Supplementary Information

Cu(OAc)$_2$-catalyzed remote benzylic C(sp$^3$)–H oxyfunctionalization for C=O formation directed by the hindered para-hydroxyl group with ambient air as terminal oxidant under ligand- and additive-free conditions

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1. General Information

All solvents and reagents were purchased from commercial suppliers and used without further purification. All reactions were carried out in oven-dried glassware and monitored by thin layer chromatography (TLC, precoated silica gel plates containing HF₂₅₄). Reaction products were purified by silica gel chromatography (300–400 mesh). Melting points were determined using an open capillaries and uncorrected, NMR spectra were determined on Bruker AV400 in CDCl₃ or DMSO-d₆, with TMS as internal standard for ¹H NMR (400 MHz) and ¹³C NMR (100 MHz), respectively. HRMS were carried out on a QSTAR Pulsar I LC/TOF MS mass spectrometer or a Micromass GCTTM gas chromatograph-mass spectrometer.

2. General Procedures and Characterization Data of Compounds

2.1 Optimizing the reaction conditions (comprehensive experiments for Table 1 in the text).

**Table S1. Copper(II)-catalyzed oxidation of 1aa to 2aa.**

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<th>Entry</th>
<th>Cu(II) salt (n mol%)</th>
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<th>Solvent</th>
<th>Yield [%]</th>
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*Reaction conditions: 1aa (1.0 mmol), copper(II) salt (n mol%), solvent (2 mL), atmos. (1 atm), 12 h. †Isolated yield. ‡Reaction time: 24 h.

**General procedure:** a mixture of 1aa (1.0 mmol, 168.2 mg) and specified copper(II) salt (n mol%) in solvent (2 mL) was stirred at specified reaction temperature under corresponding atmosphere for 12 h. Hydrochloric acid (4 mL, 2%) and methyl tert-butyl ether (MTBE, 4 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (4 mL × 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to provide the desired product 2aa.
3,5-Dimethoxy-4-hydroxybenzaldehyde (2aa): 1 yellow solid, 167.6 mg (the best yield of 92%), m.p. 108–110 °C (lit1 m.p. 110–111 °C); 1H NMR (400 MHz, CDCl₃, ppm): δ 9.81 (br s, 1H), 7.15 (s, 2H), 6.10 (br s, 1H), 3.97 (s, 6H); 13C NMR (100 MHz, CDCl₃, ppm): δ 190.8, 147.4 (2C), 140.8, 128.4, 106.7 (2C), 56.5 (2C); HRMS (ESI): m/z [M+H⁺] calcd. for C₁₉H₁₄O₄ 183.0657, found 183.0635.

2.2 General procedure for the Cu(OAc)₂-catalyzed oxidation of 2,6-disubstituted 4-cresols and 4-alkylphenols 1 (Scheme 3 in the text).
Scheme 3. Scope of I for Cu(OAc)$_2$-catalyzed oxidation. *Reaction conditions: I (1.0 mmol), Cu(OAc)$_2$ (0.01 mmol), EG (2 mL), ambient air, 12 h. "Isolated yield for the oxidation product. 'Recovery for the starting material.

**General procedure:** A mixture of substrate I (1.0 mmol) and Cu(OAc)$_2$ (0.01 mmol, 1.8 mg) in EG (2 mL) was stirred at specified temperature under ambient air for 12 h. Hydrochloric acid (4 mL, 2%) and MTBE (4 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (4 mL × 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to provide the corresponding product 2.
**3,5-Diethoxy-4-hydroxybenzaldehyde (2ab):** yellow solid, 182.9 mg (87% yield), m.p. 116–118 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta 9.79\) (br s, 1H), 7.12 (s, 2H), 6.07 (br s, 1H), 4.20 (q, \(J = 7.2\) Hz, 4H), 1.49 (t, \(J = 7.2\) Hz, 6H); \(^13\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta 190.9, 146.6\) (2C), 141.2, 128.3, 107.6 (2C), 65.1 (2C), 14.8 (2C); HRMS (ESI): \(m/z\) [M+H\(^+\)] calcd. for C\(_{14}\)H\(_{15}\)O\(_4\) 211.0970, found 211.0962.

**3,5-Dipropoxy-4-hydroxybenzaldehyde (2ac):** yellow solid, 200.2 mg (84% yield), m.p. 64–66 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta 9.79\) (br s, 1H), 7.12 (s, 2H), 6.05 (br s, 1H), 4.09 (t, \(J = 7.2\) Hz, 4H), 1.89 (sext, \(J = 7.2\) Hz, 2H), 1.06 (t, \(J = 7.2\) Hz, 6H); \(^13\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta 190.9, 146.8\) (2C), 141.4, 128.3, 107.7 (2C), 71.0 (2C), 22.5 (2C), 14.8 (2C); HRMS (ESI): \(m/z\) [M+H\(^+\)] calcd. for C\(_{15}\)H\(_{17}\)O\(_4\) 239.1282.

**3,5-Dibutoxy-4-hydroxybenzaldehyde (2ad):** yellow solid, 213.1 mg (80% yield), m.p. 90–92 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta 9.77\) (br s, 1H), 7.11 (s, 2H), 6.16 (br s, 1H), 4.10 (t, \(J = 7.2\) Hz, 4H), 1.81 (quint, \(J = 7.2\) Hz, 4H), 1.48 (sext, \(J = 7.2\) Hz, 4H), 0.96 (t, \(J = 7.2\) Hz, 6H); \(^13\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta 189.8, 149.0\) (2C), 147.0, 130.0, 108.7 (2C), 69.8 (2C), 28.0 (2C), 22.4 (2C), 14.0 (2C); HRMS (ESI): \(m/z\) [M–H\(^–\)] calcd. for C\(_{16}\)H\(_{19}\)O\(_4\) 265.1440, found 265.1446.

**3,5-Diisobutoxy-4-hydroxybenzaldehyde (2ae):** yellow solid, 221.0 mg (83% yield), m.p. 58–60 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta 9.79\) (br s, 1H), 7.11 (s, 2H), 6.03 (br s, 1H), 3.88 (d, \(J = 6.8\) Hz, 4H), 2.17 (heptet, \(J = 6.8\) Hz, 2H), 1.05 (d, \(J = 6.8\) Hz, 12H); \(^13\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta 189.9, 145.8\) (2C), 140.4, 127.2, 106.7 (2C), 74.8 (2C), 27.1 (2C), 18.2 (4C); HRMS (ESI): \(m/z\) [M–H\(^–\)] calcd. for C\(_{17}\)H\(_{21}\)O\(_4\) 265.1436.

**3-Hydroxy-4-hydroxy-5-methoxybenzaldehyde (2af):** yellow solid, 168.7 mg (86% yield), m.p. 72–74 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta 9.80\) (br s, 1H), 7.13 (d, \(J = 1.6\) Hz, 2H), 6.10 (br s, 1H), 4.21 (t, \(J = 6.8\) Hz, 2H), 3.97 (s, 3H), 1.49 (t, \(J = 6.8\) Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta 190.9, 147.4, 146.5, 141.0, 128.3, 107.6, 106.6, 65.1, 56.4, 14.8\); HRMS (ESI): \(m/z\) [M+H\(^+\)] calcd. for C\(_{16}\)H\(_{15}\)O\(_4\) 197.0814, found 197.0804.

**4-Hydroxy-3-methoxy-5-\(\alpha\)-propoxybenzaldehyde (2ag):** yellow solid, 172.4 mg (82% yield), m.p. 84–86 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta 9.80\) (br s, 1H), 7.14 (s, 2H), 6.06 (br s, 1H), 4.09 (t, \(J = 6.8\) Hz, 2H), 3.97 (s, 3H), 1.88 (sext, \(J = 6.8\) Hz, 2H), 1.06 (t, \(J = 6.8\) Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta 189.9, 146.4, 145.6, 140.0, 127.3, 106.6, 105.6, 70.0, 55.4, 21.4, 9.4\); HRMS (ESI): \(m/z\) [M+H\(^+\)] calcd. for C\(_{17}\)H\(_{17}\)O\(_4\) 211.0970, found 211.0962.

