

Supporting Information

[1]. Preparative photolyses: The light source was a medium-pressure mercury lamp (500 W) in a cooling water jacket. The solution of the substrate and an excess amount of alkene in benzene was purged with dry argon for 15 min. The solutions were then irradiated with $\lambda > 400$ nm at room temperature under continuous argon purging. At the end of the reaction (TLC monitoring), the solvent was removed *in vacuo* and the residue was separated by flash chromatography on a silica gel column with petroleum ether (b.p. 60-90 °C)/ethyl acetate as eluents.

[2]. Spectral data of typical compounds.

4a. Yellow crystals from chloroform. m.p. 228°C; IR (KBr): 3050, 1740, 1700, 1660, 1600, 1580, 1450, 1370, 1340, 1290, 1240, 1180, 1100, 1050, 940, 900, 840, 790, 760, 700 cm^{-1} ; ^1H NMR(300M, CDCl_3): 2.61 (3H, s, $-\text{CH}_3$), 6.95-7.01(2H, m), 7.31-7.62(9H, m), 8.06-8.09(2H, dd, $J=7.1, 1.4$), 8.32(1H, d, $J=8.2$); MS: 367(M^+ , 23.0), 325(38.1), 296(26.3), 248(13.4), 220(12.8), 190(9.9), 165(12.1), 105(100), 77(53.8), 43(31.6); Found: C, 78.30, H, 4.60, N, 3.79. $\text{C}_{24}\text{H}_{17}\text{O}_3\text{N}$ requires C, 78.47, H, 4.63, N, 3.81.

4b. Yellow crystals from ethyl acetate. m.p. 179°C; IR (KBr): 1730, 1700, 1670, 1600, 1580, 1500, 1450, 1380, 1340, 1300, 1250, 1170, 1050, 840, 780, 750, 700, 680, 630 cm^{-1} ; ^1H NMR(300M, CDCl_3): 2.60(3H, s, COCH_3), 3.87(3H, s, OCH_3), 6.97-7.58(10H, m, ArH), 8.02-8.46(2H, dd, $J=8.6, 1.5$), 8.31(1H, d, $J=7.9$); MS: 397(M^+ , 61.3), 355(53.4), 326(37.0), 250(49.7), 235(8.9), 219(11.6), 207(10.6), 179(8.6), 105(100), 77(57.6), 43(74.8); Found: C, 75.39, H, 5.00, N, 3.63. $\text{C}_{25}\text{H}_{19}\text{O}_4\text{N}$ requires C, 75.57, H, 4.79, N, 3.53.

4e. Green needles from ethyl acetate. m.p. 179°C; IR (KBr): 3050, 1740, 1720, 1670, 1580, 1450, 1360, 1340, 1310, 1280, 1240, 1200, 1080, 1030, 930, 840, 790, 780, 760, 700, 620, 560 cm^{-1} ; ^1H NMR(300M, CDCl_3): 2.61(3H, s, COCH_3), 6.95-7.01(2H, m, ArH), 7.30-7.62(8H, m), 7.98-8.01(2H, m), 8.30-8.33(1H, d, $J=8.1$); MS: 401(M^+ , 30.1), 359(56.9), 330(35.0), 248(8.2), 220(24.2), 191(11.9), 171(14.3), 165(23.8), 139(100), 111(41.6), 43(59.2); Found: C, 71.78, H, 3.97, N, 3.39. $\text{C}_{24}\text{H}_{16}\text{O}_3\text{NCl}$ requires C, 71.73, H, 3.99, N, 3.49.

6. Colorless prisms from ethyl acetate. m.p. 270-272°C; IR (KBr): 1805, 1750, 1718, 1600, 1460, 1365, 1335, 1310, 1270, 1170, 1015, 940, 760, 700 cm^{-1} ; ^1H NMR(500M, CDCl_3): 2.41(3H, s, COCH_3), 2.71(3H, s, COCH_3), 5.41(1H, s), 7.10(2H, d, $J=7.2$, ArH), 7.18-7.39(7H, m, ArH), 7.59(2H, m, ArH), 7.92(1H, d, $J=8.0$, ArH), 8.03(1H, d, $J=8.0$, ArH); MS: 367(0.2), 352(0.9), 291(26), 263(45), 221(100); Found: C, 70.09, H, 4.32, N, 6.02. $\text{C}_{28}\text{H}_{20}\text{N}_2\text{O}_6$ requires C, 70.00, H, 4.17, N, 5.83.

