

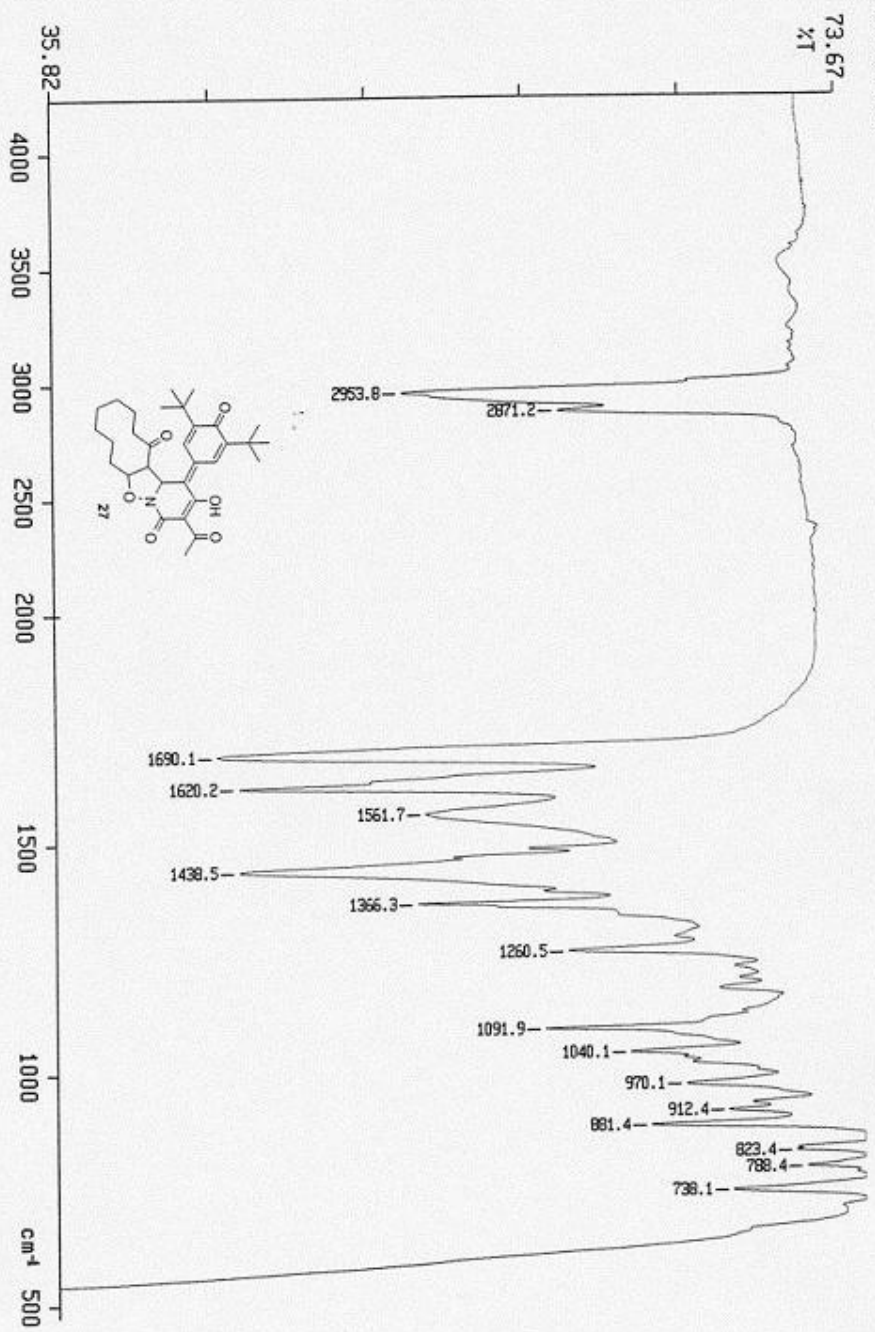
Experimental procedure for **12** and **27**.

