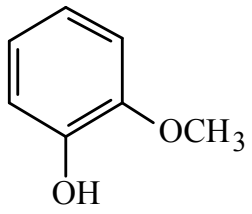
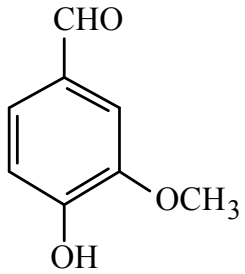
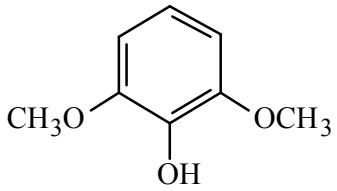
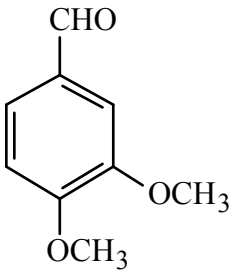
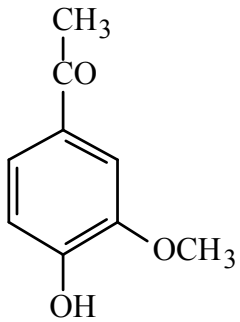
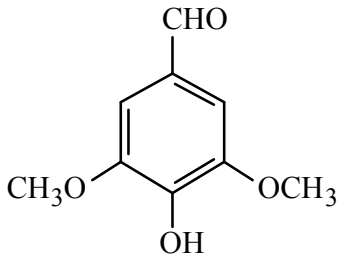
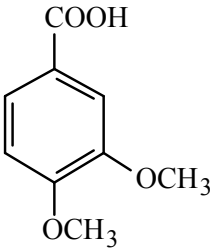
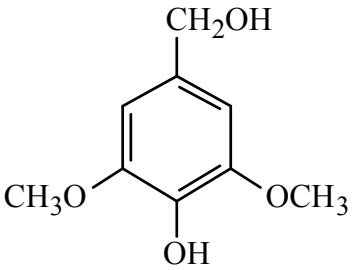
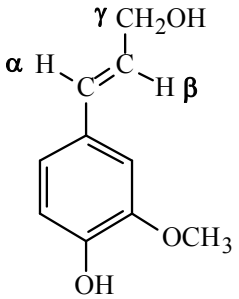
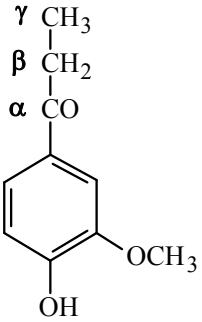
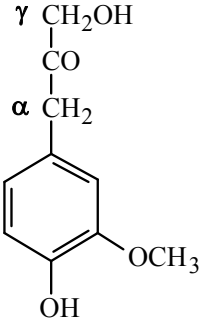
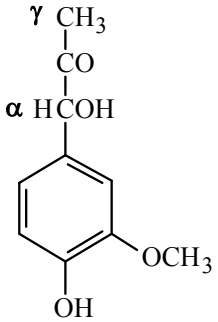
2010-04-20

# Supplementary $^{1}H$ NMR data for lignin models in $CDCl_3$

| | | |
|---|---|---|
| 1 $C_7H_8O_2$  | 2 $C_8H_8O_3$  | 3 $C_8H_{10}O_3$  |
| 4 $C_9H_{10}O_3$  | 5 $C_9H_{10}O_3$  | 6 $C_9H_{10}O_4$  |
| 7 $C_9H_{10}O_4$  | 8 $C_9H_{12}O_4$  | 9 $C_{10}H_{12}O_3$  |
| 10 $C_{10}H_{12}O_3$  | 11 $C_{10}H_{12}O_4$  | 12 $C_{10}H_{12}O_4$  |

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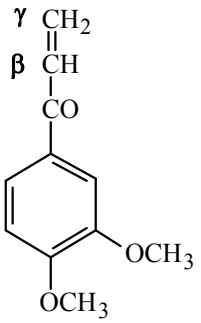
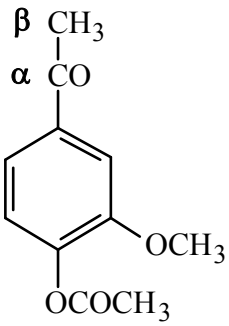
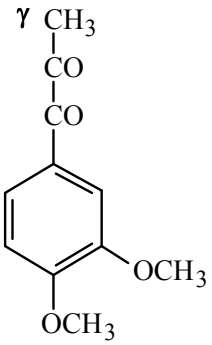
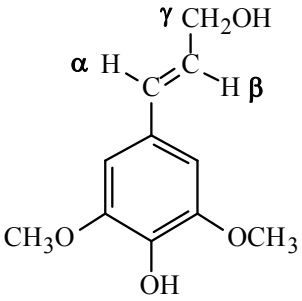
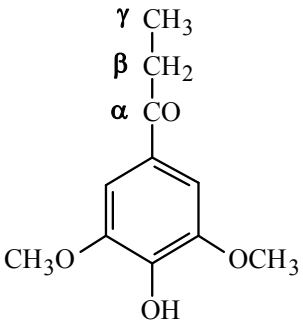
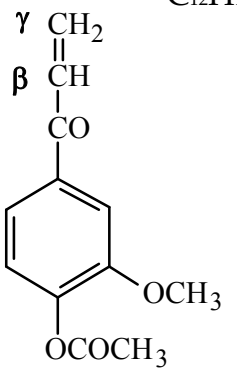
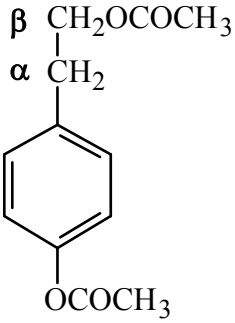
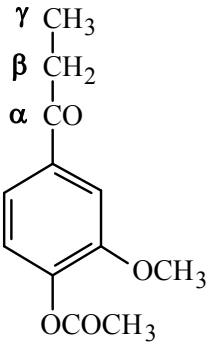
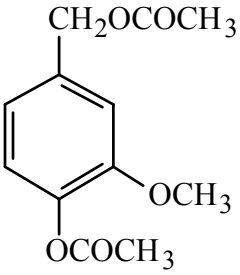
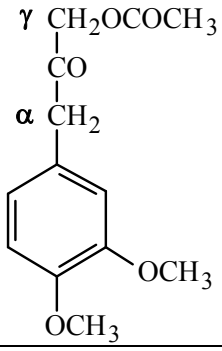
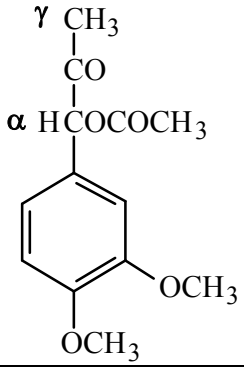
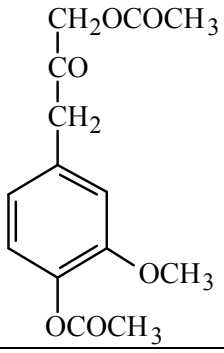
|---|---|---|
| 13 $C_{11}H_{12}O_3$  | 14 $C_{11}H_{12}O_4$  | 15 $C_{11}H_{12}O_4$  |
| 16 $C_{11}H_{14}O_4$  | 17 $C_{11}H_{14}O_4$  | 18 $C_{12}H_{12}O_4$  |
| 19 $C_{12}H_{14}O_4$  | 20 $C_{12}H_{14}O_4$  | 21 $C_{12}H_{14}O_5$  |
| 22 $C_{13}H_{16}O_5$  | 23 $C_{13}H_{16}O_5$  | 24 $C_{14}H_{16}O_6$  |

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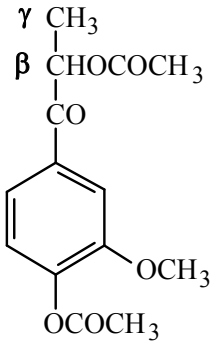
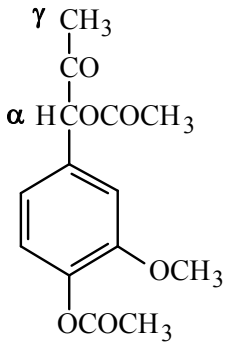
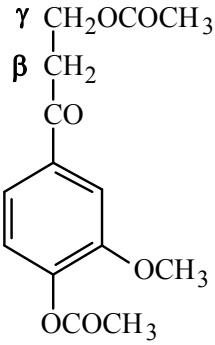
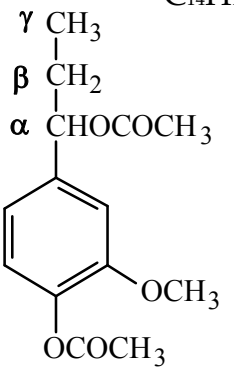
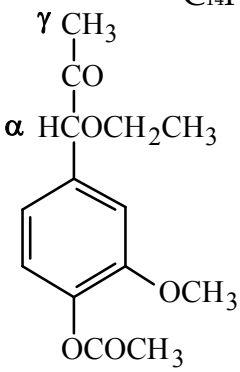
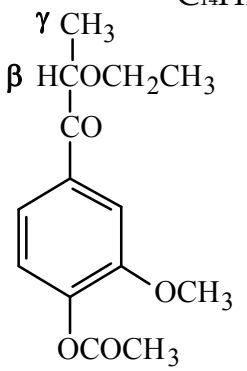
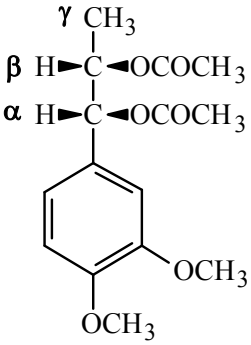
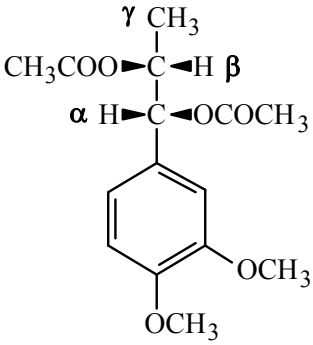
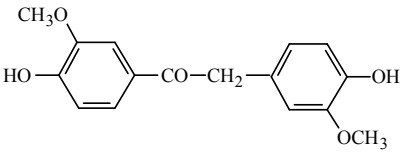
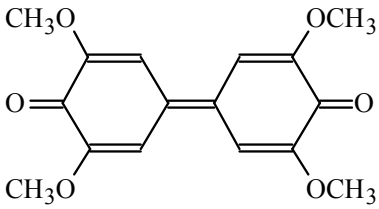
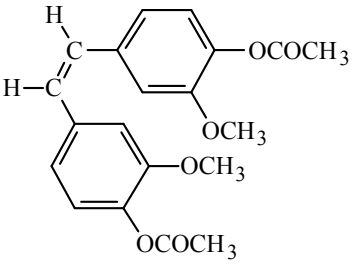
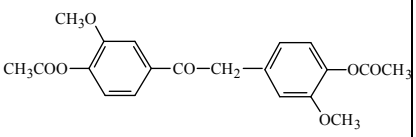
|---|---|---|
| 25 $C_{14}H_{16}O_6$  | 26 $C_{14}H_{16}O_6$  | 27 $C_{14}H_{16}O_6$  |
| 28 $C_{14}H_{18}O_5$  | 29 $C_{14}H_{18}O_5$  | 30 $C_{14}H_{18}O_5$  |
| 31 $C_{15}H_{20}O_6$  | 32 $C_{15}H_{20}O_6$  | 33 $C_{16}H_{16}O_5$  |
| 34 $C_{16}H_{16}O_6$  | 35 $C_{20}H_{20}O_6$  | 36 $C_{20}H_{20}O_7$  |

