

Supplementary Materials

Monocarbonyl Analogues of Curcumin Based on Pseudopelletierine Scaffold: Synthesis and Anti-inflammatory Activity

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Figure S1. ^1H NMR: 9-Methyl-9-azabicyclo[3.3.1]nonan-3-one (1)

Figure S2. FTIR (ATR): 9-methyl-9-azabicyclo[3.3.1]nonan-3-one (1)

Figure S3. ^1H NMR: 4-(2-(Dimethylamino)ethoxy)-3-methoxybenzaldehyde (2)

Figure S4. ^{13}C NMR: 4-(2-(Dimethylamino)ethoxy)-3-methoxybenzaldehyde (2)

Figure S5. FTIR (ATR): 4-(2-(Dimethylamino)ethoxy)-3-methoxybenzaldehyde (2)

Figure S6. ^1H NMR: 2,4-Bis((*E*)-4-fluorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (3)

Figure S7. ^{13}C NMR: 2,4-Bis((*E*)-4-fluorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (3)

Figure S8. ^{19}F NMR: 2,4-Bis((*E*)-4-fluorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (3)

Figure S9. FTIR (ATR): 2,4-Bis((*E*)-4-fluorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (3)

Figure S10. ^1H NMR: 2,4-Bis((*E*)-4-chlorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (4)

Figure S11. ^{13}C NMR: 2,4-Bis((*E*)-4-chlorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (4)

Figure S12. FTIR (ATR): 2,4-Bis((*E*)-4-chlorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (4)

Figure S13. ^1H NMR: 2,4-Bis((*E*)-4-bromobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (5)

Figure S14. ^{13}C NMR: 2,4-Bis((*E*)-4-bromobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (5)

Figure S15. FTIR (ATR): 2,4-Bis((*E*)-4-bromobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (5)

Figure S16. ^1H NMR: 9-Methyl-2,4-bis((*E*)-4-(trifluoromethyl)benzylidene)-9-azabicyclo[3.3.1]nonan-3-one (6)

Figure S17. ^{13}C NMR: 9-Methyl-2,4-bis((*E*)-4-(trifluoromethyl)benzylidene)-9-azabicyclo[3.3.1]nonan-3-one (6)

Figure S18. FTIR (ATR): 9-Methyl-2,4-bis((*E*)-4-(trifluoromethyl)benzylidene)-9-azabicyclo[3.3.1]nonan-3-one (6)

Figure S19. ^1H NMR: 9-Methyl-2,4-bis((*E*)-4-nitrobenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (7)

Figure S20. ^{13}C NMR: 9-Methyl-2,4-bis((*E*)-4-nitrobenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (7)

Figure S21. FTIR (ATR): 9-Methyl-2,4-bis((*E*)-4-nitrobenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (7)

Figure S22. ^1H NMR: 4-Bis((*E*)-4-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (8)

Figure S23. ^{13}C NMR: 4-Bis((*E*)-4-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (8)

Figure S24. FTIR (ATR): 4-Bis((*E*)-4-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**8**)

Figure S25. ¹H NMR: 4-Bis((*E*)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**9**)

Figure S26. ¹³C NMR: 4-Bis((*E*)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**9**)

Figure S27. FTIR (ATR): 4-Bis((*E*)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**9**)

Figure S28. ¹H NMR: 2,4-Bis((*E*)-3,4-dimethoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**10**)

Figure S29. ¹³C NMR: 2,4-bis((*E*)-3,4-dimethoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**10**)

Figure S30. FTIR (ATR): 2,4-bis((*E*)-3,4-dimethoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**10**)

Figure S31. ¹H NMR: 9-Methyl-2,4-bis((*E*)-3,4,5-trimethoxybenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (**11**)

Figure S32. ¹³C NMR: 9-Methyl-2,4-bis((*E*)-3,4,5-trimethoxybenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (**11**)

Figure S33. FTIR (ATR): 9-Methyl-2,4-bis((*E*)-3,4,5-trimethoxybenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (**11**)

Figure S34. ¹H NMR: 2,4-Bis((*E*)-4-(2-(dimethylamino)ethoxy)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**12**)

Figure S35. ¹³C NMR: 2,4-Bis((*E*)-4-(2-(dimethylamino)ethoxy)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**12**)

Figure S36. FTIR (ATR): 2,4-Bis((*E*)-4-(2-(dimethylamino)ethoxy)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**12**)

Figure S37. ¹H NMR: 2,4-Bis((*E*)-4-hydroxy-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**13**)

Figure S38. ¹³C NMR: 2,4-Bis((*E*)-4-hydroxy-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**13**)

Figure S39. FTIR (ATR): 2,4-Bis((*E*)-4-hydroxy-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**13**)

Figure S40. ¹H NMR (200 MHz, DMSO): CUR.

Figure S41. Analysis of the purity of CUR by HPLC.

Table S1. Crystal data and structure refinement details for **3**, **4**, **5**, **6**, **7**, **8**, **10** and **11** compounds.

Table S2. Solubility (S) in distilled water at 21 ± 1°C.

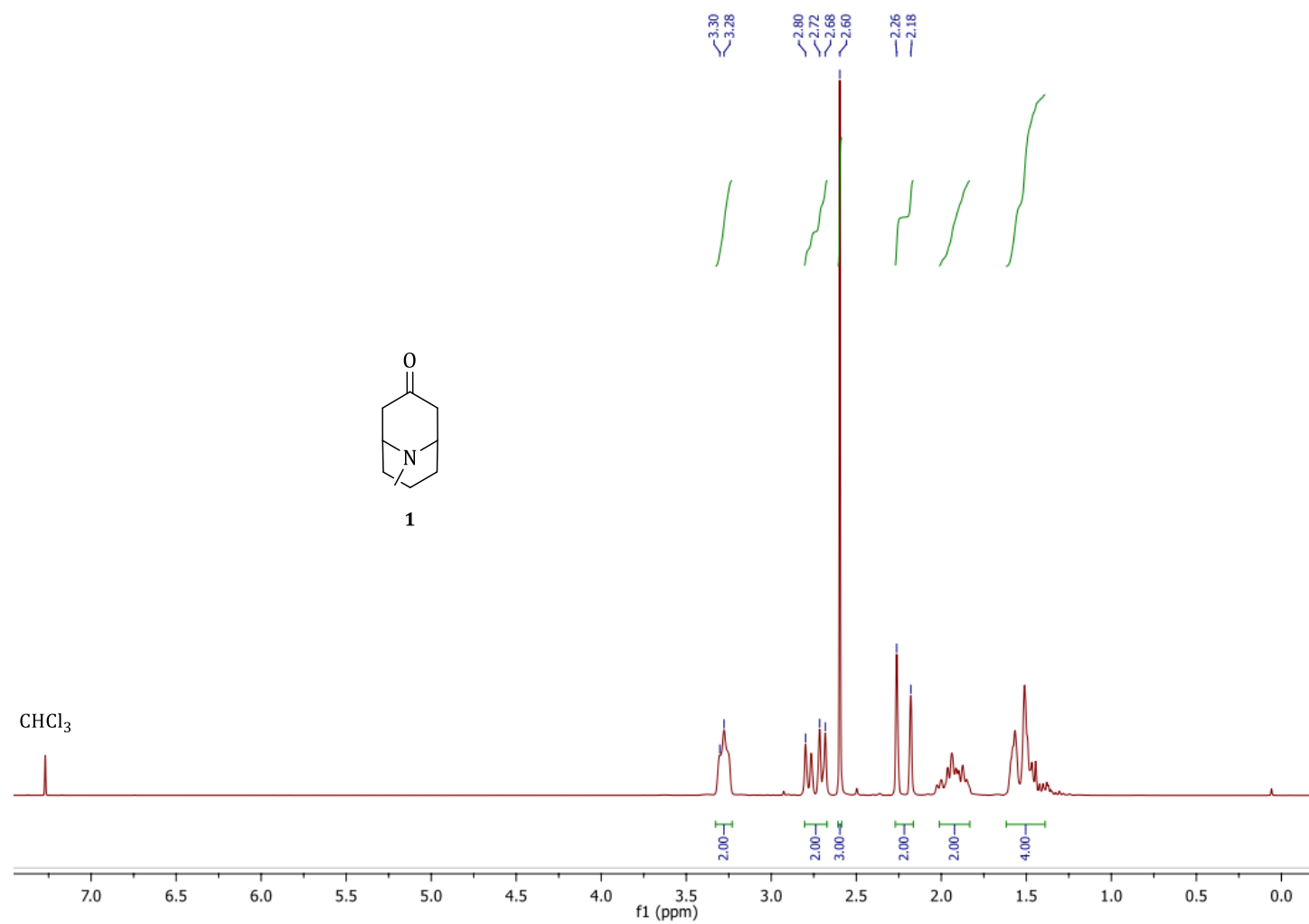


Figure S1. ^1H NMR (200 MHz, CDCl_3): 9-Methyl-9-azabicyclo[3.3.1]nonan-3-one (**1**)

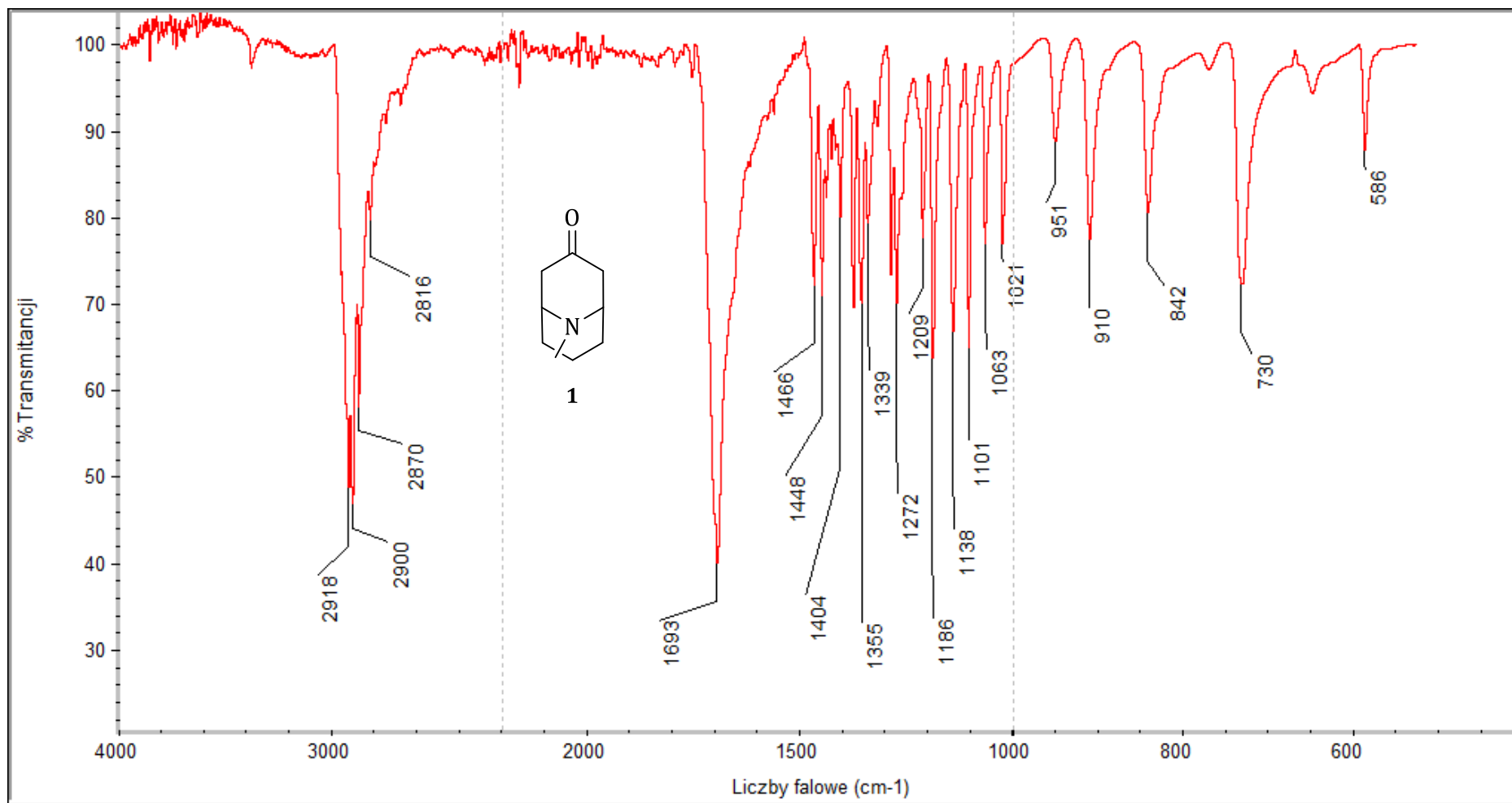


Figure S2. FTIR (ATR): 9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**1**)