To a mixture of 3-Acetyl-N-hydroxy-5-[(3,5-di-*tert*-butyl-4-hydroxy)phenyl]-4-hydroxy-2-pyridone **9** (100 mg, 0.27 mmol, 1.0 eq) and *cis*-cyclodecenone **11** (41 mg, 0.27 mmol, 1.0 eq) in DCM (3 mL) was added iodobenzene diacetate (95 mg, 0.29 mmol, 1.1 eq) all at once. Immediately the reaction turned a dark colour. After stirring for 2 h at 25 °C, the reaction was refluxed for 24 h. During the reflux the colour of the reaction turned into reddish yellow. After cooling to 25 °C, water (5 mL) was added to the reaction mixture and extracted with ethyl acetate (3×10 mL). The combined organic layers were washed with brine (10 mL), dried (MgSO₄) and filtered. The filtrate was evaporated under vacuum. The crude product was purified by flash column chromatography (silica gel, DCM, and 3 % ethyl acetate in DCM; silica gel had been prewashed by being allowed to stand as a slurry in 50 % aqueous nitric acid for 24 h followed by rinsing with doubly distilled water until the aqueous filtrates were neutral. Subsequent trituration with reagent grade acetone was followed by drying in vacuum at 25 °C.) to yield 49 mg (35 %) of quinone **27**, as a yellow solid, recrystallized from ethanol, m.p. 105-107 °C and 35 mg (25 %) of phenol **12**, as pale yellow solid, recrystallized from acetonitrile, m.p. 135-137 °C.

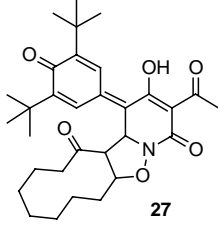
Phenol 12: ν_{\max} (Neat)/cm⁻¹ 3634w, 2953s, 2874m, 1711m, 1659s, 1609m, 1539m, 1434m, 1415m, 1364w, 1278w, 1237w, 1151m, 1121m, 978w; ¹HNMR (500 MHz, CDCl₃) δ_{H} 1.00-1.10 (m, 2H), 1.21-1.30 (m, 4H), 1.42-1.55 (m, 5H), 1.51 (s, 18H), 1.68-1.73 (m, 1H), 2.11-2.15 (m, 2H), 2.85 (s, 3H), 4.79-4.92 (m, 1H), 4.91 (d, J = 4.9 Hz, 1H), 5.42 (s, 1H), 7.04-7.15 (brs, 2H); ¹³CNMR (125 MHz, CDCl₃) δ_{C} 21.9, 22.6, 23.0, 24.2, 26.9, 29.4, 30.1, 31.3, 34.3, 45.5, 55.4, 84.0, 107.1, 108.8, 121.9, 126.1, 136.7, 145.0, 153.9, 154.1, 173.1, 204.9, 205.4; HRMS: Found 524.3005 (M+1). C₃₁H₄₂NO₆ requires 524.3012.

Quinone 27: ν_{\max} (Neat)/cm⁻¹ 2954s, 2871m, 1690s, 1620s, 1562m, 1438s, 1366m, 1260m, 1092w, 1040w; ¹HNMR (500 MHz, CDCl₃) δ_{H} 0.96-1.03 (m, 1H), 1.11-1.19 (m, 2H), 1.30-1.42 (m, 3H), 1.34 (s, 9H), 1.36 (s, 9H), 1.46-1.51 (m, 1H), 1.69-1.79 (m, 2H), 1.88-1.97 (m, 1H), 2.03-2.12 (m, 2H), 2.22-2.28 (m, 1H), 2.53 (dd, J = 10.3, 16.3 Hz, 1H), 2.81 (s, 3H), 3.69 (t, J = 9.0 Hz, 1H), 4.32 (t, J = 9.4 Hz, 1H), 5.35 (d, J = 9.0 Hz, 1H), 7.04 (d, J = 2.7 Hz, 1H), 8.47 (d, J = 2.7 Hz, 1H); ¹³CNMR (125 MHz, CDCl₃) δ_{C} 22.1, 23.1, 24.1, 24.5, 24.9, 27.3, 28.6, 29.4, 29.5, 35.8, 36.1, 48.1, 61.3, 66.0, 85.6, 106.7, 126.3, 128.6, 129.2, 141.1, 150.7, 152.7, 167.9, 182.3, 187.3, 204.1, 209.4; HRMS: Found 524.3000 (M+1). C₃₁H₄₂NO₆ requires 524.3012.