**3,5-Di-\(\alpha\)-tert-butyl-4-hydroxybenzaldehyde (2ah):** white solid, 181.5 mg (79% yield), m.p. 188–190 °C (lit\(^2\) m.p. 190–191 °C); \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta 9.85\) (br s, 1H), 7.73 (s, 2H), 5.85 (br s, 1H), 1.48 (s, 18H); \(^13\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta 191.9, 159.7\) (2C), 136.5, 128.7, 127.7 (2C), 34.4 (2C), 30.1 (6C); HRMS (ESI): \(m/z\) [M+H\(^+\)] calcd. for C\(_{15}\)H\(_{23}\)O\(_2\) 235.1698, found 235.1693.
3,5-Dimethyl-4-hydroxybenzaldehyde (2ai): white solid, 130.6 mg (87% yield), m.p. 112–114 °C (lit: m.p. 113–114 °C); ²H NMR (400 MHz, CDCl₃, ppm): δ 9.81 (br s, 1H), 7.54 (s, 2H), 5.46 (br s, 1H), 2.31 (s, 6H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 191.5, 158.1 (2C), 131.0, 129.3, 123.7 (2C), 15.8 (2C); HRMS (ESI): m/z [M+H⁺] calcd. for C₉H₁₀O₂ 151.0759, found 151.0750.

3-tert-Butyl-4-hydroxy-5-methylbenzaldehyde (2aj): white solid, 161.5 mg (84% yield), m.p. 148–150 °C (lit: m.p. 152–153 °C); ²H NMR (400 MHz, CDCl₃, ppm): δ 9.83 (br s, 1H), 7.71 (s, 1H), 7.70 (s, 1H), 5.49 (br s, 1H), 2.32 (s, 3H), 1.44 (s, 9H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 191.7, 158.6, 153.6, 136.4, 130.8, 128.0, 123.8, 34.7, 29.5 (3C), 15.9; HRMS (ESI): m/z [M+H⁺] calcd. for C₁₃H₁₇O₂ 193.1229, found 193.1234.

4-Hydroxy-3-methoxy-5-methylbenzaldehyde (2ak): white solid, 146.2 mg (88% yield), m.p. 98–100 °C; ²H NMR (400 MHz, CDCl₃, ppm): δ 9.80 (br s, 1H), 7.30 (s, 1H), 7.28 (s, 1H), 6.24 (br s, 1H), 3.96 (s, 3H), 2.32 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 191.2, 149.8, 146.7, 128.9, 128.8, 124.0, 106.7, 56.2, 15.3; HRMS (ESI): m/z [M+H⁺] calcd. for C₁₂H₁₅O₂ 167.0708, found 167.0706.

3-Ethoxy-4-hydroxy-5-methylbenzaldehyde (2al): white solid, 153.2 mg (85% yield), m.p. 86–88 °C; ²H NMR (400 MHz, CDCl₃, ppm): δ 9.78 (br s, 1H), 7.29 (s, 1H), 7.26 (s, 1H), 6.34 (br s, 1H), 4.19 (q, J = 6.8 Hz, 2H), 2.32 (s, 3H), 1.47 (t, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 191.3, 149.9, 146.1, 128.8, 128.7, 124.0, 107.5, 64.8, 15.4, 14.8; HRMS (ESI): m/z [M+H⁺] calcd. for C₁₀H₁₅O₂ 181.0865, found 181.0862.

4-Hydroxy-3-methyl-5-propoxybenzaldehyde (2am): white solid, 163.2 mg (84% yield), m.p. 110–112 °C; ²H NMR (400 MHz, CDCl₃, ppm): δ 9.79 (br s, 1H), 7.29 (s, 1H), 7.27 (s, 1H), 6.33 (br s, 1H), 4.08 (t, J = 6.8 Hz, 2H), 2.32 (s, 3H), 1.87 (sext, J = 6.8 Hz, 2H), 1.06 (t, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 191.3, 149.9, 146.1, 128.8, 128.7, 124.0, 107.5, 70.7, 22.4, 15.4, 10.5; HRMS (ESI): m/z [M+H⁺] calcd. for C₁₁H₁₃O₂ 195.1021, found 195.1020.

3-(Benzoxyl)-4-hydroxy-5-methylbenzaldehyde (2an): white solid, 203.5 mg (84% yield), m.p. 118–120 °C; ²H NMR (400 MHz, CDCl₃, ppm): δ 9.71 (br s, 1H), 7.40–7.18 (m, 7H), 6.24 (br s, 1H), 5.09 (s, 2H), 2.25 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 191.2, 149.9, 145.9, 135.6, 128.9, 128.85 (2C), 128.8, 128.7, 128.1 (2C), 124.4, 108.1, 71.3, 15.4; HRMS (ESI): m/z [M+H⁺] calcd. for C₁₇H₁₅O₃ 243.1021, found 243.1012.

5,5'-Methylenebis(3-tert-butyl-4-hydroxybenzaldehyde) (2ao): yellow solid, 283.7 mg (77% yield), m.p. 184–186 °C; ²H NMR (400 MHz, CDCl₃, ppm): δ 9.85 (br s, 2H), 7.75 (d, J = 2.0 Hz, 2H), 7.70 (d, J = 2.0 Hz, 2H), 6.73 (br s, 2H), 4.06 (s, 2H), 1.45 (s, 18H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 191.3 (2C), 157.5 (2C), 136.9 (2C), 131.0 (2C), 129.9 (2C), 128.2 (2C), 127.1 (2C), 34.5 (2C), 30.9, 29.8 (6C); HRMS (ESI): m/z [M+H⁺] calcd. for C₂₉H₂₉O₂ 369.2066, found 369.2045.
4-Hydroxy-2,3,5-trimethoxybenzaldehyde (2ap): white solid, 171.9 mg (81% yield), m.p. 112–114 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 10.25 (br s, 1H), 7.12 (s, 1H), 6.17 (br s, 1H), 3.98 (s, 3H), 3.97 (s, 3H), 3.91 (s, 3H); 13C NMR (100 MHz, CDCl3, ppm): δ 188.4, 152.5, 145.9, 144.2, 139.9, 121.0, 103.2, 62.8, 61.0, 56.4; HRMS (ESI): m/z [M+H+] calcd. for C10H13O5 213.0763, found 213.0760.

1-(4-Hydroxy-3,5-dimethoxyphenyl)ethanone (2ba): yellow solid, 164.8 mg (84% yield), m.p. 122–124 °C (liq); m.p. 121–122 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 7.26 (d, J = 4.0 Hz, 2H), 5.95 (br s, 1H), 3.96 (s, 6H), 2.58 (s, 3H); 13C NMR (100 MHz, CDCl3, ppm): δ 197.1, 147.4 (2C), 140.4, 129.5, 106.5 (2C), 57.1 (2C), 26.8; HRMS (EI): m/z [M] calcd. for C10H13O4 196.0736, found 196.0737.

1-(3,5-Diethoxy-4-hydroxyphenyl)ethanone (2bb): yellow solid, 183.9 mg (82% yield), m.p. 117–119 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 7.15 (s, 2H), 6.02 (br s, 1H), 4.10 (q, J = 6.8 Hz, 4H), 2.48 (s, 3H), 1.40 (t, J = 6.8 Hz, 6H); 13C NMR (100 MHz, CDCl3, ppm): δ 197.3, 146.7 (2C), 141.1, 129.4, 107.7 (2C), 65.8 (2C), 26.9, 15.5 (2C); HRMS (EI): m/z [M] calcd. for C12H16O4 224.1049, found 224.1047.