7. colorless needles from petroleum ether (60-90°)/ethyl acetate. m.p. 179°C; IR (KBr): 1800, 1770, 1720, 1600, 1460, 1370, 1340, 1310, 1280, 1180, 1018, 932, 770, 700 cm^{-1} ; ^1H NMR(500M, CDCl_3): 2.51(3H, s, COCH_3), 2.82(3H, s, COCH_3), 5.80(1H, s), 6.79(1H, d, $J=7.5$, ArH), 6.85(1H, t, $J=7.5$, ArH), 7.10-7.33(8H, m, ArH), 7.63(1H, d, $J=6.7$, ArH), 7.84(1H, d, $J=7.7$, ArH), 8.18(1H, d, $J=8.3$, ArH); MS: 352(0.9), 291(26), 263(41), 221(100); Found: C, 70.12, H, 4.09, N, 5.63. $\text{C}_{28}\text{H}_{20}\text{N}_2\text{O}_6$ requires C, 70.00, H, 4.17, N, 5.83.

Crystal Structure of compound 4a

$\text{C}_{24}\text{H}_{17}\text{NO}_3$, $M = 367.39$ triclinic, space group P-1 with $a = 8.2328(2)$, $b = 16.7881(5)$, $c = 12.5486(6)$ Å, $\alpha = 83.112(2)^\circ$, $\beta = 86.853(2)^\circ$, $\gamma = 79.392(2)^\circ$, $V = 919.21(6)$ Å³, $z = 2$, $D_c = 1.327$ g cm^{-3} . Absorption coefficient 0.088 mm^{-1} , $F(000) = 384$. A yellow block shaped crystal of 0.46 × 0.28 × 0.24 mm was used. Data were collected on a Siemens SMART CCD area detector diffractometer equipped with graphite-monochromatized Mo $K\alpha$ in the range of θ 2.96 -28.37°. The structure was solved by direct method SHELXL-97 and refined on F^2 by full-matrix least-squares method. A total of 4484 independent reflections [$R(\text{int}) = 0.0750$] were used in the refinement which converged with $R = 0.1357$ and $wR = 0.3221$.