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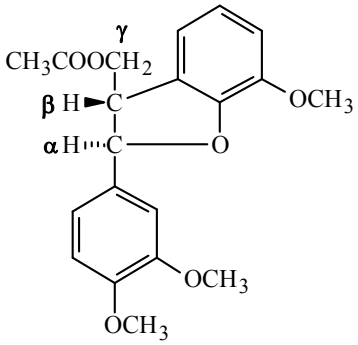
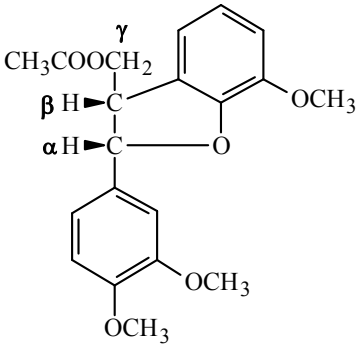
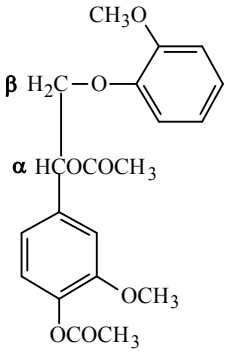
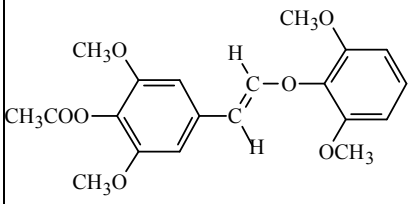
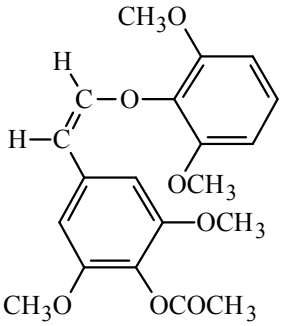
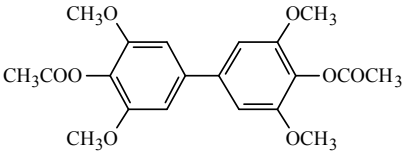
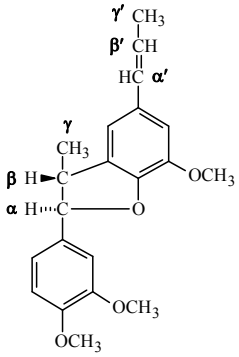
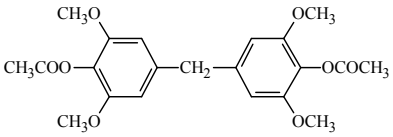
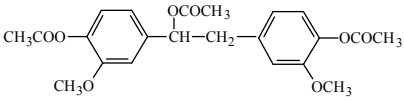
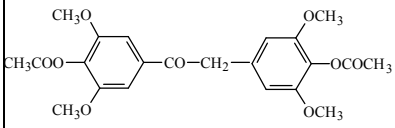
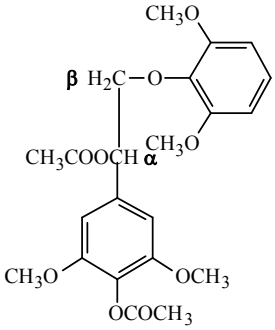
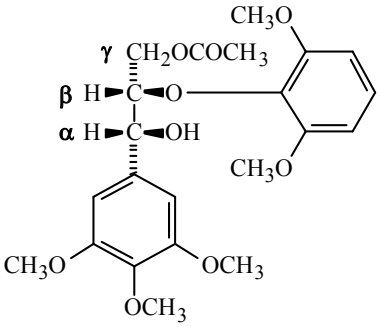
|---|---|---|
| 37 $C_{20}H_{22}O_6$  | 38 $C_{20}H_{22}O_6$  | 39 $C_{20}H_{22}O_7$  |
| 40 $C_{20}H_{22}O_7$  | 41 $C_{20}H_{22}O_7$  | 42 $C_{20}H_{22}O_8$  |
| 43 $C_{21}H_{24}O_4$  | 44 $C_{21}H_{24}O_8$  | 45 $C_{22}H_{24}O_8$  |
| 46 $C_{22}H_{24}O_9$  | 47 $C_{22}H_{26}O_9$  | 48 $C_{22}H_{28}O_9$  |

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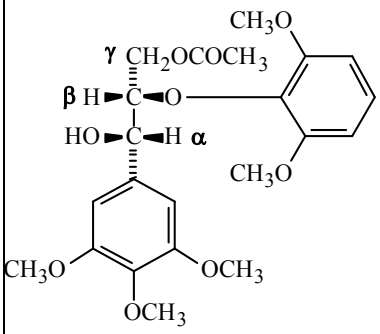
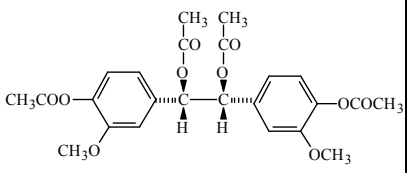
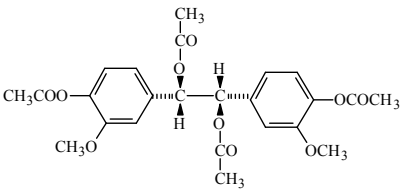
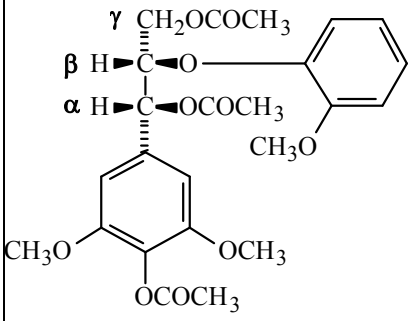
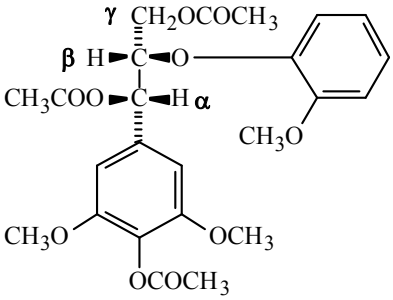
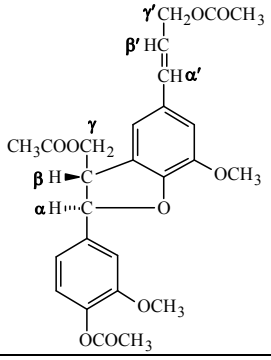
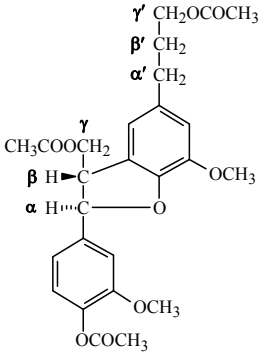
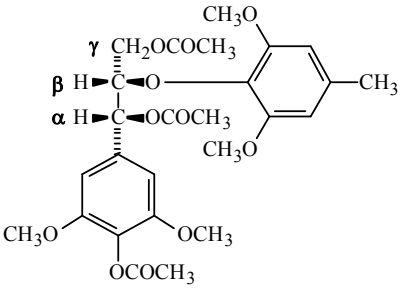
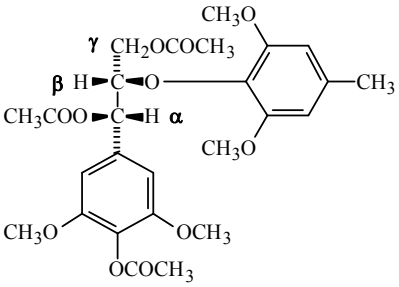
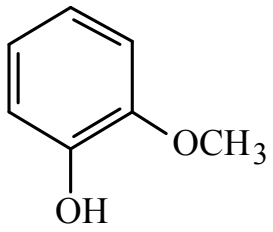
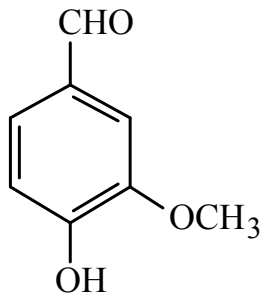
|---|---|---|
| 49 $C_{22}H_{28}O_9$  | 50 $C_{24}H_{26}O_{10}$  | 51 $C_{24}H_{26}O_{10}$  |
| 52 $C_{24}H_{28}O_{10}$  | 53 $C_{24}H_{28}O_{10}$  | 54 $C_{26}H_{28}O_9$  |
| 55 $C_{26}H_{30}O_9$  | 56 $C_{26}H_{32}O_{11}$  | 57 $C_{26}H_{32}O_{11}$  |