1-(4-Hydroxy-3,5-di-n-propoxyphenyl)ethanone (2be): white solid, 201.0 mg (80% yield), m.p. 98–100 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 7.23 (s, 2H), 5.93 (br s, 1H), 4.07 (t, J = 6.8 Hz, 4H), 2.55 (s, 3H), 1.88 (m, 4H), 1.06 (t, J = 7.2 Hz, 6H); 13C NMR (100 MHz, CDCl3, ppm): δ 196.7, 146.2 (2C), 140.4, 128.7, 107.0 (2C), 71.1 (2C), 26.3, 22.5 (2C), 10.4 (2C); HRMS (EI): m/z [M] calcd. for C16H20O4 252.1362, found 252.1361.

1-(3,5-Di-n-butoxy-4-hydroxyphenyl)ethanone (2bd): white solid, 215.9 mg (77% yield), m.p. 90–92 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 7.23 (s, 2H), 5.93 (br s, 1H), 4.11 (t, J = 6.8 Hz, 4H), 2.55 (s, 3H), 1.83 (m, 4H), 1.52 (m, 4H), 0.99 (q, J = 7.2 Hz, 6H); 13C NMR (100 MHz, CDCl3, ppm): δ 197.3, 146.8 (2C), 141.1, 129.3, 107.6 (2C), 70.0 (2C), 31.9 (2C), 26.9, 19.8 (2C), 14.4 (2C); HRMS (EI): m/z [M] calcd. for C22H28O4 280.1675, found 280.1676.

1-(3-tert-butyl-4-hydroxy-5-methoxyphenyl)ethanone (2be): yellow solid, 175.6 mg (79% yield), m.p. 82–84 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 7.58 (s, 1H), 7.43 (s, 1H), 6.47 (br s, 1H), 3.95 (s, 3H), 2.57 (s, 3H), 1.43 (s, 9H); 13C NMR (100 MHz, CDCl3, ppm): δ 197.7, 149.7, 147.3, 135.5, 129.3, 122.1, 108.3, 56.9, 35.3, 29.8 (3C), 26.7; HRMS (EI): m/z [M] calcd. for C14H14O3 222.1256, found 222.1254.

1-(3-tert-Butyl-5-ethoxy-4-hydroxyphenyl)ethanone (2bf): white solid, 181.9 mg (77% yield), m.p. 80–82 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 7.57 (s, 1H), 7.41 (s, 1H), 6.54 (br s, 1H), 4.18 (q, J = 6.8 Hz, 2H), 2.56 (s, 3H), 1.47 (t, J = 6.8 Hz, 3H), 1.43 (s, 9H); 13C NMR (100 MHz, CDCl3, ppm): δ 197.2, 149.1, 145.9, 134.8, 128.6, 121.3, 108.5, 64.9, 34.7, 29.2 (3C), 26.2, 14.8; HRMS (EI): m/z [M] calcd. for C14H14O3 236.1412, found 236.1414.
1-(3-tert-Butyl-4-hydroxy-5-\(n\)-propoxyphenyl)ethanone (2bg): yellow solid, 190.2 mg (76\% yield), m.p. 78–80 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.59 (s, 1H), 7.43 (s, 1H), 6.57 (br s, 1H), 4.09 (t, \(J = 6.8\) Hz, 2H), 2.58 (s, 3H), 1.89 (sext, \(J = 6.8\) Hz, 2H), 1.46 (s, 9H), 1.09 (t, \(J = 6.8\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta\) 197.2, 149.1, 146.0, 134.8, 128.6, 121.3, 108.5, 70.8, 34.7, 29.2 (3C), 26.2, 22.5, 10.5; HRMS (EI): \(m/z\) [M\(^+\)] calcd. for C\(_{24}\)H\(_{24}\)O\(_3\): 250.1569, found 250.1570.

1-(4-Hydroxy-3,5-dimethylphenyl)ethanone (2bh): yellow solid, 136.3 mg (83\% yield), m.p. 157–159 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.64 (s, 2H), 5.43 (br s, 1H), 2.54 (s, 3H), 2.30 (s, 6H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta\) 197.6, 157.0, 129.6 (3C), 123.1 (2C), 26.3, 16.0 (2C); HRMS (EI): \(m/z\) [M\(^+\)] calcd. for C\(_{10}\)H\(_{12}\)O\(_2\): 164.0834, found 164.0838.

1-(4-Hydroxy-3,5-dimethoxyphenyl)propan-1-one (2ca):\(^6\) white solid, 172.4 mg (82\% yield), m.p. 109–111 °C (lit\(^6\) m.p. 109–110 °C); \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.26 (s, 2H), 6.09 (br s, 1H), 3.95 (s, 6H), 2.97 (q, \(J = 7.2\) Hz, 2H), 1.22 (t, \(J = 7.2\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta\) 199.3, 146.8 (2C), 139.5, 128.5, 105.4 (2C), 56.4 (2C), 31.3, 8.5; HRMS (EI): \(m/z\) [M\(^+\)] calcd. for C\(_{11}\)H\(_{16}\)O\(_2\): 210.0892, found 210.0893.

1-(4-Hydroxy-3,5-dimethoxyphenyl)butan-1-one (2da): yellow solid, 168.2 mg (75\% yield), m.p. 89–91 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.26 (s, 2H), 5.93 (br s, 1H), 3.96 (s, 6H), 2.91 (t, \(J = 7.2\) Hz, 2H), 1.78 (sext, \(J = 7.2\) Hz, 2H), 1.01 (t, \(J = 7.2\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta\) 198.9, 146.7 (2C), 139.5, 128.7, 105.4 (2C), 56.5 (2C), 40.1, 18.0, 13.9; HRMS (EI): \(m/z\) [M\(^+\)] calcd. for C\(_{12}\)H\(_{18}\)O\(_2\): 224.1049, found 224.1047.

1-(4-Hydroxy-3,5-dimethoxyphenyl)pentan-1-one (2ea): yellow solid, 166.8 mg (70\% yield), m.p. 76–78 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.26 (s, 2H), 5.93 (br s, 1H), 3.96 (s, 6H), 2.92 (t, \(J = 7.6\) Hz, 2H), 1.71 (sext, \(J = 7.6\) Hz, 2H), 1.42 (sext, \(J = 7.6\) Hz, 2H), 0.96 (t, \(J = 7.6\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta\) 199.0, 146.7 (2C), 139.5, 128.7, 105.5 (2C), 56.5 (2C), 37.9, 26.8, 22.5, 14.0; HRMS (ESI): \(m/z\) [M\(^+\)H\(^+\)] calcd. for C\(_{13}\)H\(_{20}\)O\(_2\): 239.1283, found 239.1277.

(4-Hydroxy-3,5-dimethoxyphenyl)(phenyl)methanone (2fa): yellow solid, 170.5 mg (66\% yield), m.p. 124–126 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.77 (d, \(J = 8.0\) Hz, 2H), 7.59 (t, \(J = 7.2\) Hz, 1H), 7.49 (t, \(J = 7.2\) Hz, 2H), 7.13 (s, 2H), 5.98 (br s, 1H), 3.92 (s, 6H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta\) 195.5, 146.6 (2C), 139.4, 138.2, 132.0, 129.7 (2C), 128.6, 128.2 (2C), 107.8 (2C), 56.5 (2C); HRMS (EI): \(m/z\) [M\(^+\)] calcd. for C\(_{21}\)H\(_{19}\)O\(_3\): 358.0892, found 358.0896.

