mL/min
1TEMP0 + 4Br2 + 8NaNO2, 80C, 9atm/air, 4hours

Injection Date : 11/28/03 7:13:13 PM
Sample Name : Cyclohexanol Location : Vial 2
Acq. Operator : C Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/28/03 7:08:50 PM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/30/03 5:13:05 PM by C
(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	8.784	VB	0.0600	3369.68555	926.06061	99.42074
2	11.416	VB	0.0404	19.63291	7.52494	0.57926

Totals : 3389.31845 933.58555

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/30/03 5:13:24 PM C

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Figure 42
GC diagram of oxidation of cyclohexanol with Method B for 4 hours.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00112801.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
Acq. Operator  : C
Location       : Vial 2
Int:           : 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   : 11/28/03 3:42:53 PM by C
                (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] [pA*s] [pA] %
-----|-----|-----|-----|-----|-----|
1 9.073 PB 0.1569 6008.22510 485.35443 1.000e2

Totals : 6008.22510 485.35443

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

Instrument 1 11/28/03 3:42:58 PM C

Page 1 of 1

Fig. 43
GC diagram of 2-thiophene methanol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\0112803.D

Sample Name: 2-C4H3S-CH2OH

COLUMN: HP-5
80 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
ITEMP0 + 4Br2 + 6NaNO2, 80C, 9atm/air, 5hours

Injection Date : 11/28/03 2:24:54 PM
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 : 11/28/03 3:40:36 PM by C
(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	7.697	BP	0.0803	2317.96460	398.91727	94.67877
2	22.843	BB	0.0608	130.27641	33.16838	5.32123

Totals : 2448.24101 432.08565

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/28/03 3:40:43 PM C

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Figure 44
GC diagram of oxidation of 2-thiophene methanol with Method B for 5 hours.

Supporting Information

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 : 11/30/03 3:17:15 PM by C
(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	4.581	BB	0.0401	40.32877	15.64017	0.88579
2	8.141	VV	0.2060	4512.51562	283.10745	99.11421
Totals :				4552.84440	298.74762	

Results obtained with enhanced integrator!

=====

Instrument 1 11/30/03 3:17:19 PM C

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Figure 45
GC diagram of 3-pyridine methanol

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00112810.D

Sample Name: 3-Py-CH2OH

COLUMN: HP-5
100 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
1TEMP0+4Br2+8NaNO2, 80C, 9atm/air, 12hours

Injection Date : 11/29/03 9:03:03 AM
Sample Name : 3-Py-CH2OH
Acq. Operator : C
Location : Vial 2
Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/29/03 8:56:26 AM by C
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/29/03 9:46:19 AM by C
(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	4.606	VB	0.0380	463.08276	192.79886	50.74879
2	7.845	PB	0.0830	449.41730	80.01293	49.25121

Totals : 912.50006 272.81179

Results obtained with enhanced integrator!

Instrument 1 11/29/03 9:46:29 AM C

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Figure 46
GC diagram of oxidation of 3-pyridine methanol with Method B for 12 hours.

Data File C:\HPCHEM\1\DATA\1\00113009.D

Sample Name: 3-Py-CH2OH

COLUMN: HP-5
 100 C (20 min)--15 C/min--250 C, Flow 1.2 mL/min
 4TEMP0+8Br2+8NaNO2, 9atm/air, 100C, 5hours

```

=====
Injection Date   : 11/30/03 3:21:01 PM
Sample Name     : 3-Py-CH2OH
Acq. Operator   : C
Location        : Vial 2
Inj             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\205.M
Last changed    : 11/30/03 3:17:15 PM by C
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed    : 11/30/03 4:03:10 PM by C
                  (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	4.593	VB	0.0405	1408.83911	538.32709	94.42484
2	7.858	BB	0.0749	83.18266	16.90013	5.57516

Totals : 1492.02177 555.22721

Results obtained with enhanced integrator!

Instrument 1 11/30/03 4:03:21 PM C

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Figure 47
 GC diagram of oxidation of 3-pyridine methanol with Method B at 100 °C for 5 hours, 4 mol % of TEMPO.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00113003.D

Sample Name: i-Pentenol

COLUMN: HP-5
50 C (5 min)--10 C/min--250 C, Flow 1.2 mL/min

```

=====
Injection Date : 11/30/03 11:05:30 AM
Sample Name    : i-Pentenol                Location : Vial 2
Acq. Operator  : C                        Inj : 1
                                           Inj Volume : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/30/03 10:26:04 AM by C
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/30/03 12:09:49 PM by C
                  (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	5.658	VV	0.0512	1585.79224	510.94720	1.000e2

Totals : 1585.79224 510.94720

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/30/03 12:10:01 PM C

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Figure 48
GC diagram of 3-methyl-2-buten-1-ol

Data File C:\HPCHEM\1\DATA\1\00113005.D

Sample Name: i-Pentenol

COLUMN: HP-5
 50 C (5 min)--10 C/min--250 C, Flow 1.2 mL/min
 1TEMP0+4PyHBr3+8NaNO2, 9atm/air, 3hours

```

=====
Injection Date : 11/30/03 12:52:49 PM
Sample Name    : i-Pentenol
Acq. Operator  : C
Location       : Vial 2
Inj           : 1
Inj Volume     : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/30/03 12:29:10 PM by C
                (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/30/03 4:05:15 PM by C
                (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	5.628	VV	0.0512	1506.94104	485.58774	66.75761
2	5.839	VB	0.0391	672.78198	269.69489	29.80430
3	15.295	VB	0.0425	77.60914	30.14707	3.43809

Totals : 2257.33216 785.42970

Results obtained with enhanced integrator!

Instrument 1 11/30/03 4:43:40 PM C

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Figure 49
 GC diagram of oxidation of 3-methyl-2-buten-1-ol with Method B for 3 hours, 4 mol% of pyridine hydrobromide perbromide as Br₂ equivalent.

Supporting Information

Date File C:\HPCHEM\1\DATA\1\00112602.D

Sample Name: TEMPO

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

=====

Injection Date	: 11/26/03 11:44:59 AM	Location	: Vial 2
Sample Name	: TEMPO	Int	: 1
Acq. Operator	: C	Inj Volume	: Manually

Method : C:\HPCHEM\1\METHODS\205.M
Last changed : 11/26/03 11:48:20 AM by C
(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	15.590	BB	0.1409	438.47333	49.46960	1.000e2

Totals : 438.47333 49.46960

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/26/03 12:45:36 PM C

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Figure 50
GC diagram of TEMPO at 80 °C (initial column temperature)

Supporting Information

Data File C:\HPCHEM\1\DATA\1\00113010.D

Sample Name: TEMPO

COLUMN: HP-5
50 C (5 min)--10 C/min--250 C, Flow 1.2 mL/min

```
=====
Injection Date : 11/30/03 4:10:57 PM
Sample Name    : TEMPO
Acq. Operator  : C
Location       : Vial 2
Int           : 1
Inj Volume     : Manually

Method         : C:\HPCHEM\1\METHODS\205.M
Last changed   : 11/30/03 4:05:15 PM by C
                (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	14.251	PV	0.0409	1370.79651	517.51636	1.000e2

Totals : 1370.79651 517.51636

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 11/30/03 4:42:26 PM C

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Figure 51
GC diagram of TEMPO at 50 °C (initial column temperature)