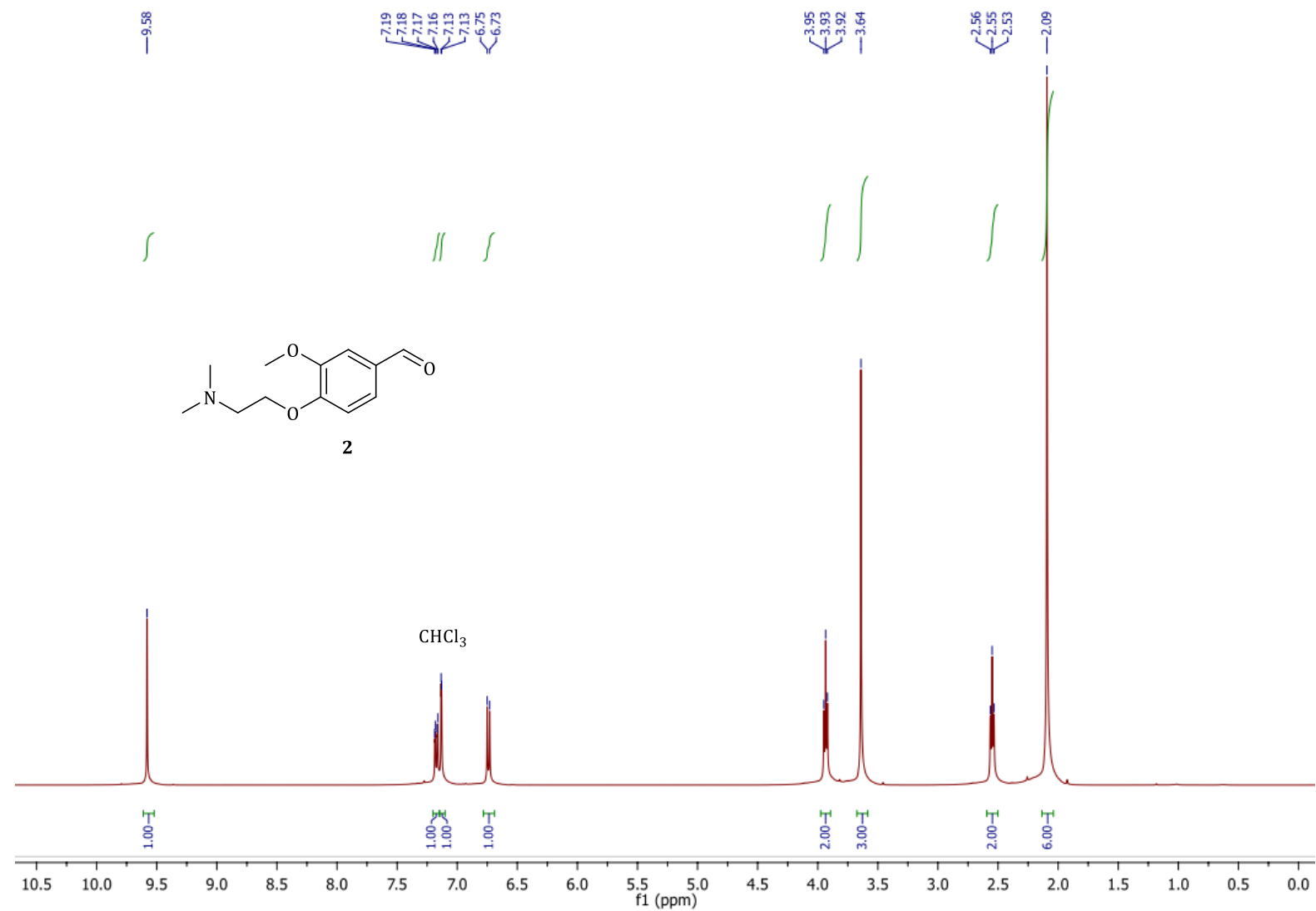


Figure S3. ¹H NMR (400 MHz, CDCl₃): 4-(2-(Dimethylamino)ethoxy)-3-methoxybenzaldehyde (**2**)

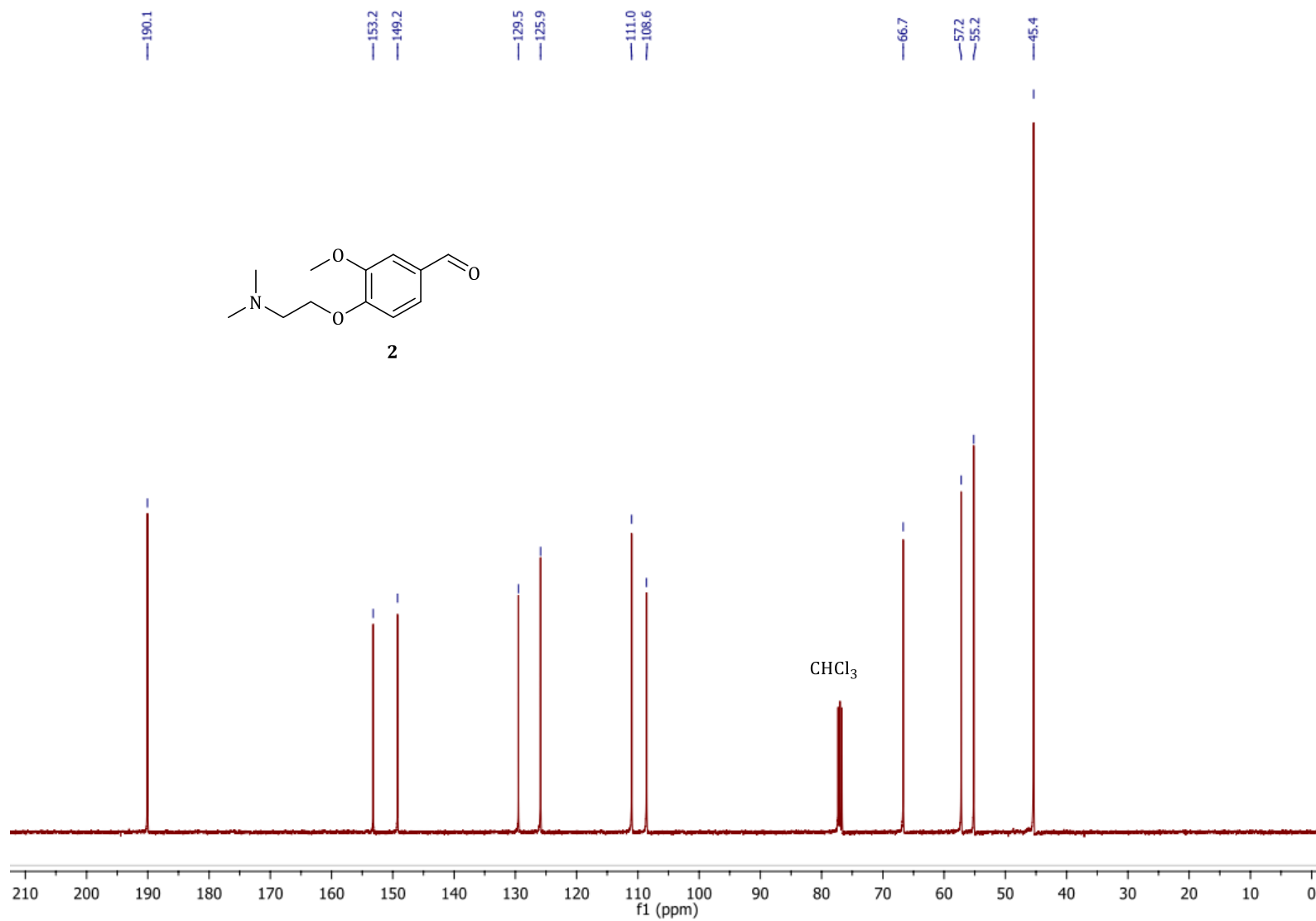


Figure S4. ¹³C NMR (101 MHz, CDCl₃): 4-(2-(Dimethylamino)ethoxy)-3-methoxybenzaldehyde (2)

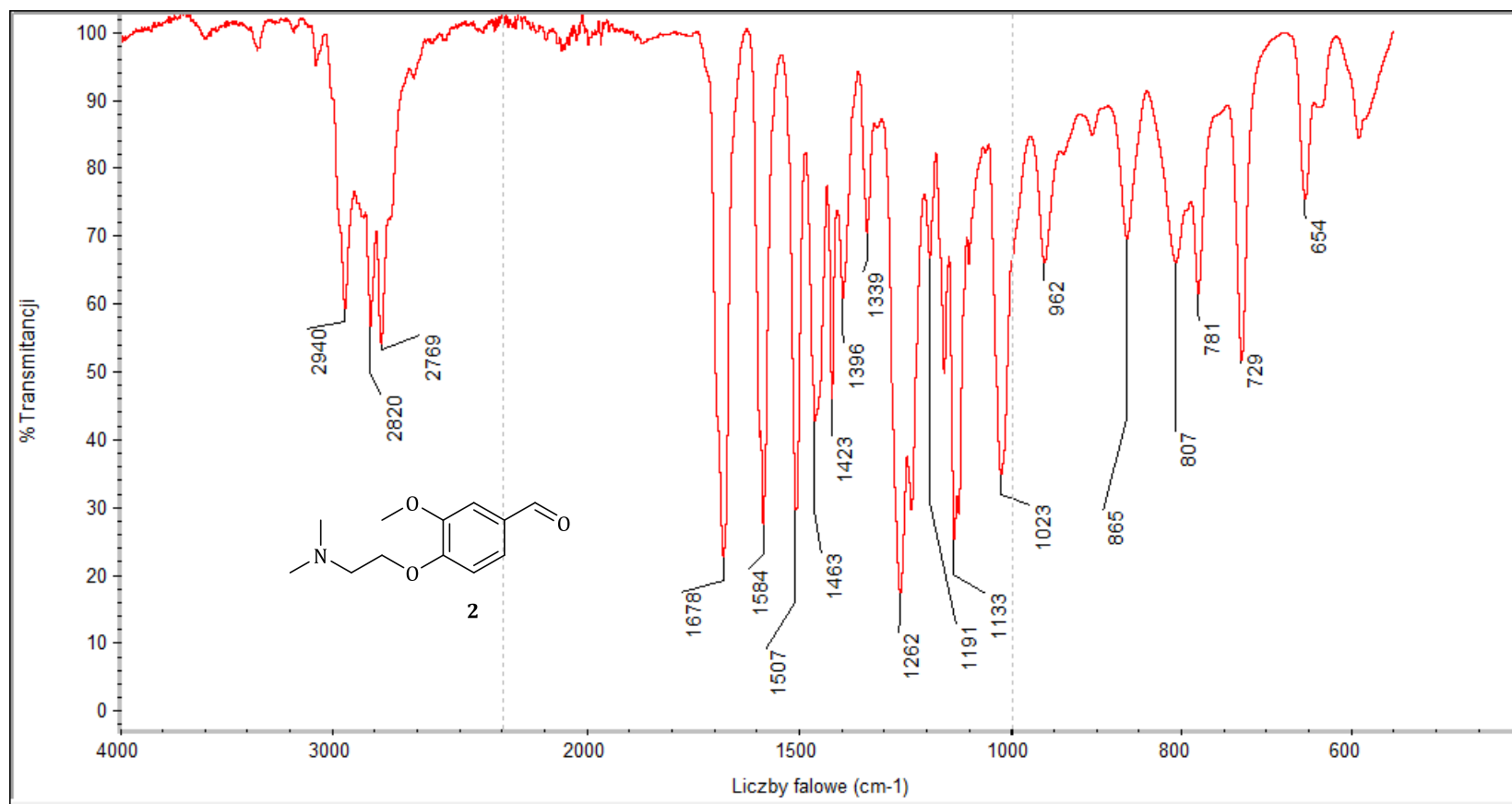


Figure S5. FTIR (ATR): 4-(2-(Dimethylamino)ethoxy)-3-methoxybenzaldehyde (2)

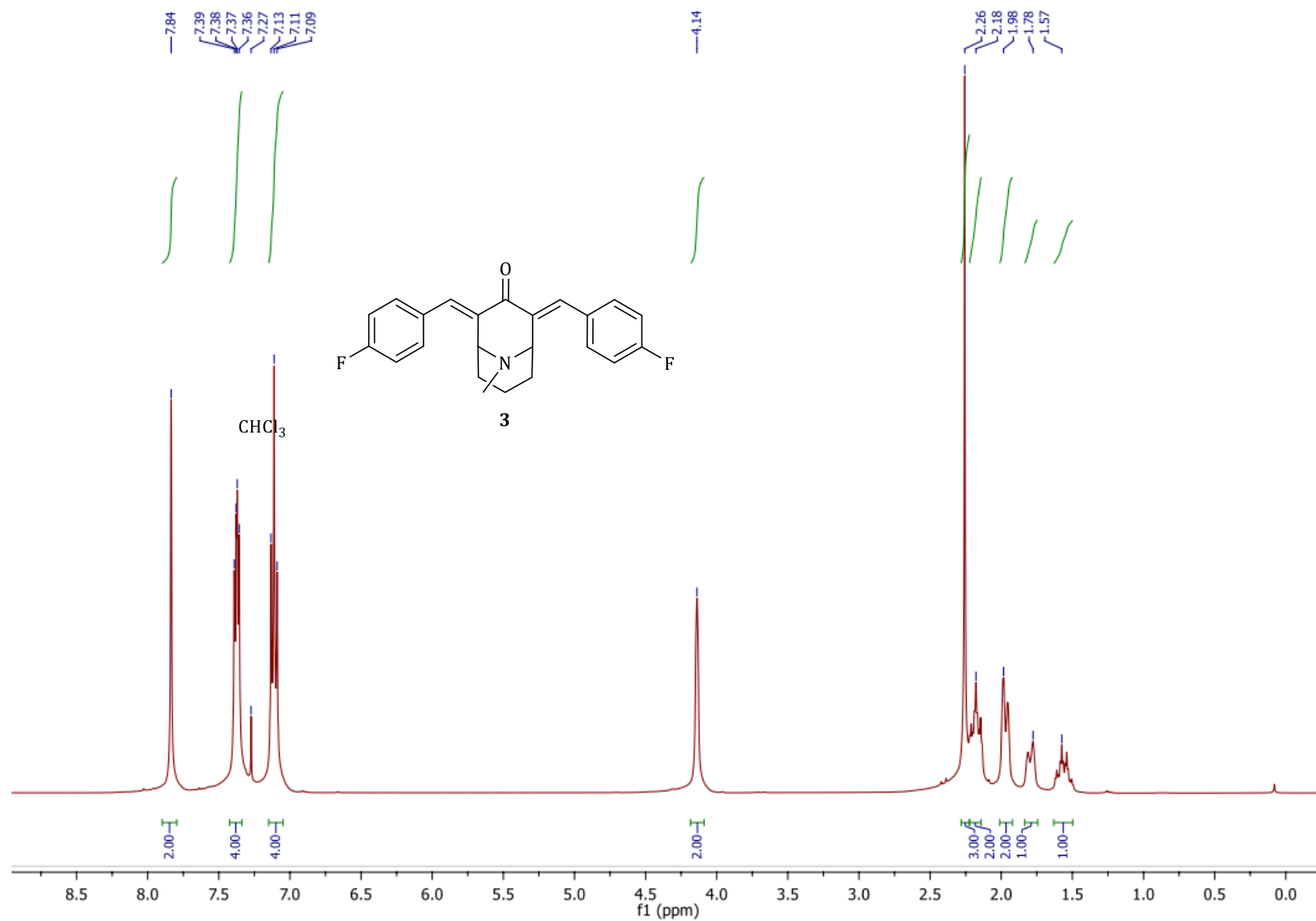


Figure S6. ¹H NMR (400 MHz, CDCl₃): 2,4-Bis((*E*)-4-fluorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**3**)