1-(4-Hydroxy-3,5-dimethylphenyl)-2-methylpropan-1-one (2gh): pale yellow solid, 132.7 mg (69\% yield), m.p. 104–106 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.65 (s, 2H), 5.24 (br s, 1H), 3.52 (heptet, \(J = 6.8\) Hz, 1H), 2.29 (s, 6H), 1.19 (d, \(J = 6.8\) Hz, 6H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): \(\delta\) 203.9, 156.8, 129.5 (2C), 128.5 (2C), 123.1, 34.8, 19.4 (2C), 16.0 (2C); HRMS (EI): \(m/z\) [M\(^+\)] calcd. for C\(_{12}\)H\(_{16}\)O\(_2\): 192.1150, found 192.1151.
1-(4-Hydroxy-3,5-dimethylphenyl)-2,2-dimethylpropan-1-one (2hh): yellow oil, 127.9 mg (62% yield); $^1$H NMR (400 MHz, CDCl$_3$, ppm): $\delta$ 7.51 (s, 2H), 5.30 (br s, 1H), 2.27 (s, 6H), 1.36 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 207.3, 155.2, 129.8 (4C), 122.5, 44.0, 28.5 (3C), 16.0 (2C); HRMS (EI): m/z [M$^+$] calcd. for C$_{13}$H$_{16}$O$_2$ 206.1307, found 206.1304.

1-(4-Hydroxy-3,5-dimethylphenyl)-2-phenylethanone (2ih): white solid, 168.2 mg (70% yield), m.p. 110–112 °C; $^1$H NMR (400 MHz, CDCl$_3$, ppm): $\delta$ 7.69 (s, 2H), 7.37–7.19 (m, 5H), 5.23 (br s, 1H), 4.22 (s, 2H), 2.27 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 197.1, 157.1, 135.1, 130.0 (2C), 129.4 (2C), 129.0, 128.6 (2C), 126.7 (2C), 123.2, 45.1, 16.0 (2C); HRMS (EI): m/z [M$^+$] calcd. for C$_{19}$H$_{18}$O$_2$ 240.1150, found 240.1149.

1-(4-Hydroxy-3,5-dimethylphenyl)-3-phenylpropan-1-one (2jh): white solid, 185.6 mg (73% yield), m.p. 99–101 °C; $^1$H NMR (400 MHz, CDCl$_3$, ppm): $\delta$ 7.64 (s, 2H), 7.32–7.18 (m, 5H), 5.25 (br s, 1H), 3.24 (t, $J = 7.2$ Hz, 2H), 3.04 (t, $J = 7.2$ Hz, 2H), 2.27 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 198.6, 156.9, 141.5, 129.4 (2C), 129.3, 128.5 (2C), 128.4 (2C), 126.1 (2C), 123.1, 40.1, 30.5, 16.0 (2C); HRMS (EI): m/z [M$^+$] calcd. for C$_{23}$H$_{20}$O$_2$ 254.1307, found 254.1309.

3,5-Dibromo-4-hydroxybenzaldehyde (2aq)$^8$: white solid, 72.8 mg (26% yield), m.p. 182–184 °C (lit$^8$ m.p. 183 °C); $^1$H NMR (400 MHz, CDCl$_3$, ppm): $\delta$ 9.80 (br s, 1H), 8.00 (s, 2H), 6.40 (br s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 188.2, 154.4, 133.7 (2C), 131.3, 110.7 (2C); HRMS (ESI): m/z [M–H$^+$] calcd. for C$_{13}$H$_{12}$Br$_2$O$_2$ 276.8500, found 276.8490. Recovery of the starting material: 172.2 mg (65%).

3,5-Dichloro-4-hydroxybenzaldehyde (2ar): white solid, 34.4 mg (18% yield), m.p. 160–162 °C; $^1$H NMR (400 MHz, CDCl$_3$, ppm): $\delta$ 9.82 (s, 1H), 7.83 (s, 2H), 6.43 (br s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 188.5, 152.9, 130.1 (2C), 129.8 (2C), 122.2; HRMS (ESI): m/z [M–H$^+$] calcd. for C$_{13}$H$_{12}$Cl$_2$O$_2$ 188.9510, found 188.9502. Recovery of the starting material: 132.8 mg (75%).

3-Bromo-5-fluoro-4-hydroxybenzaldehyde (2as): white solid, 19.7 mg (9% yield), m.p. 138–140 °C; $^1$H NMR (400 MHz, CDCl$_3$, ppm): $\delta$ 9.74 (d, $J = 2.0$ Hz, 1H), 7.78 (t, $J = 1.6$ Hz, 1H), 7.54 (dd, $J = 9.6, 2.0$ Hz, 1H), 6.33 (br s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 188.7, 151.1 (d, $J = 247.3$), 147.2 (d, $J = 15.0$), 130.6 (d, $J = 2.7$), 130.0 (d, $J = 5.4$), 115.6 (d, $J = 18.8$), 111.4 (d, $J = 1.4$); HRMS (ESI): m/z [M–H$^+$] calcd. for C$_{13}$H$_{12}$BrFO$_2$ 216.9300, found 216.9281. Recovery of the starting material: 179.6 mg (82%).

The substrate failed to undergo the oxidation. Recovery of the starting material: 151.8 mg (96%).
3-Bromo-4-hydroxy-5-methoxybenzaldehyde (2au): white solid, 78.6 mg (34% yield), m.p. 162–164 °C (lit8 m.p. 163–166 °C); 1H NMR (400 MHz, CDCl3, ppm): δ 9.79 (br s, 1H), 7.64 (d, J = 1.6 Hz, 1H), 7.36 (d, J = 1.6 Hz, 1H), 6.50 (br s, 1H), 3.99 (s, 3H); 13C NMR (100 MHz, CDCl3, ppm): δ 189.8, 148.9, 147.7, 130.2, 130.0, 108.2, 108.0, 56.6; HRMS (ESI): m/z [M–H+] calcd. for C13H9BrO2 228.9500, found 228.9473. Recovery of the starting material: 130.2 mg (60%).

3-Fluoro-4-hydroxy-5-methoxybenzaldehyde (2av): white solid, 44.2 mg (26% yield), m.p. 116–118 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 9.81 (s, 1H), 7.33–7.25 (m, 2H), 5.98 (br s, 1H), 4.00 (s, 3H); 13C NMR (100 MHz, CDCl3, ppm): δ 190.0 (d, J = 2.3), 150.4 (d, J = 243.8), 148.7 (d, J = 5.3), 140.0 (d, J = 13.5), 128.1 (d, J = 6.3), 113.0 (d, J = 18.5), 106.0 (d, J = 1.7), 56.7; HRMS (ESI): m/z [M+H+] calcd. for C13H8FNO 171.0457, found 171.0447. Recovery of the starting material: 104.6 mg (67%).

5-Formyl-2-hydroxy-3-methoxybenzonitrile (2aw): white solid, 28.3 mg (16% yield), m.p. 196–198 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 9.84 (s, 1H), 7.66 (s, 1H), 7.58 (s, 1H), 6.90 (br s, 1H), 4.04 (s, 3H); 13C NMR (100 MHz, DMSO-d6, ppm): δ 190.6, 156.1, 148.9, 129.6, 129.2, 116.3, 113.6, 99.7, 56.8; HRMS (EI): m/z [M+I+] calcd. for C14H11ON2 228.9400, found 228.9426. Recovery of the starting material: 125.6 mg (77%).

1-(3,5-Dibromo-4-hydroxyphenyl)ethane (2bi): white solid, 67.6 mg (23% yield), m.p. 185–187 °C; 1H NMR (400 MHz, CDCl3, ppm): δ 8.08 (s, 2H), 6.33 (br s, 1H), 2.56 (s, 3H); 13C NMR (100 MHz, CDCl3, ppm): δ 194.3, 153.3, 132.6 (2C), 131.9, 110.1 (2C), 26.3; HRMS (ESI): m/z [M+H+] calcd. for C14H11Br2O2 292.8813, found 292.8817. Recovery of the starting material: 207.2 mg (74%).