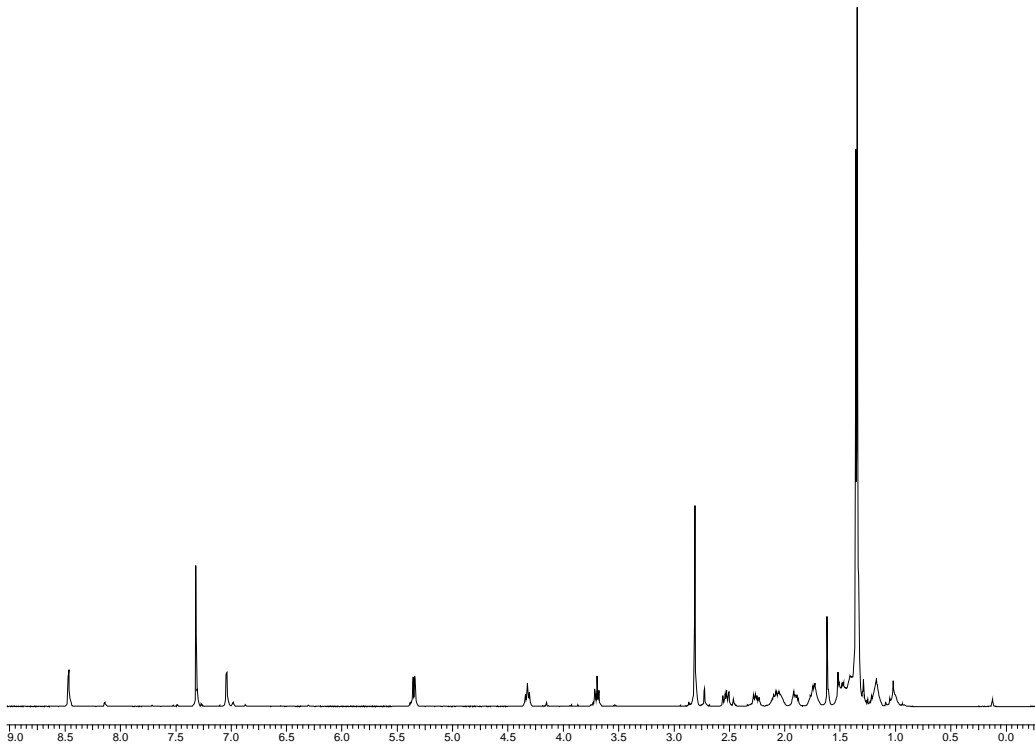
PERKIN ELMER

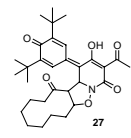
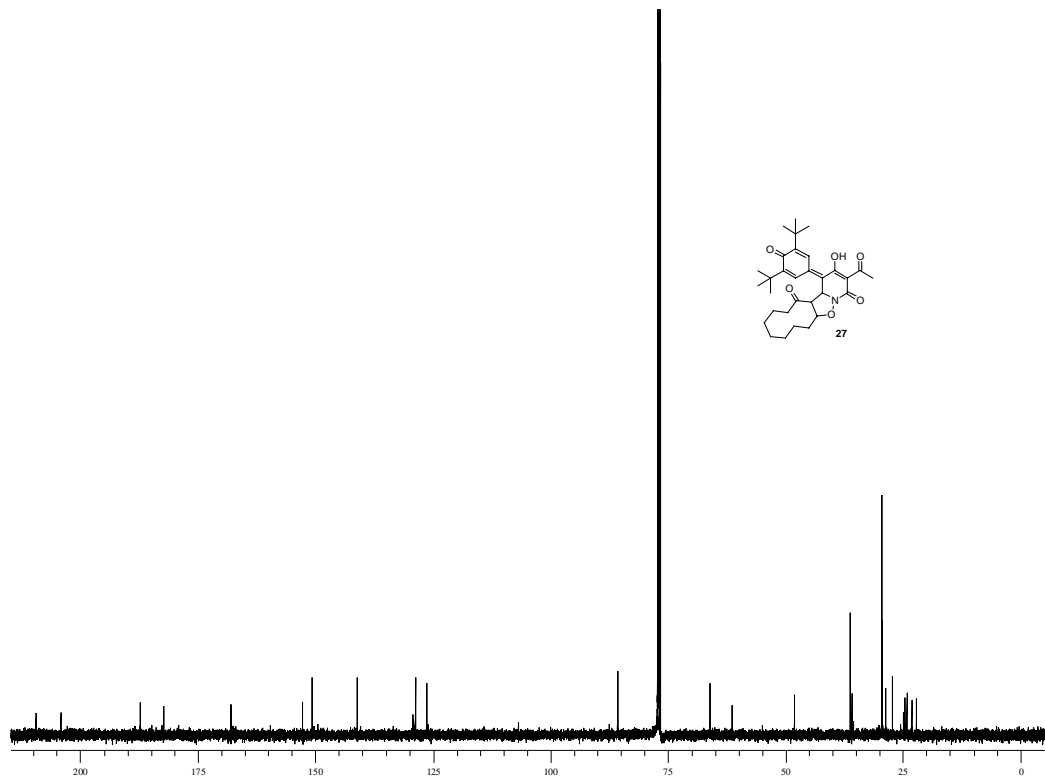