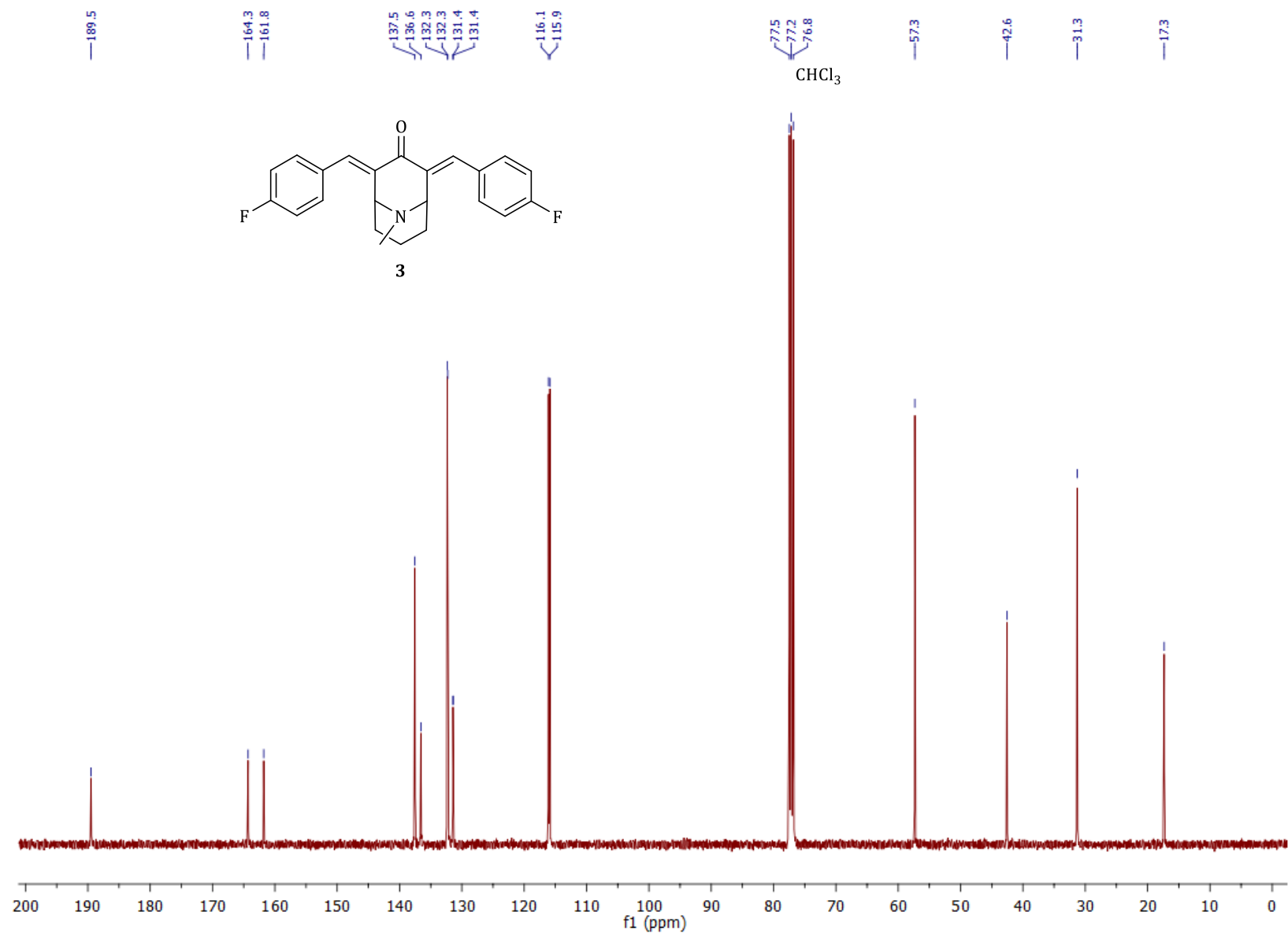


Figure S7. ¹³C NMR (101 MHz, CDCl₃): 2,4-Bis((E)-4-fluorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (3)

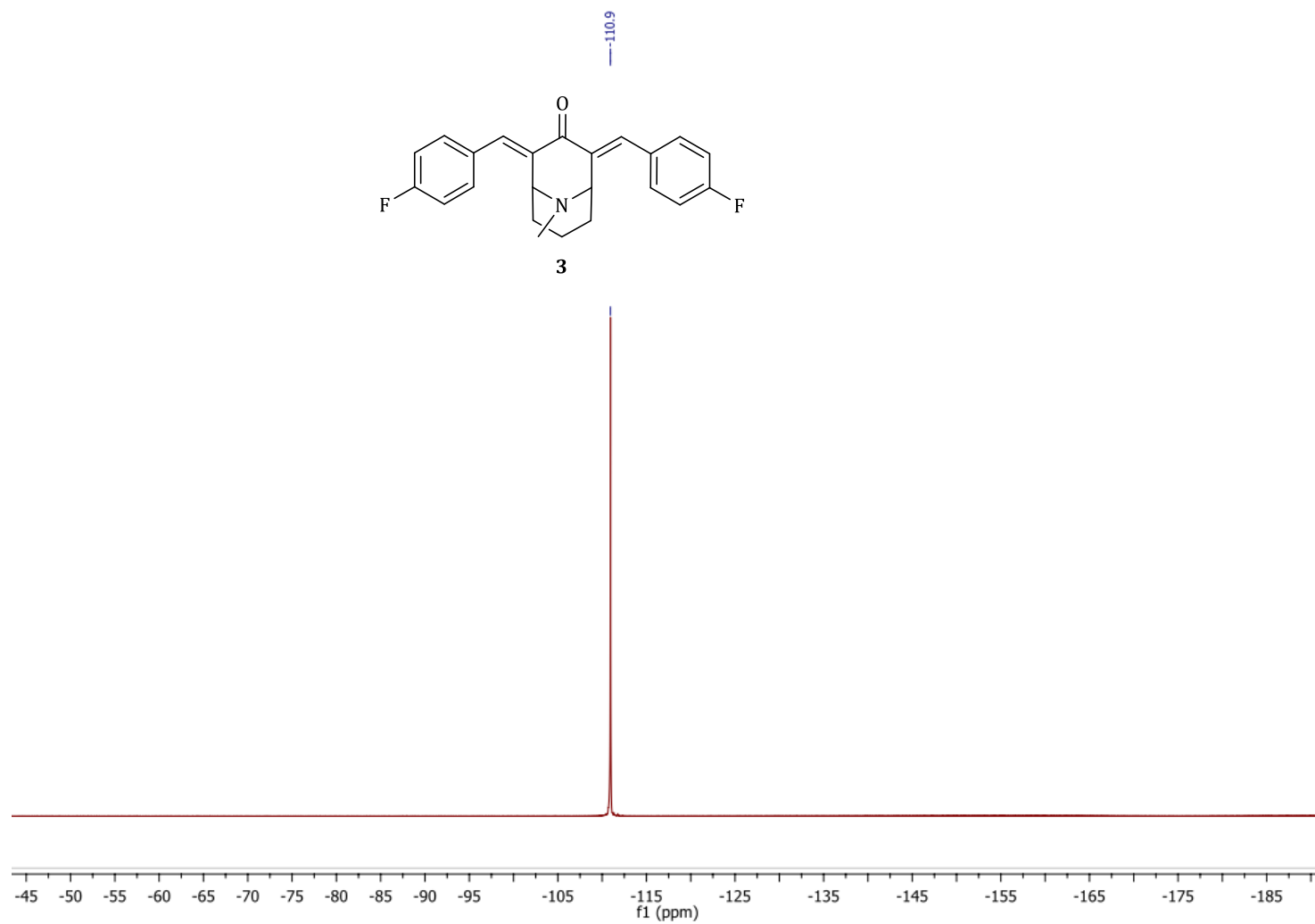


Figure S8. ^{19}F NMR (377 MHz, CDCl_3): 2,4-Bis((*E*)-4-fluorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**3**)

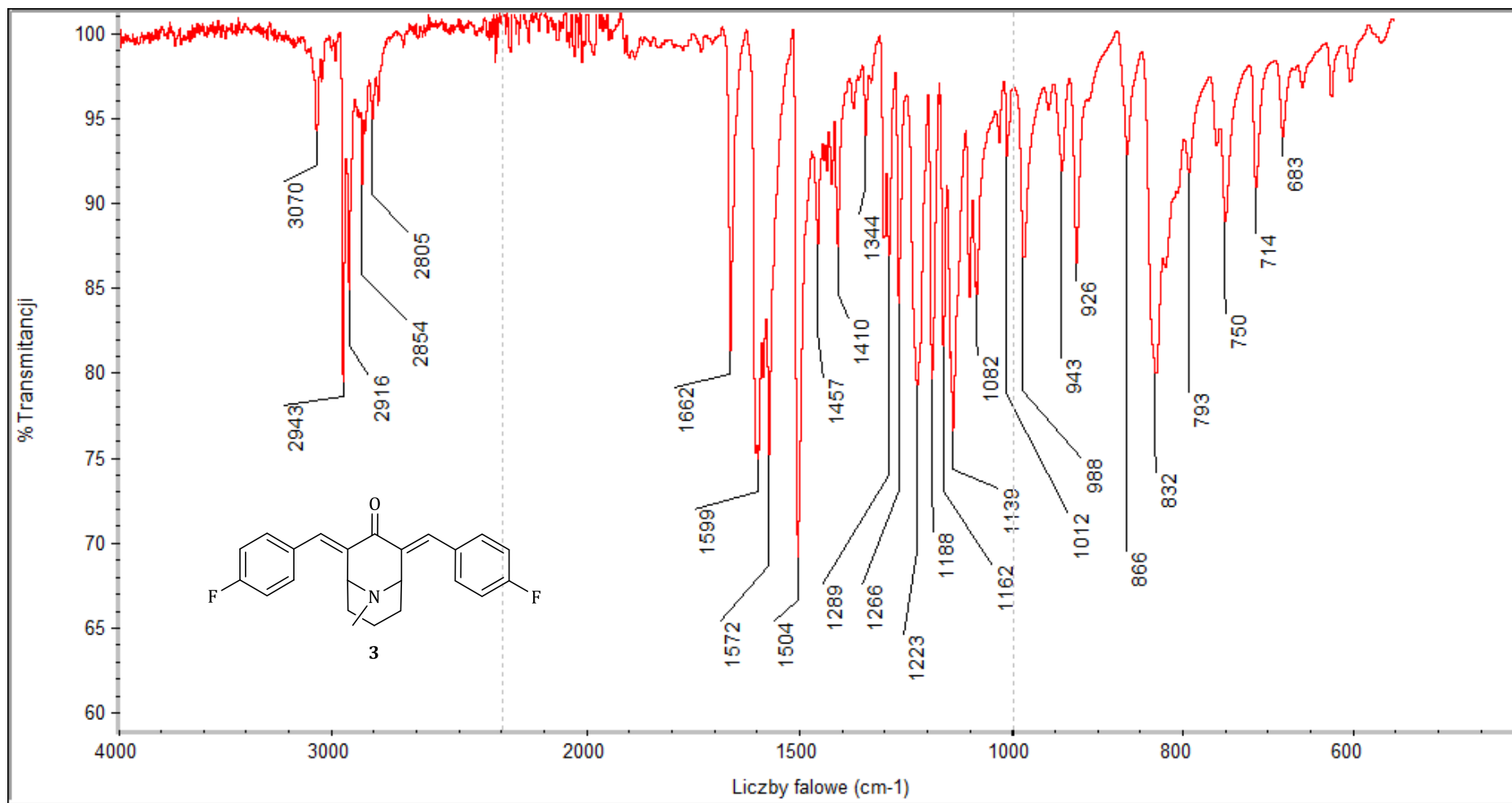


Figure S9. FTIR (ATR): 2,4-Bis((*E*)-4-fluorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**3**)

