

Supplementary Information

Activated carbon for aerobic oxidation: Benign approach toward 2-benzoylbenzimidazoles and 2-benzoylbenzoxazoles synthesis

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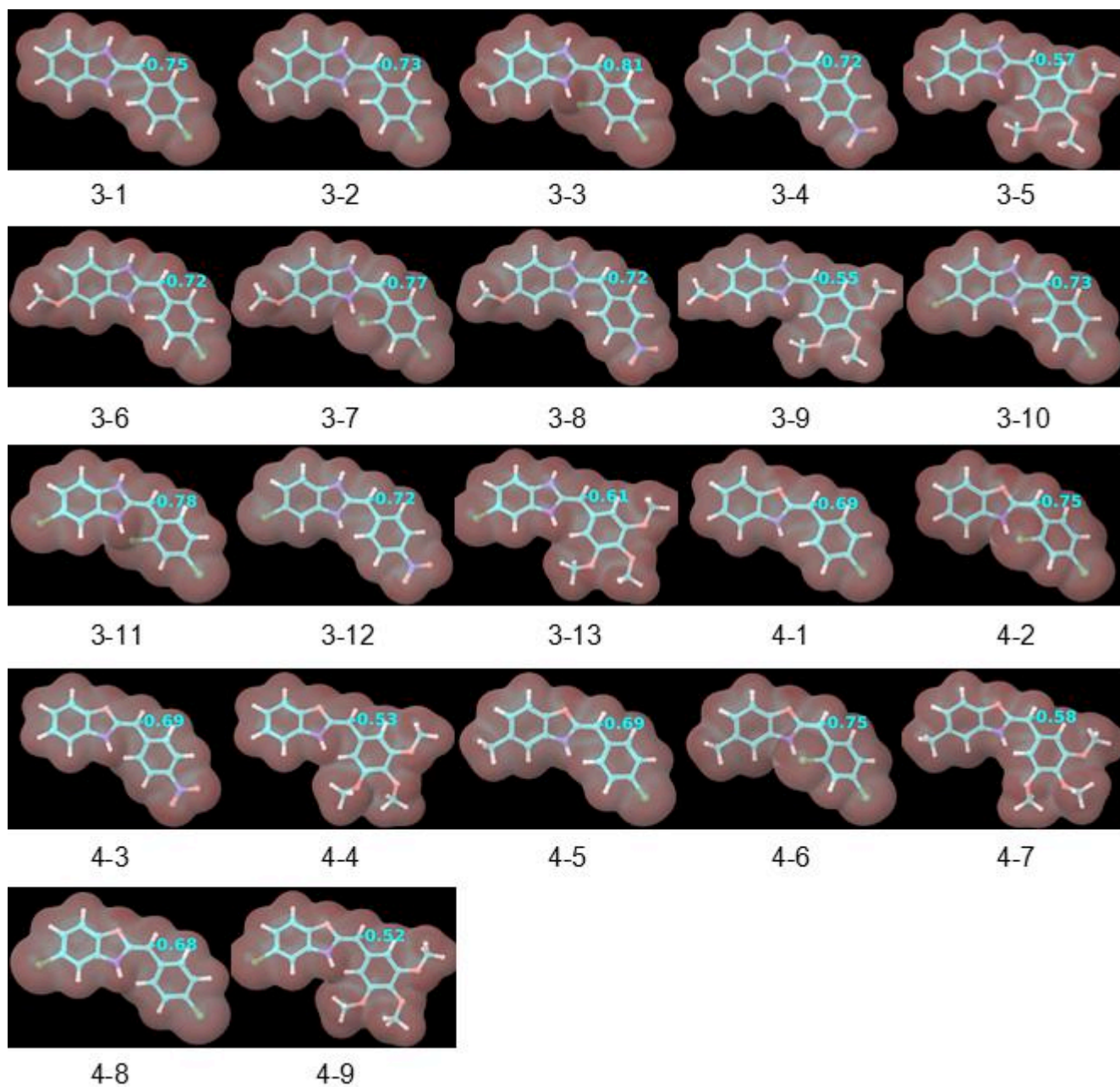
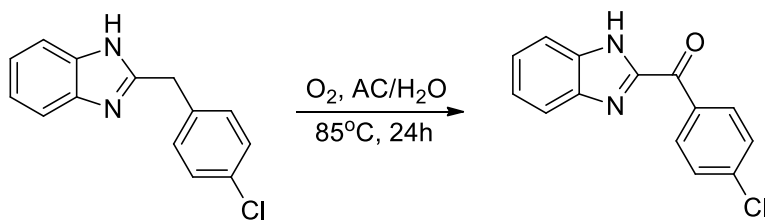


Figure S1: Electric density of the substrates in Table 3 and 4 in the form of the enamine tautomer.

Method: Calculations of the electric density were carried out with the Jaguar 4.0 software within Schrodinger (2014.1) using the B3LYP hybrid density functional method. Geometries were optimized using the 6-31G (d) basis set, and single point energies were calculated using the 6-311+G (d,p) basis set.

Table S1. Comparison of reported procedures for the benzylic oxidation of 2-benzylbenzimidazoles

a.

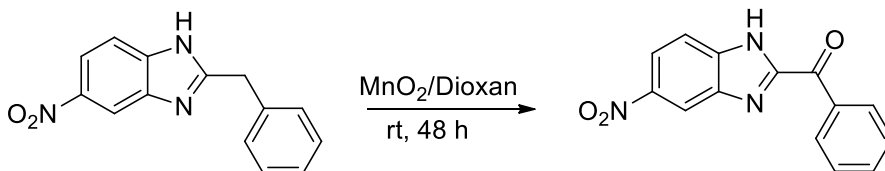


Raw Materials Used		Product and Waste	
Compound 4	485.40 mg	Compound 6	446.64 mg
AC III	60.00 mg	Aqueous waste	5.00 g
Water	5.00 g (5 mL)		
Total	5.54 g	Total	5.45 g

E Factor (E) = (5 g of waste produced / 446.64 mg of product) = 11.19

Mass Intensity = (5.54 g of raw material used / 446.64 mg of product) = 12.40

b.



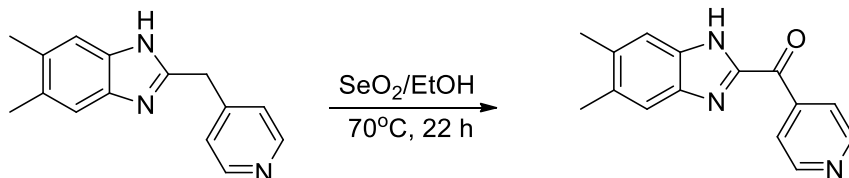
H. C. Ooi and H. Suschitzky, *J. Chem. Soc., Perkin Trans. 1*, 1982, 2871.

Raw Materials Used		Product and Waste	
Compound 5	0.20 g	Compound 4	0.16 g
MnO ₂	1.00 g	Solid waste	1.00 g
Dioxan	10.30 g (10 mL)	Organic waste	10.30 g
Total	11.50 g	Total	11.46 g

E Factor (E) = (11.46 g of waste produced / 0.16 g of product) = 71.63

Mass Intensity = (11.50 g of raw material used / 0.16 g of product) = 71.88

c.



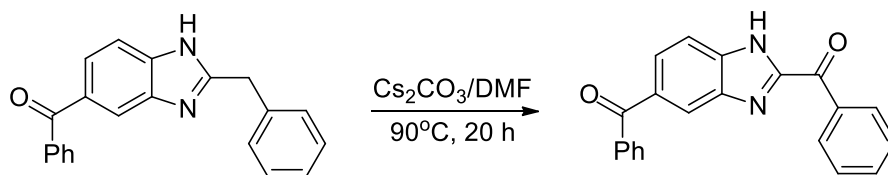
E. Alcalde, M. Gisbert, J. M. Pons and L. Perez-Garcia, *Tetrahedron*, 1996, 52, 15197.

Raw Materials Used		Product and Waste	
Compound 8b	3.30 g	Compound 11b	1.32 g
SeO ₂	1.76 g	Organic waste	60.94 g
EtOH	59.18 g (75 mL)		
Total	64.24 g	Total	62.26 g

E Factor (E) = (60.94 g of waste produced / 1.32 g of product) = 46.17

Mass Intensity = (64.24 g of raw material used / 1.32 g of product) = 48.67

d.



A. D. Santos, L. E. Kaïm and L. Grimaud, *Org. Biomol. Chem.*, 2013, 11, 3282.

Raw Materials Used		Product and Waste	
Compound 3d	62.47 mg	Compound 4d	47.65 mg
Cs_2CO_3	65.16 mg	Organic waste	1.00 g
DMF	0.94 g (1 mL)		
Total	1.07 g	Total	1.05 g

E Factor (E) = (1.00 g of waste produced / 47.65 mg of product) = 20.99

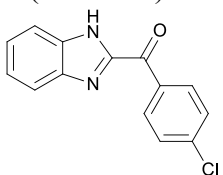
Mass Intensity = (1.07 g of raw material used / 47.65 mg of product) = 22.46

Note:

1. >98 % catalyst recovered in our method.
2. Calculation did not consider for solvent used for column chromatography.

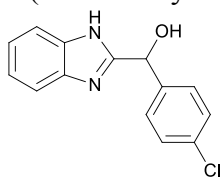
2-Benzoylbenzimidazoles

2-(4-Chloro)benzoylbenzimidazole (6, Table 3, entry 1)



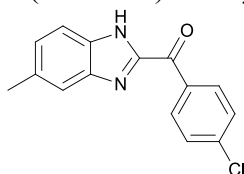
M. p. 226-228°C; $^1\text{H-NMR}$ (600MHz, DMSO- d_6): δ =13.54 (s, 1H), 8.61 (d, J=8.58 Hz, 2H), 7.87 (s, 1H), 7.66 (d, J=8.58 Hz, 2H), 7.60 (s, 1H), 7.40 (s, 1H), 7.32 (s, 1H); $^{13}\text{C-NMR}$ (DMSO- d_6): δ =182.6, 148.2, 143.6, 139.2, 134.7, 133.2, 129.1, 126.4, 123.8, 121.8, 113.3; MS: m/z (%): 257.0 ($[\text{M}+\text{H}]^+$).

2-(4-Chlorohydroxybenzyl)-benzo[d]imidazole (5)



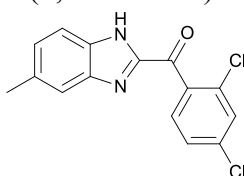
$^1\text{H-NMR}$ (600MHz, DMSO- d_6): δ =7.41 (m, 2H), 7.23 (d, J=7.15 Hz, 2H), 7.18 (m, 2H), 7.13 (d, J=7.15 Hz, 2H); MS: m/z (%): 259.0 ($[\text{M}+\text{H}]^+$).

2-(4-Chloro)benzoyl-5-methylbenzimidazole (Table 3, entry 2)



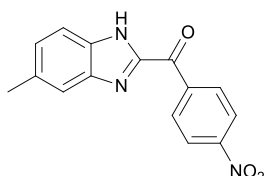
M. p. 200-203°C; $^1\text{H-NMR}$ (300MHz, DMSO- d_6): δ =10.54 (s, 1H), 8.66 (d, J=8.60 Hz, 2H), 7.82 (d, J=8.45 Hz, 1H), 7.53 (d, J=8.60 Hz, 2H), 7.20 (d, J=8.45 Hz, 1H), 2.52 (s, 3H); $^{13}\text{C-NMR}$ (DMSO- d_6): δ =182.4, 147.9, 144.1, 142.0, 139.1, 136.2, 134.7, 133.1, 129.0, 125.7, 121.4, 112.6, 22.0; MS: m/z (%): 268.9 ($[\text{M}-\text{H}]^-$).

2-(2,4-Dichloro)benzoyl-5-methylbenzimidazole (Table 3, entry 3)



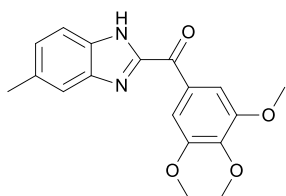
M. p. 223-225°C; $^1\text{H-NMR}$ (300MHz, DMSO- d_6): δ =13.61 (s, 1H), 7.84 (m, 2H), 7.63 (m, 2H), 7.46 (m, 1H), 7.19 (m, 1H), 2.44 (s, 3H); $^{13}\text{C-NMR}$ (DMSO- d_6): δ =185.3, 147.7, 136.5, 136.2, 136.1, 133.2, 132.5, 132.4, 129.9, 129.0, 127.6, 126.4, 121.2, 112.9, 21.9; MS: m/z (%): 305.0 ($[\text{M}+\text{H}]^+$), 327.0 ($[\text{M}+\text{Na}]^+$).

2-(4-Nitro)benzoyl-5-methylbenzimidazole (Table 3, entry 4)



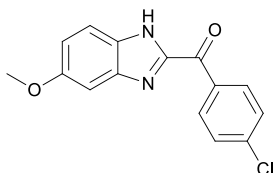
M. p. 212-214°C; $^1\text{H-NMR}$ (300MHz, DMSO- d_6): δ =13.53 (s, 1H), 8.72 (d, J=8.60 Hz, 2H), 8.41 (d, J=8.60 Hz, 2H), 7.19-7.70(m, 3H), 2.45 (s, 3H); $^{13}\text{C-NMR}$ (DMSO- d_6): δ =182.4, 150.3, 147.6, 144.1, 142.0, 141.0, 136.6, 135.1, 132.5, 125.9, 123.7, 121.5, 112.7, 22.0; MS: m/z (%): 279.9 ($[\text{M}-\text{H}]^-$).

2-(3,4,5-Trimethoxy)benzoyl-5-methylbenzimidazole (Table 3, entry 5)



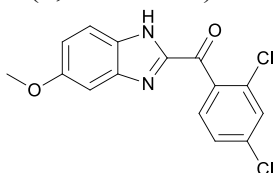
M. p. 195-197°C; $^1\text{H-NMR}$ (300MHz, DMSO-d_6): δ =13.33 (s, 1H), 8.05 (s, 2H), 7.73 (d, $J=8.45$ Hz, 2H), 7.39 (s, 1H), 7.15 (dd, $J=8.45$ Hz, $J=1.12$ Hz), 3.89 (s, 6H), 3.81 (s, 3H), 2.46 (s, 3H); $^{13}\text{C-NMR}$ (DMSO-d_6): δ =182.1, 152.9, 148.2, 143.1, 134.8, 131.9, 129.1, 125.6, 121.4, 112.5, 109.3, 60.7, 56.6, 19.1; MS: m/z (%): 327.1 ($[\text{M}+\text{H}]^+$), 349.1 ($[\text{M}+\text{Na}]^+$).