1



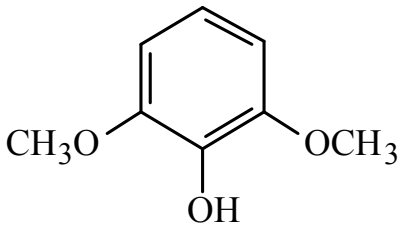

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.90 | 3H, s; $CH_3O$ |
| 5.63 | 1H, s; Ar-OH |
| 6.82-6.95 | 4H, m; H-Ar |

2

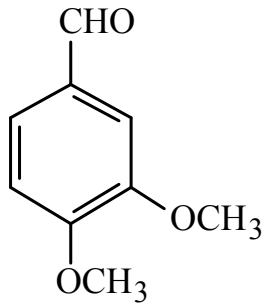



| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.98 | 3H, s; $CH_3O$ |
| ≈6.2 | 1H, s; Ar-OH |
| 7.05 | 1H, d, *J*=8.4 Hz; H-Ar |
| 7.42-7.45 | 2H, m; H-Ar |
| 9.83 | 1H, s; CHO |

3

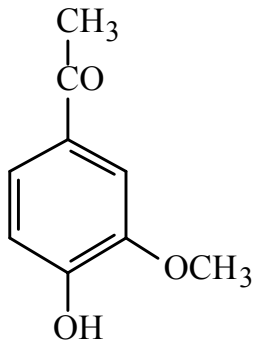



| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.90 | 6H, s; $CH_3O$ |
| 5.53 | 1H, s; Ar-OH |
| 6.59 | 2H, d, *J*=8.4 Hz; H-Ar |
| 6.81 | 1H, t, *J*=8.4 Hz; H-Ar |



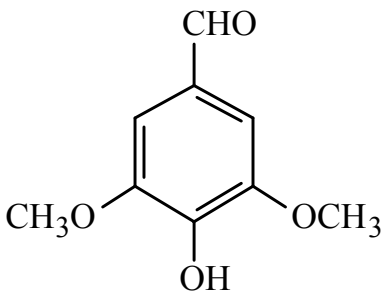

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.95 | 3H, s; $CH_3O$ |
| 3.98 | 3H, s; $CH_3O$ |
| 6.99 | 1H, d, *J*=8 Hz; H-Ar |
| 7.42 | 1H, d, *J*=2.0 Hz; H-Ar |
| 7.47 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 9.86 | 1H, s; CHO |

5



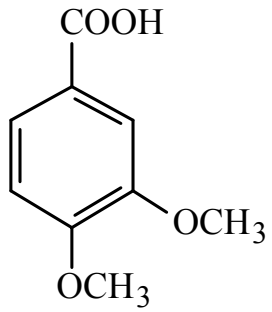

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.57 | 3H, s; $CH_3$-C |
| 3.96 | 3H, s; $CH_3O$ |
| 6.13 | 1H, s; Ar-OH |
| 6.95 | 1H, d, *J*=8.8 Hz; H-Ar |
| 7.52-7.56 | 2H, m; H-Ar |

6



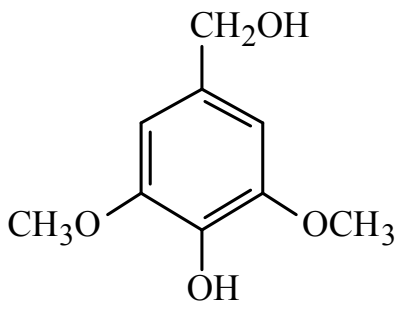

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.98 | 6H, s; $CH_3O$ |
| 6.09 | 1H, broad s; Ar-OH |
| 7.16 | 2H, s; H-Ar |
| 9.82 | 1H, s; CHO |

7



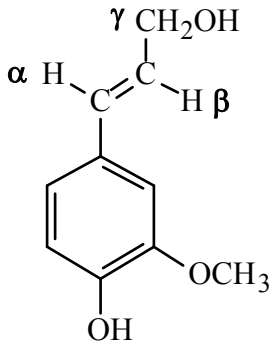

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.96 | 3H, s; $CH_3O$ |
| 3.97 | 3H, s; $CH_3O$ |
| 6.93 | 1H, d, *J*=8.6 Hz; H-Ar |
| 7.61 | 1H, d, *J*=1.8 Hz; H-Ar |
| 7.79 | 1H, dd, *J*=1.8 and 8.6 Hz; H-Ar |
| 12.0 | 1H, broad s; COOH |

# 8

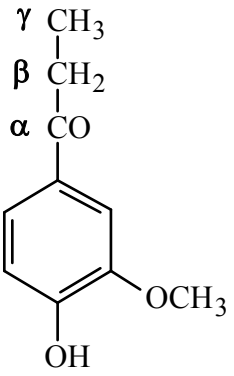



| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.90 | 6H, s; $CH_3O$ |
| 4.61 | 2H, s; $CH_2$ |
| 5.51 | 1H, s; Ar-OH |
| 6.62 | 2H, s; H-Ar |

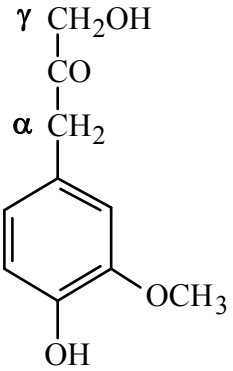
9



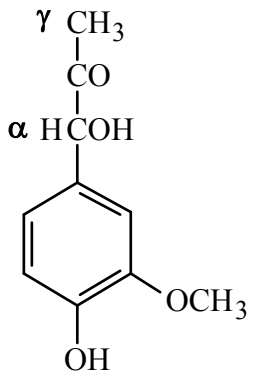

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.90 | 3H, s; $CH_3O$ |
| 4.30 | 2H, dd, *J*=1.4 and 6.0 Hz; $H_\gamma$ |
| 5.70 | 1H, s; Ar-OH |
| 6.22 | 1H, td, *J*=6.0 and 16.0 Hz; $H_\beta$ |
| 6.53 | 1H, d, *J*=16.0 Hz; $H_\alpha$ |
| 6.86 | 1H, d, *J*=8.0 Hz; H-Ar |
| 6.88 | 1H, d, *J*=1.6 Hz; H-Ar |
| 6.91 | 1H, dd, *J*=1.6 and 8.0 Hz; H-Ar |



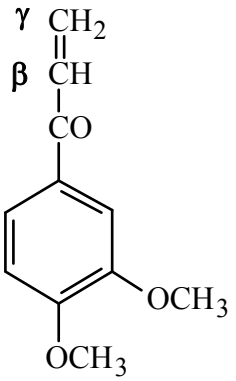

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.22 | 3H, t, *J*=6.8 Hz; $H_\gamma$ |
| 2.96 | 2H, q, *J*=6.8 Hz; $H_\beta$ |
| 3.96 | 3H, s; $CH_3O$ |
| 6.04 | 1H, s; Ar-OH |
| 6.9-7.6 | 3H, m; H-Ar |



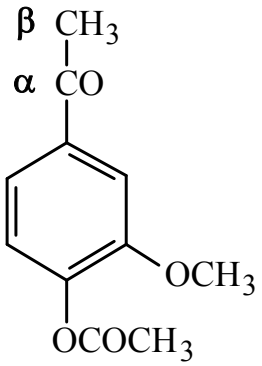

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.01 | 1H, broad s; $C_\gamma$-OH |
| 3.65 | 2H, s; $H_\alpha$ |
| 3.88 | 3H, s; $CH_3O$ |
| 4.29 | 2H, s; $H_\gamma$ |
| 5.60 | 1H, broad s; Ar-OH |
| 6.68-6.73 | 2H, m; H-Ar |
| 6.80 | 1H, d, *J*=8.4 Hz; H-Ar |



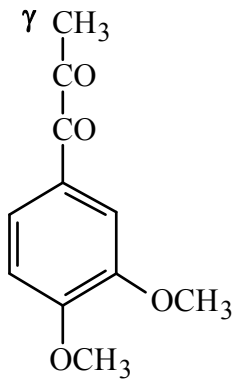

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.09 | 3H, s; $H_\gamma$ |
| 3.89 | 3H, s; $CH_3O$ |
| 4.25 | 1H, d, *J*=4.0 Hz; $C_\alpha$-OH |
| 5.03 | 1H, d, *J*=4.0 Hz; $H_\alpha$ |
| 5.67 | 1H, s; Ar-OH |
| 6.75 | 1H, d, *J*=2.0 Hz; H-Ar |
| 6.87 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.93 | 1H, d, *J*=8.0 Hz; H-Ar |



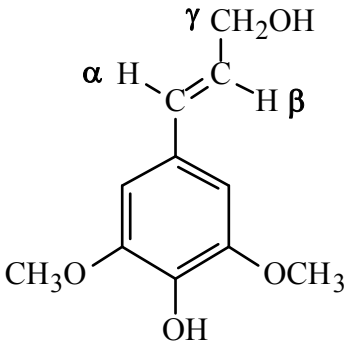

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.96 | 3H, s; $CH_3O$ |
| 3.97 | 3H, s; $CH_3O$ |
| 5.88 | 1H, dd, *J*=2 and 10.6 Hz; $H_\gamma$ |
| 6.44 | 1H, dd, *J*=2 and 16.8 Hz; $H_\gamma$ |
| 6.92 | 1H, d, *J*=8 Hz; H-Ar |
| 7.20 | 1H, dd, *J*=10.6 and 16.8 Hz; $H_\beta$ |
| 7.57 | 1H, d, *J*=2.0 Hz; H-Ar |
| 7.59 | 1H, dd, *J*=2 and 8 Hz; H-Ar |