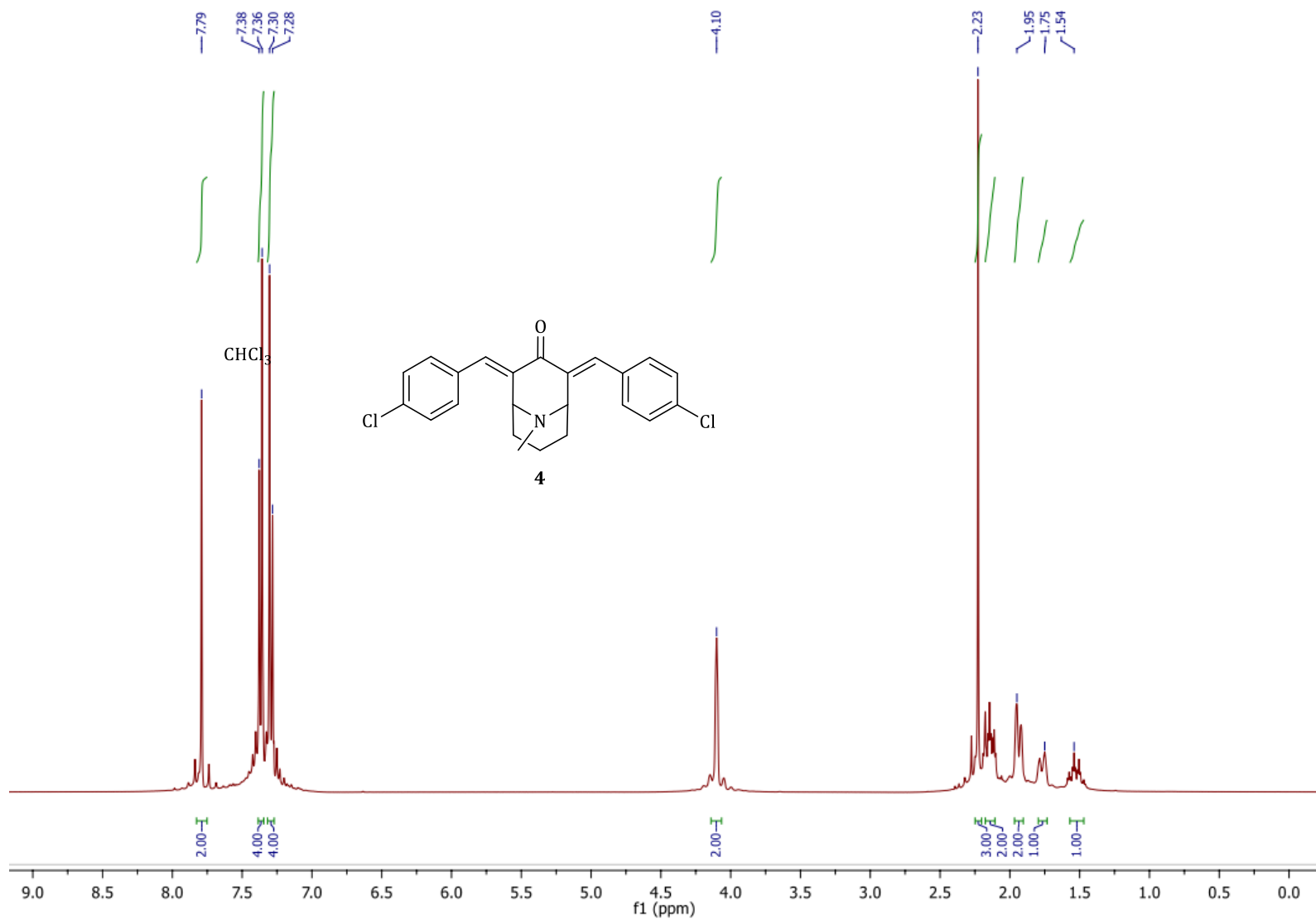


Figure S10. ¹H NMR (400 MHz, CDCl₃): 2,4-Bis((*E*)-4-chlorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**4**)

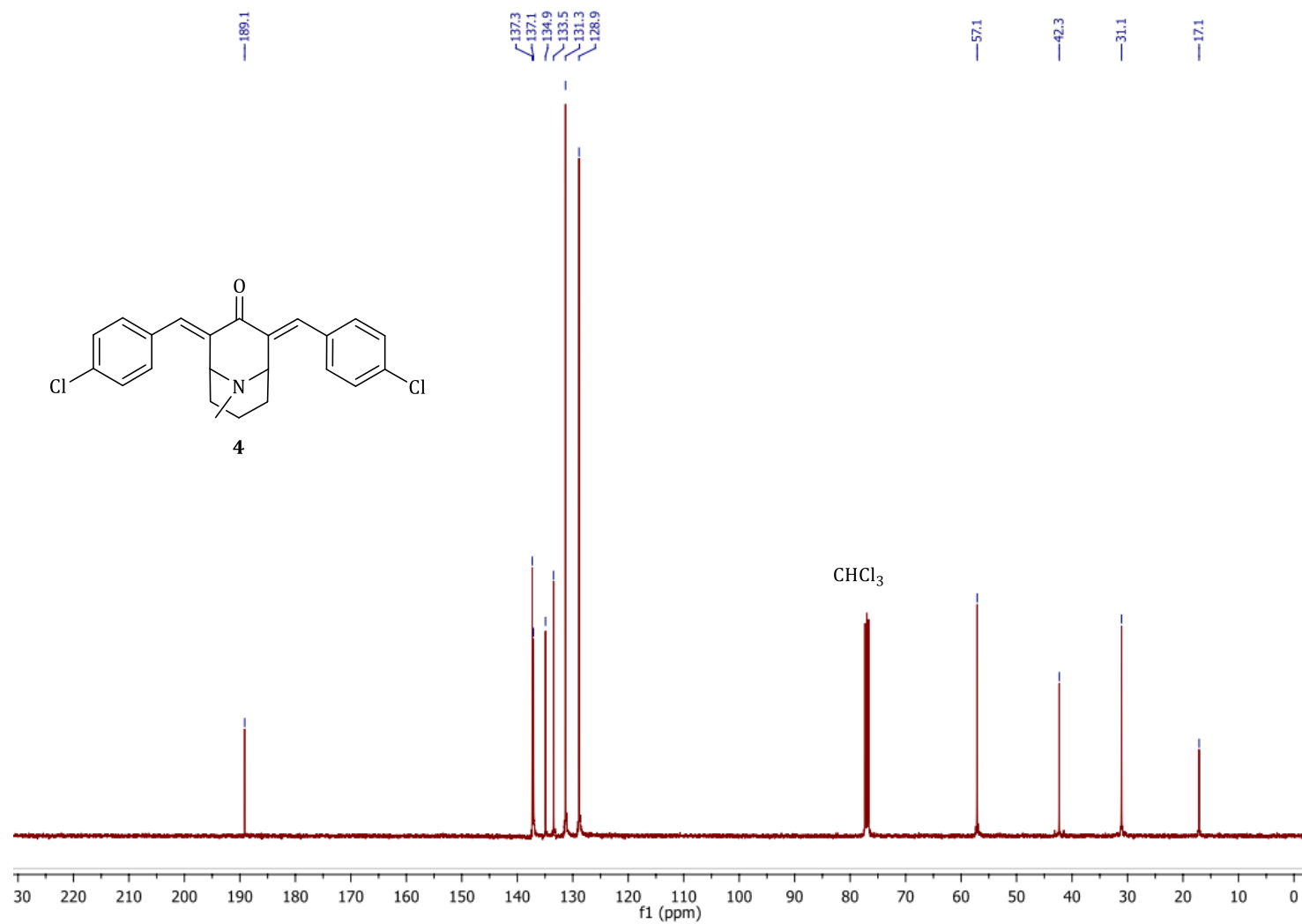


Figure S11. ¹³C NMR (101 MHz, CDCl₃): 2,4-Bis((*E*)-4-chlorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**4**)

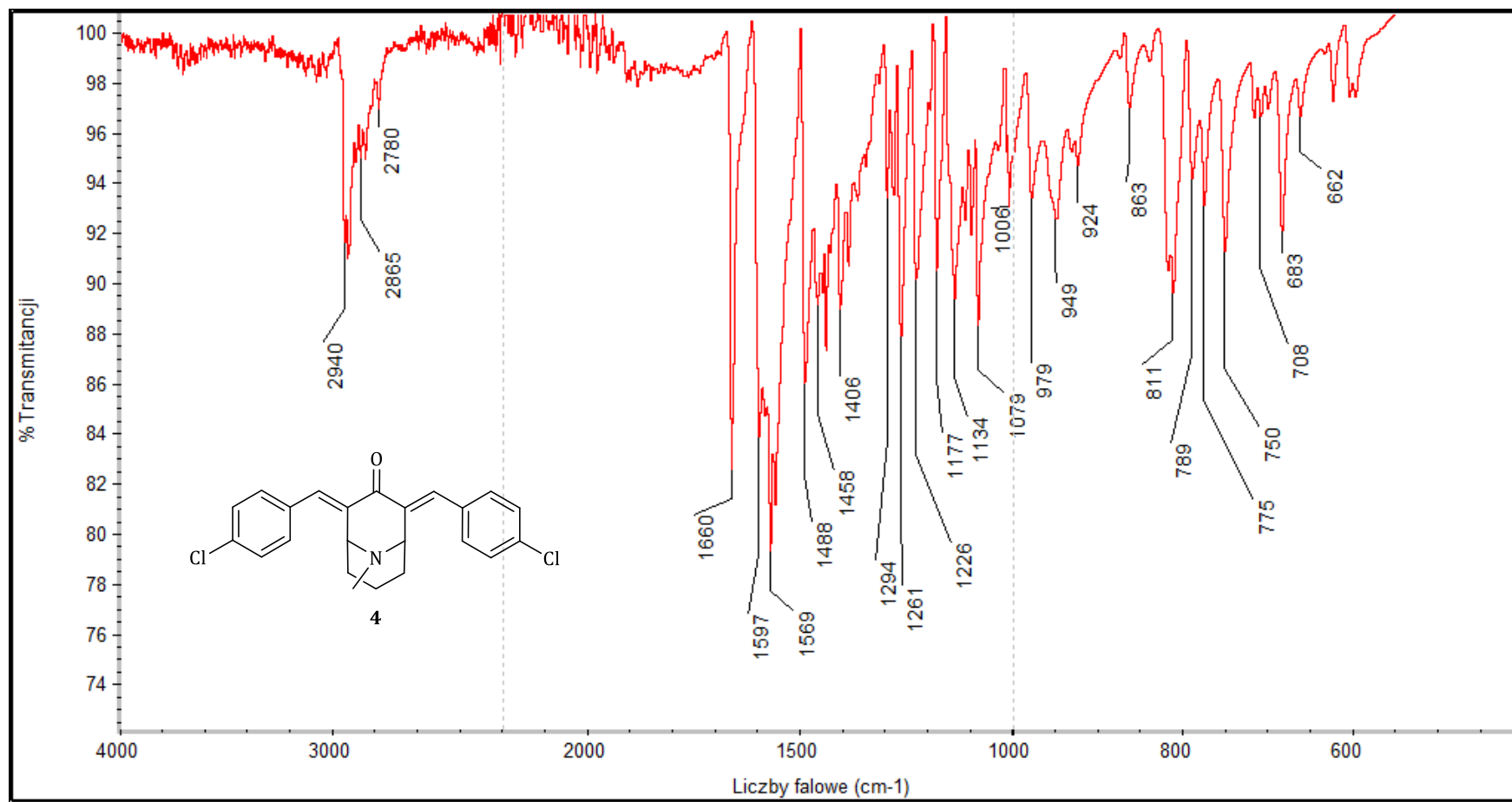


Figure S12. FTIR (ATR): 2,4-Bis((*E*)-4-chlorobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**4**)