2-(4-Chloro)benzoyl-5-methoxybenzimidazole (Table 3, entry 6)



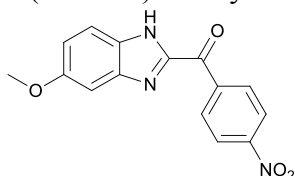
M. p. 169-171°C; $^1\text{H-NMR}$ (300MHz, DMSO-d_6): δ =13.40 (s, 1H), 8.58 (d, $J=7.05$ Hz, 2H), 7.65 (m, 3H), 6.98 (m, 1H), 3.8 (s, 3H); $^{13}\text{C-NMR}$ (DMSO-d_6): δ =182.0, 159.3, 150.3, 147.3, 141.2, 138.6, 136.0, 132.5, 123.8, 122.8, 115.6, 94.2, 60.0; MS: m/z (%): 284.9 ($[\text{M}-\text{H}]^-$).

2-(2,4-Dichloro)benzoyl-5-methoxybenzimidazole (Table 3, entry 7)



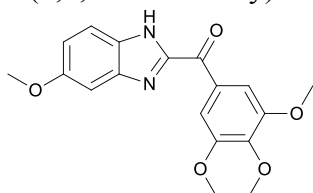
M. p. 202-203°C; $^1\text{H-NMR}$ (300MHz, DMSO-d_6): δ =13.56 (s, 1H), 7.84 (d, $J=8.30$, 1H), 7.79 (d, $J=1.91$, 1H), 7.62 (m, 2H), 7.02 (m, 2H), 3.82 (s, 3H); $^{13}\text{C-NMR}$ (DMSO-d_6): δ =184.6, 159.4, 147.3, 138.6, 136.3, 132.3, 131.9, 129.8, 129.1, 127.5, 122.9, 115.6, 94.2, 56.0; MS: m/z (%): 318.9 ($[\text{M}-\text{H}]^-$).

2-(4-Nitro)benzoyl-5-methoxybenzimidazole (Table 3, entry 8)



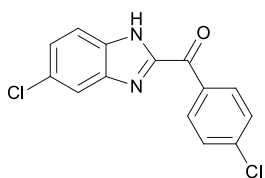
M. p. 216-218°C; $^1\text{H-NMR}$ (300MHz, DMSO-d_6): δ =13.52 (s, 1H), 8.70 (d, $J=8.67$ Hz, 2H), 8.41 (d, $J=8.67$ Hz, 2H), 7.70 (s, 1H), 7.00 (m, 2H), 3.83 (s, 3H); $^{13}\text{C-NMR}$ (DMSO-d_6): δ =181.8, 159.0, 147.6, 138.9, 138.5, 135.7, 134.8, 133.0, 128.9, 122.6, 115.2, 94.3, 55.9; MS: m/z (%): 295.9 ($[\text{M}-\text{H}]^-$).

2-(3,4,5-Trimethoxy)benzoyl-5-methoxybenzimidazole (Table 3, entry 9)



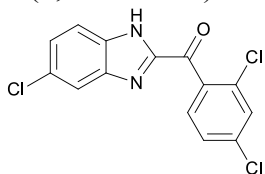
M. p. 150-153°C; $^1\text{H-NMR}$ (300MHz, DMSO-d_6): δ =13.32 (s, 1H), 8.03 (s, 2H), 7.73 (d, $J=8.81$ Hz, 1H), 7.00 (s, 1H), 6.95 (d, $J=8.81$ Hz, 1H), 3.88 (s, 9H), 3.83 (s, 3H); $^{13}\text{C-NMR}$ (DMSO-d_6): δ =181.6, 167.4, 158.8, 152.9, 142.9, 138.4, 135.5, 131.1, 129.1, 122.6, 114.9, 109.3, 94.3, 60.7, 56.5, 55.9; MS: m/z (%): 343.1 ($[\text{M}+\text{H}]^+$), 365.0 ($[\text{M}+\text{Na}]^+$).

2-(4-Chloro)benzoyl-5-chlorobenzimidazole (Table 3, entry 10)



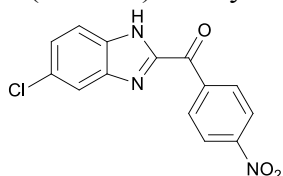
M. p. 224-226°C; $^1\text{H-NMR}$ (300MHz, DMSO- d_6): δ =8.57 (d, J=8.52 Hz, 2H), 7.77 (s, 1H), 7.74 (d, J=8.81 Hz, 1H), 7.66 (d, J=8.52 Hz, 2H), 7.37 (dd, J=8.81 Hz, J=1.69 Hz, 1H); $^{13}\text{C-NMR}$ (DMSO- d_6): δ =182.5, 149.2, 142.3, 139.4, 135.2, 134.4, 133.2, 130.7, 129.1, 124.4, 120.9, 112.9; MS: m/z (%): 288.8 ($[\text{M-H}]^-$).

2-(2,4-Dichlorobenzoyl)-5-chlorobenzimidazole (Table 3, entry 11)



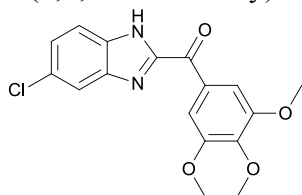
M. p. 182-184°C; $^1\text{H-NMR}$ (300MHz, DMSO- d_6): δ =13.76 (s, 1H), 7.78-7.90 (m, 2H), 7.63 (m, 3H), 7.40 (s, 1H); $^{13}\text{C-NMR}$ (DMSO- d_6): δ =185.0, 149.0, 144.3, 136.9, 135.6, 132.7, 132.5, 131.2, 130.0, 128.3, 127.5, 124.5, 121.2, 112.9; MS: m/z (%): 322.8 ($[\text{M-H}]^-$)

2-(4-Nitrobenzoyl)-5-chlorobenzimidazole (Table 3, entry 12)



M. p. 234-235°C; $^1\text{H-NMR}$ (300MHz, DMSO- d_6): δ =13.61 (s, 1H), 8.68 (d, J=8.52 Hz, 2H), 8.37 (d, J=8.52 Hz, 2H), 7.75 (s, 2H), 7.36 (m, 1H); $^{13}\text{C-NMR}$ (DMSO- d_6): δ =181.9, 150.2, 148.6, 140.3, 132.4, 131.8, 130.5, 129.0, 124.4, 120.7, 114.6, 111.1; MS: m/z (%): 299.8 ($[\text{M-H}]^-$)

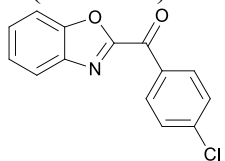
2-(3,4,5-Trimethoxybenzoyl)-5-chlorobenzimidazole (Table 3, entry 13)



M. p. 201-203°C; $^1\text{H-NMR}$ (300MHz, DMSO- d_6): δ =13.64 (s, 1H), 8.01 (s, 2H), 7.39-7.83 (m, 3H), 3.89 (s, 6H), 3.81 (s, 3H); $^{13}\text{C-NMR}$ (DMSO- d_6): δ =180.0, 152.9, 149.6, 143.3, 132.2, 131.9, 130.7, 129.1, 125.4, 118.3, 109.3, 60.8, 56.6; MS: m/z (%): 347.1 ($[\text{M+H}]^+$), 369.0 ($[\text{M+Na}]^+$).

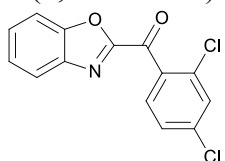
2-Benzoylbenzoxazoles

2-(4-Chloro)benzoylbenzoxazole (Table 4, entry 1)



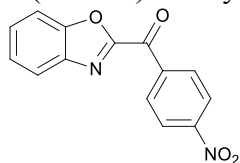
M.p. 106-108°C; ¹H-NMR (300MHz, CDCl₃): δ =8.57 (d, J=8.63 Hz, 2H), 7.94 (d, J=7.79 Hz, 1H), 7.70 (d, J=7.79, 1H), 7.45-7.58 (m, 4H); ¹³C-NMR (CDCl₃): δ =179.1, 156.9, 150.4, 141.1, 140.7, 133.30, 132.5, 128.9, 128.6, 125.9, 122.4, 111.9; MS: *m/z* (%): 257.9 ([M+H]⁺), 279.9 ([M+Na]⁺)

2-(2,4-Dichloro)benzoylbenzoxazole (Table 4, entry 2)



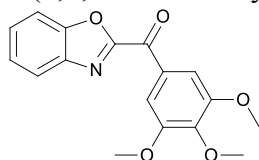
M.p. 102-103°C; ¹H-NMR (300MHz, CDCl₃): δ =7.90 (d, J=8.08, 1H), 7.78 (d, J=8.23, 1H), 7.70 (d, J=8.08 Hz, 1H), 7.59 (dd, J=7.41 Hz, J=1.22 Hz, 1H), 7.54 (dd, J=7.41 Hz, J=1.22 Hz, 1H), 7.49 (dd, J=8.23 Hz, J=1.79 Hz, 1H), 7.43 (m, 1H); ¹³C-NMR (CDCl₃): δ =180.9, 157.0, 150.9, 140.8, 138.8, 134.0, 133.9, 131.9, 130.7, 129.0, 127.2, 126.1, 122.7, 112.0; MS: *m/z* (%): 291.9 ([M+H]⁺), 313.9 ([M+Na]⁺)

2-(4-Nitro)benzoylbenzoxazole (Table 4, entry 3)



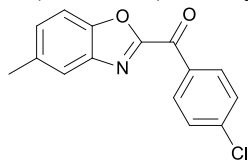
M.p. 106-108°C; ¹H-NMR (300MHz, CDCl₃): δ =8.78 (d, J=9.07 Hz, 2H), 8.41 (d, J=9.07 Hz, 2H), 7.98 (m, 1H), 7.73 (m, 1H), 7.61 (m, 1H), 7.52 (m, 1H); ¹³C-NMR (CDCl₃): δ =178.7, 156.5, 150.8, 150.5, 140.6, 139.5, 132.1, 129.2, 126.2, 123.6, 122.7, 112.0; *m/z* (%): 269.1 ([M+H]⁺)

2-(3,4,5-Trimethoxy)benzoylbenzoxazole (Table 4, entry 4)



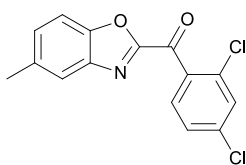
M.p. 89-91°C; ¹H-NMR (300MHz, CDCl₃): δ =7.96 (s, 2H), 7.94 (d, J=8.14 Hz, 1H), 7.73 (d, J=8.14 Hz, 1H), 7.53 (m, 2H), 4.00 (d, 9H); ¹³C-NMR (CDCl₃): δ =178.9, 157.3, 152.9, 150.4, 144.0, 140.8, 129.9, 128.4, 125.7, 122.3, 111.9, 108.8, 61.1, 56.4; MS: *m/z* (%): 314.1 ([M+H]⁺), 336.0 ([M+Na]⁺)

2-(4-Chloro)benzoyl-5-methylbenzoxazole (Table 4, entry 5)



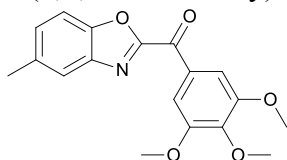
M.p. 107-108°C; ¹H-NMR (300MHz, CDCl₃): δ =8.54 (d, J=8.88 Hz, 2H), 7.70 (m, 1H), 7.57 (d, J=8.47 Hz, 1H), 7.52 (d, J=8.88 Hz, 2H), 7.36 (dd, J=8.47 Hz, J=1.30 Hz, 1H), 2.51 (s, 3H); ¹³C-NMR (CDCl₃): δ =179.1, 157.0, 148.7, 141.0, 140.9, 135.9, 133.4, 132.4, 130.1, 128.9, 121.9, 111.2, 21.6; MS: *m/z* (%): 272.0 ([M+H]⁺)

2-(2,4-Dichloro)benzoyl-5-methylbenzoxazole (Table 4, entry 6)



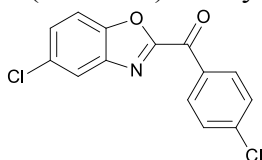
M.p. 105-107°C; $^1\text{H-NMR}$ (300MHz, CDCl_3): δ =7.76 (d, $J=8.37$ Hz, 1H), 7.66 (s, 1H), 7.55 (d, $J=8.52$ Hz, 1H), 7.53 (d, $J=1.87$ Hz, 1H), 7.34 (dd, $J=8.37$ Hz, $J=1.87$ Hz, 1H), 7.36(m, 1H), 2.50(s, 3H); $^{13}\text{C-NMR}$ (CDCl_3): δ =180.9, 157.2, 149.2, 141.0, 138.7, 136.2, 134.1, 133.9, 131.9, 130.7, 130.5, 127.2, 122.1, 111.3, 21.6; MS: m/z (%): 306.0 ($[\text{M}+\text{H}]^+$), 338.0 ($[\text{M}+\text{Na}]^+$).

2-(3,4,5-Trimethoxy)benzoyl-5-methylbenzoxazole (Table 4, entry 7)



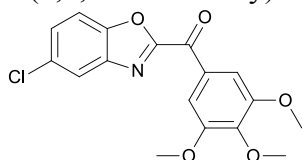
M. p. 100-102°C; $^1\text{H-NMR}$ (300MHz, CDCl_3): δ =7.94 (s, 2H), 7.72 (s, 1H), 7.58 (d, $J=8.55$ Hz, 1H), 7.37 (dd, $J=8.55$ Hz, $J=1.03$, 1H), 3.99 (m, 9H), 2.52 (s, 3H); $^{13}\text{C-NMR}$ (CDCl_3): δ =179.0, 157.4, 152.9, 148.7, 143.9, 141.0, 135.8, 130.0, 129.8, 121.9, 111.2, 108.8, 61.1, 56.3, 21.5; MS: m/z (%): 328.1 ($[\text{M}+\text{H}]^+$), 350.0 ($[\text{M}+\text{Na}]^+$).

