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Fig1. ¹H NMR of the sample of 4-(2,2,6,6-Tetramethyl-1-oxyl-4-piperidoxyl) butyl bromide reduced by phenylhydrazine (CDCl₃)

¹H NMR (400 MHz; CDCl₃; Me₄Si) δ: 1.14 (6H, s, Piperidine-Me), 1.20 (6H, s, Piperidine-Me), 1.42 (2H, t, *J* = 11.6 Hz, Piperidine-C*H*H), 1.66-1.70 (2H, m, O-CH₂-C*H*₂-CH₂-CH₂-Br), 1.87-1.94 (4H, m, O-CH₂-CH₂-CH₂-CH₂-Br, Piperidine-CH*H*), 3.42-3.47 (4H, m, OCH₂, BrCH₂), 3.50-3.57 (1H, m, Piperidine-CH).



Fig2. ¹H NMR of the sample of [Quaternium-TEMPO]⁺Br⁻ reduced by phenylhydrazine (CDCI₃)

¹H NMR (400 MHz; CDCl₃; Me₄Si) δ : 0.88 (3H, t, J = 6.8 Hz, Me), 1.26 (12H, s, Piperidine-Me), 1.28-1.48 (18H, m, N-CH₂-CH₂-(CH₂)₉-CH₃), 1.65-1.88 (8H, m, Piperidine-CH₂, O-CH₂-CH₂-CH₂-CH₂-N, N-CH₂-CH₂-C₉H₁₈-CH₃), 2.00-2.13 (2H, m, O-CH₂-CH₂-CH₂-CH₂-N), 3.35 (6H, s, NMe), 3.43-3.47 (2H, m, N-CH₂-CH₂-C₉H₁₈-CH₃), 3.55 (2H, t, J = 6 Hz, O-CH₂-CH₂-CH₂-CH₂-CH₂-N), 3.63-3.68 (2H, m, OCH₂), 3.71-3.76 (1H, m, Piperidine-CH).



Fig3. ¹³C NMR of the sample of [Quaternium-TEMPO]*Br⁻ reduced by phenylhydrazine (CDCl₃)

¹³C NMR (100MHz, CDCl₃) δ: 14.11 (Me), 19.89 (O-CH₂-CH₂-CH₂-CH₂-CH₂-N), 20.69 (N-CH₂-CH₂-C₈H₁₆-CH₃), 22.64 (N-CH₂-CH₂-C₉H₁₈-CH₃), 22.66 (N-CH₂-CH₂-CH₂-C₈H₁₆-CH₃), 26.22 (O-CH₂-CH₂-CH₂-CH₂-CH₂-N), 26.54 (Piperidine-Me), 29.22, 29.28, 29.41, 29.46, 29.56, 31.85, 31.99 (N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₃), 44.60 (Piperidine-CH₂), 51.26 (NMe), 59.29 (Piperidine-C), 63.50 (N-CH₂-CH₂-CH₂-C₉H₁₈-CH₃), 63.84 (O-CH₂-CH₂-CH₂-CH₂-N), 66.80 (Piperidine-CH),70.71(OCH₂).

*10 2 *ESI Scan (2.196-2.406 min, 14 scans) Frag=175.0V LFL-519.d Subtract (3) 440.4336 9- 0.85 0.8 0.75 0.7 0.65 0.6 0.55 0.5 0.5 0.5 0.5 0.5 0.5 0.	ssed 1:29:56 A	Some Ions Miss 11/14/2013 11:	atus	IRM Calibration Status Acquired Time	Sample	SampleType Comment	chen-ms.m	InjPosition ACQ Method	2 LFL-519.d	Inj Vol Data Filename
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	,	70 580 590	560 570	530 540 550 56	90 500 510 5 Charge (m/z)	(%) vs. Mass-to-0	0 430 440 4 Coun	90 400 410 42	0 370 380 3	36

Fig4. HR-MS (ESI) of [Quaternium-TEMPO]*









Fig7. ¹H NMR of 2-Methoxybenzaldehyde (entry 3) (CDCl₃)



----0.00

---3.87

8.8 V

F666666666888

Fig9. ¹H NMR of 4-Methylbenzaldehyde (entry 5) (CDCI₃)



Fig10. ¹H NMR of 4-Chlorobenzaldehyde (entry 6) (CDCl₃)



----9.99



Fig12. ¹H NMR of 4-Fluorobenzaldehyde (entry 8) (CDCl₃)







Fig14. ¹H NMR of 3,5-Difluorobenzaldehyde (entry 10) (CDCI₃)