Crystal Structure of compound 4d

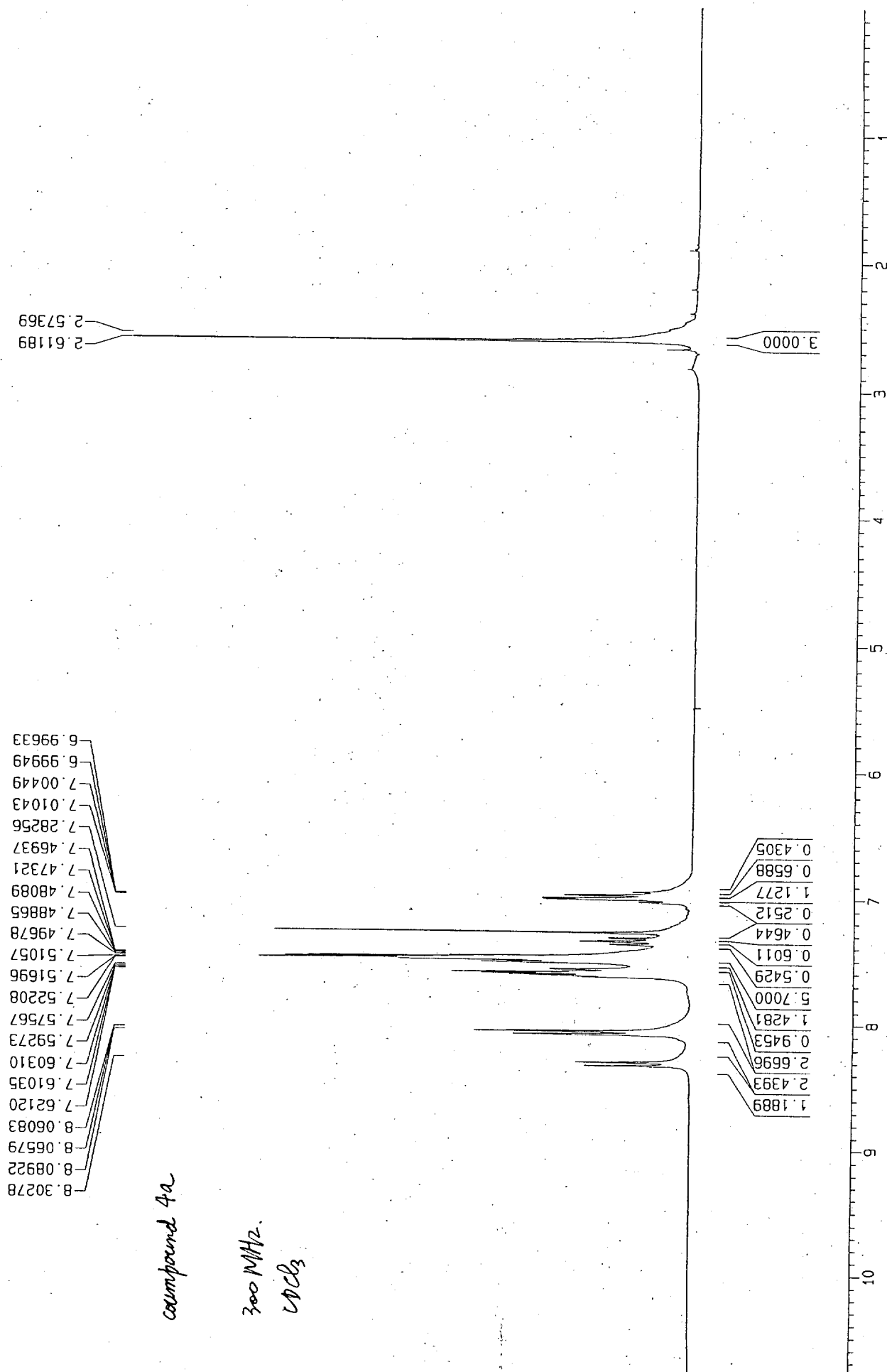
$\text{C}_{24}\text{H}_{17}\text{NO}_3$, $M = 397.41$ monoclinic, space group P 21/c with $a = 8.0395(3)$, $b = 9.1233(4)$, $c = 14.8438(5)$ Å, $\alpha = 90^\circ$, $\beta = 96.954(1)^\circ$, $\gamma = 90^\circ$, $V = 1988.70(12)$ Å³, $z = 4$, $D_c = 1.327$ g cm^{-3} . Absorption coefficient 0.090 mm^{-1} , $F(000) = 832$. A yellow block shaped crystal of 0.34 × 0.26 × 0.22 mm was used. Data were collected on a Siemens SMART CCD area detector diffractometer equipped with graphite-monochromatized Mo $K\alpha$ in

the range of θ 1.84-28.35°. The structure was solved by direct method SHELXL-97 and refined on F^2 by full-matrix least-squares method. A total of 13747 independent reflections [R (int) = 0.0693] were used in the refinement which converged with R = 0.0566 and wR = 0.1393.

Crystal Structure of compound 6

$C_{28}H_{20}N_2O_6$, M = 480.48. Monoclinic, space group P21/c with a = 13.20(2), b = 12.246(4), c = 14.176(3) Å, α = 90°, β = 90.90(5)°, γ = 90°, V = 2290.7400 Å³, z = 4, D_c = 1.393 g cm⁻³. Absorption coefficient 0.099 mm⁻¹, $F(000)$ = 1000. A colorless prismatic shaped crystal of 0.400×0.300×0.300 mm was used. Data were collected on an Rigaku RAXISIV detector diffractometer equipped with graphite-monochromatized Mo K α in the range of θ 2-27°. The structure was solved by direct method (SHELXS86) and refined on F^2 by full-matrix least-squares method. A total of 4222 independent reflections [R (int) = 0.086] were used in the refinement which converged with R = 0.0722 and wR = 0.1043.