2-(4-Chloro)benzoyl-5-chlorobenzoxazole (Table 4, entry 8)



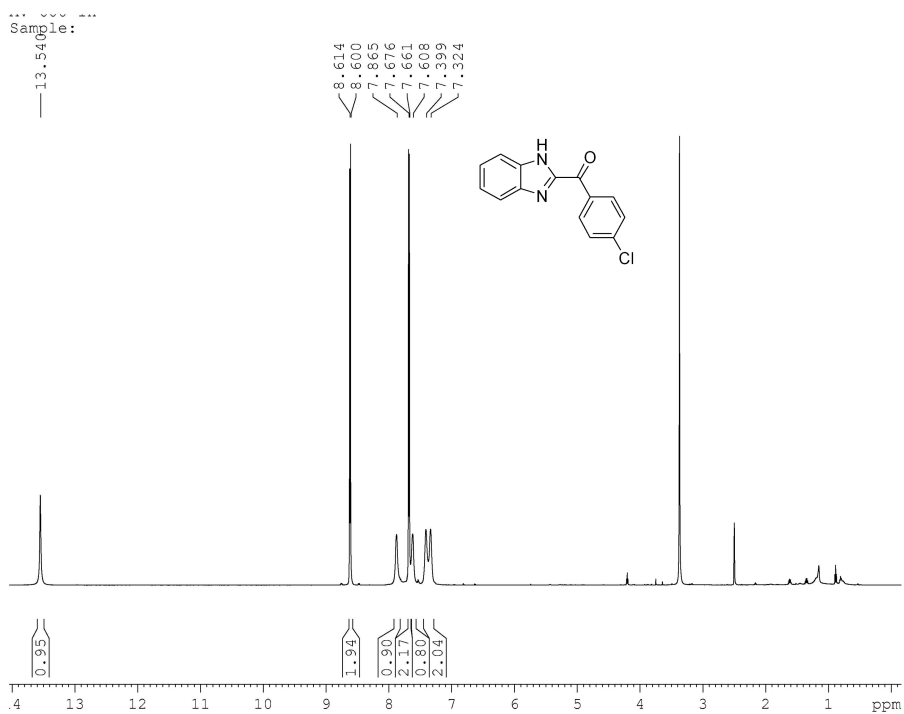
M. p. 112-114°C; $^1\text{H-NMR}$ (300MHz, CDCl_3): δ =8.50 (d, $J=8.63$ Hz, 2H), 7.90 (s, 1H), 7.61 (d, $J=8.71$, 1H), 7.51(m, 3H); $^{13}\text{C-NMR}$ (CDCl_3): δ =178.7, 157.8, 148.9, 141.6, 141.4, 133.0, 132.5, 131.5, 129.1, 129.0, 122.1, 112.7; MS: m/z (%): 292.1 ($[\text{M}+\text{H}]^+$), 313.9 ($[\text{M}+\text{Na}]^+$).

2-(3,4,5-Trimethoxy)benzoyl-5-chlorobenzoxazole (Table 4, entry 9)

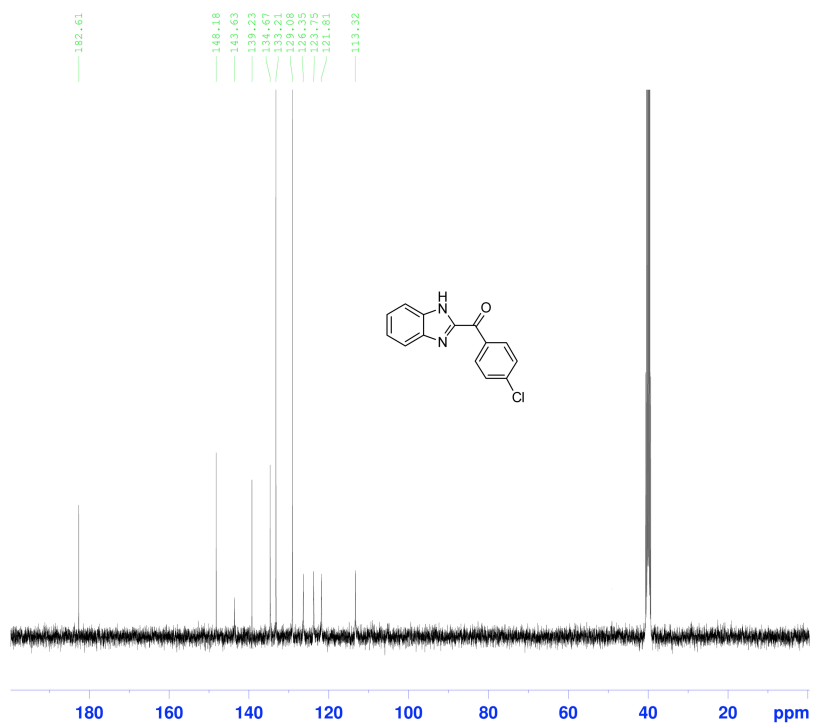


M. p. 107-108°C; $^1\text{H-NMR}$ (300MHz, CDCl_3): δ =7.94 (m, 3H), 7.66 (d, $J=8.88$ Hz, 1H), 7.53 (dd, $J=8.88$ Hz, $J=1.95$ Hz, 1H), 4.00 (m, 9H); $^{13}\text{C-NMR}$ (CDCl_3): δ =178.5, 158.2, 153.0, 148.9, 144.3, 141.7, 131.3, 129.5, 128.8, 122.1, 112.7, 108.8, 61.1, 56.4; MS: m/z (%): 348.0 ($[\text{M}+\text{H}]^+$), 370.0 ($[\text{M}+\text{Na}]^+$).

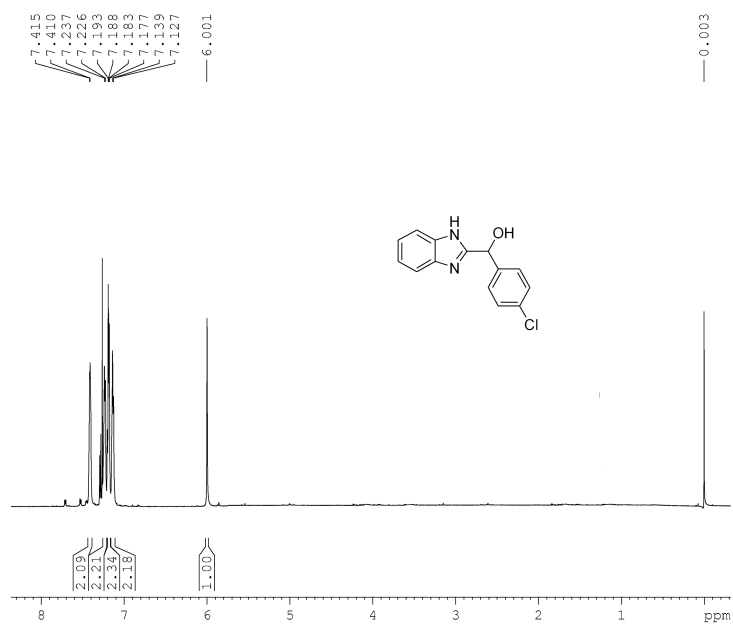
2-(4-Chloro)benzoylbenzimidazole (6)



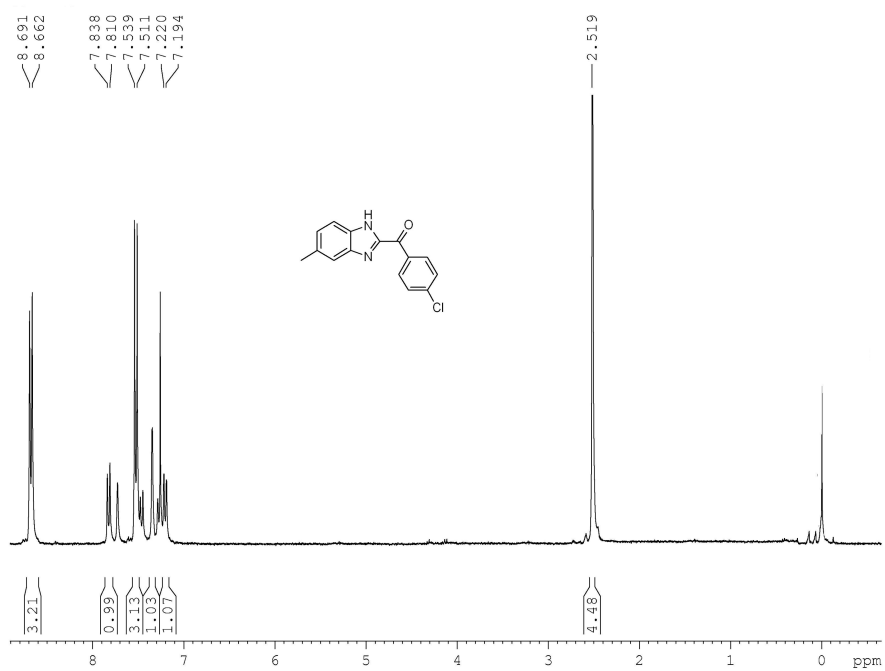
2-(4-Chloro)benzoylbenzimidazole (6)



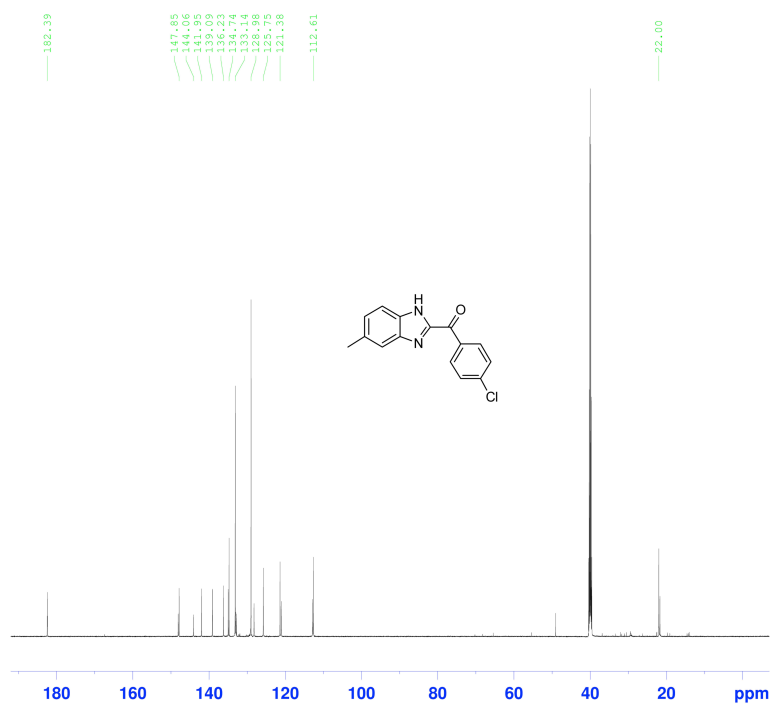
2-(4-Chlorohydroxybenzyl)-benzo[d]imidazole(5)



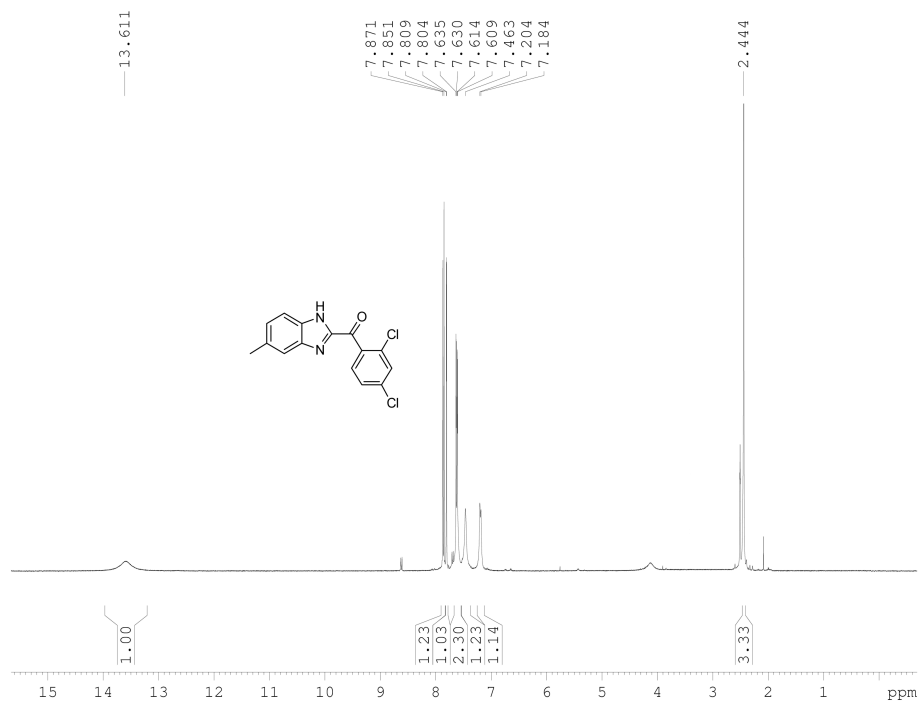
2-(4-Chloro)benzoyl-5-methylbenzimidazole