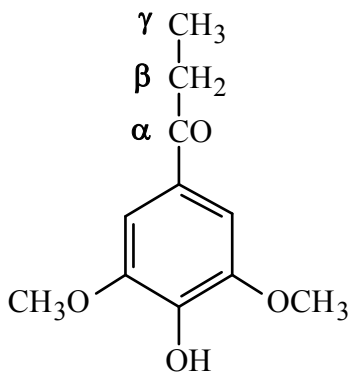

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.34 | 3H, s; $CH_3COO$ |
| 2.60 | 3H, s; $H_\beta$ |
| 3.89 | 3H, s; $CH_3O$ |
| 7.12 | 1H, d, *J*=8.4 Hz; H-Ar |
| 7.55 | 1H, dd, *J*=2.0 and 8.4 Hz; H-Ar |
| 7.60 | 1H, d, *J*=2.0 Hz; H-Ar |



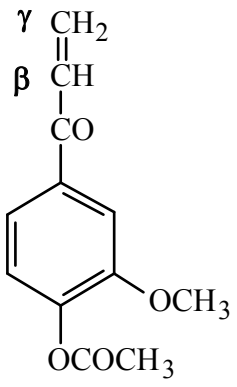

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.52 | 3H, s; $H_\gamma$ |
| 3.95 | 3H, s; $CH_3O$ |
| 3.97 | 3H, s; $CH_3O$ |
| 6.92 | 1H, d, *J*=8.4 Hz; H-Ar |
| 7.57 | 1H, d, *J*=2.0 Hz; H-Ar |
| 7.66 | 1H, dd, *J*=2.0 and 8.4 Hz; H-Ar |



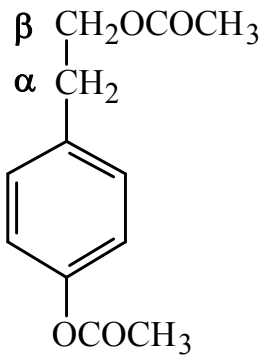

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.91 | 6H, s; $CH_3O$ |
| 4.32 | 2H, broad s; $H_\gamma$ |
| 5.55 | 1H, s; Ar-OH |
| 6.25 | 1H, td, *J*=6.0 and 16.0 Hz; $H_\beta$ |
| 6.53 | 1H, d, *J*=16.0 Hz; $H_\alpha$ |
| 6.64 | 2H, s; H-Ar |



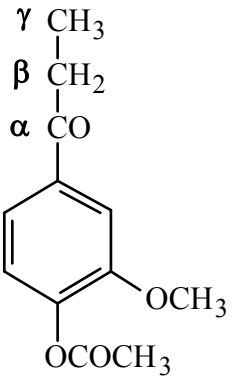

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.23 | 3H, t, *J*=6.8 Hz; $H_\gamma$ |
| 2.97 | 1H, q, *J*=6.8 Hz; $H_\beta$ |
| 3.95 | 6H, s; $CH_3O$ |
| 5.99 | 1H, s; Ar-OH |
| 6.58 | 2H, s; H-Ar |



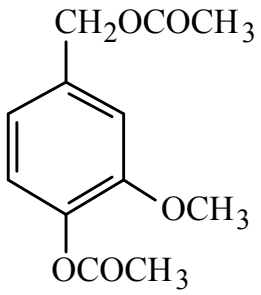

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.35 | 3H, s; $CH_3CO$ |
| 3.91 | 3H, s; $CH_3O$ |
| 5.94 | 1H, dd, *J*=1.6 and 10.4 Hz; $H_\gamma$ |
| 6.46 | 1H, dd, *J*=1.6 and 17.2 Hz; $H_\gamma$ |
| 7.15 | 1H, dd, *J*=10.4 and 17.2 Hz; $H_\beta$ |
| 7.15 | 1H, d, *J*=8 Hz; H-Ar |
| 7.55 | 1H, d, *J*=2 Hz; H-Ar |
| 7.61 | 1H, dd, *J*=2 and 8 Hz; H-Ar |



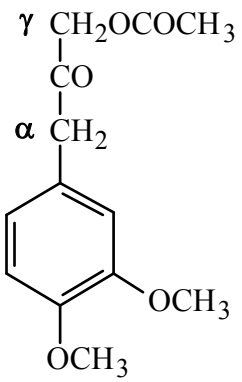

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.04 | 3H, s; $CH_3CO$ |
| 2.30 | 3H, s; $CH_3CO$ |
| 2.93 | 2H, t, *J*=7.2 Hz; $H_\alpha$ |
| 4.27 | 2H, t, *J*=7.2 Hz; $H_\beta$ |
| 7.02 | 2H, ≈d, *J*=8.4 Hz; H-Ar |
| 7.23 | 2H, ≈d, *J*=8.4 Hz; H-Ar |



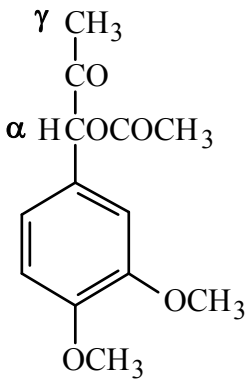

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.23 | 3H, t, *J*=6.8 Hz; $H_\gamma$ |
| 2.34 | 3H, s; $CH_3CO$ |
| 2.99 | 2H, q, *J*=6.8 Hz; $H_\beta$ |
| 3.90 | 3H, s; $CH_3O$ |
| 7.12 | 1H, d, *J*=8.0 Hz; H-Ar |
| 7.57 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 7.62 | 1H, d, *J*=2.0 Hz; H-Ar |



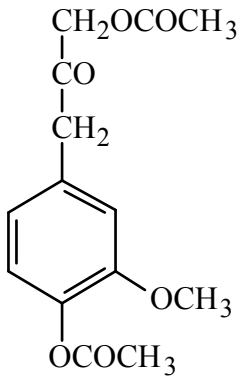

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.11 | 3H, s; $CH_3CO$ |
| 2.32 | 3H, s; $CH_3CO$ |
| 3.85 | 3H, s; $CH_3O$ |
| 5.08 | 2H, s; $CH_2$ |
| 6.94 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.97 | 1H, d, *J*=2.0 Hz; H-Ar |
| 7.02 | 1H, d, *J*=8.0 Hz; H-Ar |



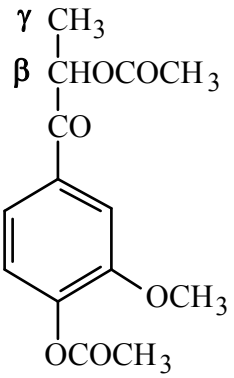

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.16 | 3H, s; $CH_3CO$ |
| 3.68 | 2H, s; $H_\alpha$ |
| 3.87 | 6H, s; $CH_3O$ |
| 4.70 | 2H, s; $H_\gamma$ |
| 6.72 | 1H, d, *J*=2 Hz; H-Ar |
| 6.76 | 1H, dd, *J*=2 and 8 Hz; H-Ar |
| 6.84 | 1H, d, *J*=8 Hz; H-Ar |



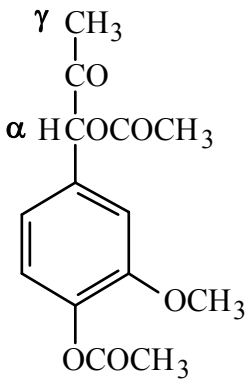

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.12 | 3H, s; $H_\gamma$ or $CH_3CO$ |
| 2.19 | 3H, s; $H_\gamma$ or $CH_3CO$ |
| 3.89 | 3H, s; $CH_3O$ |
| 3.90 | 3H, s; $CH_3O$ |
| 5.92 | 1H, s; $H_\alpha$ |
| 6.86 | 1H, d, *J*=2 Hz; H-Ar |
| 6.88 | 1H, d, *J*=8 Hz; H-Ar |
| 6.97 | 1H, dd, *J*=2 and 8 Hz; H-Ar |



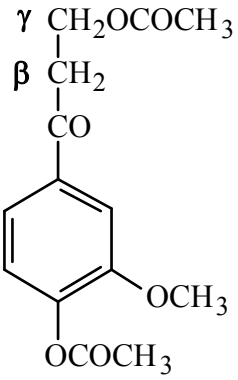

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.16 | 3H, s; $CH_3CO$ |
| 2.31 | 3H, s; $CH_3CO$ |
| 3.72 | 2H, s; $H_\alpha$ |
| 3.82 | 3H, s; $CH_3O$ |
| 4.72 | 2H, s; $H_\gamma$ |
| 6.79 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.82 | 1H, d, *J*=2.0 Hz; H-Ar |
| 6.99 | 1H, d, *J*=8.0 Hz; H-Ar |



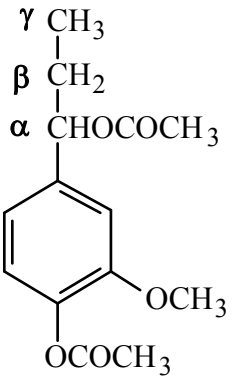

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.53 | 3H, d, *J*=6.8 Hz; $H_\gamma$ |
| 2.15 | 3H, s; $CH_3CO$ |
| 2.34 | 3H, s; $CH_3CO$ |
| 3.89 | 3H, s; $CH_3O$ |
| 5.93 | 1H, q, *J*=6.8 Hz; $H_\beta$ |
| 7.14 | 1H, d, *J*=8 Hz; H-Ar |
| 7.55 | 1H, dd, *J*=2 and 8 Hz; H-Ar |
| 7.59 | 1H, d, *J*=2 Hz; H-Ar |