2.3 General procedure for the Cu(OAc)2-catalyzed oxidation of 4-hydroxybenzyl alcohols and 4-hydroxybenzyl ethers 3 (Table 2 in the text).

Table 2. Cu(OAc)2-catalyzed oxygenation of 3.

<table>
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<th>Entry</th>
<th>Substrate 3</th>
<th>Product 2</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
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<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td>96</td>
</tr>
</tbody>
</table>
General procedure: a mixture of substrate 3 (1.0 mmol) and Cu(OAc)$_2$ (0.01 mmol, 1.8 mg) in EG (2 mL) was stirred at 50 °C under ambient air for 8 h. Hydrochloric acid (4 mL, 2%) and MTBE (4 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (4 mL × 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to provide the corresponding product 2.

3,5-Dimethoxy4-hydroxybenzaldehyde (2aa): yellow solid, 174.9 mg (96% yield, from 3a); 156.7 mg (86% yield, from 3b); 171.2 mg (94% yield, from 3e). The spectral data see 2.1 section.

3-Ethoxy-4-hydroxy-5-methoxybenzaldehyde (2af): yellow solid, 182.5 mg (93% yield, from 3d); 164.8 mg (84% yield, from 3e); 160.9 mg (82% yield, from 3f). The spectral data see 2.2 section.

3,5-dimethyl-4-hydroxybenzaldehyde (2ai): white solid, 138.2 mg (92% yield, from 3g); 127.6 mg (85% yield, from 3h). The spectral data see 2.2 section.

4-Hydroxy-3-methoxy-5-methylbenzaldehyde (2ak): white solid, 154.5 mg (93% yield, from 3i); 144.6 mg (87% yield, from 3j). The spectral data see 2.2 section.

1-(4-Hydroxy-3,5-dimethoxyphenyl)ethanone (2ba): yellow solid, 178.5 mg (91% yield, from 3k); 162.8 mg (83% yield, from 3l). The spectral data see 2.2 section.

1-(4-Hydroxy-3,5-dimethoxyphenyl)propan-1-one (2ca): white solid, 189.2 mg (90% yield, from 3m); 170.3 mg (81% yield, from 3n). The spectral data see 2.2 section.

2.4 The gram-scale oxidations of 1aa and 1ba (Scheme 4 in the text).
General procedure: a mixture of substrate 1aa or 1ba (10 mmol) and Cu(OAc)$_2$ (0.1 mmol, 18 mg) in EG (8 mL) was stirred at 50 °C under ambient air for 18 h. Hydrochloric acid (15 mL, 2%) and MTBE (15 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (15 mL $\times$ 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to provide the corresponding product 2.

3,5-Dimethoxy-4-hydroxybenzaldehyde (2aa):$^1$ yellow solid, 1.585 g (87% yield).

1-(4-Hydroxy-3,5-dimethoxyphenyl)ethanone (2ba):$^5$ yellow solid, 1.570 g (80% yield).

2.5 Limitations of Cu(OAc)$_2$-catalyzed oxygenation (Scheme 5 in the text).
Scheme 5. Limitations of Cu(OAc)₂-catalyzed oxygenation due to (A) undesired coupling or (B) inhibited oxygenation.

**Procedure for Scheme 5(A):** a mixture of substrate (1.0 mmol) and Cu(AcO)₂ (0.01 mmol, 1.8 mg) in EG (2 mL) was stirred at 75 °C under ambient air for 12 h. Hydrochloric acid (4 mL, 2%) and MTBE (4 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (4 mL × 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to provide the corresponding products.

**Recovery of the starting material:** 20.5 mg (19%).

**5,5'-Dimethylbiphenyl-2,2'-diol (4):** white solid, 79.3 mg (37% yield), m.p. 148–150 °C; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.11 (dd, J = 8.0, 1.6 Hz, 2H), 7.06 (d, J = 1.6 Hz, 2H), 6.92 (d, J = 8.0 Hz, 2H), 5.43 (br s, 2H), 2.32 (s, 6H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 150.6 (2C), 131.6 (2C), 130.8 (2C), 130.3 (2C), 123.7 (2C), 116.5 (2C), 20.5 (2C); HRMS (EI): m/z [M⁺] calcd. for C₁₄H₁₄O₂ 214.0994, found 214.0992.

**4-Hydroxybenzaldehyde:** yellow solid, 11.0 mg (9% yield), m.p. 116–118 °C; ¹H NMR (400 MHz, CDCl₃, ppm): δ 9.87 (br s, 1H), 7.83 (d, J = 8.8 Hz, 2H), 7.98 (d, J = 8.8 Hz, 2H), 6.29 (br s, 1H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 191.2, 161.5, 132.5 (2C), 129.9, 116.0 (2C); HRMS (EI): m/z [M⁺] calcd. for C₇H₆O₂ 122.0368, found 122.0367.
Recycling of the starting material: 78.6 mg (42%).

3-Bromo-4-hydroxybenzaldehyde: white solid, 14.1 mg (7% yield), m.p. 130–132 °C; ¹H NMR (400 MHz, CDCl₃, ppm): δ 9.83 (br s, 1H), 8.04 (s, 1H), 7.77 (d, J = 8.4 Hz, 1H), 7.15 (d, J = 8.0 Hz, 1H), 6.43 (s, 1H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 192.7, 151.8, 132.9, 130.3, 128.1, 127.5, 127.4; HRMS (ESI): m/z [M-H⁺] calcd. for C₇H₄BrO₂ 198.9395, found 198.9359.

3,3'-Dimethoxy-5,5'-dimethylbiphenyl-2,2'-diol (5): brown solid, 133.0 mg (97% yield), m.p. 132–134 °C (lit. m.p. 133–135 °C); ¹H NMR (400 MHz, CDCl₃, ppm): δ 6.73 (s, 2H), 6.72 (s, 2H), 5.96 (br s, 2H), 3.91 (s, 6H), 2.33 (s, 6H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 147.1 (2C), 140.3 (2C), 129.6 (2C), 124.4 (2C), 123.4 (2C), 111.3 (2C), 56.0 (2C), 21.2 (2C); HRMS (ESI): m/z [M+H⁺] calcd. for C₁₆H₁₉O₄ 275.1283, found 275.1283.

Procedure for Scheme 5(B): a mixture of corresponding substrate (1.0 mmol) and Cu(AcO)₂ (0.01 mmol, 1.8 mg) in EG (2 mL) was stirred at 95 °C under ambient air. No reaction occurred after 12 h monitored by TLC.

2.5 Mechanistic studies (Scheme 6 in the text).

Scheme 6. Mechanistic studies.

Procedure for Scheme 6(a): a mixture of substrate 1aa (1.0 mmol, 168.2 mg) and Cu(AcO)₂ (0.01 mmol, 1.8 mg) in EG (2 mL) was stirred at 50 °C under ambient air for 4 h. Hydrochloric acid (5.0 mL, 2%) and MTBE (4 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (4 mL × 2). The combined organic layers were dried over anhydrous...
sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to provide intermediate 3c and product 2aa.

**4-(2-Hydroxyethoxy)methyl-2,6-dimethoxyphenol (3c):** yellow solid, 61.6 mg (27% yield), m.p. 78–80 °C; $^1$H NMR (400 MHz, CDCl$_3$, ppm): $\delta$ 6.58 (s, 2H), 5.52 (br s, 1H), 4.48 (s, 2H), 3.90 (s, 6H), 3.77 (t, $J$ = 4.4 Hz, 2H), 3.60 (t, $J$ = 4.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 147.0 (2C), 134.3, 128.9, 104.7 (2C), 73.7, 71.2, 61.9, 56.3 (2C); HRMS (ESI): m/z [M+Na]$^+$ calcld. for C$_{11}$H$_{16}$O$_3$Na 251.0895, found 251.0894.