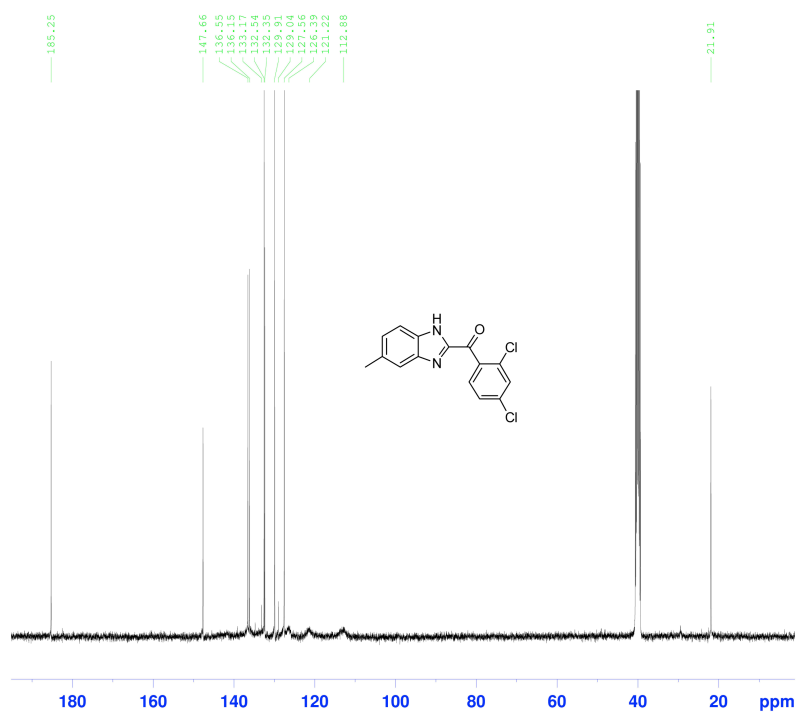
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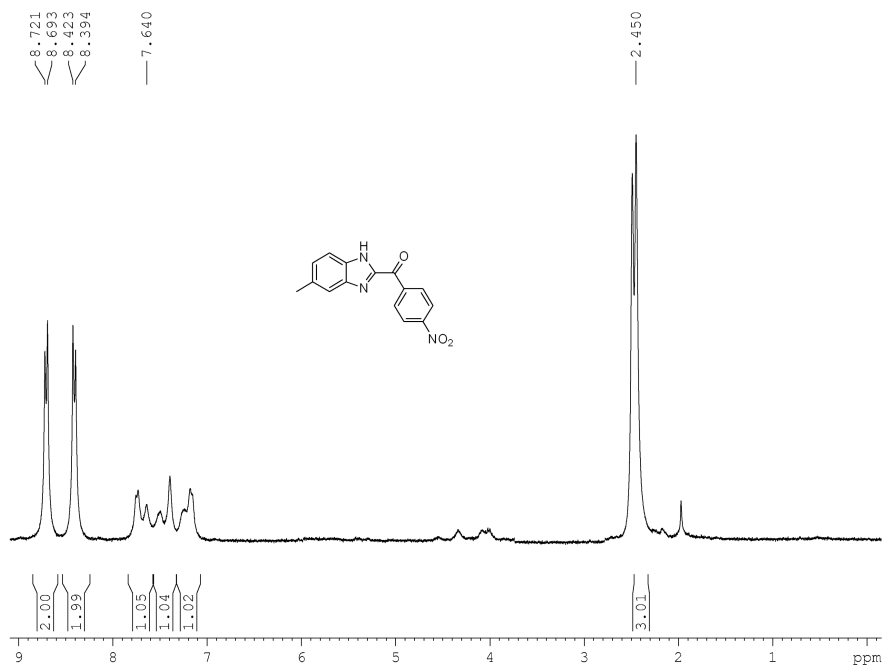
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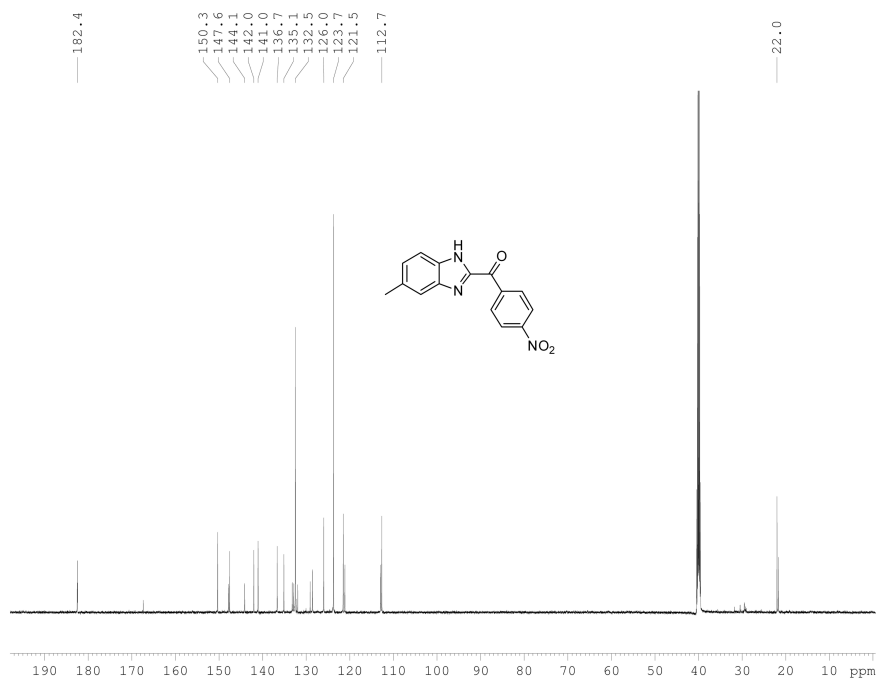
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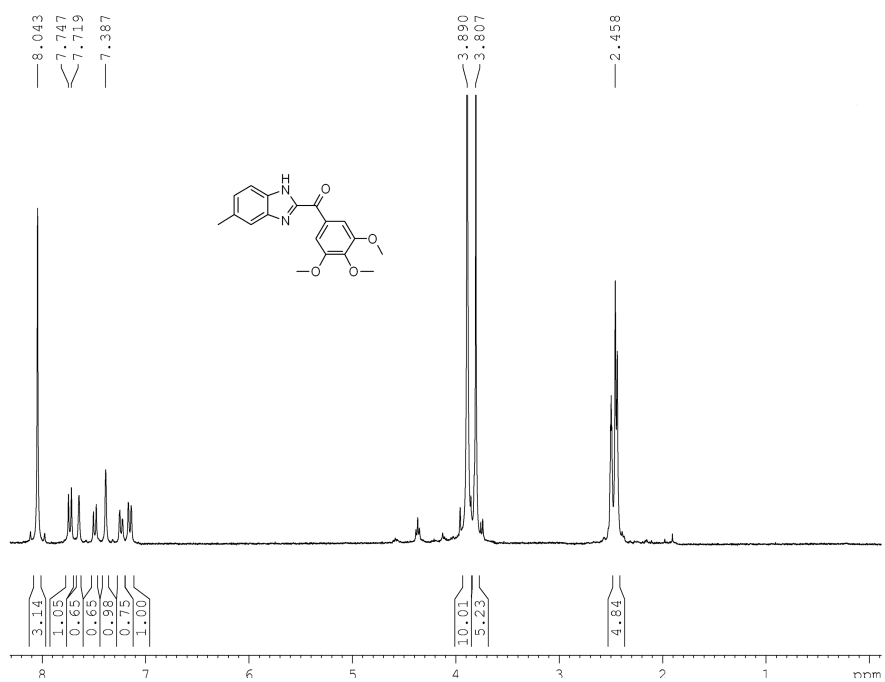
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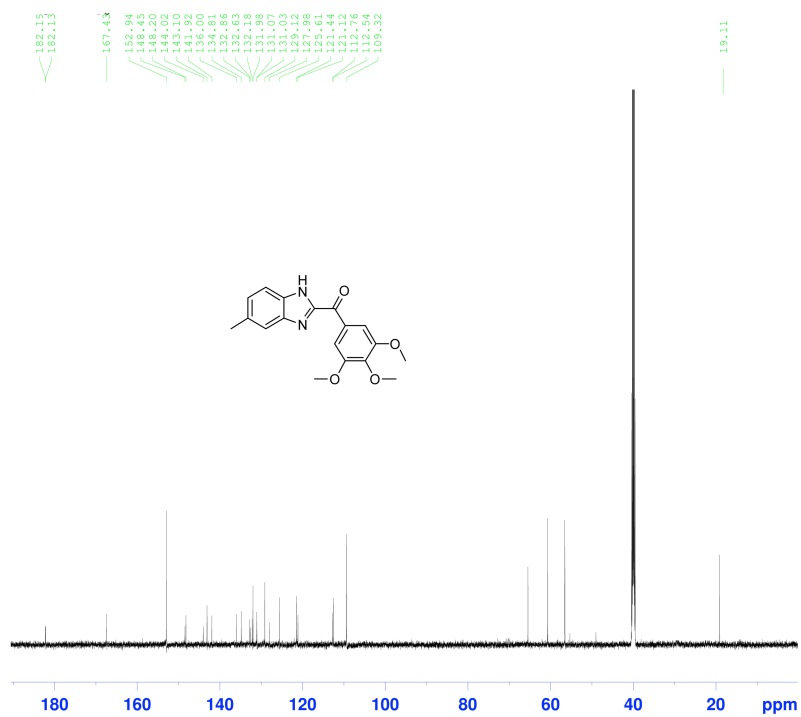
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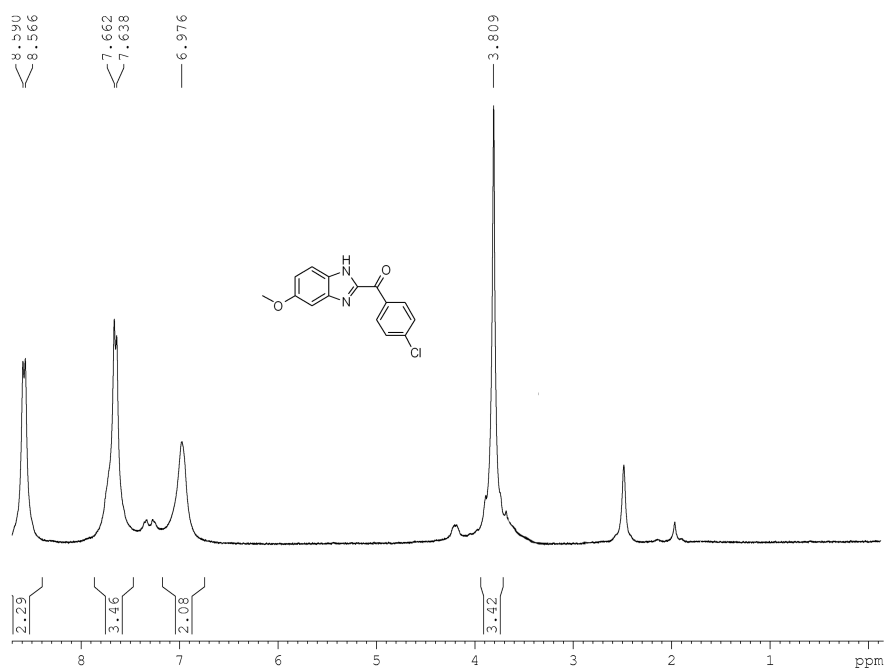
2-(3,4,5-Trimethoxybenzoyl)-5-methylbenzimidazole