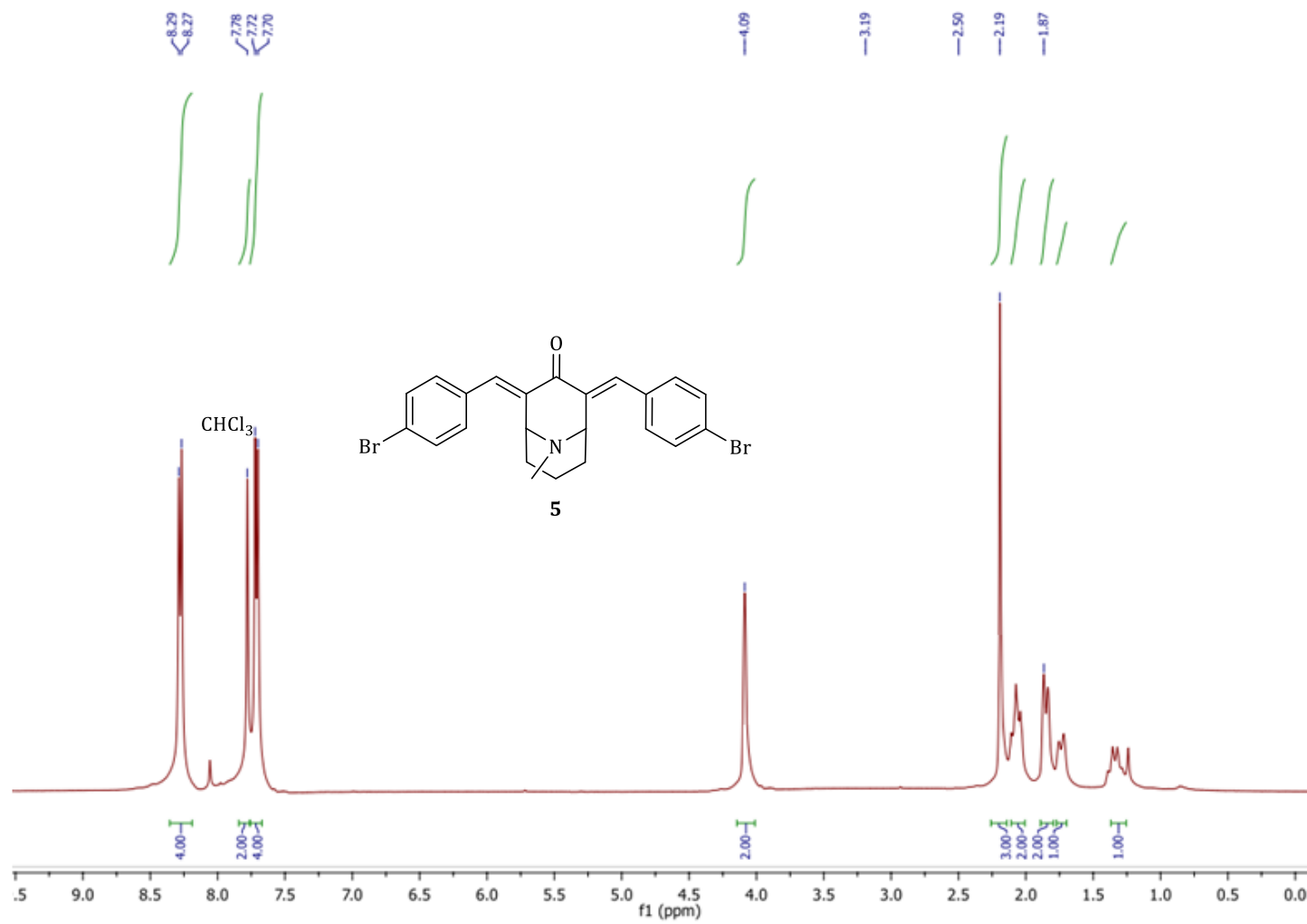


Figure S13. ^1H NMR (400 MHz, CDCl_3): 2,4-Bis((*E*)-4-bromobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (5)

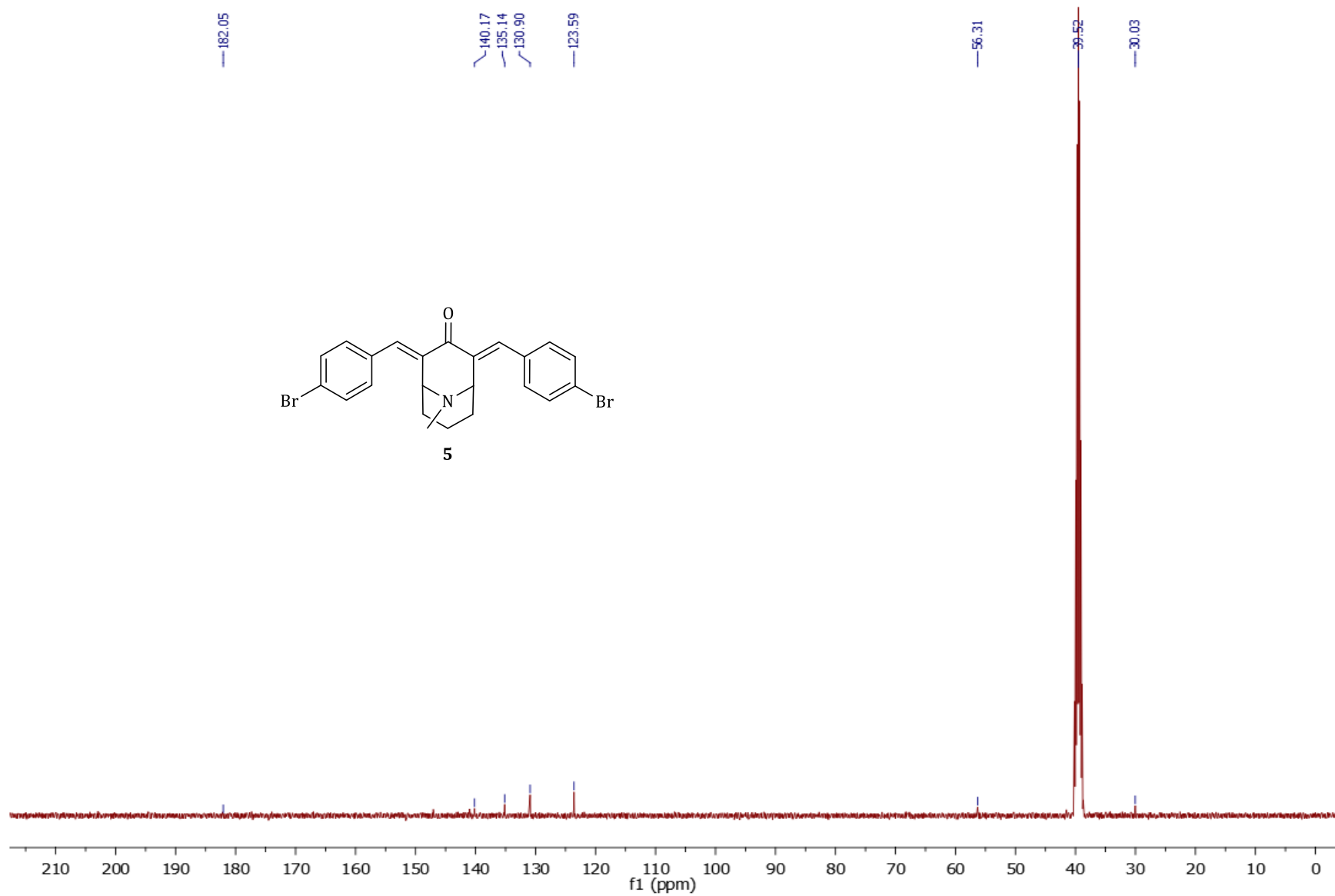


Figure S14. ¹³C NMR (101 MHz, DMSO-d₆): 2,4-Bis((E)-4-bromobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (5)

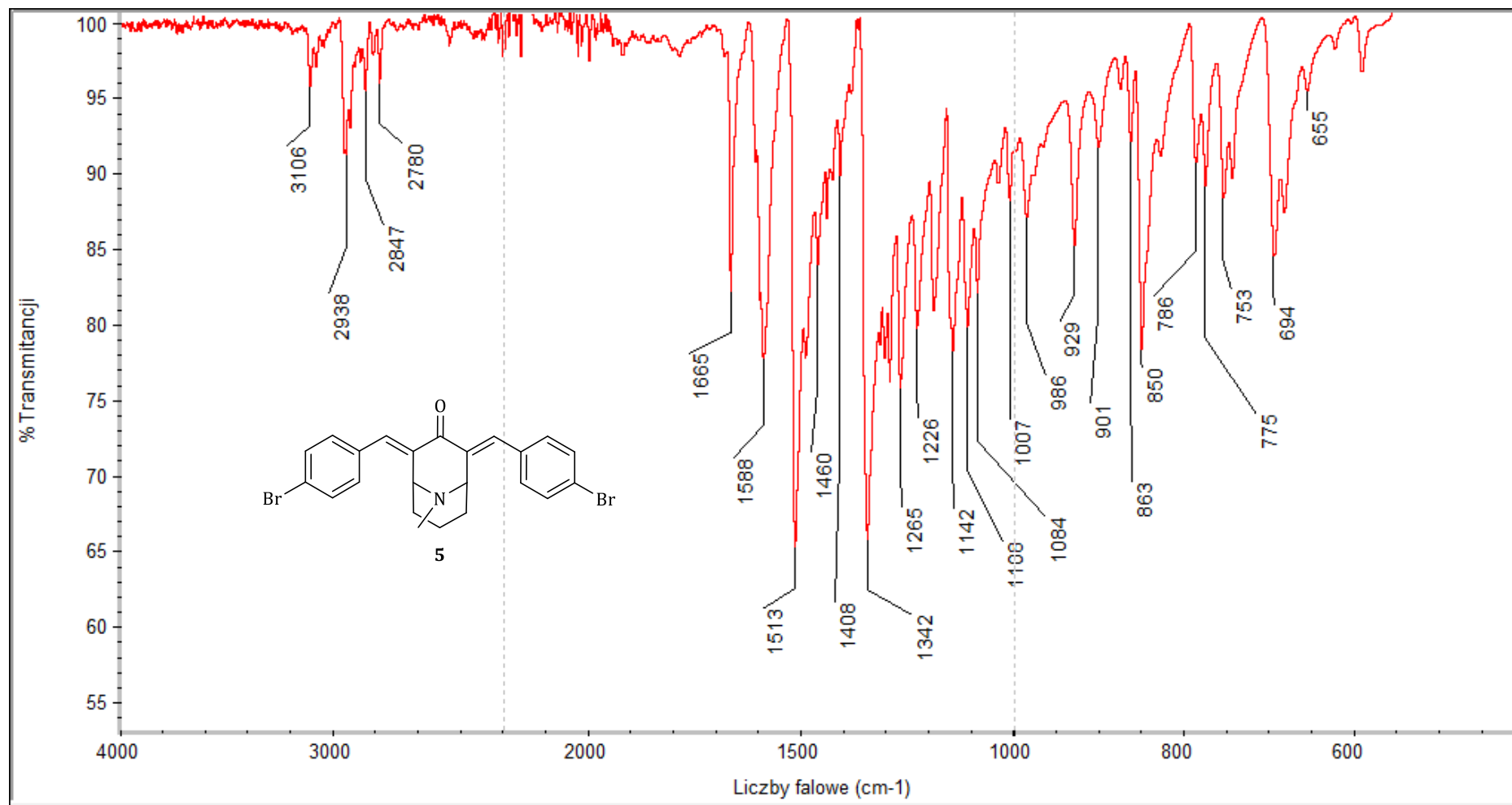


Figure S15. FTIR (ATR): 2,4-Bis((*E*)-4-bromobenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (5)

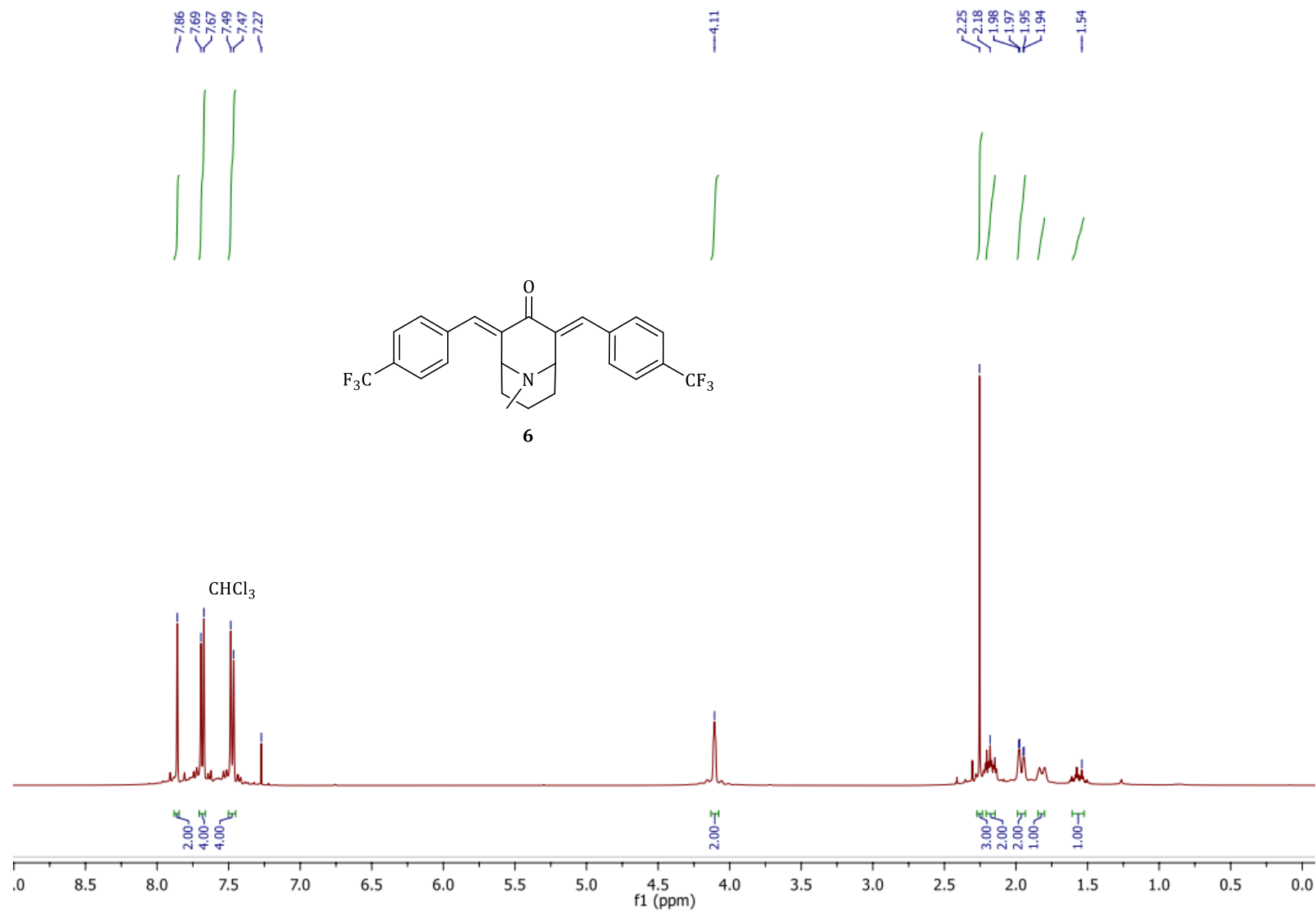


Figure S16. ¹H NMR (400 MHz, CDCl₃): 9-Methyl-2,4-bis((*E*)-4-(trifluoromethyl)benzylidene)-9-azabicyclo[3.3.1]nonan-3-one (**6**)