2223 2223

818 V



---0.00

---0.00



Fig16. ¹H NMR of 2-Thenaldehyde (entry 12) (CDCl₃)



Fig17. ¹H NMR of 2-pyridinecarboxaldehyde (entry 13) (CDCl₃)



Fig18. ¹H NMR of Butanal (entry 14) (CDCl₃)



Fig19. ¹H NMR of Acetophenone (entry 15) (CDCI₃)



Fig21. ¹H NMR of 2-Chloroacetophenone (entry 17) (CDCI₃)



Fig22. ¹H NMR of 4-Chloroacetophenone (entry 18) ($(CD_3)_2SO$)



Fig23. ¹H NMR of Cyclohexanone (entry 19) (CDCl₃)



Fig25. ¹H NMR of 1,2,3,4-Tetrahydro-1-naphthalenone (entry 21) ((CD₃)₂SO)

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Attention: The first peak in the GC chromatograms below is from the diluent (acetonitrile) in the analysis.



Fig.1 GC chromatogram of Benzyl alcohol during the oxidation reaction (entry 1)

Column temperature 190 °C, pressure of the carrier gas 0.07Mpa.





Column temperature 190 °C, pressure of the carrier gas 0.07Mpa.



Fig.3 GC chromatogram of 4-Methoxybenzyl alcohol during the oxidation reaction (Entry 2)

Column temperature 220 °C, pressure of the carrier gas 0.07Mpa.



Fig.4 GC chromatogram of 4-Methoxybenzyl alcohol at the end of the oxidation reaction (entry 2) Column temperature 220 °C, pressure of the carrier gas 0.07Mpa.



Fig.5 GC chromatogram of 2-Methoxybenzyl alcohol during the oxidation reaction (entry 3)

Column temperature 220 °C, pressure of the carrier gas 0.05Mpa.



Fig.6 GC chromatogram of 2-Methoxybenzyl alcohol at the end of the oxidation reaction (entry 3)

Column temperature 220 °C, pressure of the carrier gas 0.05Mpa.





Column temperature 220 °C, pressure of the carrier gas 0.07Mpa.



Fig.8 GC chromatogram of 3-Methoxybenzyl alcohol at the end of the oxidation reaction (entry 4)

Column temperature 220 °C, pressure of the carrier gas 0.07Mpa.



Fig.9 GC chromatogram of 4-Methxybenzyl alcohol during the oxidation reaction (entry 5)

Column temperature 190 °C, pressure of the carrier gas 0.07Mpa.



Fig.10 GC chromatogram of 4-Methxybenzyl alcohol at the end of the oxidation reaction (entry 5)

Column temperature 190 °C, pressure of the carrier gas 0.07Mpa.



Fig.11 GC chromatogram of 4-Chlorobenzyl alcohol during the oxidation reaction (entry 6)

Column temperature 200 °C, pressure of the carrier gas 0.07Mpa.



Fig.12 GC chromatogram of 4-Chlorobenzyl alcohol at the end of the oxidation reaction (entry 6)

Column temperature 200 °C, pressure of the carrier gas 0.07Mpa.



Fig.13 GC chromatogram of 2-Chlorobenzyl alcohol during the oxidation reaction (entry 7)

Column temperature 200 °C, pressure of the carrier gas 0.07Mpa.



Fig.14 GC chromatogram of 2-Chlorobenzyl alcohol at the end of the oxidation reaction (entry 7)

Column temperature 200 °C, pressure of the carrier gas 0.07Mpa.



Fig.15 GC chromatogram of 4-Fluorobenzyl alcohol during the oxidation reaction (entry 8)

Column temperature 190 °C, pressure of the carrier gas 0.07Mpa.



Fig.16 GC chromatogram of 4-Fluorobenzyl alcohol at the end of the oxidation reaction (entry 8)

Column temperature 190 °C, pressure of the carrier gas 0.07Mpa.





Column temperature 190 °C, pressure of the carrier gas 0.06Mpa.



Fig.18 GC chromatogram of 2-Fluorobenzyl alcohol at the end of the oxidation reaction (entry 9)

Column temperature 190 °C, pressure of the carrier gas 0.06Mpa.



Fig.19 GC chromatogram of 3,5-Difluorobenzyl alcohol during the oxidation reaction (entry 10)

Column temperature 180 °C, pressure of the carrier gas 0.07Mpa.



Fig.20 GC chromatogram of 3,5-Difluorobenzyl alcohol at the end of the oxidation reaction(entry 10)

Column temperature 180 °C, pressure of the carrier gas 0.07Mpa.