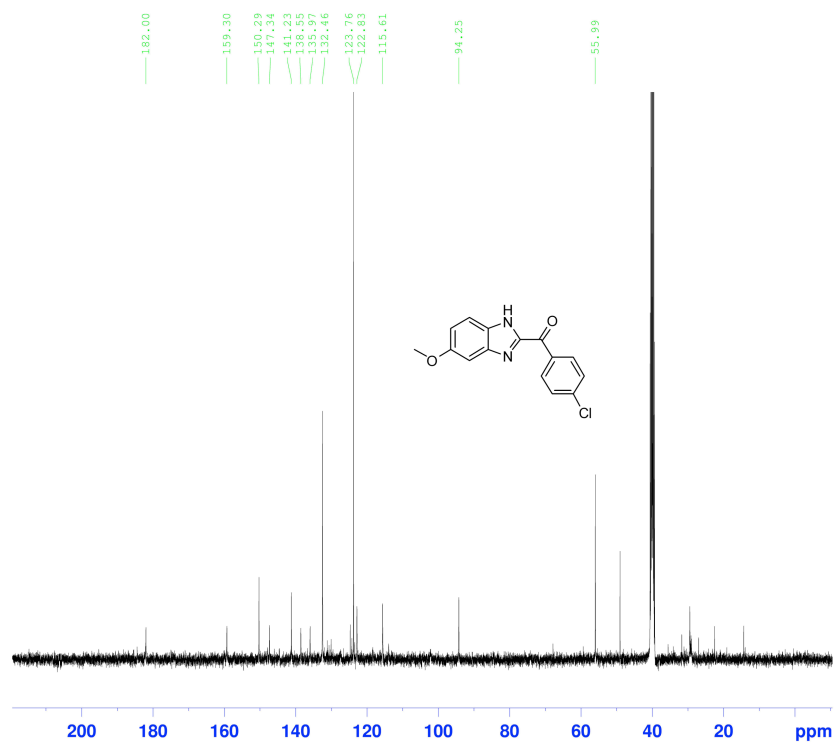
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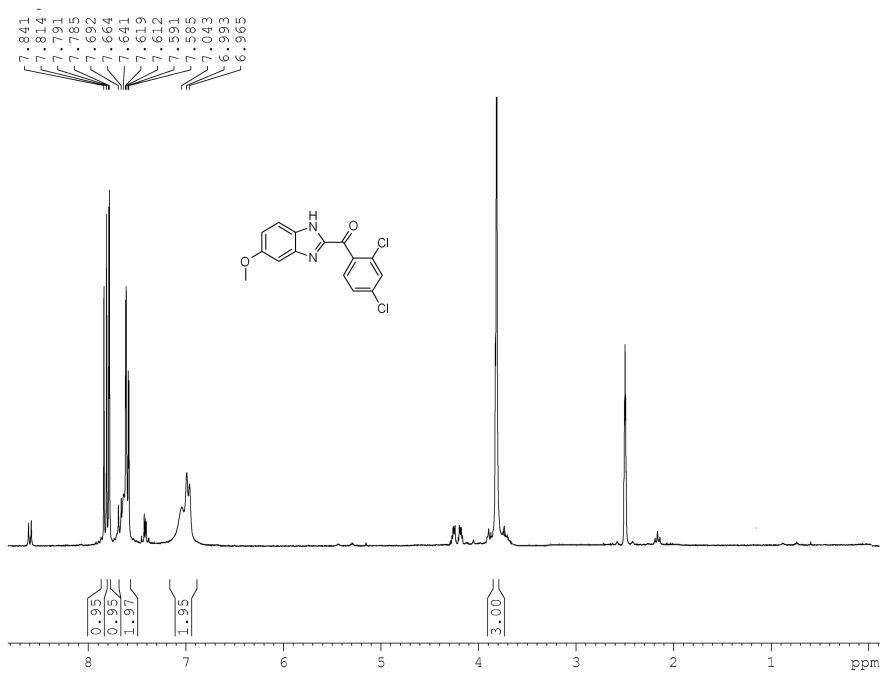
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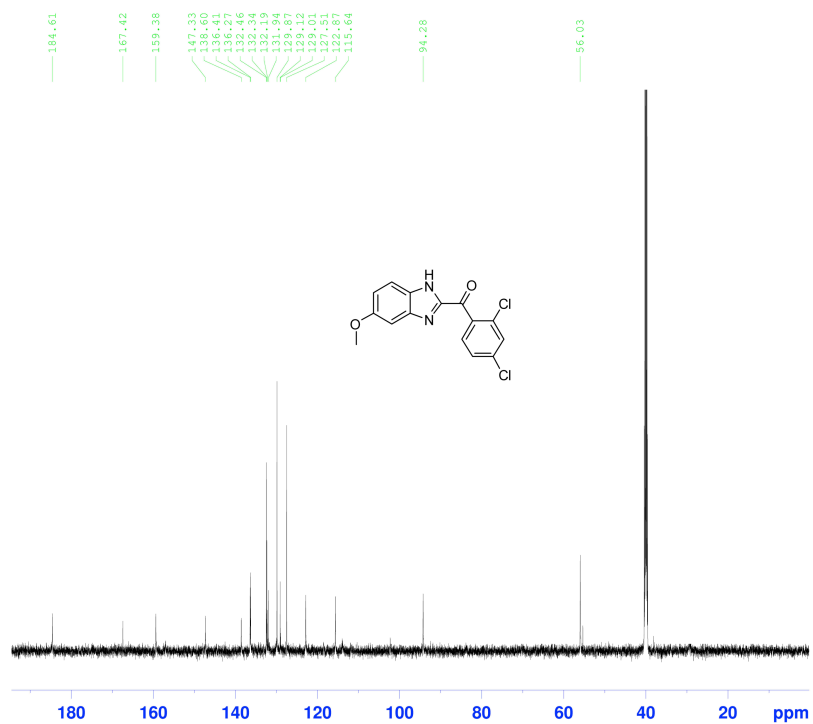
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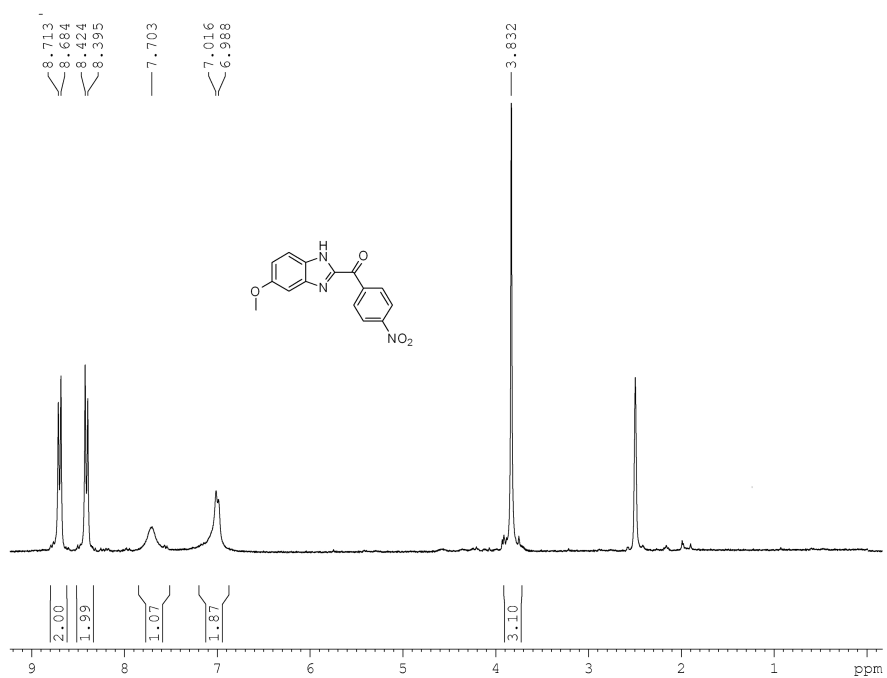
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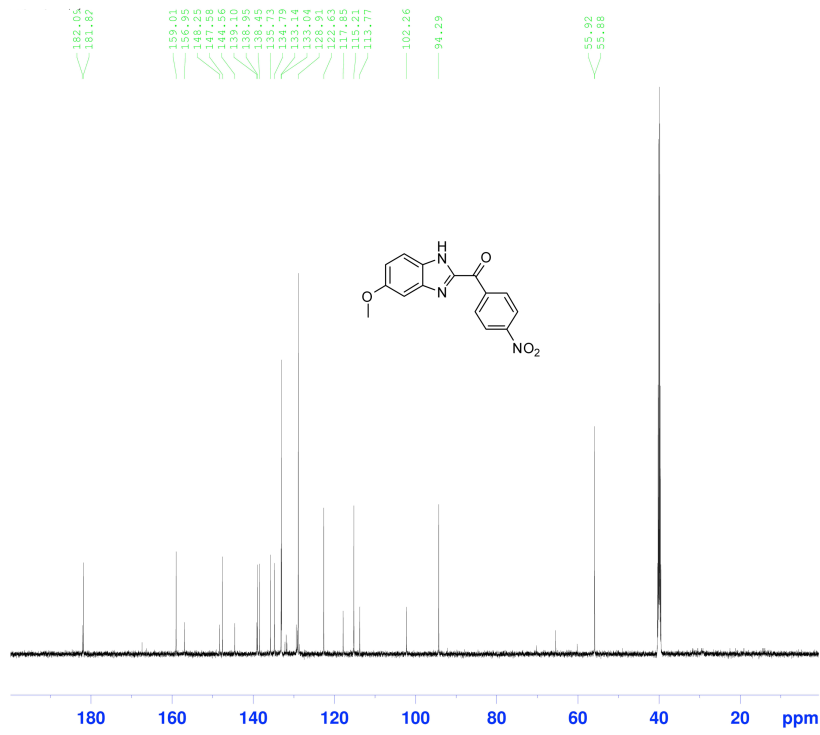
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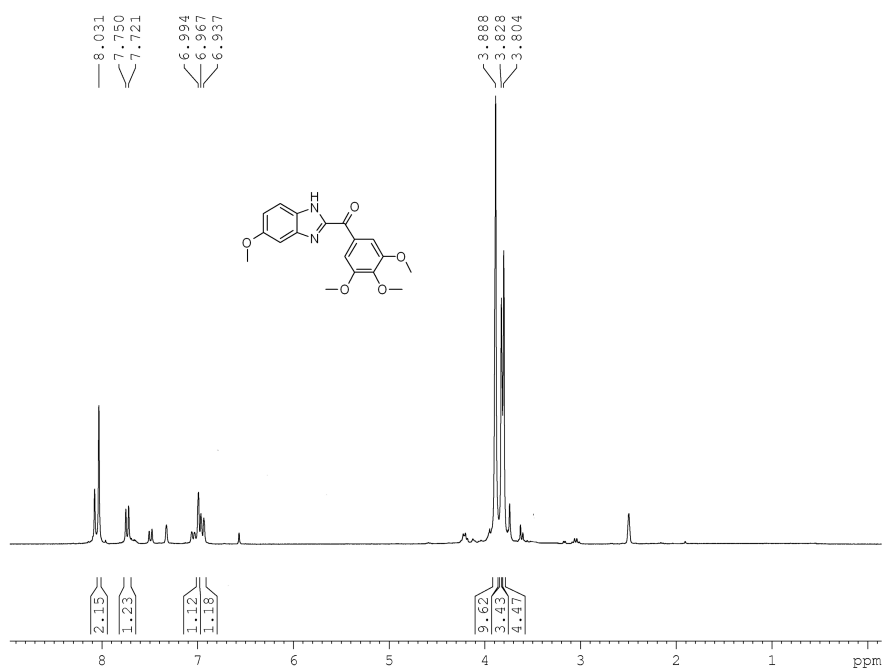
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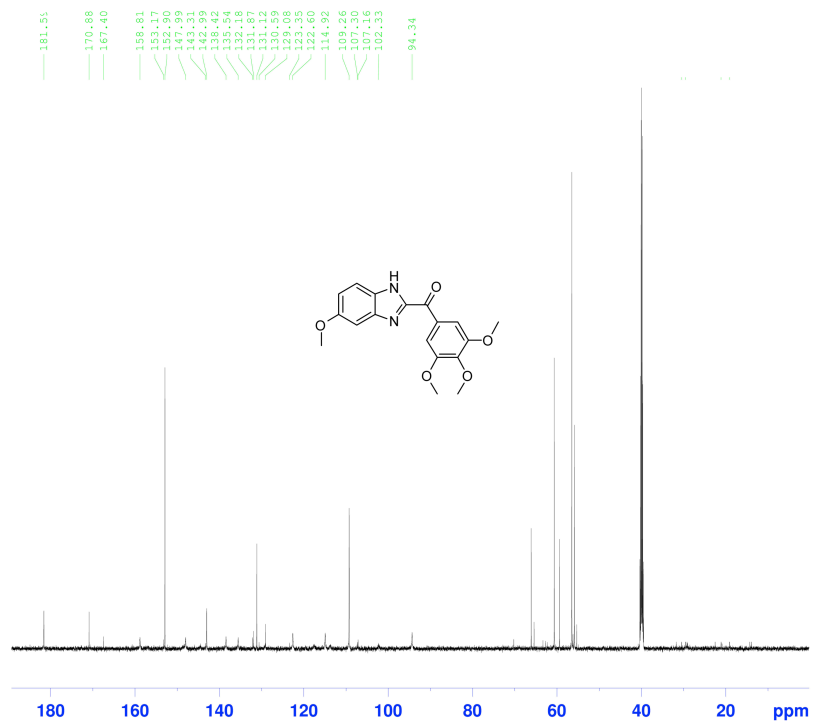
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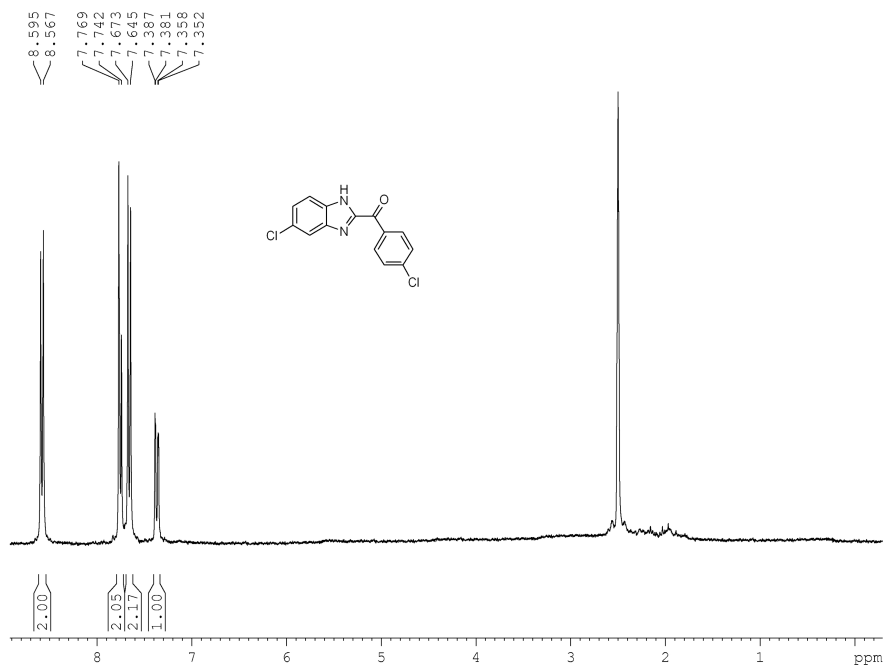
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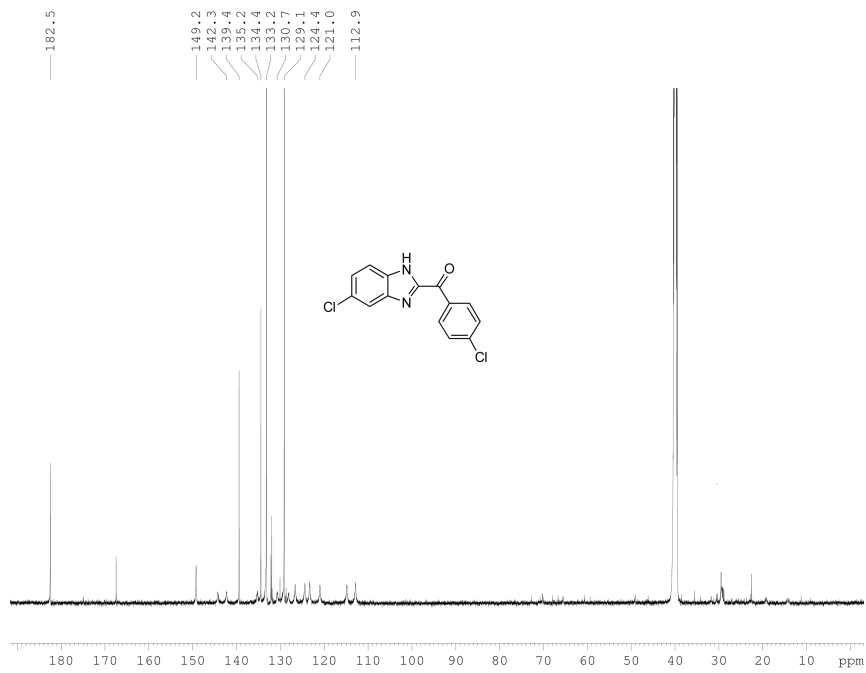