[3]. ¹H NMR spectra of compounds 4a, 4b, 4e, 6 and 7.



compound 4a

300 MHz

CDCl₃

ZY-2

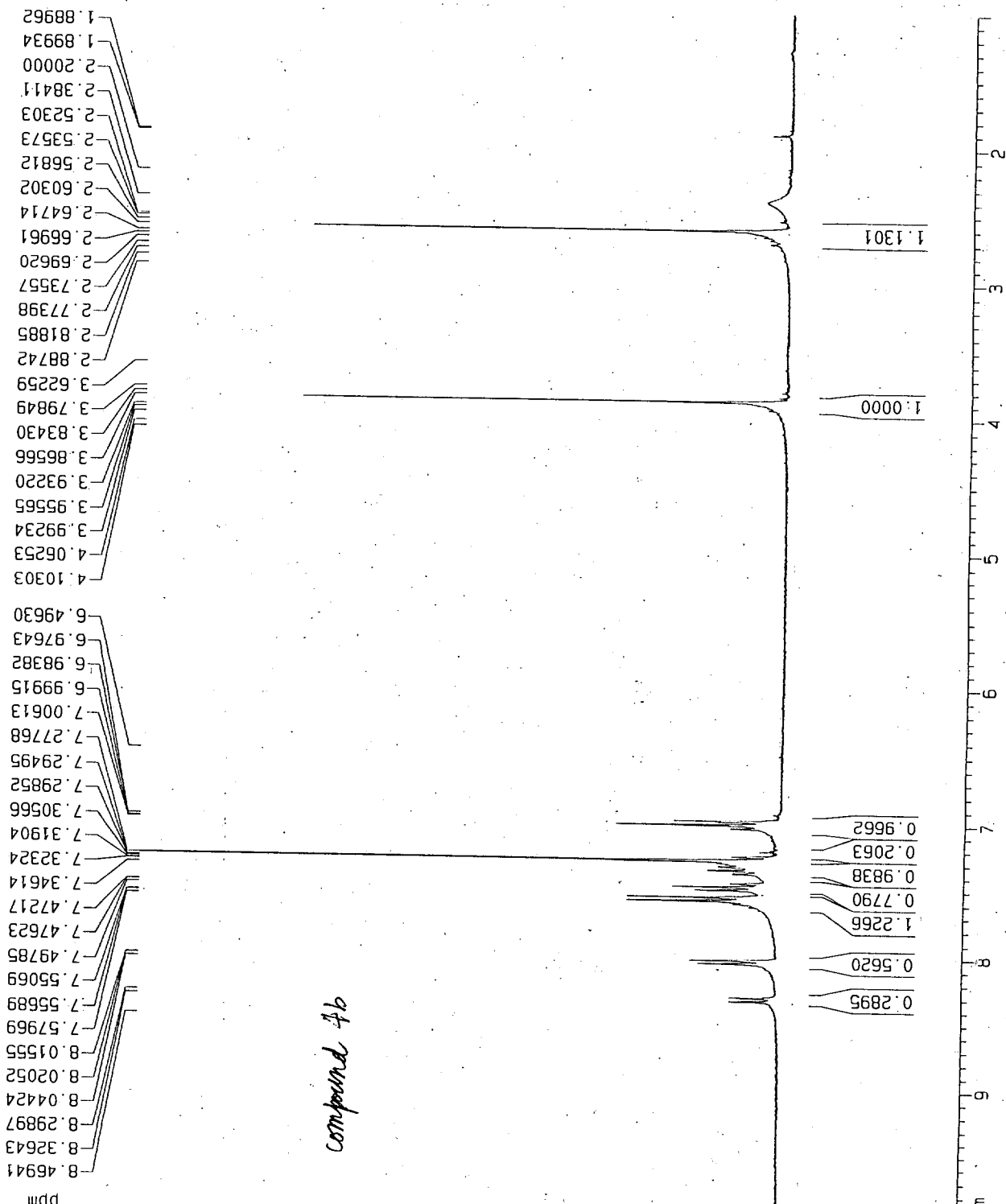