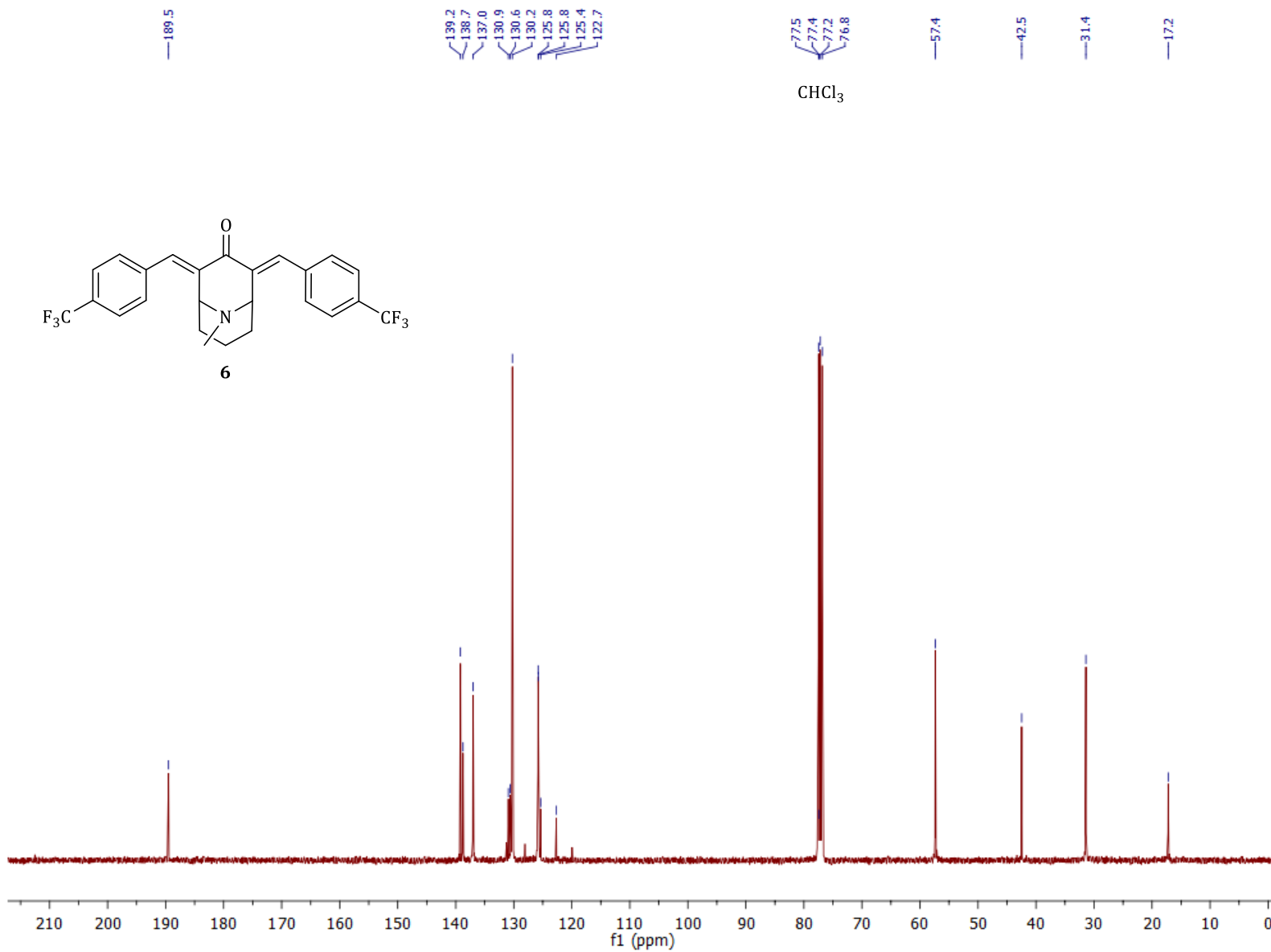


Figure S17. ¹³C NMR (101 MHz, CDCl₃): 9-Methyl-2,4-bis((*E*)-4-(trifluoromethyl)benzylidene)-9-azabicyclo[3.3.1]nonan-3-one (**6**)

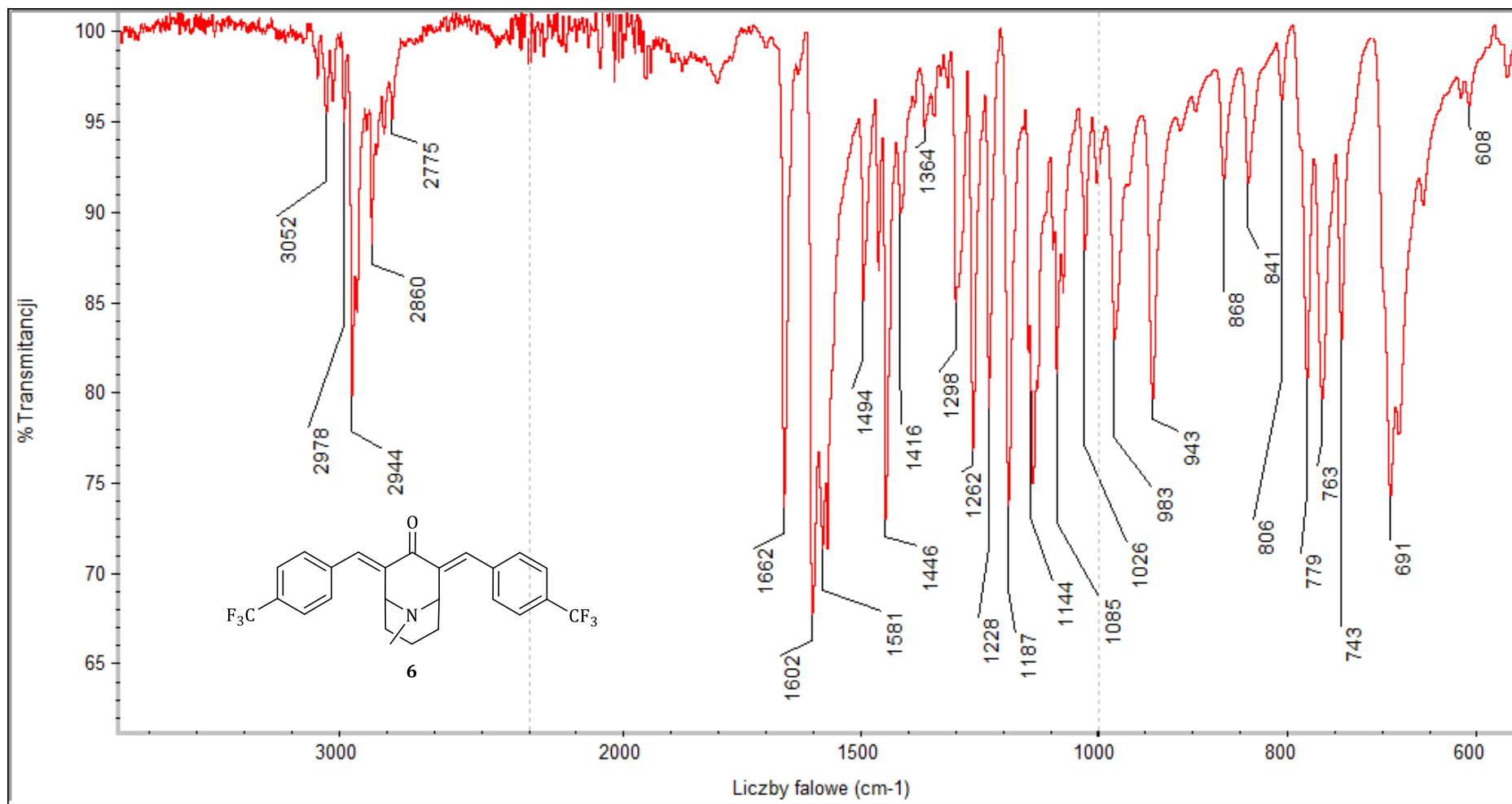


Figure S18. FTIR (ATR): 9-Methyl-2,4-bis((*E*)-4-(trifluoromethyl)benzylidene)-9-azabicyclo[3.3.1]nonan-3-one (6)

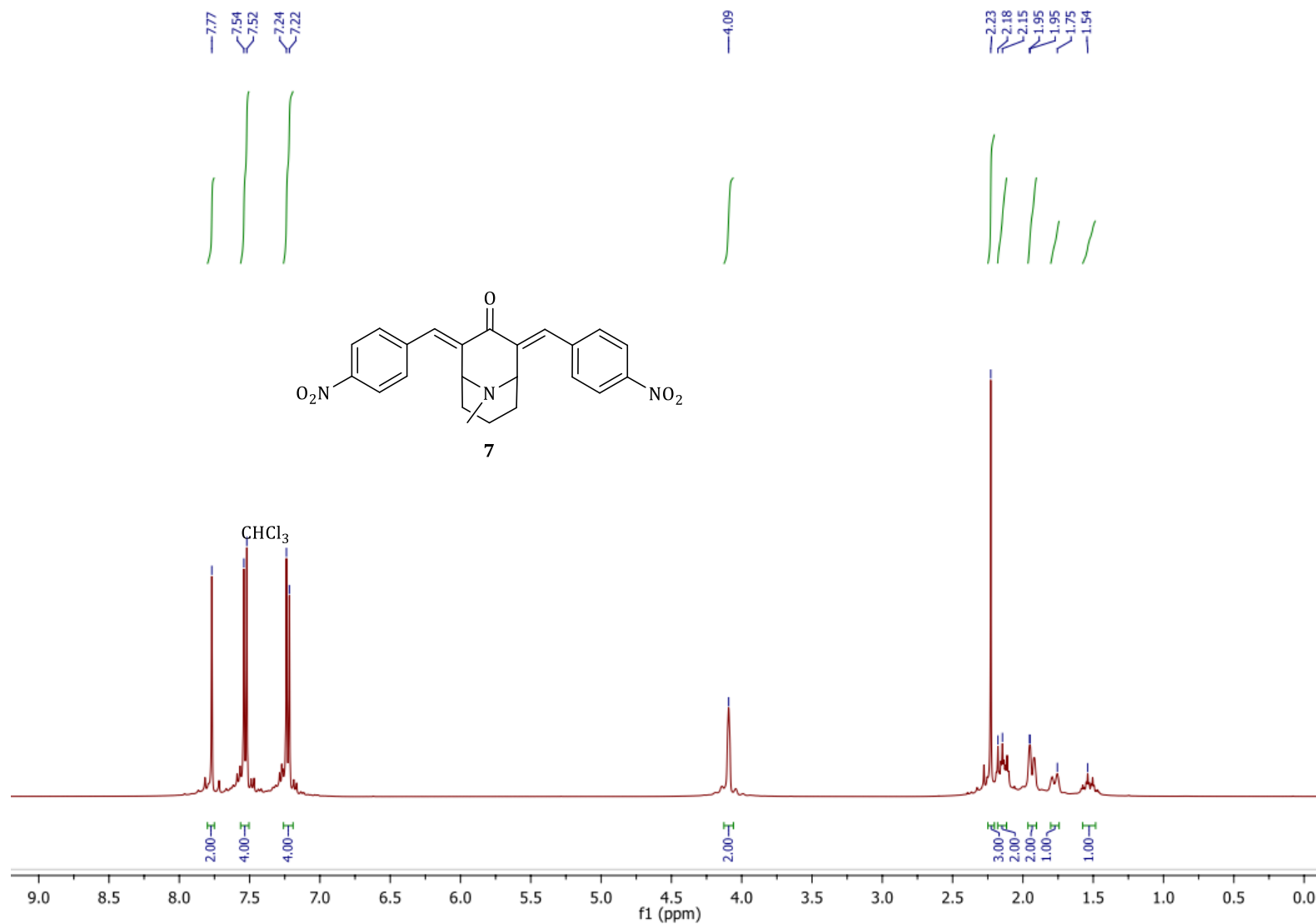


Figure S19. ¹H NMR (400 MHz, CDCl₃): 9-Methyl-2,4-bis((E)-4-nitrobenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (7)