03/02/05 17:57 BOOK4-NR-63T
X: 16 scans, 4.0cm-1



DRX500 1H acquisition, TBI probe RAO IRLAPATI 2 3/01/03





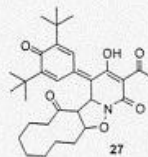
Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.0, max = 50.0

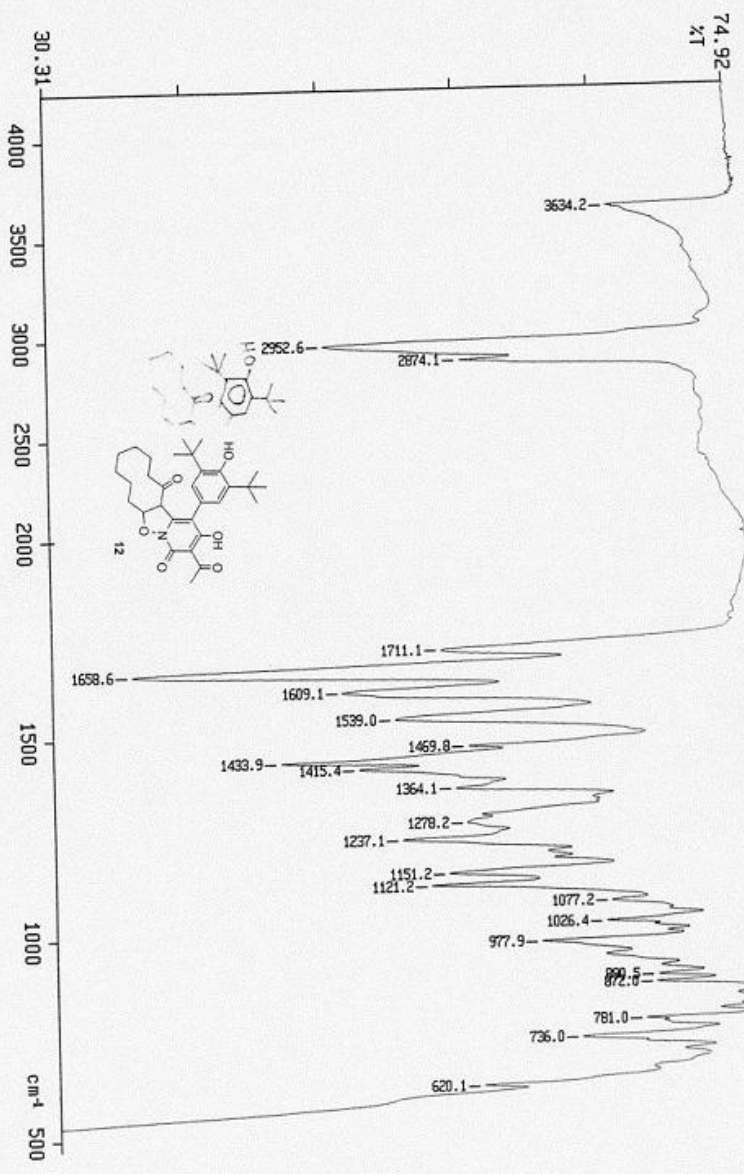
Monoisotopic Mass, Odd and Even Electron Ions

24 formula(e) evaluated with 1 results within limits (up to 45 closest results for each mass)

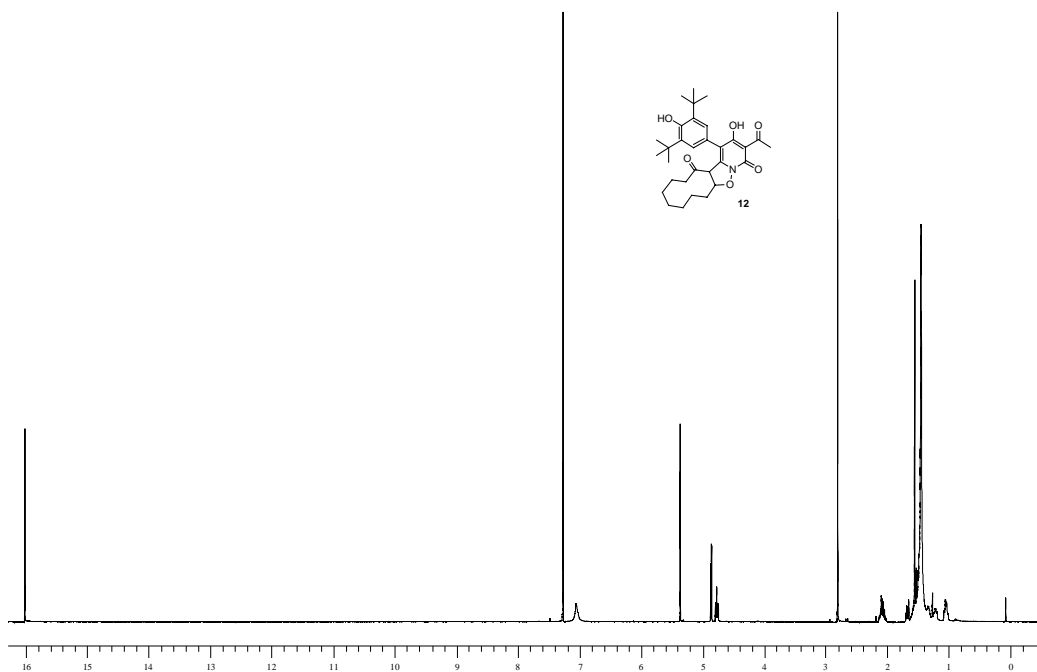
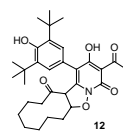
| | | | | | |
|----------|------------|------|------|------|--------------|
| Minimum: | | | | -1.0 | |
| Maximum: | 200.0 | 10.0 | | 50.0 | |
| Mass | Calc. Mass | mDa | PPM | DBE | Formula |
| 524.3000 | 524.3012 | -1.2 | -2.3 | 11.5 | C31 H42 N O6 |