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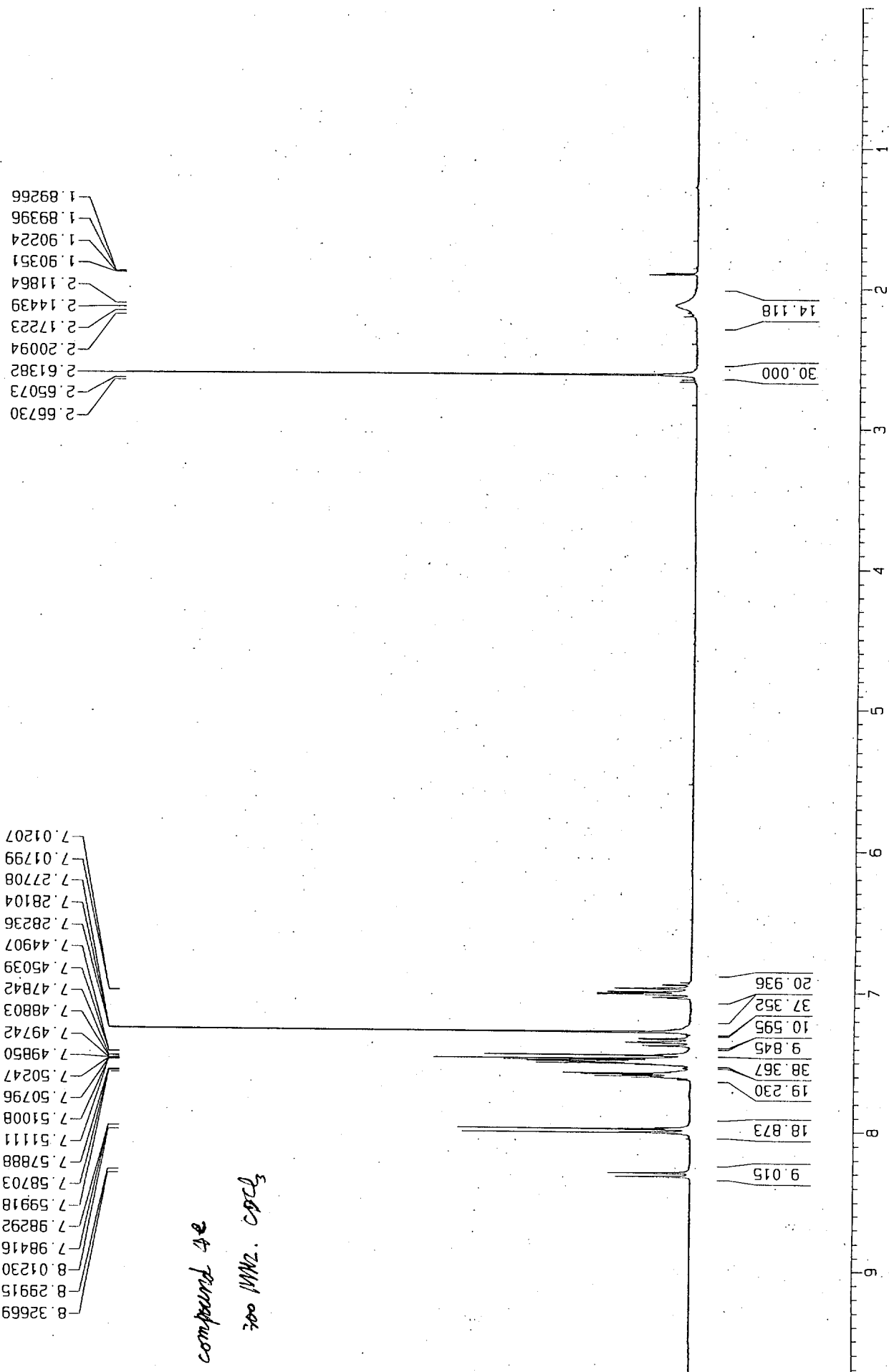
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