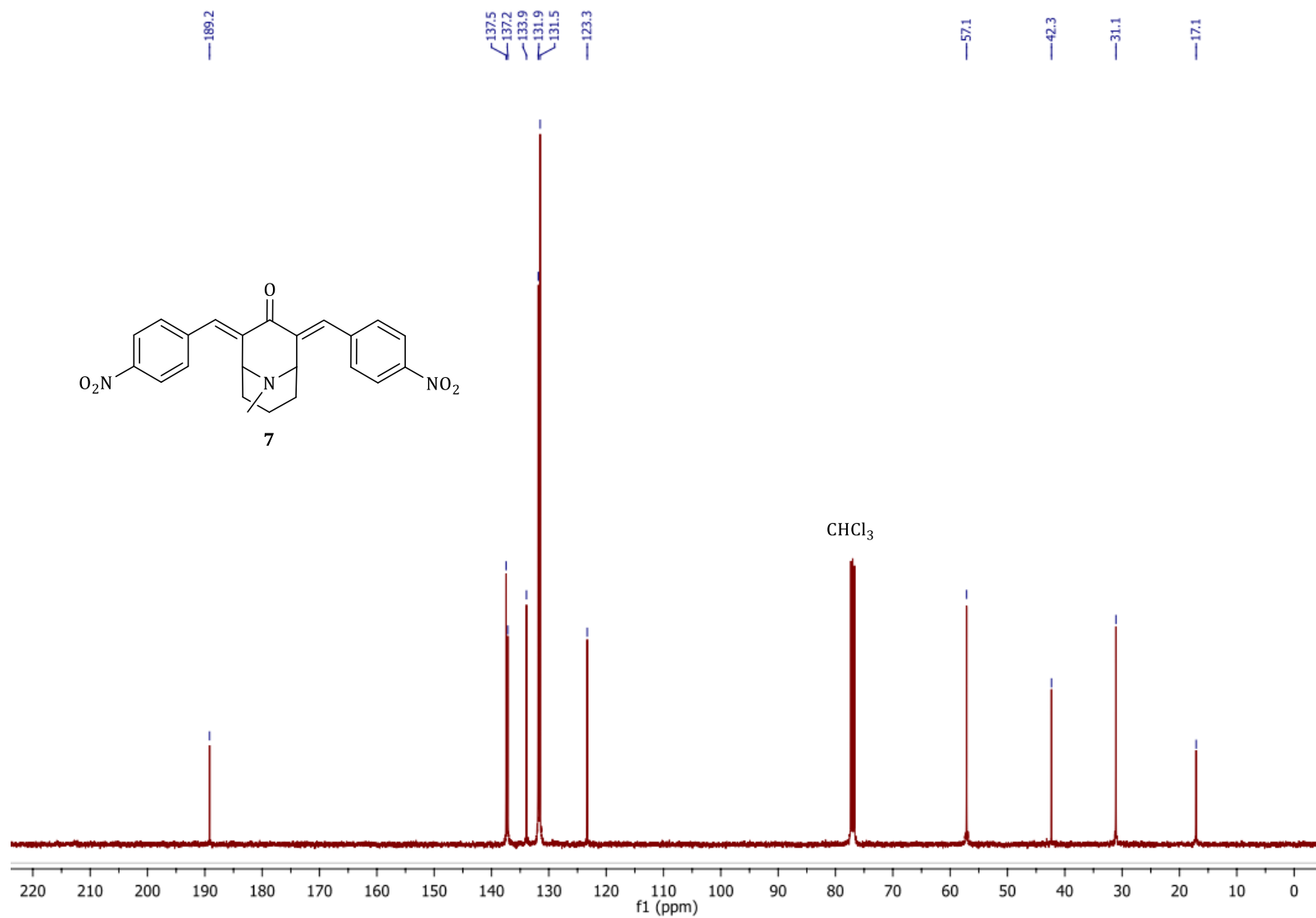


Figure S20. ¹³C NMR (101 MHz, CDCl₃): 9-Methyl-2,4-bis((*E*)-4-nitrobenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (7)

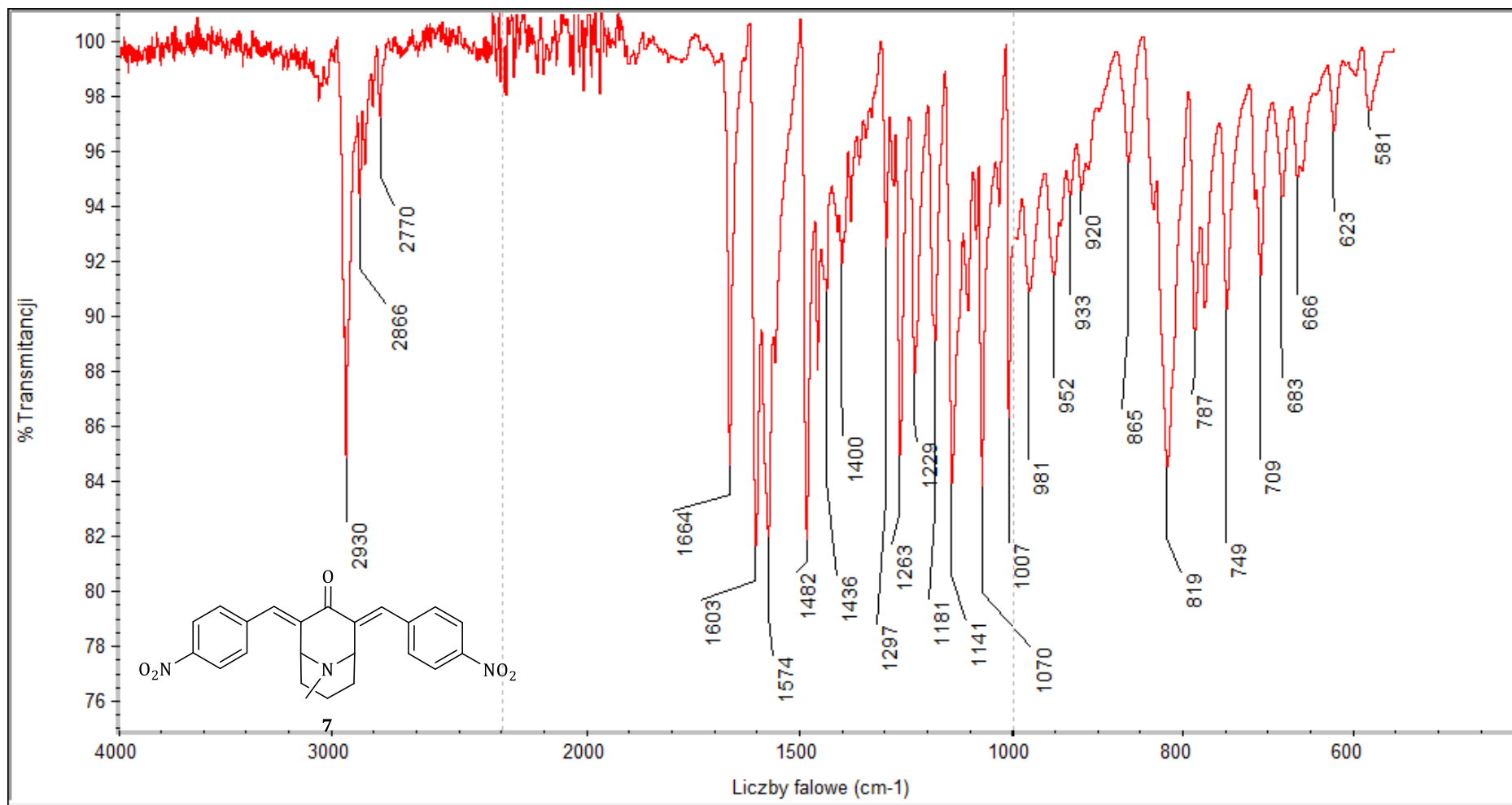


Figure S21. FTIR (ATR): 9-Methyl-2,4-bis((*E*)-4-nitrobenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (7)

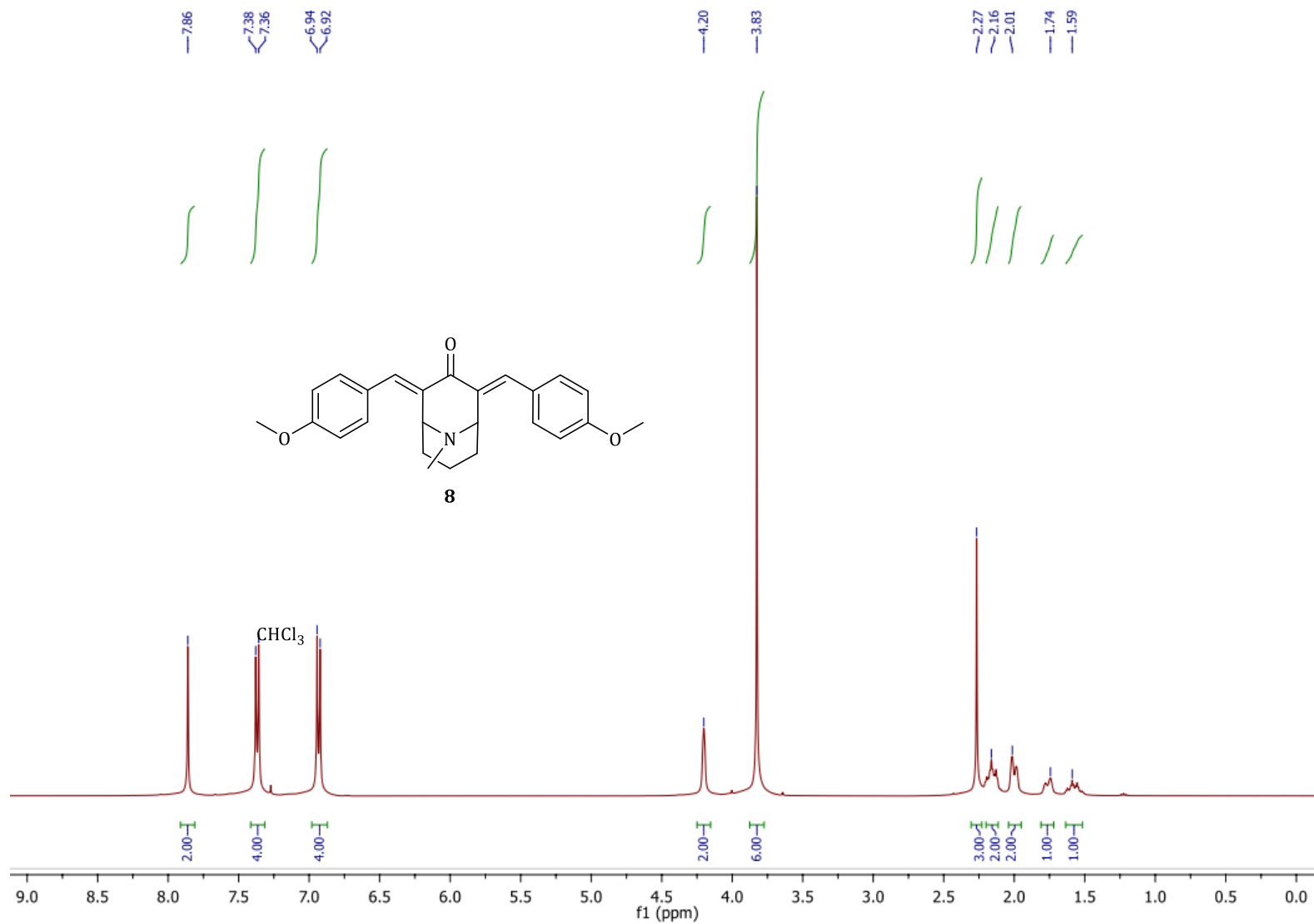


Figure S22. ^1H NMR (400 MHz, CDCl_3): 4-Bis((*E*)-4-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**8**)

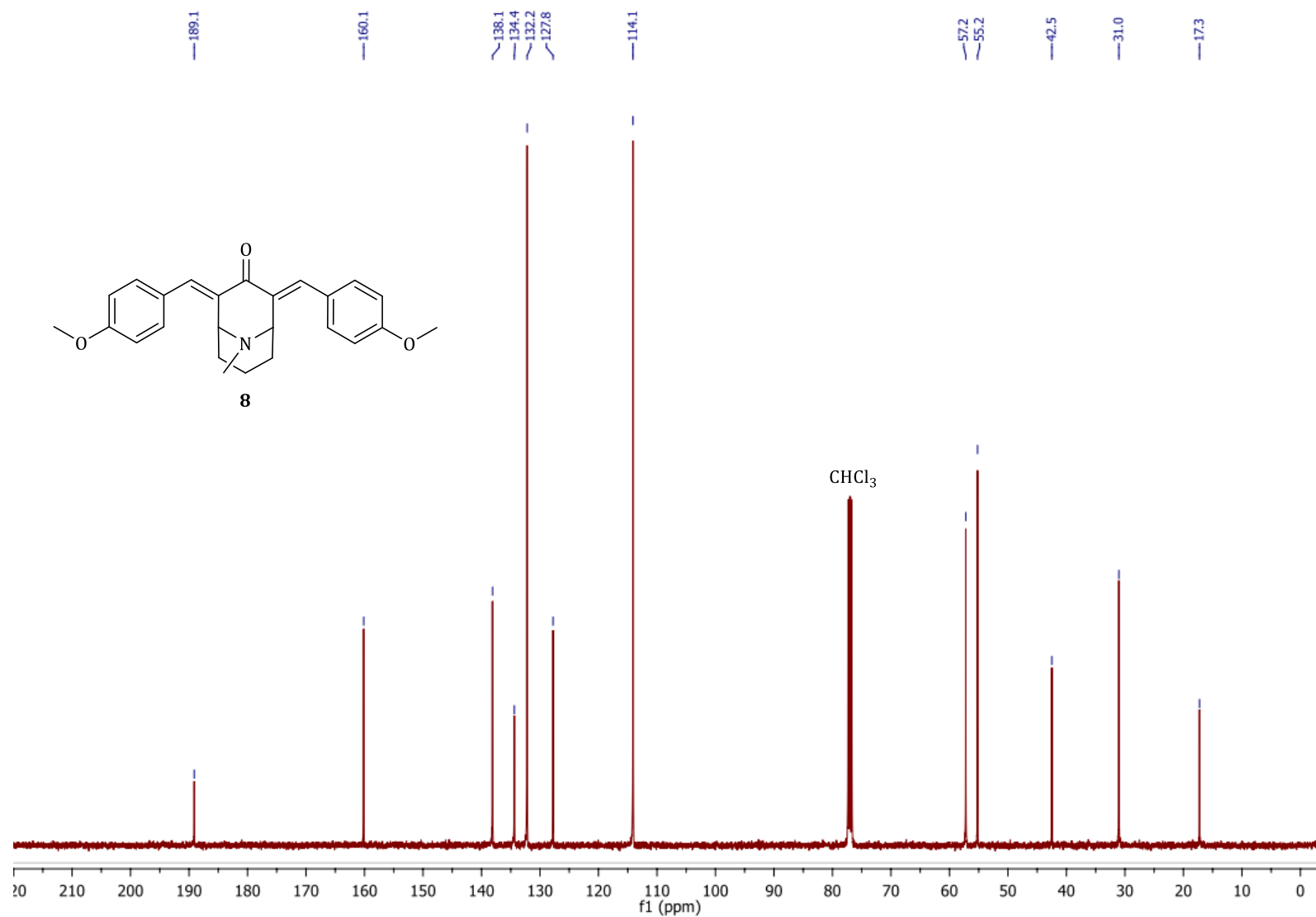


Figure S23. ¹³C NMR (101 MHz, CDCl₃): 4-Bis((*E*)-4-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**8**)