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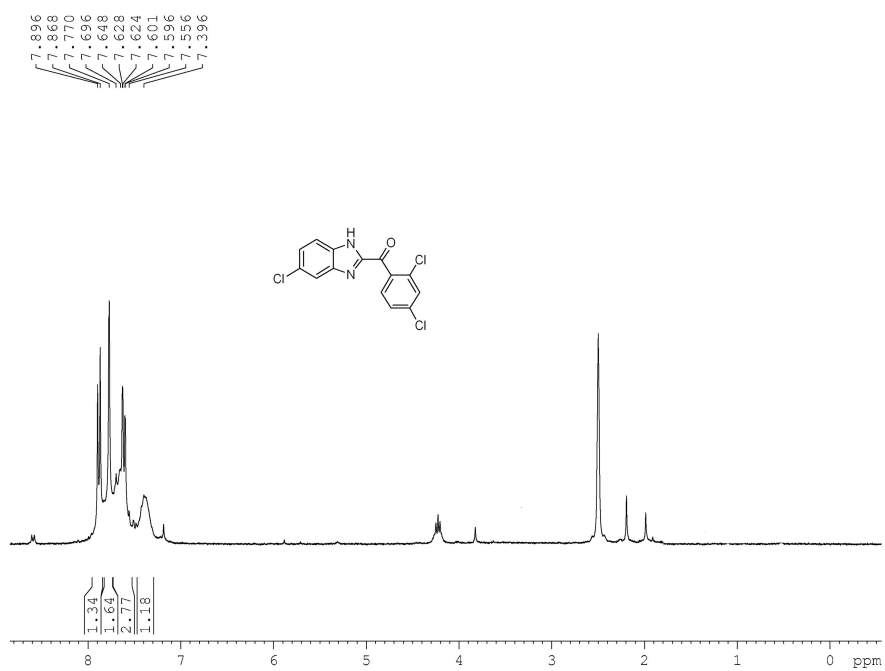
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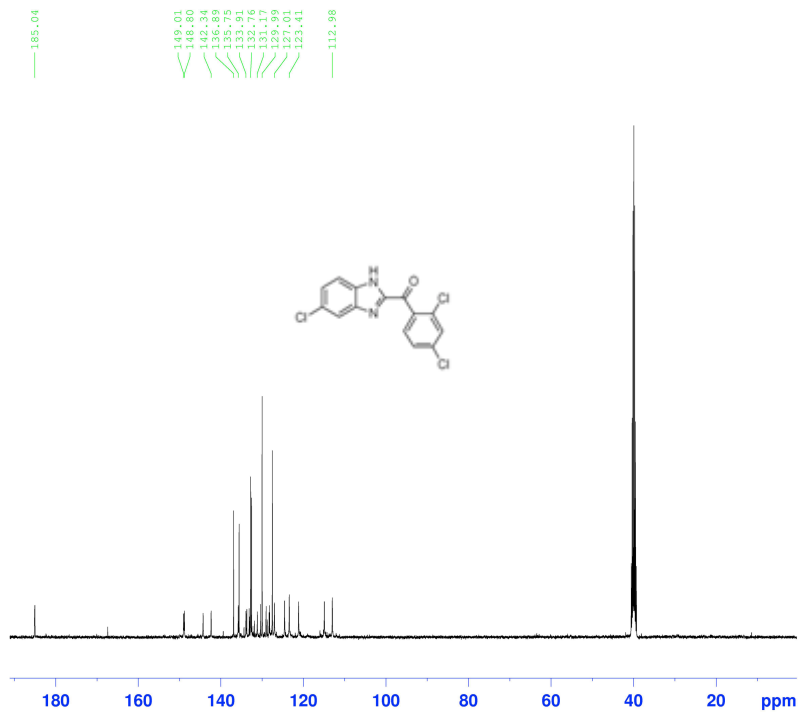
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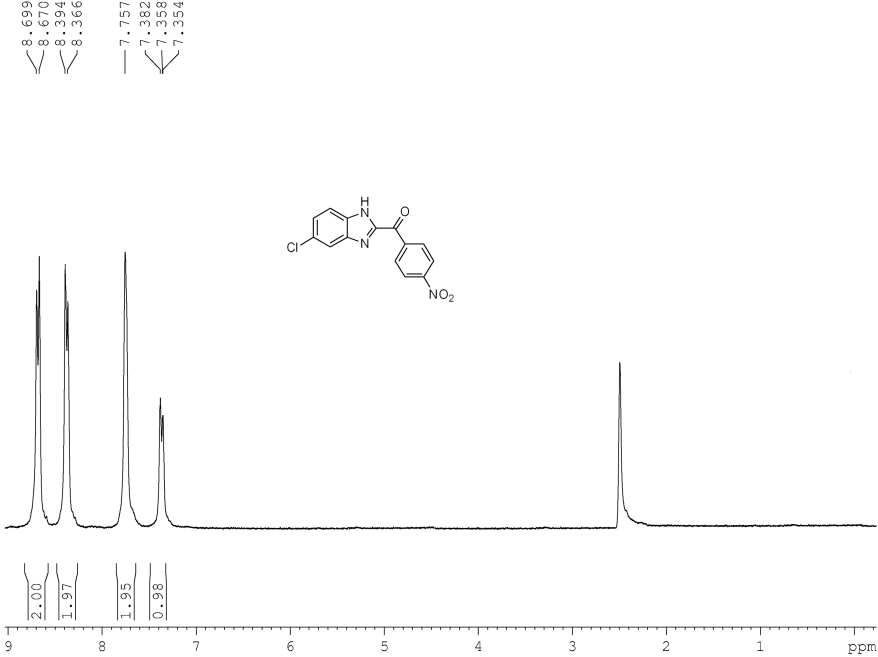
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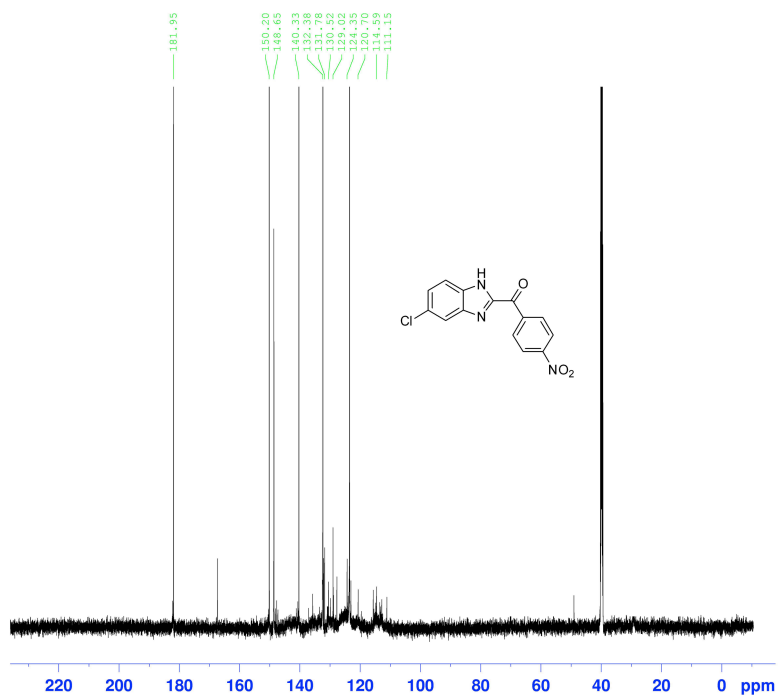
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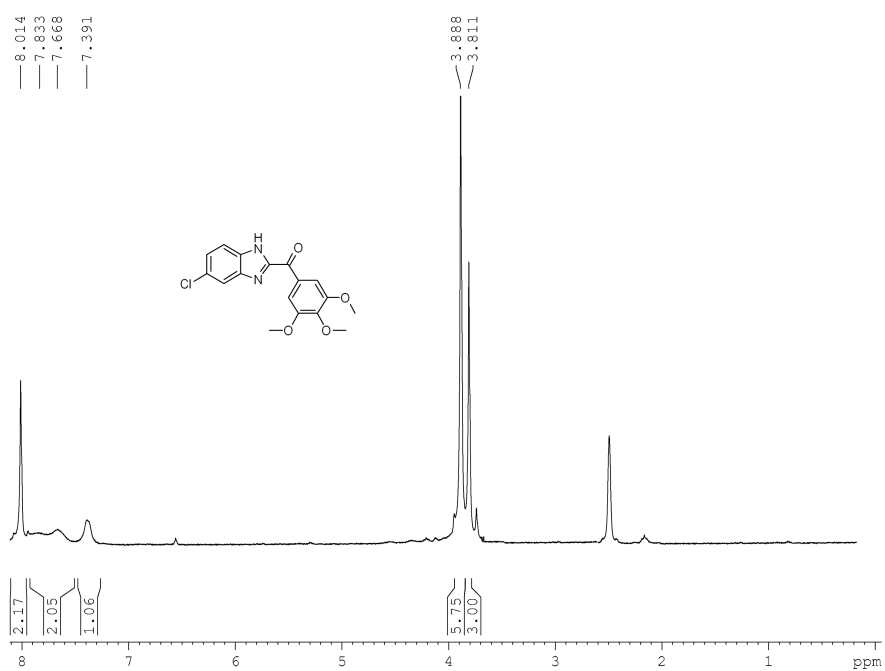
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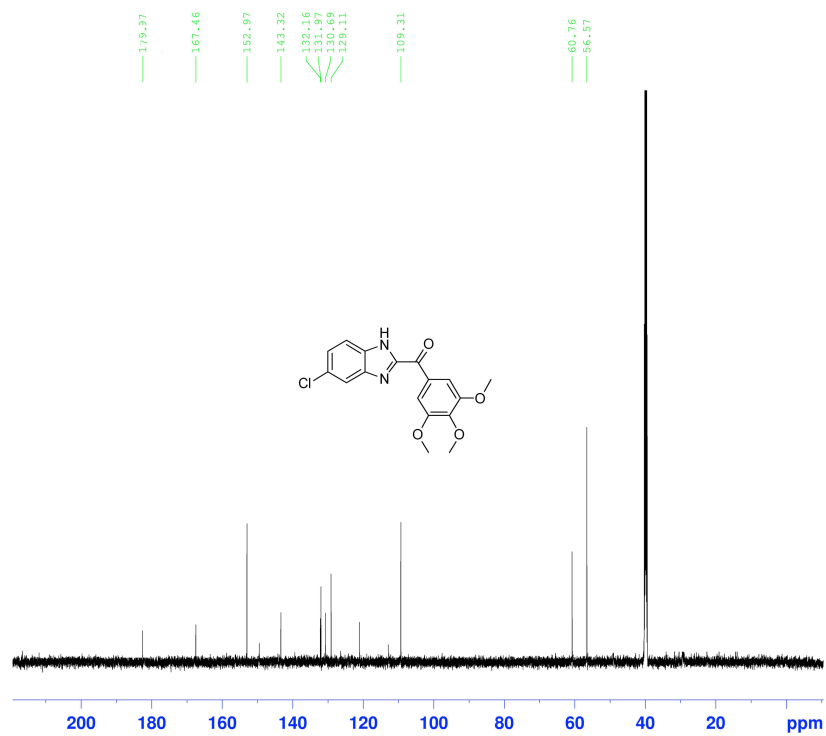
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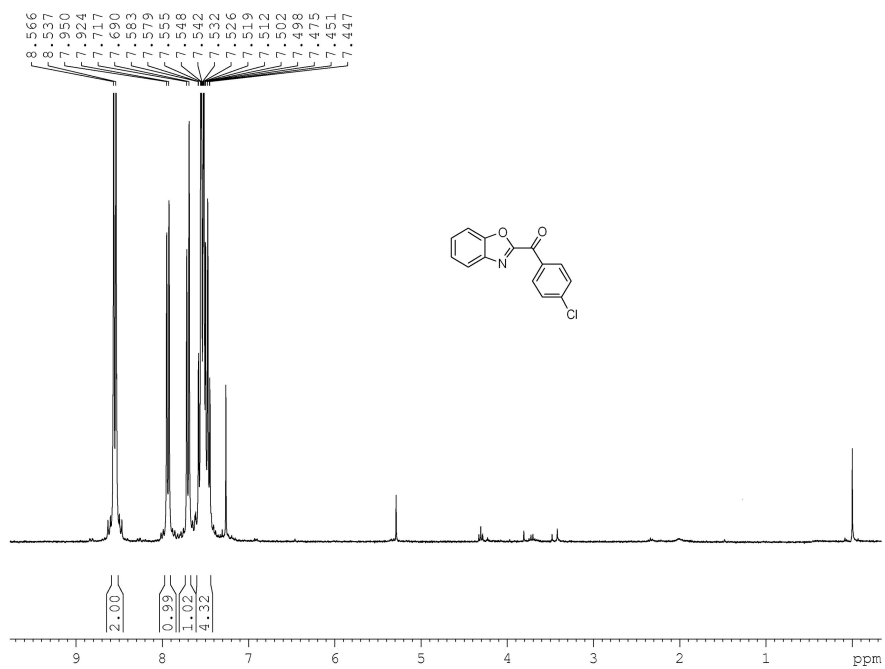
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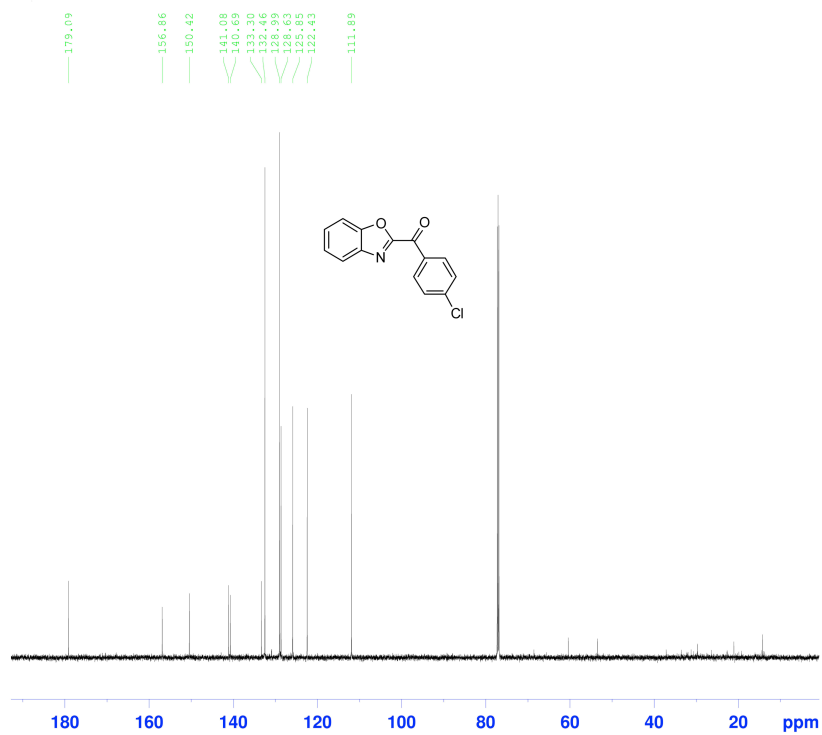
2-(3,4,5-Trimethoxybenzoyl)-5-chlorobenzimidazole