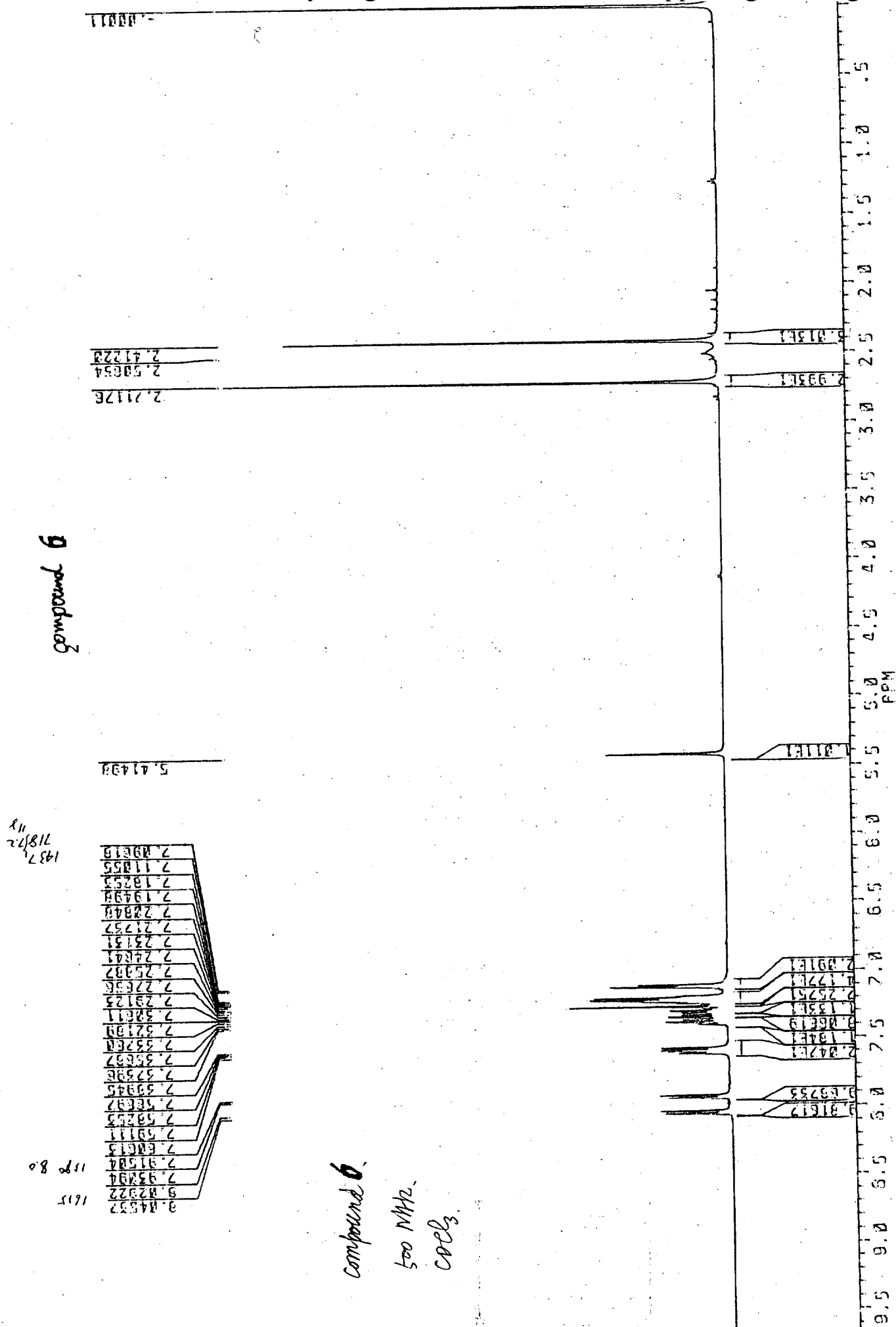
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