![Structure of 4-(2-Hydroxyethoxy)methyl-2,6-dimethoxyphenol (3c)](image)

**3,5-Dimethoxy-4-hydroxybenzaldehyde (2aa):** yellow solid, 125.7 mg (69% yield). The spectral data see 2.1 section.

**Procedure for Scheme 6(b):** a mixture of substrate 1aa (1.0 mmol) and dried Cu(AcO)$_2$ (dried in a vacuum oven at 60 °C for 10 h, 4.0 mmol, 726.5 mg) in dry EG (15 mL, dried over 4 Å molecular sieve for 24 h) was stirred at 50 °C under argon atmosphere for 2 h. Hydrochloric acid (10 mL, 1%) and MTBE (10 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (10 mL × 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to provide intermediate 3c and the desired 2aa.

**3,5-Dimethoxy-4-hydroxybenzaldehyde (2aa):** yellow solid, 154.8 mg (85% yield). The spectral data see 2.1 section.

**Procedure for Scheme 6(c):** a mixture of substrate 1aa (1.0 mmol) and dried Cu(AcO)$_2$ (dried in a vacuum oven at 60 °C for 10 h) and H$_2$O (4.0 mmol, 72 mg) in dry EG (15 mL, dried over 4 Å molecular sieve for 24 h) was stirred at 50 °C under argon atmosphere for 2 h. Hydrochloric acid (10 mL, 1%) and MTBE (10 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (10 mL × 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to provide intermediate 3c and the desired 2aa.

**3,5-Dimethoxy-4-hydroxybenzaldehyde (2aa):** yellow solid, 154.8 mg (85% yield). The spectral data see 2.1 section.

**Procedure for Scheme 6(d):** a mixture of substrate 1aa (1.0 mmol, 168.2 mg) and dried Cu(AcO)$_2$ (0.01 mmol, 1.8 mg, dried in a vacuum oven at 60 °C for 10 h) in dry EG (2 mL, dried over 4 Å molecular sieve for 24 h) was stirred at 50 °C under $^{18}$O$_2$ for 8 h. Hydrochloric acid (4 mL, 2%) and MTBE (4 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (4 mL × 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1).
4-((2-Hydroxyethoxy)methyl)-2,6-dimethoxyphenol (3c): yellow solid, 20.5 mg (9% yield). HRMS (EI): m/z [M⁺] calcd. for C₁₉H₁₅O₅ 228.0998, found 228.0997 (herein HRMS determined again for the mechanistic studies). Other spectral data see 2.5 section.

3,5-Dimethoxy-4-hydroxybenzaldehyde (2aa): yellow solid, 152.9 mg (83% yield), HRMS (EI): m/z [M⁺] calcd. for C₉H₁₀O₃ 184.0622, found 184.0623. Other spectral data see 2.1 section.

Procedure for Scheme 6(e): a mixture of substrate 1aa (1.0 mmol, 168.2 mg), Cu(AcO)₂ (0.01 mmol, 1.8 mg) and TEMPO (1.0 mmol, 156.3 mg) in EG (2 mL) was stirred at 50 °C under ambient air for 4 h. Hydrochloric acid (4 mL, 2%) and MTBE (4 mL) were added to the reaction mixture successively. The MTBE phase was separated, and the aqueous phase was further extracted with MTBE (4 mL × 2). The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 5:1) to recover 1aa.

2,6-dimethoxy-4-methylphenol (1aa): recovery, 161.5 mg (96%).

3. References
4. Copies of Spectra for All Compounds
# Elemental Composition Report

## Single Mass Analysis

Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron ions
15 formula(e) evaluated with 8 results within limits (up to 1 closest results for each mass)
Elements Used:
- C: 0.21
- H: 0.50
- O: 0.4

**YF-JI**

![Chemical Structure](image)

**JYF-JA-01** 1 (0.128) Cm (1:2)

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ECUST Institute of Fine Chem

02-Jan-2013
20/20/35
1: TCPS ES+ 5.36e+002
Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
25 formula(e) evaluated with 15 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-39  H: 0-60  O: 0-8

YF-11

EUST institute of Fine Chem

02-Jan-2013
20:35:36
1: TOF MS ES+
5.08e+002

Minimum:      100.0  50.0  -1.5
Maximum:      100.0  50.0  100.0

Mass  Calc. Mass  mDa  FTM  DBE  i-FIT  i-FIT (Norm)  Formula
211.0962  211.0970  -0.8  -3.8  4.5  5.5  0.0  C11 H15 O4
Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
18 formula(e) evaluated with 10 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-21    H: 0-50    O: 0-4

YF-II
JYF-JA-C3 8 (0.323) Cm (8:10)
ECUST institute of Fine Chem

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
239.1292 239.1293  -0.1  -0.4  4.5  15.0  0.0  C13 H19 O4
Elemental Composition Report

Single Mass Analysis

Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron ion

30 formula(e) evaluated with 17 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-39  H: 0-60  O: 0-8

YF-JI

ECUST Institute of Fine Chem

JYF-JA-04 12 (0.465) Cm (7.14)

02-Jan-2013
20:40:12
2: TOF MS ESI:
1.30e+003

Minimum: -1.5
Maximum: 100.0 50.0 100.0

Mass  Calc. Mass  mDa  FFN  DBE  i-FIT  i-FIT (Norm)  Formula
265.1446  265.1440  0.6  2.3  5.5  9.3  0.0  C15 H21 O4
Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
30 formula(e) evaluated with 17 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-39  H: 0-60  O: 0-8

YF-JI

ECUST Institute of Fine Chem

JYF-JA-05 9 (0.394) Crn (8.12)

02-Jan-2013
20:42:48
2: TOF MS ES-
3.48e+003

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Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa
DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron ions
22 formula(s) evaluated with 16 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-39  H: 0-60  O: 0-8

YF-JI

ECUST Institute of Fire Chem

JYF-JA-065 (0.274) Cm (5.6)

02-Jan-2013
20:45:03
1: TOF MS ESI+
1.40e+003

Mass  Calc. Mass  mDa  PPB  DBE  i-FIT  F-HIT (Norm) Formula
197.0804  197.0814  -0.0  -5.1  4.5  14.0  0.0  Cl0 H13 O4
Elemental Composition Report

Single Mass Analysis

Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
25 formula(e) evaluated with 16 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-39  H: 0-60  O: 0-8

YF-JI
ECUST institute of Fine Chem

JYF-JA-02 19 (0.668) Cm (19/20)

1: TDF MSE+  5.08e+002

Minimum:  -1.5
Maximum:   100.0  50.0  100.0

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Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
29 formula(c) evaluated with 13 results within limits (up to 1 closest results for each mass)
Elements Used
C: 0-39    H: 0-60    O: 0-8
YF-JI
ECUST institute of Fine Chem

JVF-JA-06 B (0.323) Cm (7.10)

02-Jan-2013
20:57:01
1: TOP MS ES+
9.47e+002

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Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
20 formula(e) evaluated with 12 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-39  H: 0-60  O: 0-8

YPJI
ECUST Institute of Fine Chem

JYF-JA-08 33 (1.120) Cm (21:39)

Minimum: 100.0  80.0  100.0
Maximum: -1.5

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02-Jan-2013
20:46:42
1: TOF MS ES+ 1.23e+003
Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron ions
15 formula(e) evaluated with 10 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-37  H: 0-50  O: 0-4

YF-JI
ECUST institute of Fine Chem

JYF-JA-10 10 (0.396) Cm (9.10)

02-Jan-2013
21 00:40
1: TOF MS ES-
2.95e+003

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
193.1234  193.1229  0.5  2.6  4.5  24.0  0.0  C12 H17 O2
**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for I-FIT = 2