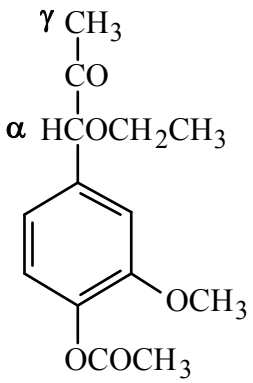

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.14 | 3H, s; $H_\gamma$ or $CH_3CO$ |
| 2.20 | 3H, s; $H_\gamma$ or $CH_3CO$ |
| 2.32 | 3H, s; $CH_3CO$ |
| 3.85 | 3H, s; $CH_3O$ |
| 5.95 | 1H, s; $H_\alpha$ |
| 6.97 | 1H, d, *J*=2 Hz; H-Ar |
| 7.01 | 1H, dd, *J*=2 and 8 Hz; H-Ar |
| 7.07 | 1H, d, *J*=8 Hz; H-Ar |



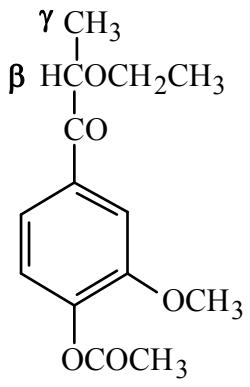

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.04 | 3H, s; $CH_3CO$ |
| 2.34 | 3H, s; $CH_3CO$ |
| 3.30 | 2H, t, *J*=6.4 Hz; $H_\beta$ |
| 3.90 | 3H, s; $CH_3O$ |
| 4.52 | 2H, t, *J*=6.4 Hz; $H_\gamma$ |
| 7.14 | 1H, d, *J*=8 Hz; H-Ar |
| 7.56 | 1H, dd, *J*=2 and 8 Hz; H-Ar |
| 7.61 | 1H, d, *J*=2 Hz; H-Ar |



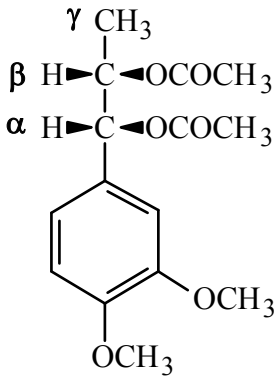

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 0.90 | 3H, t, *J*=6.8 Hz; $H_\gamma$ |
| ≈1.85 | 2H, m; $H_\beta$ |
| 2.08 | 3H, s; $CH_3CO$ |
| 2.31 | 3H, s; $CH_3CO$ |
| 3.84 | 3H, s; $CH_3O$ |
| 5.65 | 1H, ≈t, *J*=6.8 Hz; $H_\alpha$ |
| 6.91 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.92 | 1H, d, *J*=2.0 Hz; H-Ar |
| 6.99 | 1H, d, *J*=8.0 Hz; H-Ar |



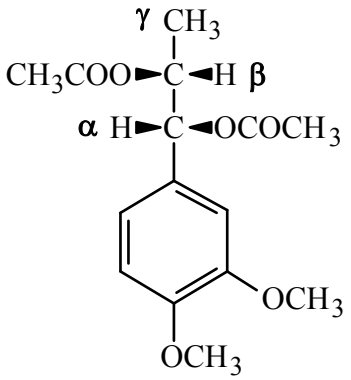

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.28 | 3H, t, *J*=7.2 Hz; $CH_2$**$CH_3$** |
| 2.13 | 3H, s; $H_\gamma$ |
| 2.31 | 3H, s; $CH_3CO$ |
| 3.53 | 2H, m; **$CH_2$**$CH_3$ |
| 3.84 | 3H, s; $CH_3O$ |
| 4.72 | 1H, s; $H_\alpha$ |
| 6.98-7.05 | 3H, m; H-Ar |



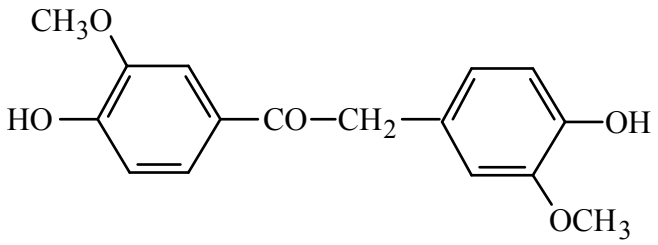

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.23 | 3H, t, *J*=6.8 Hz; $CH_2$**$CH_3$** |
| 1.50 | 3H, d, *J*=6.8 Hz; $H_\gamma$ |
| 2.34 | 3H, s; $CH_3CO$ |
| 3.52 | 2H, m; **$CH_2$**$CH_3$ |
| 3.90 | 3H, s; $CH_3O$ |
| 4.63 | 1H, q, *J*=6.8 Hz; $H_\beta$ |
| 7.12 | 1H, d, *J*=8.0 Hz; H-Ar |
| 7.74 | 1H, d, *J*=2.0 Hz; H-Ar |
| 7.77 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |



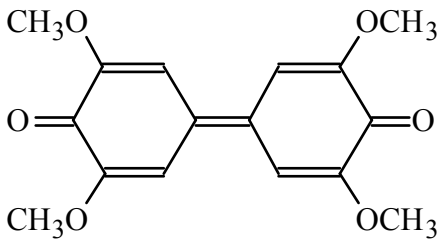

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.19 | 3H, d, *J*=6.8 Hz; $H_\gamma$ |
| 2.01 | 3H, s; $CH_3CO$ |
| 2.13 | 3H, s; $CH_3CO$ |
| 3.88 | 3H, s; $CH_3O$ |
| 3.90 | 3H, s; $CH_3O$ |
| 5.21 | 1H, m; $H_\beta$ |
| 5.84 | 1H, d, *J*=4.4 Hz; $H_\alpha$ |
| 6.8-7.0 | 3H, m; H-Ar |



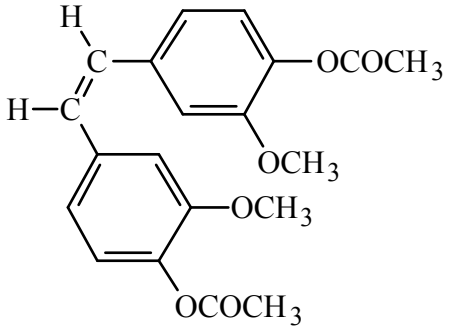

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.08 | 3H, d, *J*=6.4 Hz; $H_\gamma$ |
| 2.05 | 3H, s; $CH_3CO$ |
| 2.07 | 3H, s; $CH_3CO$ |
| 3.88 | 3H, s; $CH_3O$ |
| 3.90 | 3H, s; $CH_3O$ |
| 5.26 | 1H, m; $H_\beta$ |
| 5.69 | 1H, d, *J*=8.0 Hz; $H_\alpha$ |
| 6.8-7.1 | 3H, m; H-Ar |



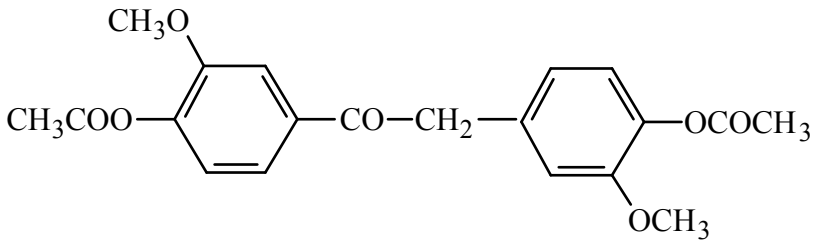

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.86 | 3H, s; $CH_3O$ |
| 3.93 | 3H, s; $CH_3O$ |
| 4.15 | 2H, s; $CH_2$ |
| ≈5.6 | 1H, s; Ar-OH |
| ≈6.1 | 1H, s; Ar-OH |
| 6.74-6.80 | 2H, m; H-Ar |
| 6.86 | 1H, d, *J*=8.0 Hz; H-Ar |
| 6.94 | 1H, d, *J*=8.4 Hz; H-Ar |
| 7.56 | 1H, d, *J*=2.0 Hz; H-Ar |
| 7.63 | 1H, dd, *J*=2.0 and 8.4 Hz; H-Ar |



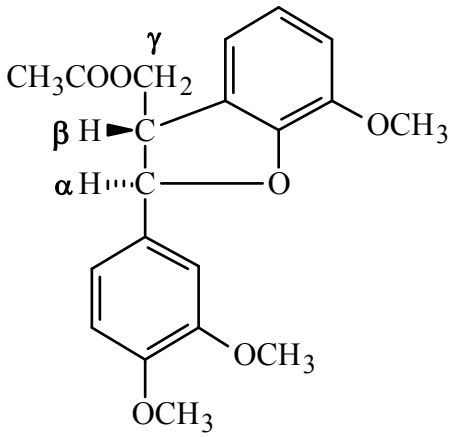

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 3.98 | 6H, s; $CH_3O$ |
| 6.92 | 4H, s; vinyl H |



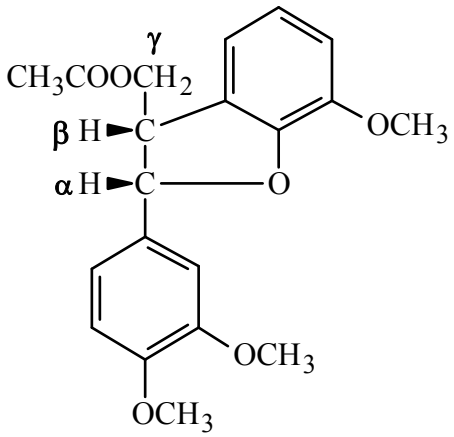

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.29 | 6H, s; $CH_3CO$ |
| 3.61 | 6H, s; $CH_3O$ |
| 6.57 | 2H, s; vinyl H |
| 6.84 | 2H, d, *J*=2.0 Hz; H-Ar |
| 6.86 | 2H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.93 | 2H, d, *J*=8.0 Hz; H-Ar |