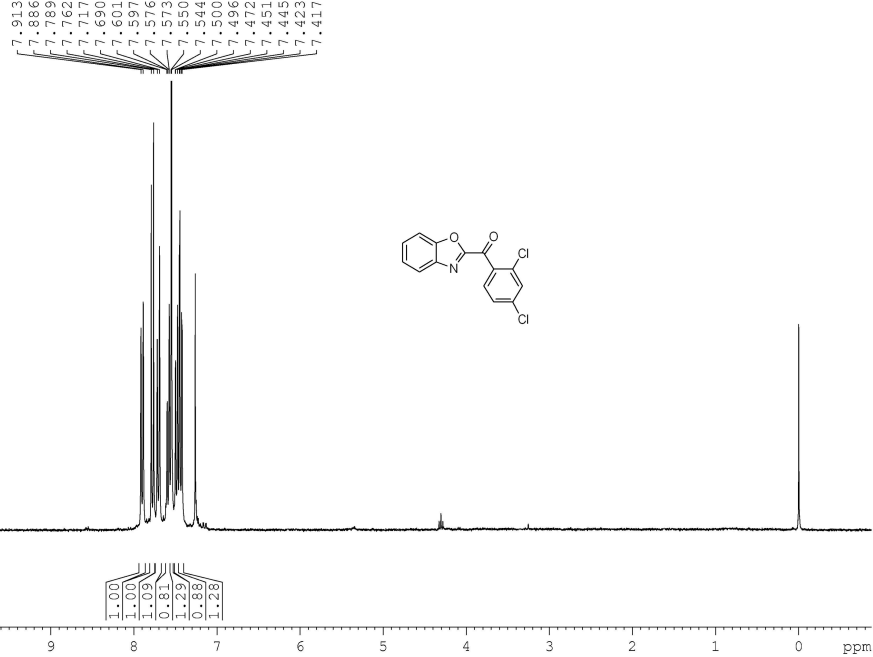
2-(4-Chloro)benzoylbenzoxazole



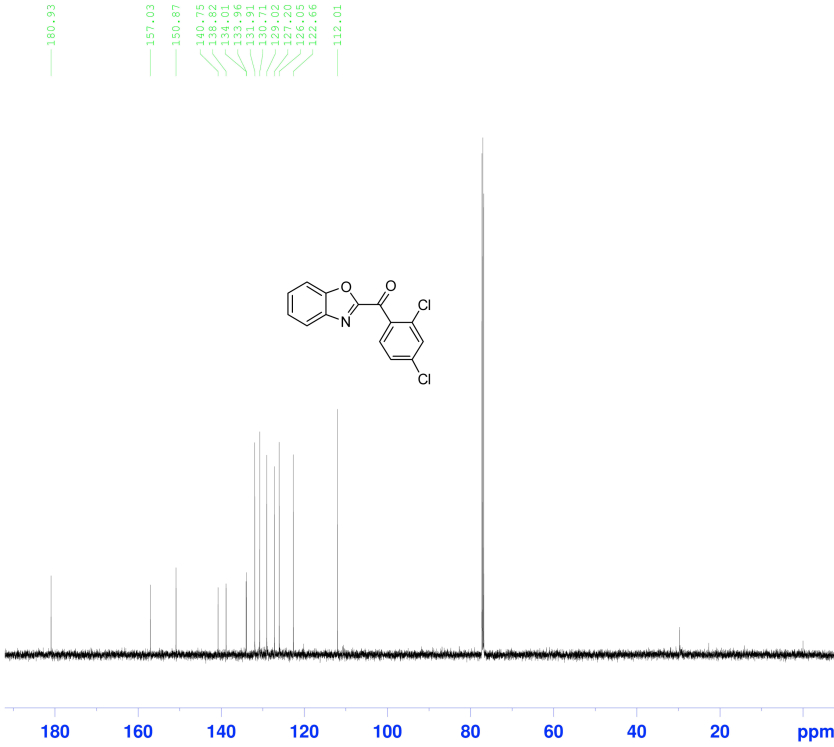
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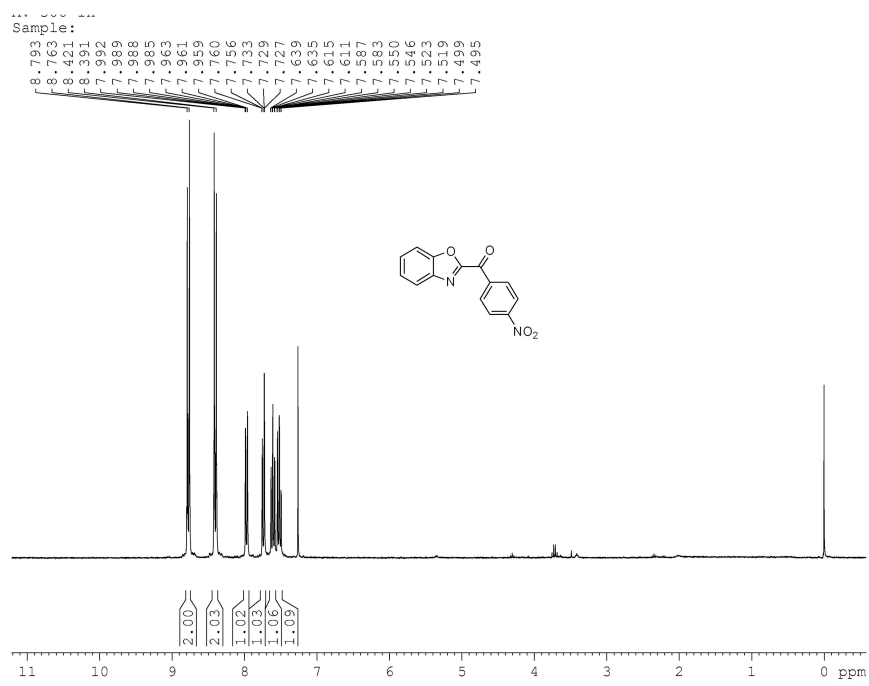
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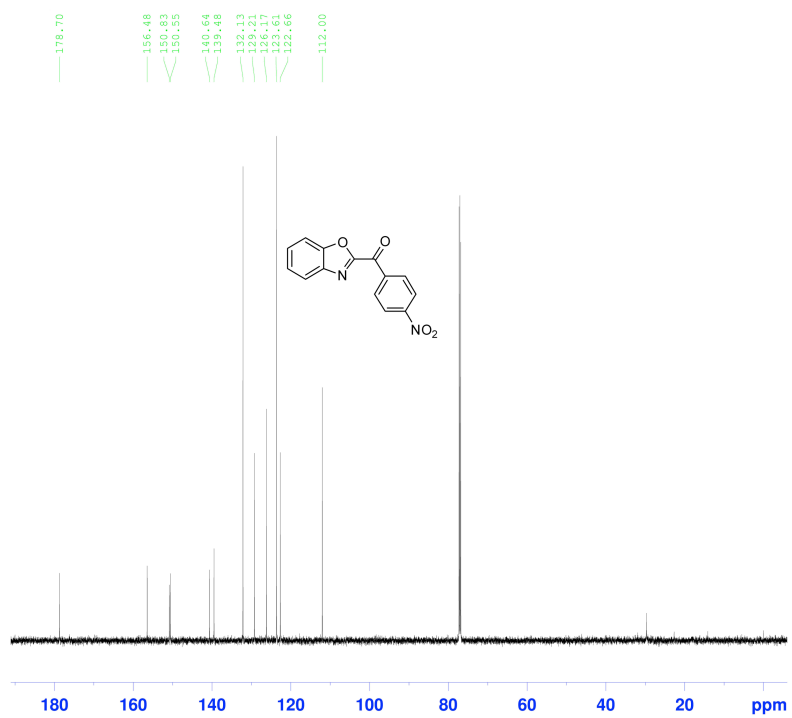
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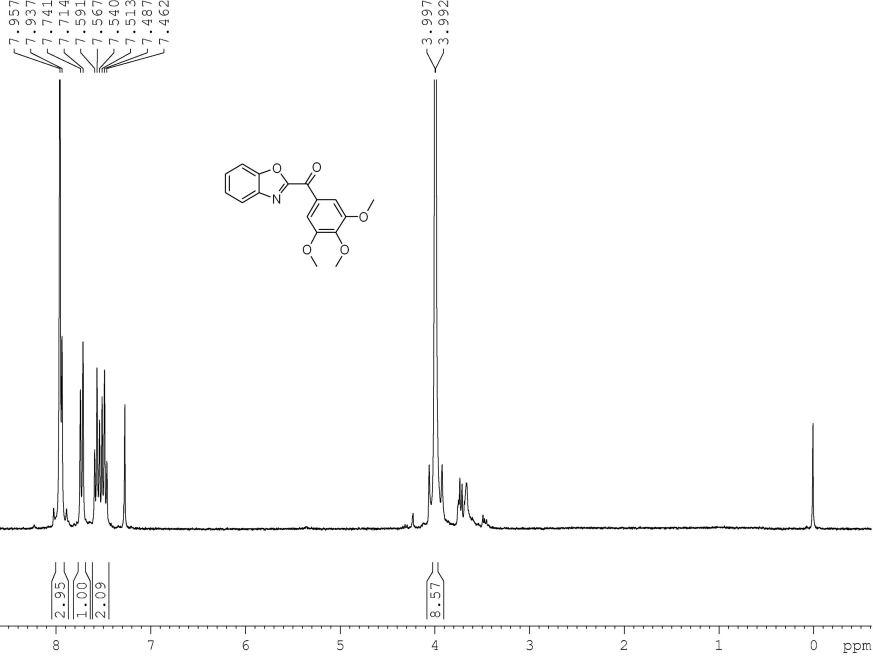
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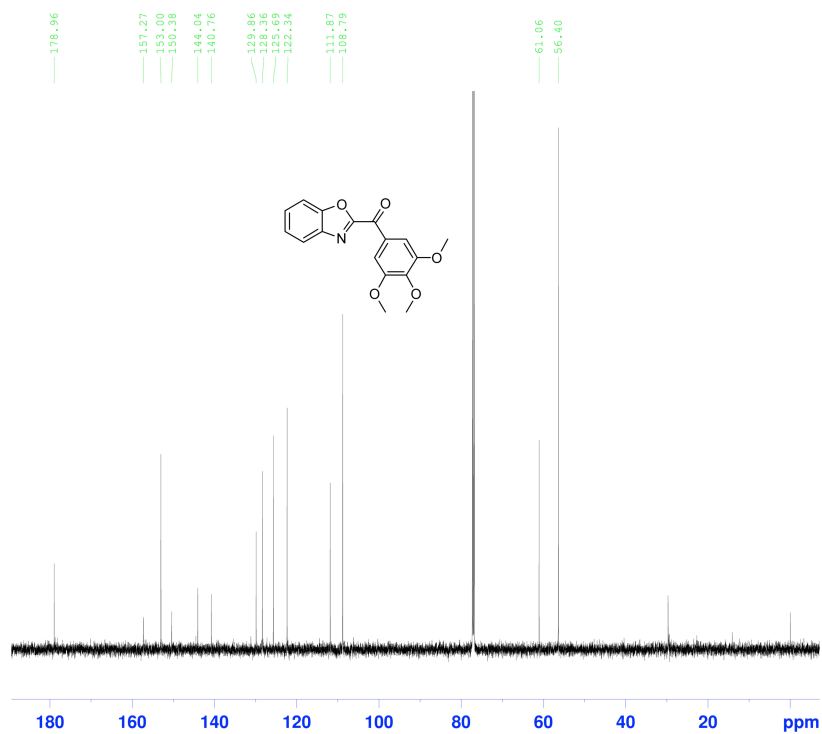
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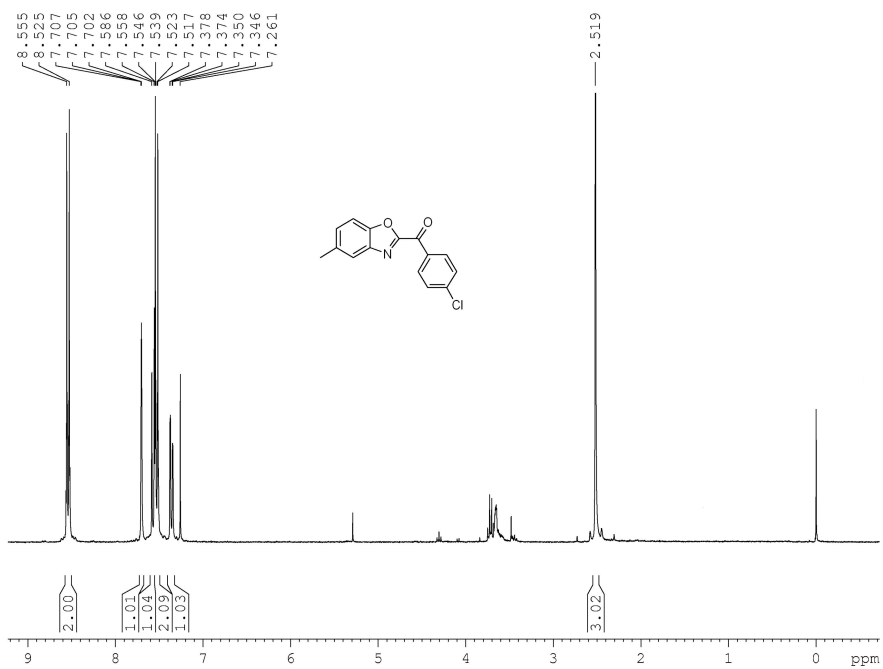
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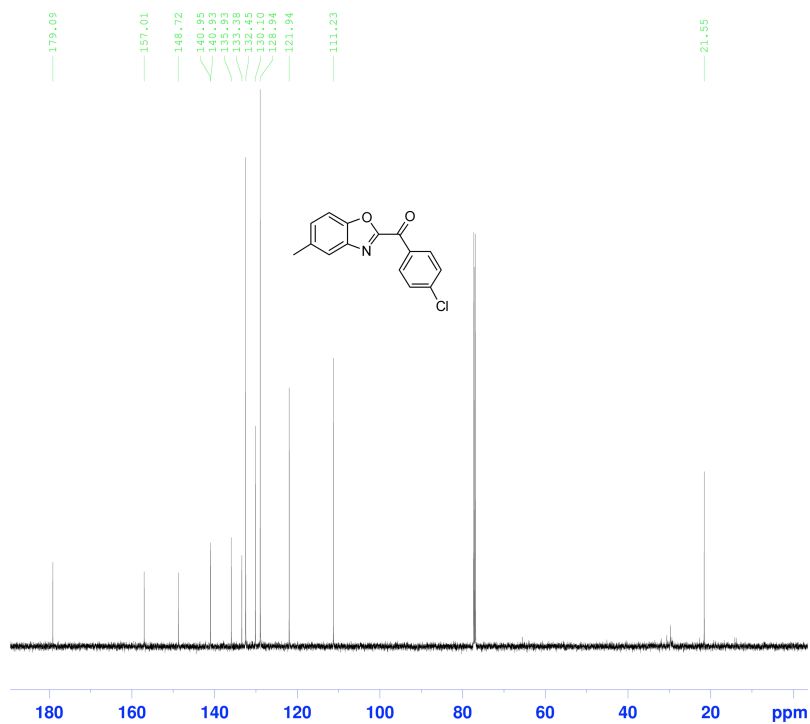
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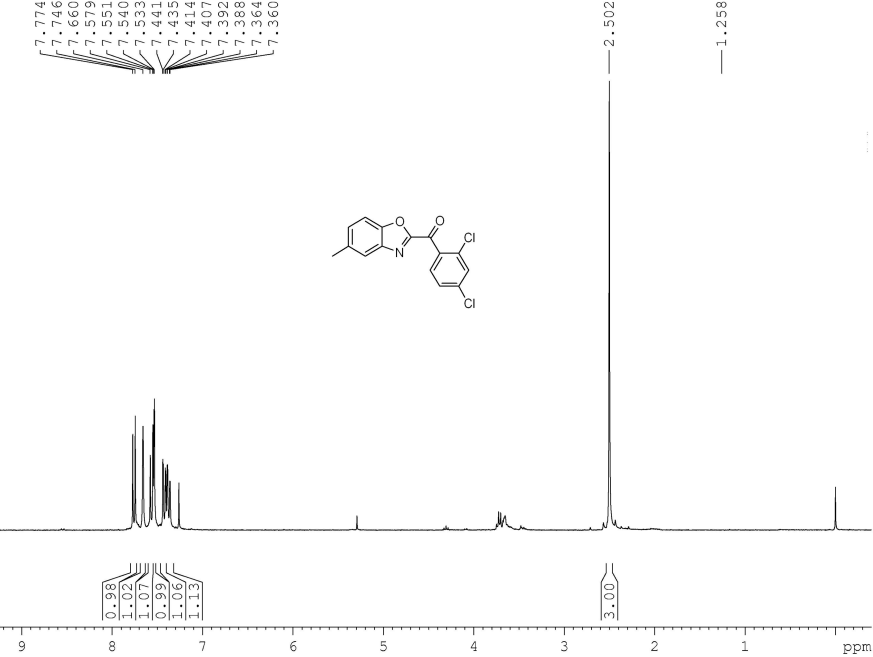