Monoisotopic Mass, Even Electron ions
21 formula(c) evaluated with 13 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-39 H: 0-60 O: 0-8

ECUST institute of Fine Chem

JYF-JA-11 1B (0.643) Cm (17.21)

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02-Jan-2013
21:03:40
1: TOF MS ES+
1.53e+003
Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa  /  DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
21 formula(e) evaluated with 15 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-39  H: 0-60  O: 0-8

ECUST institute of Fine Chem

02-Jan-2013
21:06:26
1: TOF MS ES+
2.52e+003

Minimum:
181.0857
181.0865

Maximum:
181.0862
182.0901

Mass  Calc. Mass  mDa  FPP  DBE  i-FIT  i-FIT (Norm)  Formula
181.0862  181.0865  -0.3  -1.7  4.5  28.7  0.0  C10 H13 O3
**Elemental Composition Report**

**Single Mass Analysis**

- Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
- Element prediction: Off
- Number of isotope peaks used for i-FIT = 2

**Monoisotopic Mass, Even Electron Ions**

24 formula(e) evaluated with 15 results within limits (up to 1 closest results for each mass)

**Elements Used:**

- C: 0-39
- H: 0-60
- O: 0-8

**YF-J1**

JYF-JA-13 12 (0.44B) Cm (10:14)

---

**Minimum:**

- Tolerance: 100.0
- DBE: min = -1.5

**Maximum:**

- Tolerance: 100.0
- DBE: max = 100.0

**Mass**

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**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
29 formula(c) evaluated with 16 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-39  H: 0-60  O: 0-8

YF-JI

ECUST institute of Fine Chem

JYF-JA-1422 (0.766) Cm (21.22)

02-Jan-2013
21:11:34
1: TOF MS ES+
3.57e+003

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Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
21 formula(e) evaluated with 11 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-37  H: 0-50  O: 0-4

YF-JI

ECUST institute of Fine Chem

JYF-JA-17 7 (0.269) Cm: (5:7)

02-Jan-2013
21:16:48
1: TOF MS ES+
3.00e+003

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Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
24 formula(s) evaluated with 17 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-39  H: 0-60  O: 0-8

YF-JI

ECUST Institute of Fine Chem

JYF-JA-16 6 (0.275) Cm (5.5)

Minimum: 100.0  50.0  100.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
213.0763  213.0763  -0.3  -1.4  4.5  14.1  0.0  C10 H13 O5

02-Jan-2013  21:14:32
1: TOF MS ES+
1.00e+003
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa  /  DAE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:
C: 0-10  H: 0-12  O: 0-4

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Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa  /  DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-12  H: 0-16  O: 0-4

Minimum:  -1.5
Maximum:  100.00  5.0  10.0  50.0

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Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-14    H: 0-20    O: 0-4

Minimum:   -1.5
Maximum:  100.00    5.0    10.0    50.0

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Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa  /  DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

| C | 0-16 | H | 0-24 | O | 0-4 |

Mininum:  

-1.5

Maximum:  

100.00  5.0  10.0  50.0

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Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-13  H: 0-18  O: 0-3

Minimum:  -1.5

Maximum:   100.00  5.0  10.0  50.0

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Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-14  H: 0-20  O: 0-3

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>236.1414</td>
<td>43.88</td>
<td>236.1412</td>
<td>0.2</td>
<td>0.8</td>
<td>5.0</td>
<td>1</td>
<td>C14 H20 O3</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

2.1 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-15  H: 0-22  O: 0-8

Minimum: -1.5

Maximum: 100.00  5.0  10.0  50.0

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>250.1570</td>
<td>44.79</td>
<td>250.1560</td>
<td>0.1</td>
<td>0.4</td>
<td>5.0</td>
<td>1</td>
<td>C15 H22 O3</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

23 formula(e) evaluated with 6 results within limits (up to 50 closest results for each mass)

Elements Used:

<table>
<thead>
<tr>
<th>C: 0-10</th>
<th>H: 0-12</th>
<th>O: 0-2</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
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<tbody>
<tr>
<td>164.0838</td>
<td>40.80</td>
<td>164.0837</td>
<td>0.1</td>
<td>0.4</td>
<td>5.0</td>
<td>1</td>
<td>C10H12O2</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa  /  DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-11     H: 0-14     O: 0-4

<table>
<thead>
<tr>
<th>Mass</th>
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<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
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</thead>
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<tr>
<td>210.0893</td>
<td>29.89</td>
<td>210.0892</td>
<td>0.1</td>
<td>0.5</td>
<td>5.0</td>
<td>1</td>
<td>C11 H14 O4</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis:

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-12   H: 0-16   O: 0-4

Micromass GCT

<table>
<thead>
<tr>
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<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
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</thead>
<tbody>
<tr>
<td>224.1047</td>
<td>0.94</td>
<td>224.1049</td>
<td>-0.2</td>
<td>-0.7</td>
<td>5.0</td>
<td>1</td>
<td>C12 H16 O4</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis
Tolerance = 30.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
6 formula(s) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used
C: 0-13  H: 0-80  O: 0-4

ECUST institute of Fine Chem

JYF-JG-4 36 (0.311) Cn (50.37)

Minimum:
Maximum:

Mass  Calc. Mass  mDa  PPm  DBE  i-FIT  i-FIT (Norm)  Formula
239.1277  239.1283  -0.6  -2.5  4.5  19.7  0.0  C13 H15 O4
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-15     H: 0-14     O: 0-4

<table>
<thead>
<tr>
<th>Mass</th>
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<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>258.0896</td>
<td>88.21</td>
<td>258.0892</td>
<td>0.4</td>
<td>1.5</td>
<td>9.0</td>
<td>1</td>
<td>C15 H14 O4</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

18 formula(e) evaluated with 7 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-12    H: 0-16    O: 0-2

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>192.1151</td>
<td>6.35</td>
<td>192.1150</td>
<td>0.1</td>
<td>0.4</td>
<td>5.0</td>
<td>1</td>
<td>C12 H16 O2</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

27 formula(e) evaluated with 6 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-13 H: 0-18 O: 0-2

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>206.1304</td>
<td>3.12</td>
<td>206.1307</td>
<td>-0.3</td>
<td>-1.4</td>
<td>5.0</td>
<td>1</td>
<td>C13 H18 O2</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa  /  DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

54 formula(e) evaluated with 10 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-16  H: 0-16  O: 0-2

Minima:

Mass  RA  Calc. Mass  mDa  PPM  DBE  Score  Formula
240.1149  0.64  240.1150  -0.1  -0.5  9.0  1  C16 H16 O2
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

27 formula(e) evaluated with 6 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-17  H: 0-18  O: 0-2

Minimum: 3.00  -1.5

Maximum: 100.00  5.0  5.0  50.0

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>Score</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>254.1309</td>
<td>8.68</td>
<td>254.1307</td>
<td>0.2</td>
<td>0.9</td>
<td>9.0</td>
<td>1</td>
<td>C17 H18 O2</td>
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</tbody>
</table>
Elemental Composition Report

**Single Mass Analysis**

Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
67 formula(e) evaluated with 11 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-30  H: 0-80  O: 0-8  Br: 0-2

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<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>276.9490</td>
<td>276.9500</td>
<td>-1.0</td>
<td>-3.6</td>
<td>5.5</td>
<td>26.6</td>
<td>0.0</td>
<td>C7 H3 Cl2 Br2</td>
</tr>
</tbody>
</table>
### Elemental Composition Report

#### Single Mass Analysis

- **Tolerance**: 30.0 mDa
- **DBE**: min = -1.5, max = 100.0
- **Element prediction**: Off
- **Number of isotope peaks used for i-FIT**: 2

**Monoisotopic Mass, Even Electron Ions**

48 formula(e) evaluated with 7 results within limits (up to 1 closest result for each mass)