The peaks before that of cinnamaldehyde are from the impurities of cinnamic alcohol, not from oxidation by-products.

Column temperature 220 °C, pressure of the carrier gas 0.07Mpa.





The peaks before that of cinnamaldehyde are from the impurities of cinnamic alcohol, not from oxidation by-products.

Column temperature 220 °C, pressure of the carrier gas 0.07Mpa.





Column temperature 180 °C, pressure of the carrier gas 0.05Mpa.



Fig.24 GC chromatogram of 2-Thienylmethyl alcohol at the end of the oxidation reaction (entry 12)

Column temperature 180 °C, pressure of the carrier gas 0.05Mpa.





Column temperature 200 °C, pressure of the carrier gas 0.06Mpa.



Fig.26 GC chromatogram of 2-Pyridinemethanol at the end of the oxidation reaction (entry 13)

Column temperature 200 °C, pressure of the carrier gas 0.06Mpa.





Column temperature 80 °C for nine minutes, and then heated up to 130 for ten minutes, pressure of the carrier gas 0.05Mpa.



Fig.28 GC chromatogram of Butyl alcohol at the end of the oxidation reaction (entry 14)

Column temperature 80 °C for nine minutes, and then heated up to 130 for ten minutes, pressure of the carrier gas 0.05Mpa.





Column temperature 190 °C, pressure of the carrier gas 0.05Mpa.



Fig.30 GC chromatogram of 1-Phenyl ethanol at the end of the oxidation reaction (entry 15)

Column temperature 190 °C, pressure of the carrier gas 0.05Mpa.





Column temperature 190 °C, pressure of the carrier gas 0.05Mpa.



Fig.32 GC chromatogram of 1-(4-Methylphenyl) ethanol at the end of the oxidation reaction (entry 16)

Column temperature 190 °C, pressure of the carrier gas 0.05Mpa.





Column temperature 200 °C, pressure of the carrier gas 0.05Mpa.



Fig.34 GC chromatogram of 1-(2-Chlorobenzyl) ethanol at the end of the oxidation reaction (entry 17)

Column temperature 200 °C, pressure of the carrier gas 0.05Mpa.



Fig.35 GC chromatogram of 1-(4-Chlorobenzyl) ethanol during the oxidation reaction (entry 18)

Column temperature 220 °C, pressure of the carrier gas 0.05Mpa.



Fig.36 GC chromatogram of 1-(4-Chlorobenzyl) ethanol at the end of the oxidation reaction (entry 18)

Column temperature 220 °C, pressure of the carrier gas 0.05Mpa.





Column temperature 130 °C, pressure of the carrier gas 0.05Mpa.



Fig.38 GC chromatogram of Cyclohexanol at the end of the oxidation reaction (entry 19)

Column temperature 130 °C, pressure of the carrier gas 0.05Mpa.





Column temperature 130 °C, pressure of the carrier gas 0.05Mpa.



Fig.40 GC chromatogram of 2-Octanol at the end of the oxidation reaction (entry 20)

Column temperature 130 °C, pressure of the carrier gas 0.05Mpa.





Column temperature 210 °C, pressure of the carrier gas 0.05Mpa.



Fig.42 GC chromatogram of 1,2,3,4-Tetrahydro-1-naphthol at the end of the oxidation reaction (entry 21)

Column temperature 210 °C, pressure of the carrier gas 0.05Mpa.

Effect of temperature on catalytic activity

T(°C)	Time(h)	Conv. ^{<i>b</i>} (%)	Select. ^b (%)	TON ^c	$TOF^{d}(h^{-1})$
80	0.5	>99	>99	39.6	79.2
70	1	>99	>99	39.6	39.6
60	1.5	>99	>99	39.6	26.4
50	2	>99	>99	39.6	19.8
45	2.25	>99	>99	39.6	17.6
40	9	31.5	>99	12.6	1.4
25	1	8.1	>99	3.2	3.2

Table 6. Effect of temperature on the DES-TEMPO/Fe(NO₃)₃ catalyzed aerobic oxidation of benzyl alcohol^a.

^{*a*} Reaction conditions: benzyl alcohol 10 mmol, 1.25%DES-TEMPO, 3%Fe(NO₃)₃, atmospheric oxygen pressure. ^{*b*} Conversions and selectivity were determined by GC (area normalization method). ^{*c*} TON = moles of product/2(moles of DES-TEMPO). ^{*d*} TOF = TON/ reaction time.