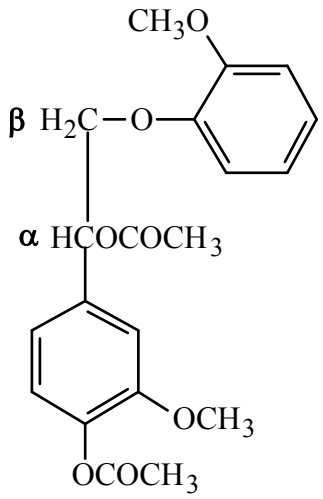

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.31 | 3H, s; $CH_3CO$ |
| 2.34 | 3H, s; $CH_3CO$ |
| 3.81 | 3H, s; $CH_3O$ |
| 3.88 | 3H, s; $CH_3O$ |
| 4.24 | 2H, s; $CH_2$ |
| 6.83 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.87 | 1H, d, *J*=2.0 Hz; H-Ar |
| 6.99 | 1H, d, *J*=8.0 Hz; H-Ar |
| 7.13 | 1H, d, *J*=8.4 Hz; H-Ar |
| 7.63 | 1H, d, *J*=2.0 Hz; H-Ar |
| 7.63 | 1H, dd, *J*=2.0 and 8.4 Hz; H-Ar |



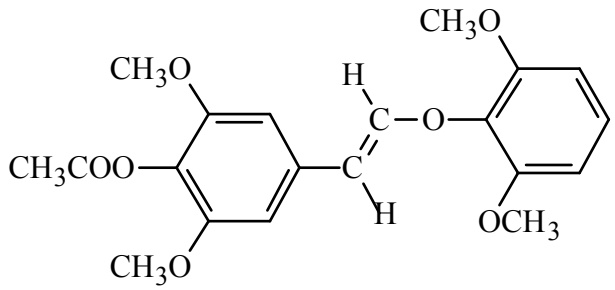

| 400 MHz, $CDCl_3$, 28° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.03 | 3H, s; $CH_3CO$ |
| 3.81 | 1H, m; $H_\beta$ |
| 3.86 | 3H, s; $CH_3O$ |
| 3.87 | 3H, s; $CH_3O$ |
| 3.90 | 3H, s; $CH_3O$ |
| 4.33 | 1H, dd, *J*=7.5 and 11.4 Hz; $H_\gamma$ |
| 4.45 | 1H, dd, *J*=5.7 and 11.4 Hz; $H_\gamma$ |
| 5.47 | 1H, d, *J*=7.3 Hz; $H_\alpha$ |
| 6.8-7.0 | 6H, m; H-Ar |



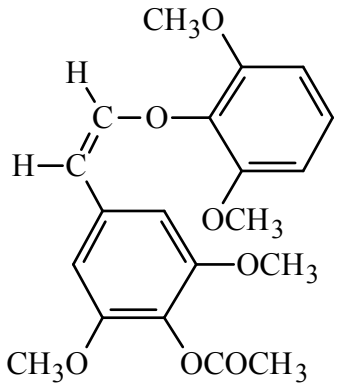

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.89 | 3H, s; $CH_3CO$ |
| 3.78-3.96 | 2H, m; $H_\gamma$ |
| 3.84 | 1H, m; $H_\beta$ |
| 3.87 | 3H, s; $CH_3O$ |
| 3.88 | 3H, s; $CH_3O$ |
| 3.93 | 3H, s; $CH_3O$ |
| 5.87 | 1H, d, $J$=8.2 Hz; $H_\alpha$ |
| 6.82-6.96 | 6H, m; H-Ar |



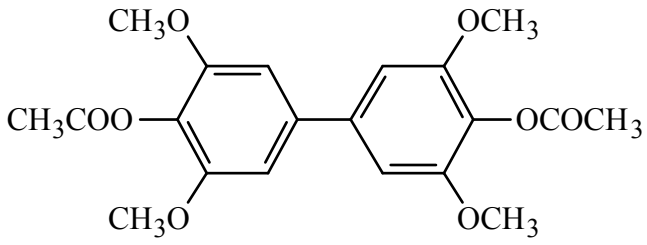

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.12 | 3H, s; $CH_3CO$ |
| 2.31 | 3H, s; $CH_3CO$ |
| 3.83 | 3H, s; $CH_3O$ |
| 3.85 | 3H, s; $CH_3O$ |
| 4.22 | 1H, dd, *J*=4.0 and 11.0 Hz; $H_\beta$ |
| 4.28 | 1H, dd, *J*=7.8 and 11.0 Hz; $H_\beta$ |
| 6.03 | 1H, dd, *J*=4.0 and 7.8 Hz; $H_\alpha$ |
| 6.86-7.05 | 5H, m; H-Ar |
| 7.02 | 1H, d, *J*=8.4 Hz; H-Ar |
| 7.04 | 1H, d, *J*=1.6 Hz; H-Ar |



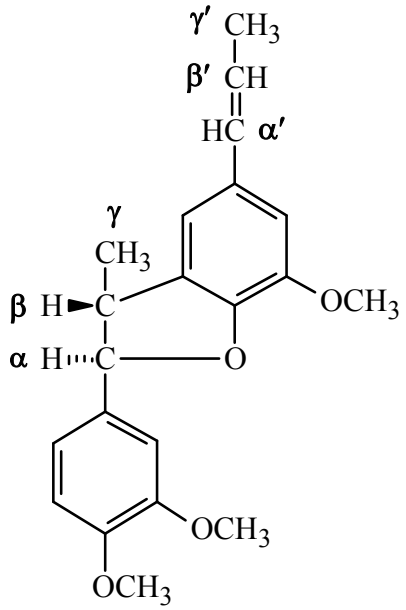

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.32 | 3H, s; $CH_3CO$ |
| 3.79 | 6H, s; $CH_3O$ |
| 3.86 | 6H, s; $CH_3O$ |
| 5.96 | 1H, d, *J*=12.4 Hz; vinyl H |
| 6.46 | 2H, s; H-Ar |
| 6.65 | 2H, d, *J*=8.4 Hz; H-Ar |
| 7.06 | 1H, d, *J*=12.4 Hz; vinyl H |
| 7.13 | 1H, t, *J*=8.4 Hz; H-Ar |



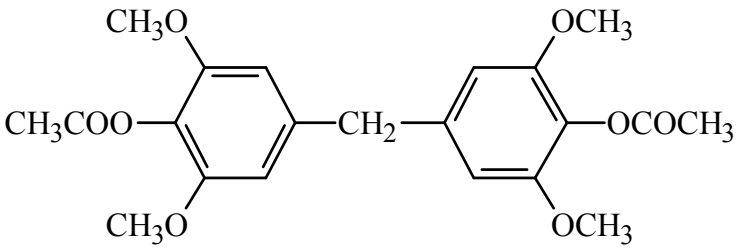

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.34 | 3H, s; $CH_3CO$ |
| 3.83 | 6H, s; $CH_3O$ |
| 3.84 | 6H, s; $CH_3O$ |
| 5.35 | 1H, d, *J*=6.8 Hz; vinyl H |
| 6.40 | 1H, d, *J*=6.8 Hz; vinyl H |
| 6.63 | 2H, d, *J*=8.4 Hz; H-Ar |
| 7.08 | 1H, t, *J*=8.4 Hz; H-Ar |
| 7.12 | 2H, s; H-Ar |



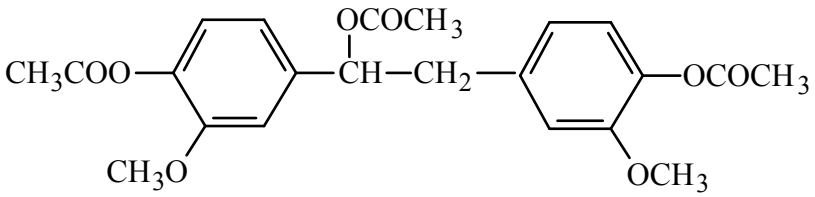

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.37 | 6H, s; $CH_3CO$ |
| 3.88 | 12H, s; $CH_3O$ |
| 6.72 | 4H, s; H-Ar |



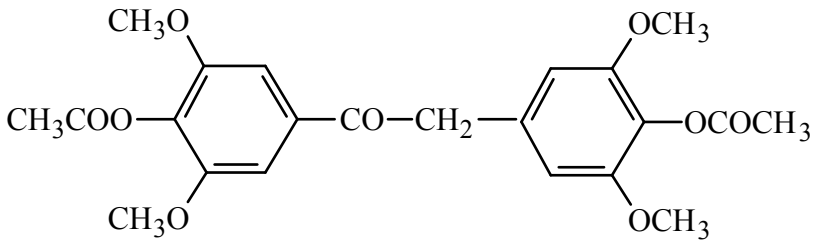

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.38 | 3H, d, *J*=6.8 Hz; $H_{\gamma}$ |
| 1.87 | 3H, dd, *J*=1.6 and 6.4 Hz; $H_{\gamma'}$ |
| 3.46 | 1H, dq, *J*=6.8 and 9.6 Hz; $H_{\beta}$ |
| 3.87 | 3H, s; $CH_3O$ |
| 3.88 | 3H, s; $CH_3O$ |
| 3.90 | 3H, s; $CH_3O$ |
| 5.12 | 1H, d, *J*=9.6 Hz; $H_{\alpha}$ |
| 6.11 | 1H, qd, *J*=6.8 and 15.6 Hz; $H_{\beta'}$ |
| 6.36 | 1H, qd, *J*=1.6 and 15.6 Hz; $H_{\alpha'}$ |
| 6.77 | 1H, s; H-Ar |
| 6.79 | 1H, s; H-Ar |
| 6.84 | 1H, d, *J*=8.0 Hz; H-Ar |
| 6.96 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.99 | 1H, d, *J*=2.0 Hz; H-Ar |