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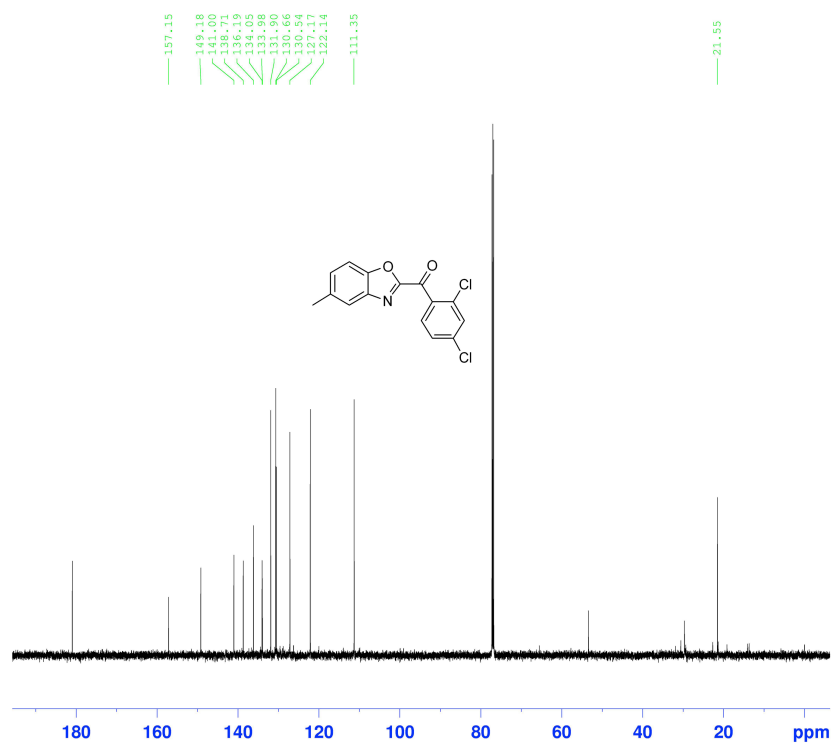
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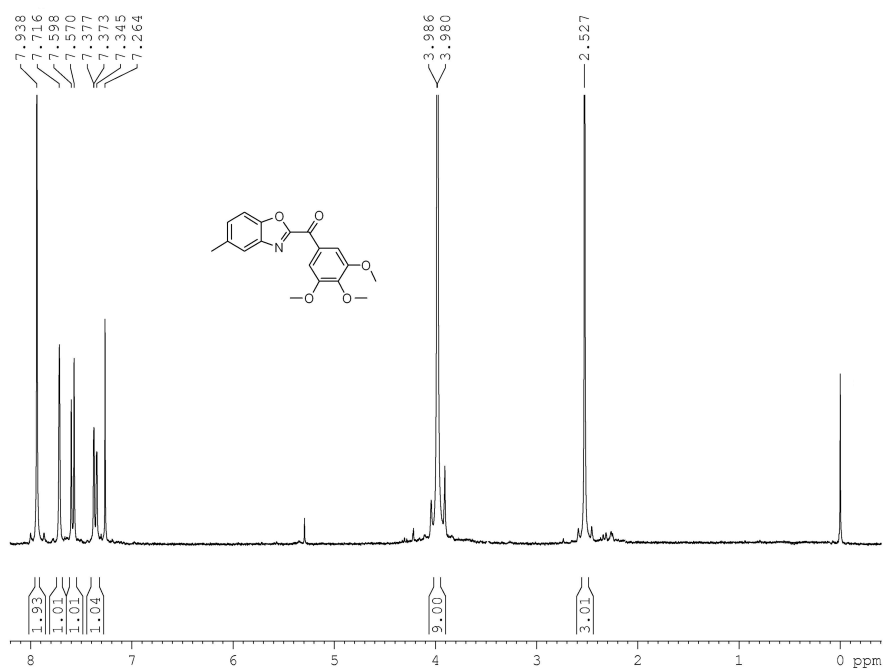
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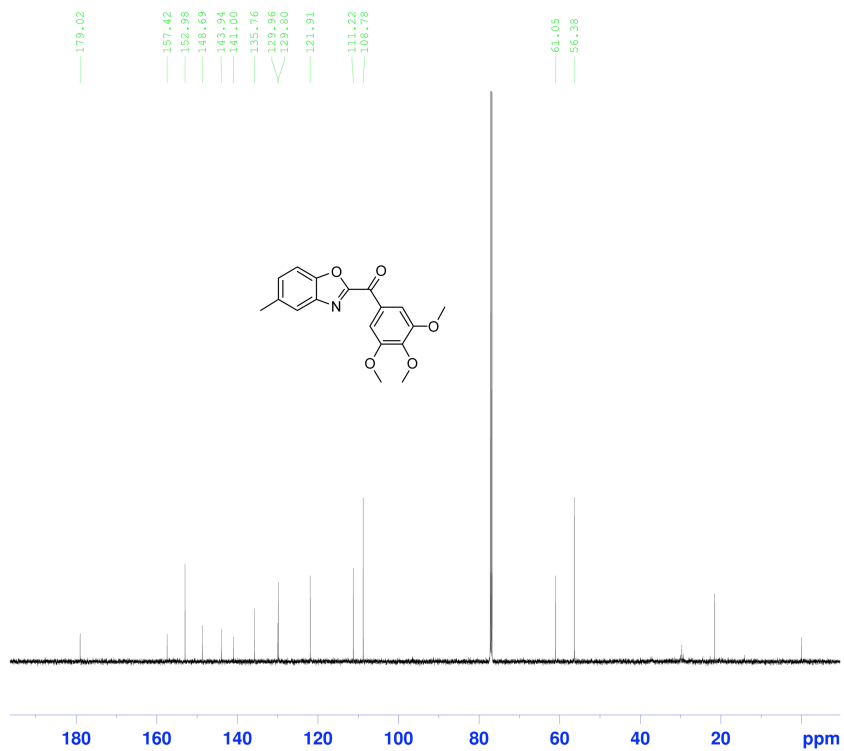
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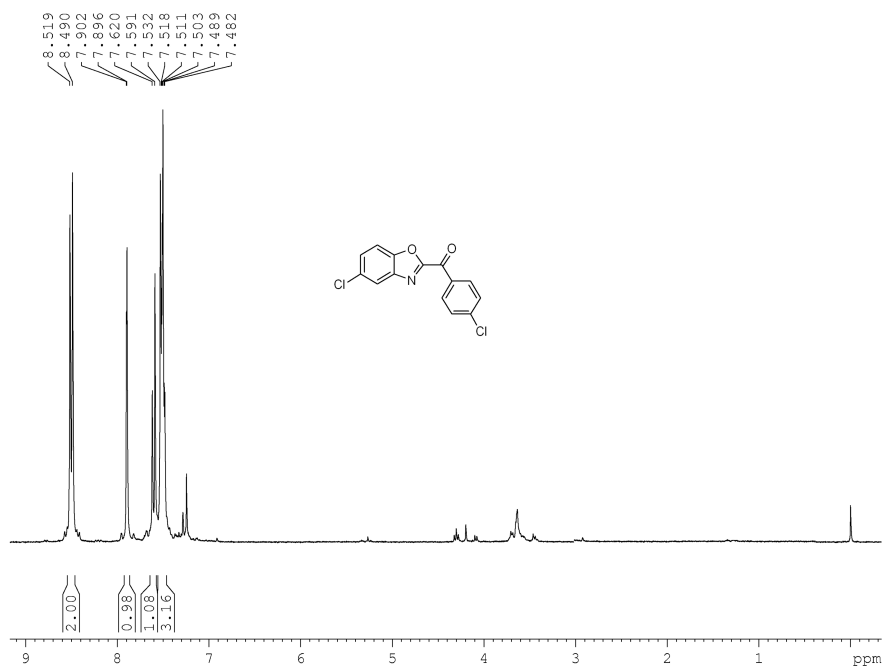
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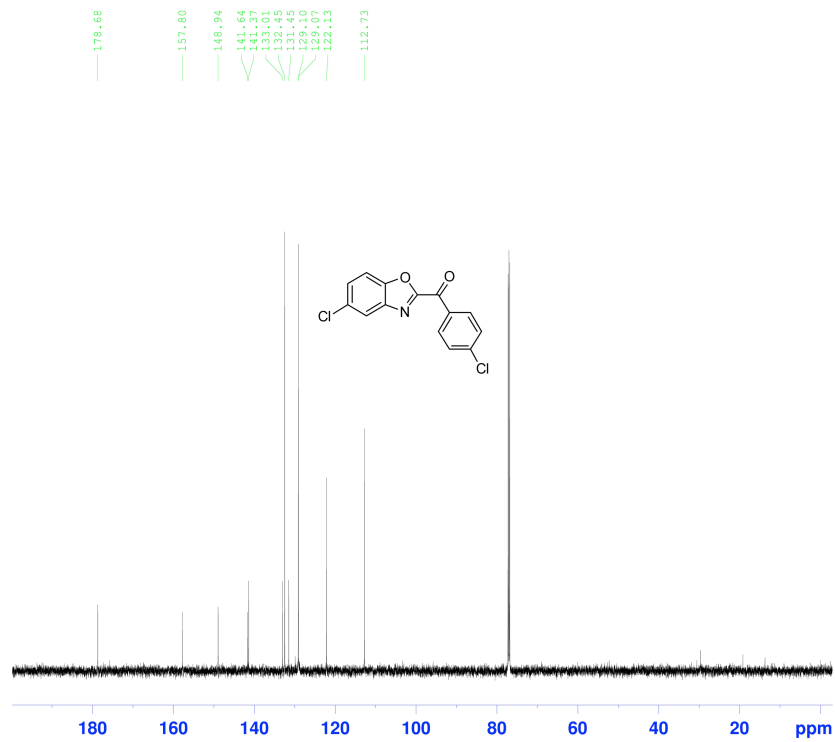
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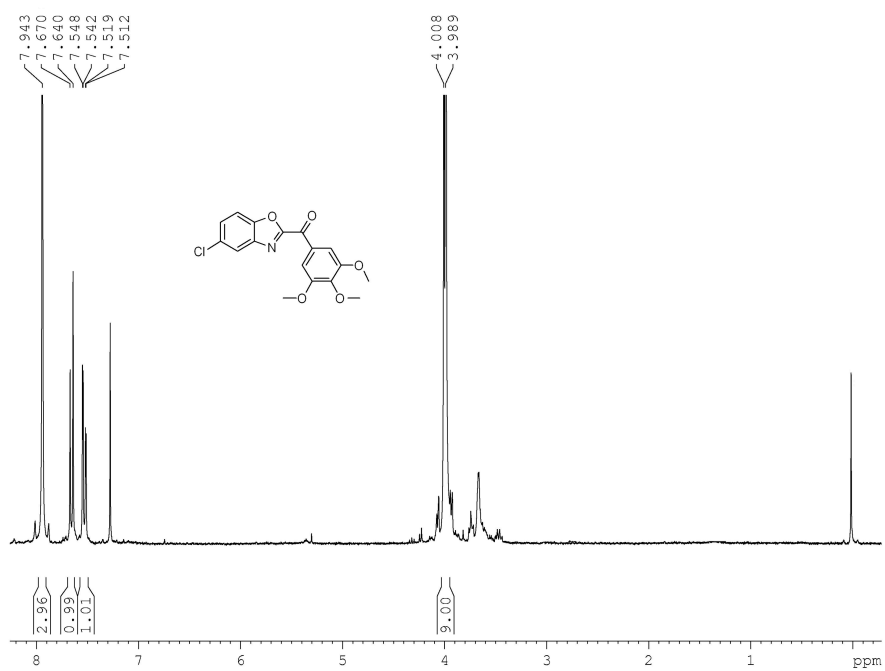
2-(4-Chloro)benzoyl-5-chlorobenzoxazole



2-(4-Chloro)benzoyl-5-chlorobenzoxazole



2-(3,4,5-Trimethoxy)benzoyl-5-chlorobenzoxazole



2-(3,4,5-Trimethoxy)benzoyl-5-chlorobenzoxazole