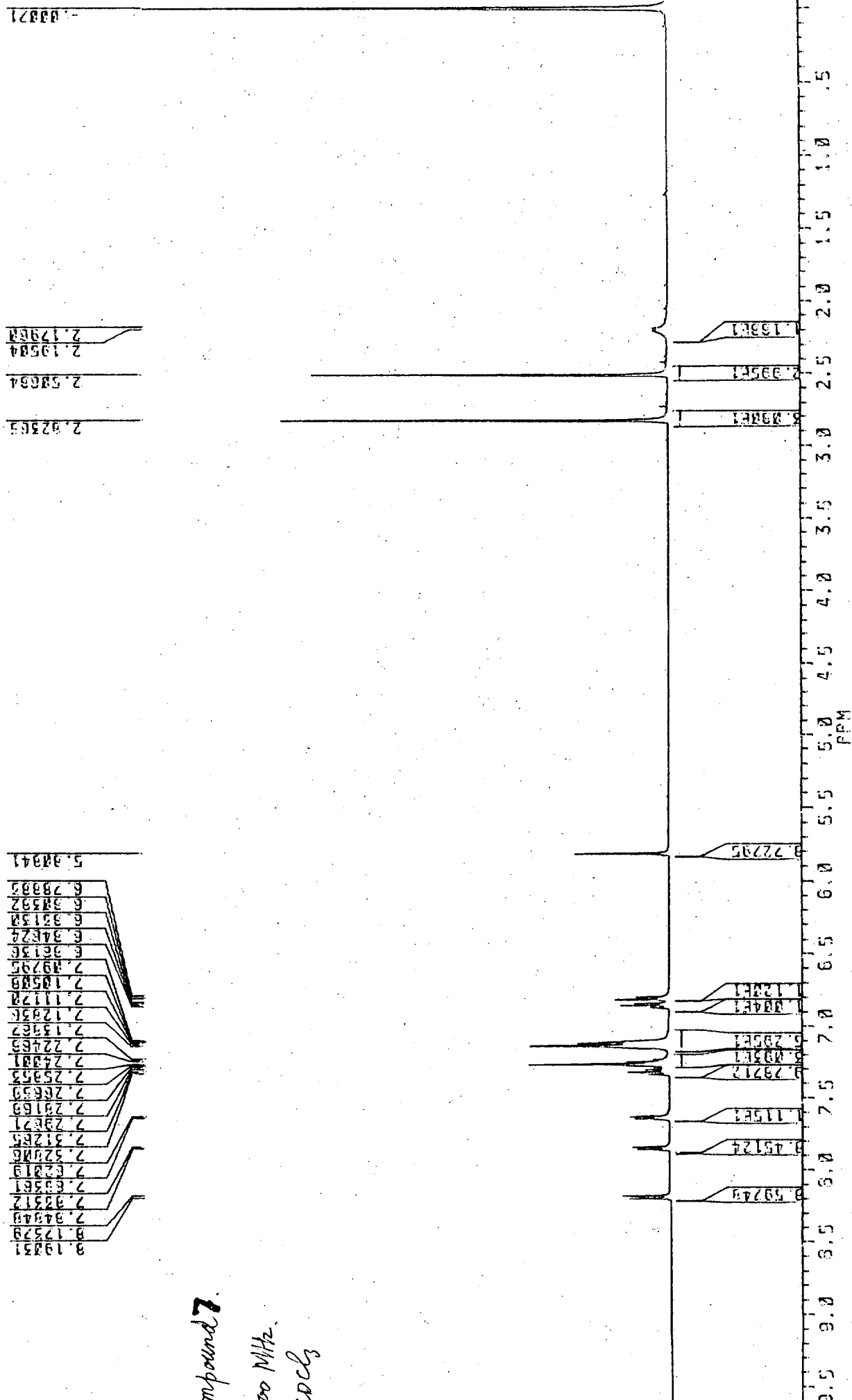
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compound 4e
300 MHz, CDCl₃





compound 7
 100 MHz
 CDCl₃