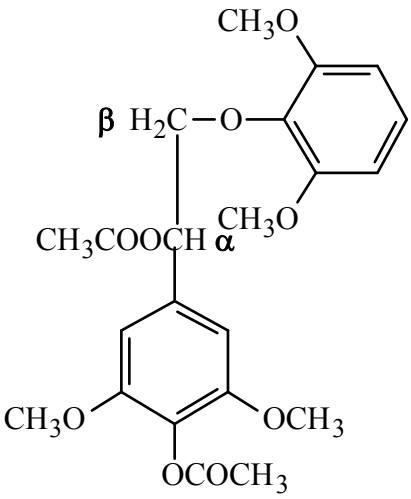

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.34 | 6H, s; $CH_3CO$ |
| 3.78 | 12H, s; $CH_3O$ |
| 3.93 | 2H, s; $CH_2$ |
| 6.43 | 4H, s; H-Ar |



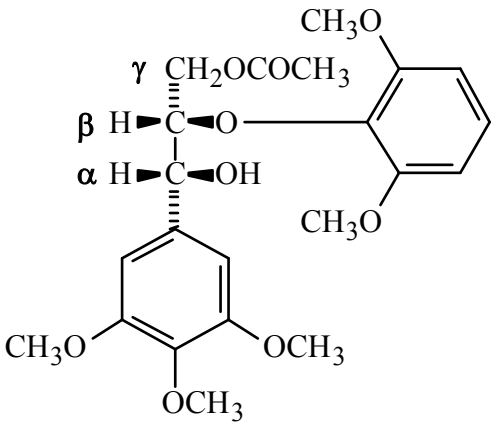

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.06 | 3H, s; $CH_3CO$ |
| 2.29 | 3H, s; $CH_3CO$ |
| 2.30 | 3H, s; $CH_3CO$ |
| 3.01 | 1H, dd, *J*=6.8 and 13.6 Hz; $CH_2$ |
| 3.16 | 1H, dd, *J*=7.2 and 13.6 Hz; $CH_2$ |
| 3.72 | 3H, s; $CH_3O$ |
| 3.75 | 3H, s; $CH_3O$ |
| 5.90 | 1H, ≈t, *J*=6.8 Hz; CH-Ar |
| 6.56 | 1H, d, *J*=2.0 Hz; H-Ar |
| 6.69 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.73 | 1H, d, *J*=2.0 Hz; H-Ar |
| 6.89 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.91 | 1H, d, *J*= 8.0 Hz; H-Ar |
| 6.99 | 1H, d, *J*=8.0 Hz; H-Ar |




| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.33 | 3H, s; $CH_3CO$ |
| 2.35 | 3H, s; $CH_3CO$ |
| 3.80 | 6H, s; $CH_3O$ |
| 3.87 | 6H, s; $CH_3O$ |
| 4.22 | 2H, s; $CH_2$ |
| 6.51 | 2H, s; H-Ar |
| 7.27 | 2H, s; H-Ar |

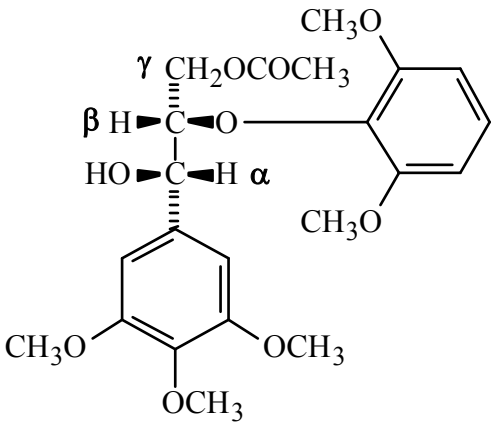



| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.11 | 3H, s; $CH_3CO$ |
| 2.33 | 3H, s; $CH_3CO$ |
| 3.81 | 6H, s; $CH_3O$ |
| 3.82 | 6H, s; $CH_3O$ |
| 4.24 | 1H, dd, *J*=4.0 and 11.6 Hz; $H_\beta$ |
| 4.28 | 1H, dd, *J*=7.8 and 11.6 Hz; $H_\beta$ |
| 6.03 | 1H, dd, *J*=4.0 and 7.8 Hz; $H_\alpha$ |
| 6.58 | 2H, d, *J*=8.4 Hz; H-Ar |
| 6.62 | 2H, s; H-Ar |
| 7.00 | 1H, t, *J*=8.4 Hz; H-Ar |



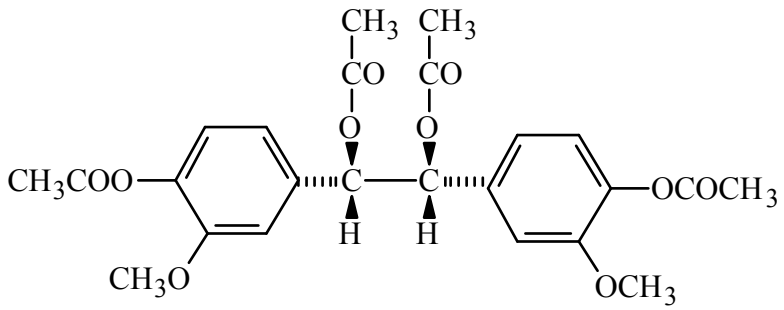

| 400 MHz, $CDCl_3$ (small amount of $CD_3COOD$ added), 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.01 | 3H, s; $CH_3CO$ |
| 3.83 | 3H, s; $CH_3O$ |
| 3.85 | 6H, s; $CH_3O$ |
| 3.89 | 6H, s; $CH_3O$ |
| 4.17 | 1H, dd, *J*=3.7 and 12.0 Hz; $H_\gamma$ |
| 4.41 | 1H, dd, *J*=8.1 and 12.0 Hz; $H_\gamma$ |
| 4.54 | 1H, ddd, *J*=2.9, 3.7 and 8.1 Hz; $H_\beta$ |
| 4.85 | 1H, d, *J*=2.9 Hz; $H_\alpha$ |
| 6.58 | 2H, s; H-Ar |
| 6.65 | 2H, d, *J*=8.4 Hz; H-Ar |
| 7.09 | 1H, t, *J*=8.4 Hz; H-Ar |

**Note**: Before addition of $CD_3COOD$, δ 4.32 (1H, d, *J*=2.9 Hz; α-OH), 4.85 (1H, m, $H_\alpha$)

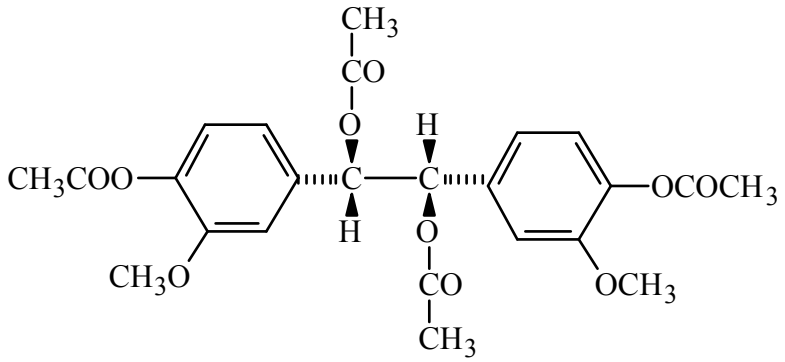



| 400 MHz, $CDCl_3$ (small amount of $CD_3COOD$ added), 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.07 | 3H, s; $CH_3CO$ |
| 3.81 | 3H, s; $CH_3O$ |
| 3.83 | 6H, s; $CH_3O$ |
| 3.88 | 6H, s; $CH_3O$ |
| 3.96 | 1H, dd, *J*=2.9 and 12.2 Hz; $H_\gamma$ |
| 4.07 | 1H, ddd, *J*=2.9, 3.4 and 8.3 Hz; $H_\beta$ |
| 4.56 | 1H, dd, *J*=3.4 and 12.2 Hz; $H_\gamma$ |
| 5.01 | 1H, d, *J*=8.3 Hz; $H_\alpha$ |
| 6.59 | 2H, s; H-Ar |
| 6.61 | 2H, d, *J*=8.4 Hz; H-Ar |
| 7.04 | 1H, t, *J*=8.4 Hz; H-Ar |

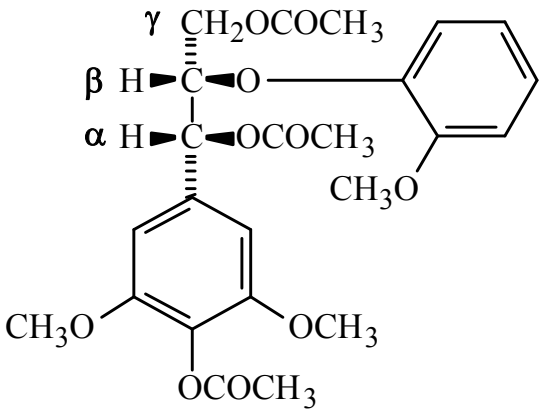
**Note**: Before addition of $CD_3COOD$, δ 4.80 (1H, d, *J*=2.0 Hz; α-OH), 5.01 (1H, m, $H_\alpha$)



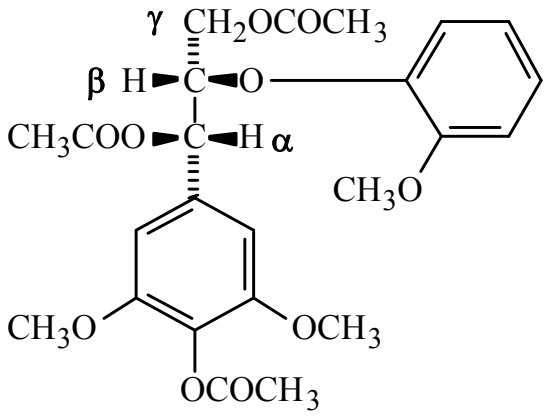

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.07 | 6H, s; $CH_3CO$ |
| 2.30 | 6H, s; $CH_3CO$ |
| 3.70 | 6H, s; $CH_3O$ |
| 6.08 | 2H, s; CH-Ar |
| 6.61 | 2H, d, *J*=2.0 Hz; H-Ar |
| 6.84 | 2H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.97 | 2H, d, *J*=8.0 Hz; H-Ar |