**Elements Used:**

<table>
<thead>
<tr>
<th>C</th>
<th>0-35</th>
</tr>
</thead>
<tbody>
<tr>
<td>H</td>
<td>0-80</td>
</tr>
<tr>
<td>O</td>
<td>0-5</td>
</tr>
<tr>
<td>Cl</td>
<td>0-3</td>
</tr>
</tbody>
</table>

**ECUST Institute of Fine Chem**

**JYF-DJL-11 66 (2.237) Cm (69.72)**

---

**Minimum:** -1.5

**Maximum:** 30.0 50.0 100.0

<table>
<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>188.9502</td>
<td>188.9510</td>
<td>-0.8</td>
<td>-4.2</td>
<td>5.1</td>
<td>73.2</td>
<td>0.0</td>
<td>C7 H3 O2 Cl2</td>
</tr>
</tbody>
</table>

---

**16-Sep-2013**

15:42:28

2. Tof N8 E5- E794-603
Elemental Composition Report

Single Mass Analysis
Tolerance = 30.0 mDa / DBE: min = -8.8, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
27 formula(s) evaluated with 4 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-7  H: 0-80  O: 0-2  Br: 0-1  F: 0-3

VF-JI
ECUST Institute of Fine Chem

JYF-JG-1 189 (1.307) 184/186

Mass Calc. Mass mDa DBE i-FIT i-FIT (Norm) Formula
216.9281 216.9300 -1.9 -8.8 5.5 5.8 0.0 C7 H7 O2 Br F

Minimum: 30.0  50.0  100.0
Maximum: 15-Sep-2013 19:32:01

Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa  /  DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron ions
50 formula(e) evaluated with 21 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-39  H: 0-60  O: 0-8  Br: 0-2

YF-JI
ECUST institute of Fine Chem
02-Jan-2013
21:47:30
2: TOF MS ES-
5.51e+003

JYF-JA-102 47 (1.550) Cm (41:47)

<table>
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<tr>
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<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>228.9473</td>
<td>228.9500</td>
<td>-2.7</td>
<td>-11.8</td>
<td>5.5</td>
<td>14.0</td>
<td>0.0</td>
<td>C8 H6 Br</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis
Tolerance = 30.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
20 formula(e) evaluated with 4 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-8 H: 0-80 O: 0-3 F: 0-3

YF-JI
15-Sep-2013
15:53:36
1: TOF MS ES+ 1.88e+003

JYF-JG-2 55 (0.425) Cm (52/62)

164.0669 165.0637 166.0651 168.0726 169.0106 170.0378 171.0092 171.0447 171.0457 171.1492 172.0478 173.0287

Minimum: 164.0669
Maximum: 173.0287

Mass Calc. Mass mDa ppm DBE i-FIT i-FIT (Norm) Formula
171.0447 171.0457 -1.0 -5.8 4.5 21.5 0.0 C8 H8 O3 F
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

102 formula(e) evaluated with 22 results within limits (up to 50 closest results for each mass)

Elements Used:
C: 0-9  H: 0-7  N: 0-1  O: 0-2

Minimum:  3.00  -1.5
Maximum:  100.00  5.0  5.0  50.0

Mass  RA  Calc. Mass  mDa  PPM  DBE  Score  Formula
177.0424  100.00  177.0426  -0.2  -1.1  7.0  1  C9 H7 N O3
Elemental Composition Report

Single Mass Analysis
Tolerance = 30.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
17 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-10   H: 0-60   O: 0-3   Br: 0-2

YF-JL
ECUST Institute of Fine Chem
15-Sep-2013
15:59:47

JVF-JG-3 109 (0.759) Cm (105-116)

Minimum:
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
292.9817  292.9813  0.4  1.4  4.5  6.2  0.0  C9 H7 O2 Br2

Maximum:
30.0  30.0  100.0

Page 1
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

68 formula(e) evaluated with 20 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-14  H: 0-14  O: 0-2

Minimum:  3.00  -1.5

Maximum:  100.00  5.0  5.0  50.0

Mass  RA  Calc. Mass  mDa  PPM  DBE  Score  Formula
214.0992  100.00  214.0994  -0.2  -0.8  8.0  1  C14 H14 O2
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

19 formula(e) evaluated with 9 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-7    H: 0-6    O: 0-2

Minimum:  3.00    -1.5

Maximum:  100.00  5.0    10.0    50.0

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>I-FIT</th>
<th>Formula</th>
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<tbody>
<tr>
<td>122.0367</td>
<td>84.79</td>
<td>122.0368</td>
<td>-0.1</td>
<td>-0.8</td>
<td>5.0</td>
<td>3.5</td>
<td>C7 H6 O2</td>
</tr>
</tbody>
</table>
**Elemental Composition Report**

**Single Mass Analysis**
- **Tolerance**: 30.0 mDa / **DBE**: min = -1.5, max = 100.0
- **Element prediction**: Off
- **Number of isotope peaks used for i-FIT**: 2

**Monoisotopic Mass, Even Electron Ions**
- 7 formula(e) evaluated with 1 results within limits (up to 1 best isotopic matches for each mass)
- **Elements Used**:
  - C: 0-7
  - H: 0-5
  - O: 0-2
  - Br: 0-2

**Ji-YF**

**ECUST institute of Fine Chem**

**JYF-JA-19 235 (1.746) Cm (253/253)**

![Graph with m/z values and mass calculations]

**Minimum**: 196.9359
**Maximum**: 212.8351

<table>
<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>ΔDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>196.9359</td>
<td>196.9395</td>
<td>-3.6</td>
<td>-18.1</td>
<td>5.5</td>
<td>16.9</td>
<td>0.0</td>
<td>C7 H4 O2 Br</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
33 formula(e) evaluated with 17 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0.39    H: 0.50    O: 0.8

YF-JI
JYF-JA-105 16 (0.509) Cm (1:16)

Minimum: 100.0  50.0  100.0
Maximum: -1.5

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
275.1293  275.1293  0.0  0.0  7.5  16.4  0.0  Cl6 H19 O1
**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 100.0 mDa  
DBE: min = -1.5, max = 100.0  
Element prediction: Off  
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions

65 formula(s) evaluated with 35 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-39  
H: 0-80  
O: 0-8  
N: 0-1

YF-Jl  
ECUST Institute of Fine Chem

JYF-JA-21 14 (0.220) Cm (13.20)

<table>
<thead>
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<th>Mass</th>
<th>Calc. Mass</th>
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<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>251.0894</td>
<td>251.0895</td>
<td>-0.1</td>
<td>-0.4</td>
<td>3.5</td>
<td>9.4</td>
<td>0.0</td>
<td>Cl1 K16 O5 Na</td>
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</tbody>
</table>
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa  /  DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron ions

138 formula(e) evaluated with 16 results within limits (up to 50 closest results for each mass)

Elements Used:

<table>
<thead>
<tr>
<th>C</th>
<th>O-11</th>
<th>H: O-15</th>
<th>O: 0-5</th>
</tr>
</thead>
</table>

Minimum:  -1.5

Maximum:   100.0  5.0  10.0  50.0

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
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<th>Score</th>
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</thead>
<tbody>
<tr>
<td>228.0997</td>
<td>53.31</td>
<td>228.0998</td>
<td>-0.1</td>
<td>-0.4</td>
<td>4.0</td>
<td>8.2</td>
<td>C11 H16 O5</td>
</tr>
</tbody>
</table>
Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

21 formula(e) evaluated with 5 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0.9  H: 0-10  16O: 0-4  18O: 0-1

Minimum:  2.00  -1.5

Maximum:  100.00  7.1  10.0  50.0

<table>
<thead>
<tr>
<th>Mass</th>
<th>RA</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>I - FIT</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
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<td>182.0579</td>
<td>-0.1</td>
<td>-0.5</td>
<td>5.0</td>
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