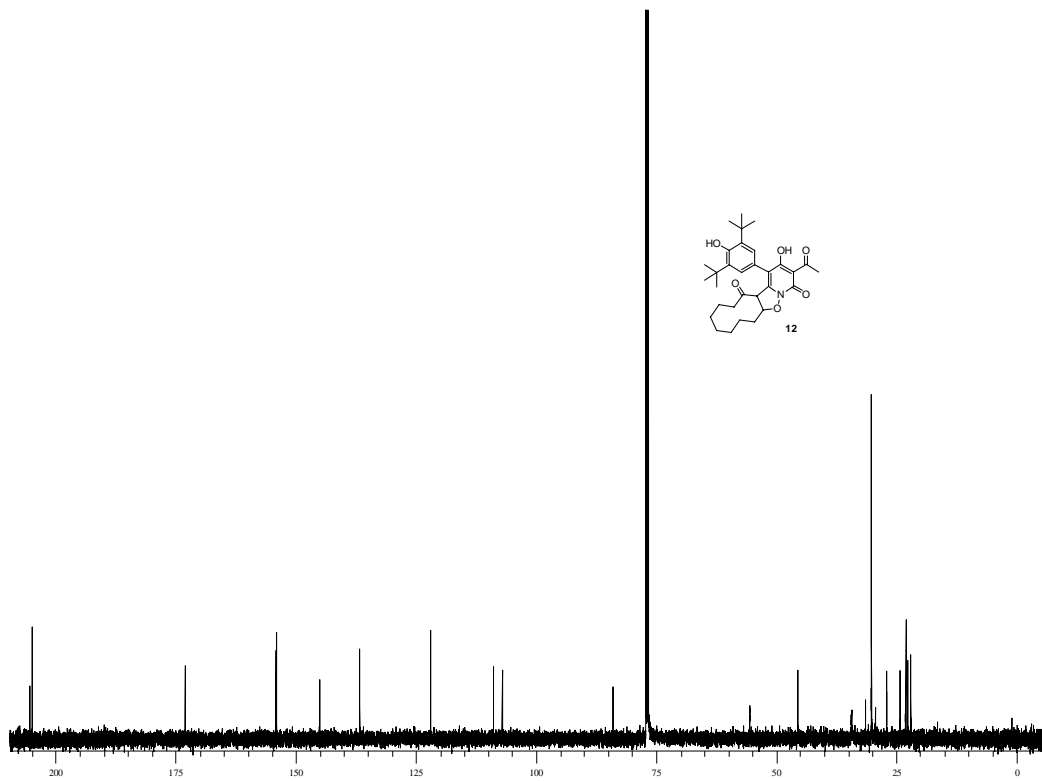


PERKIN ELMER



03/02/05 18:00 BOOK4-NR-63B
X: 16 scans, 4.0cm-1





Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.0, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

75 formula(e) evaluated with 1 results within limits (up to 45 closest results for each mass)

| | | | | | |
|----------|------------|-------|------|------|--------------|
| Minimum: | | | | | |
| Maximum: | | 200.0 | 5.0 | -1.0 | 50.0 |
| Mass | Calc. Mass | mDa | PPM | DBE | Formula |
| 524.3005 | 524.3012 | -0.7 | -1.3 | 11.9 | C31 H42 N O6 |