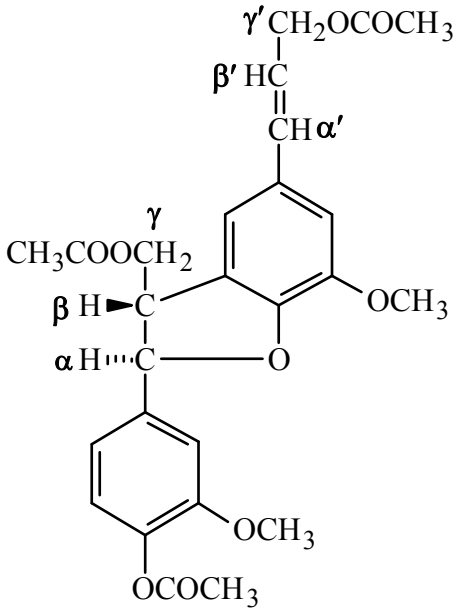

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.12 | 6H, s; $CH_3CO$ |
| 2.27 | 6H, s; $CH_3CO$ |
| 3.66 | 6H, s; $CH_3O$ |
| 5.96 | 2H, s; CH-Ar |
| 6.53 | 2H, d, *J*=2.0 Hz; H-Ar |
| 6.78 | 2H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.92 | 2H, d, *J*=8.0 Hz; H-Ar |



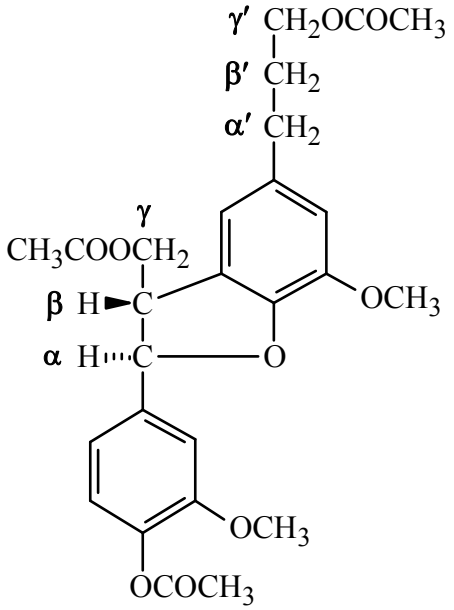

| 400 MHz, $CDCl_3$, 28° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.03 | 3H, s; $CH_3CO$ |
| 2.09 | 3H, s; $CH_3CO$ |
| 2.31 | 3H, s; $CH_3CO$ |
| 3.77 | 6H, s; $CH_3O$ |
| 3.79 | 3H, s; $CH_3O$ |
| 4.29 | 1H, dd, *J*=4.0 and 11.7 Hz; $H_\gamma$ |
| 4.46 | 1H, dd, *J*=5.5 and 11.7 Hz; $H_\gamma$ |
| 4.68 | 1H, m; $H_\beta$ |
| 6.06 | 1H, d, *J*=6.2 Hz; $H_\alpha$ |
| 6.69 | 2H, s; H-Ar |
| 6.8-7.1 | 4H, m; H-Ar |



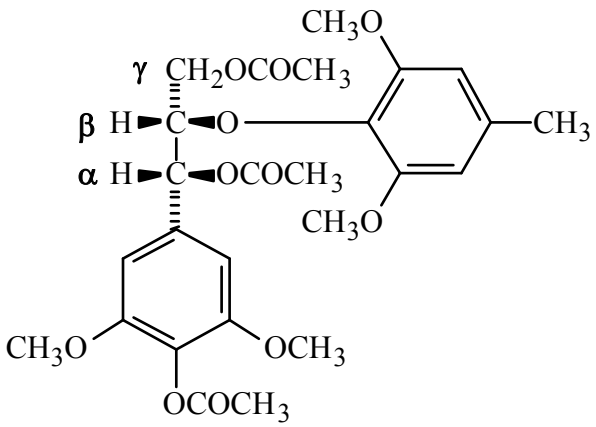

| 400 MHz, $CDCl_3$, 28° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.00 | 3H, s; $CH_3CO$ |
| 2.06 | 3H, s; $CH_3CO$ |
| 2.31 | 3H, s; $CH_3CO$ |
| 3.80 | 6H, s; $CH_3O$ |
| 3.81 | 3H, s; $CH_3O$ |
| 4.09 | 1H, dd, *J*=5.9 and 11.7 Hz; $H_\gamma$ |
| 4.33 | 1H, dd, *J*=4.6 and 11.7 Hz; $H_\gamma$ |
| 4.63 | 1H, m; $H_\beta$ |
| 6.10 | 1H, d, *J*=6.2 Hz; $H_\alpha$ |
| 7.01 | 2H, s; H-Ar |
| 6.84-7.05 | 4H, m; H-Ar |



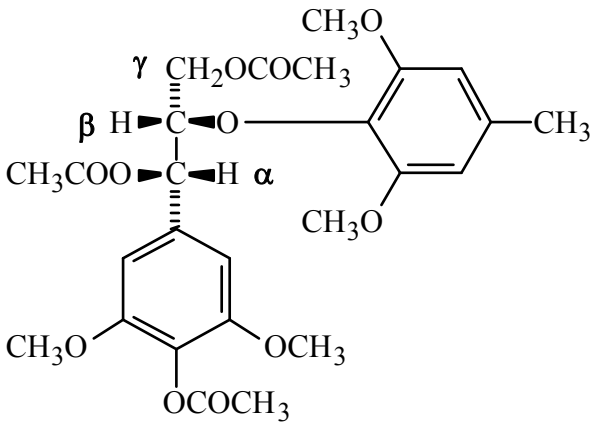

| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.06 | 3H, s; $CH_3CO$ |
| 2.10 | 3H, s; $CH_3CO$ |
| 2.31 | 3H, s; $CH_3CO$ |
| 3.78 | 1H, m; $H_\beta$ |
| 3.81 | 3H, s; $CH_3O$ |
| 3.92 | 3H, s; $CH_3O$ |
| 4.30 | 1H, dd, *J*=7.6 and 11.2 Hz; $H_\gamma$ |
| 4.45 | 1H, dd, *J*=5.4 and 11.2 Hz; $H_\gamma$ |
| 4.71 | 2H, dd, *J*=1.2 and 6.6 Hz; $H_{\gamma'}$ |
| 5.55 | 1H, d, *J*=6.8 Hz; $H_\alpha$ |
| 6.16 | 1H, td, *J*=6.6 and 15.9 Hz; $H_{\beta'}$ |
| 6.60 | 1H, ≈d, *J*=15.9 Hz; $H_{\alpha'}$ |
| 6.89 | 2H, m; H-Ar |
| 6.95 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 6.99 | 1H, d, *J*=2.0 Hz; H-Ar |
| 7.01 | 1H, d, *J*=8.0 Hz; H-Ar |




| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 1.94 | 2H, m; $H_{\beta'}$ |
| 2.05 | 3H, s; $CH_3CO$ |
| 2.07 | 3H, s; $CH_3CO$ |
| 2.31 | 3H, s; $CH_3CO$ |
| 2.64 | 2H, ≈t, *J*=7.6 Hz; $H_{\alpha'}$ |
| 3.76 | 1H, m; $H_{\beta}$ |
| 3.82 | 3H, s; $CH_3O$ |
| 3.90 | 3H, s; $CH_3O$ |
| 4.10 | 2H, t, *J*=6.4 Hz; $H_{\gamma'}$ |
| 4.29 | 1H, dd, *J*=8.0 and 11.2 Hz; $H_{\gamma}$ |
| 4.45 | 1H, dd, *J*=5.6 and 11.2 Hz; $H_{\gamma}$ |
| 5.52 | 1H, d, *J*=7.6 Hz; $H_{\alpha}$ |
| 6.64 | 1H, broad s; H-Ar |
| 6.65 | 1H, broad s; H-Ar |
| 6.91 | 1H, d, *J*=8.0 Hz; H-Ar |
| 6.95 | 1H, dd, *J*=2.0 and 8.0 Hz; H-Ar |
| 7.02 | 1H, d, *J*=2.0 Hz; H-Ar |




| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.00 | 3H, s; $CH_3CO$ |
| 2.15 | 3H, s; $CH_3CO$ |
| 2.30 | 3H, broad s; $CH_3$-Ar |
| 2.32 | 3H, s; $CH_3CO$ |
| 3.72 | 6H, s; $CH_3O$ |
| 3.79 | 6H, s; $CH_3O$ |
| 4.27 | 1H, dd, *J*=3.2 and 12.0 Hz; $H_\gamma$ |
| 4.48 | 1H, dd, *J*=5.2 and 12.0 Hz; $H_\gamma$ |
| 4.56 | 1H, m; $H_\beta$ |
| 6.07 | 1H, d, *J*=5.2 Hz; $H_\alpha$ |
| 6.35 | 2H, d, *J*=0.4 Hz; H-Ar |
| 6.63 | 2H, s; H-Ar |




| 400 MHz, $CDCl_3$, 20° C, $(CH_3)_4Si$ | |
|---|---|
| **δ (ppm)** | |
| 2.02 | 3H, s; $CH_3CO$ |
| 2.02 | 3H, s; $CH_3CO$ |
| 2.30 | 3H, broad s; $CH_3$-Ar |
| 2.33 | 3H, s; $CH_3CO$ |
| 3.74 | 6H, s; $CH_3O$ |
| 3.80 | 6H, s; $CH_3O$ |
| 3.96 | 1H, dd, *J*=4.0 and 12.0 Hz; $H_\gamma$ |
| 4.37 | 1H, dd, *J*=5.2 and 12.0 Hz; $H_\gamma$ |
| 4.46 | 1H, m; $H_\beta$ |
| 6.12 | 1H, d, *J*=6.0 Hz; $H_\alpha$ |
| 6.36 | 2H, d, *J*=0.4 Hz; H-Ar |
| 6.71 | 2H, s; H-Ar |