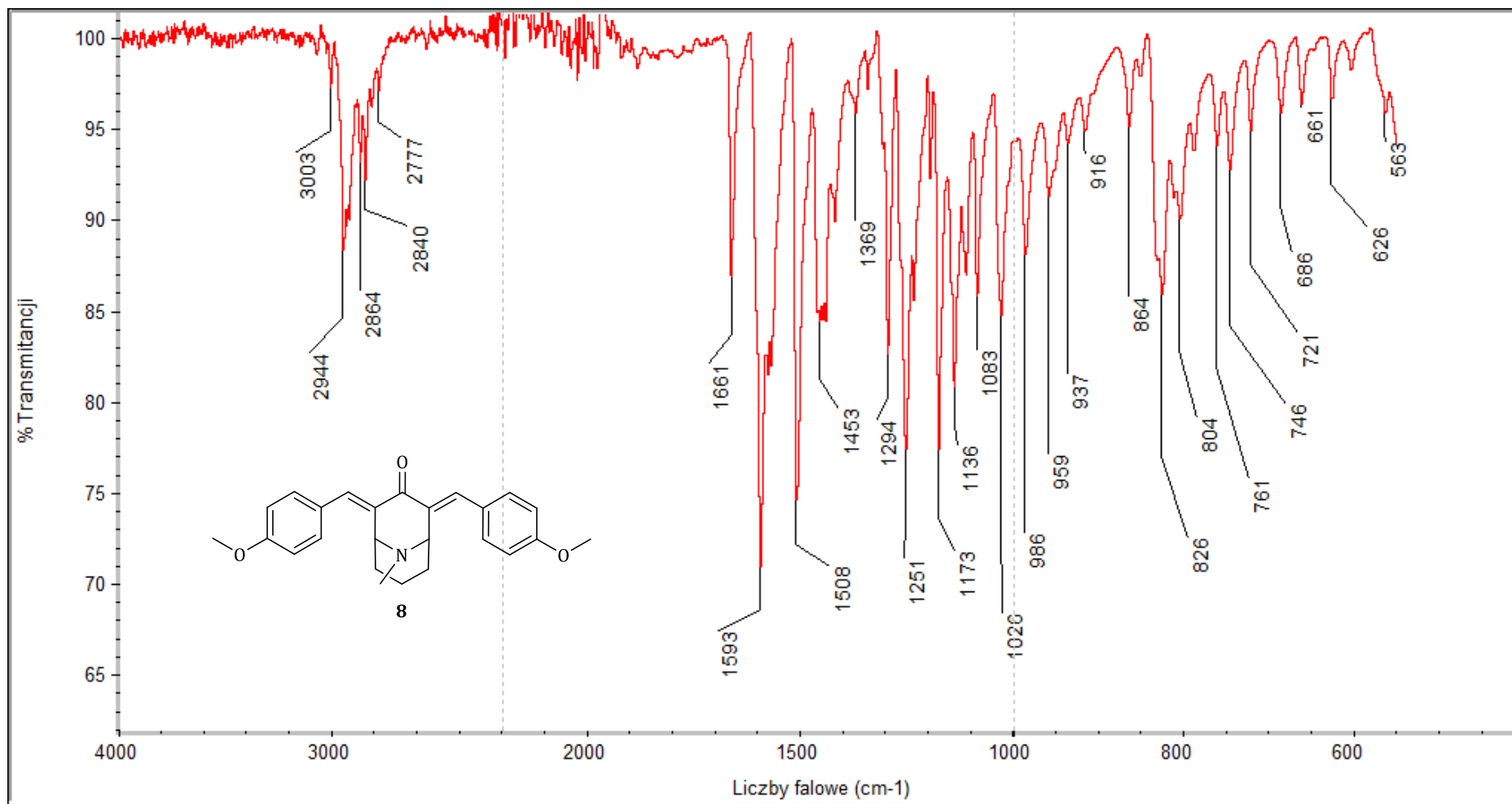


Figure S24. FTIR (ATR): 4-Bis((*E*)-4-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**8**)

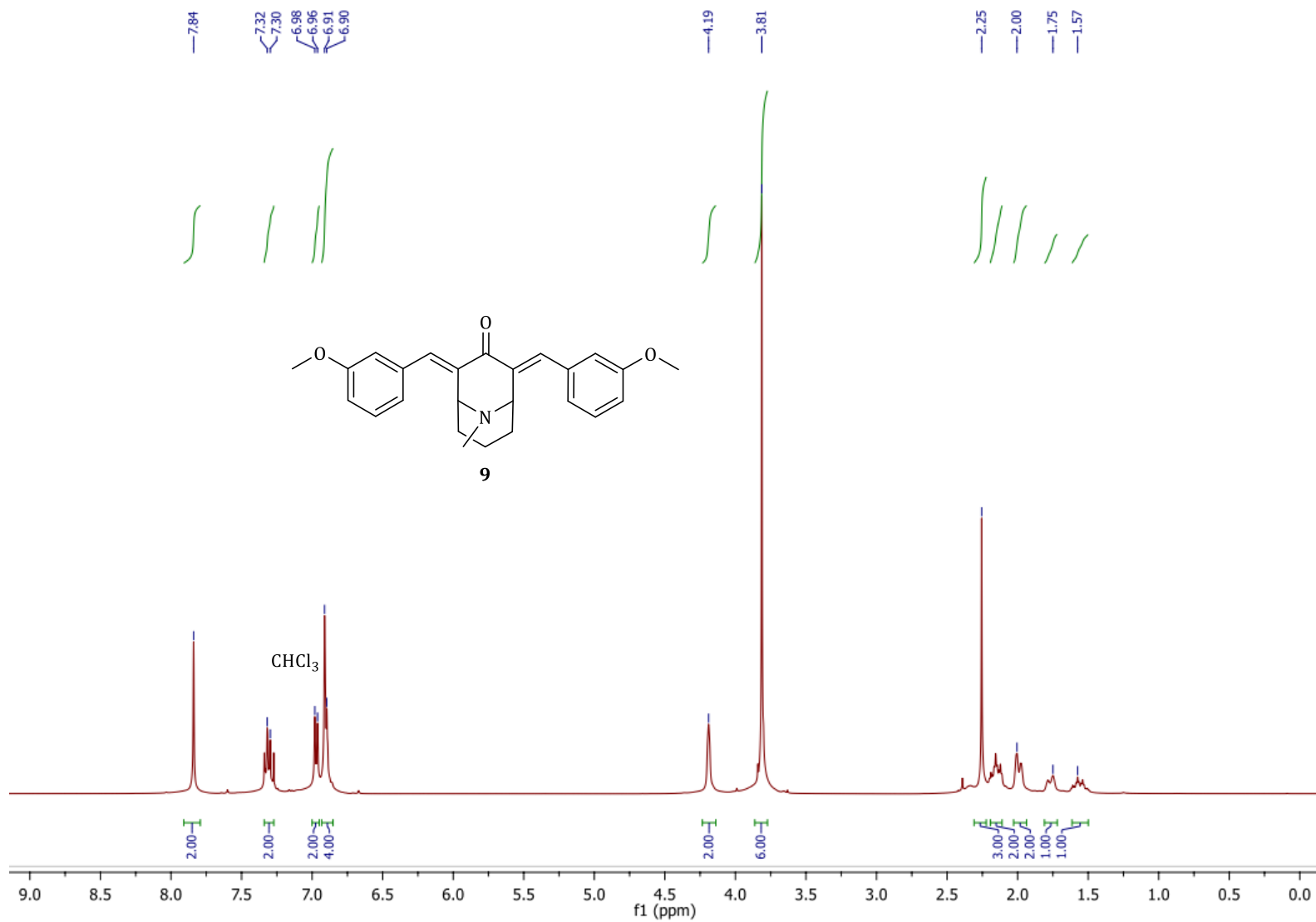


Figure S25. ¹H NMR (400 MHz, CDCl₃): 4-Bis((*E*)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**9**)

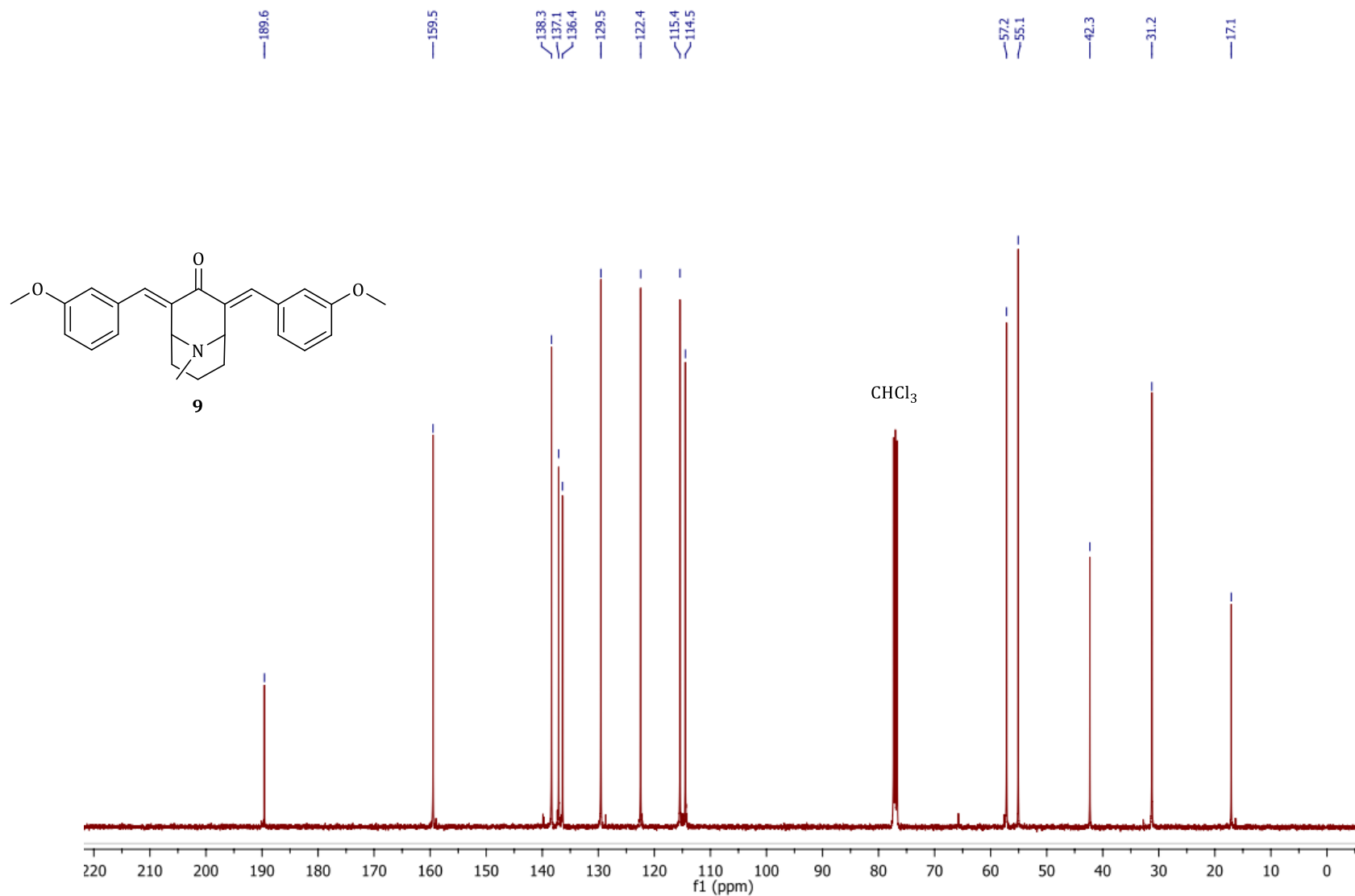


Figure S26. ^{13}C NMR (101 MHz, CDCl_3): 4-Bis((*E*)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (9)

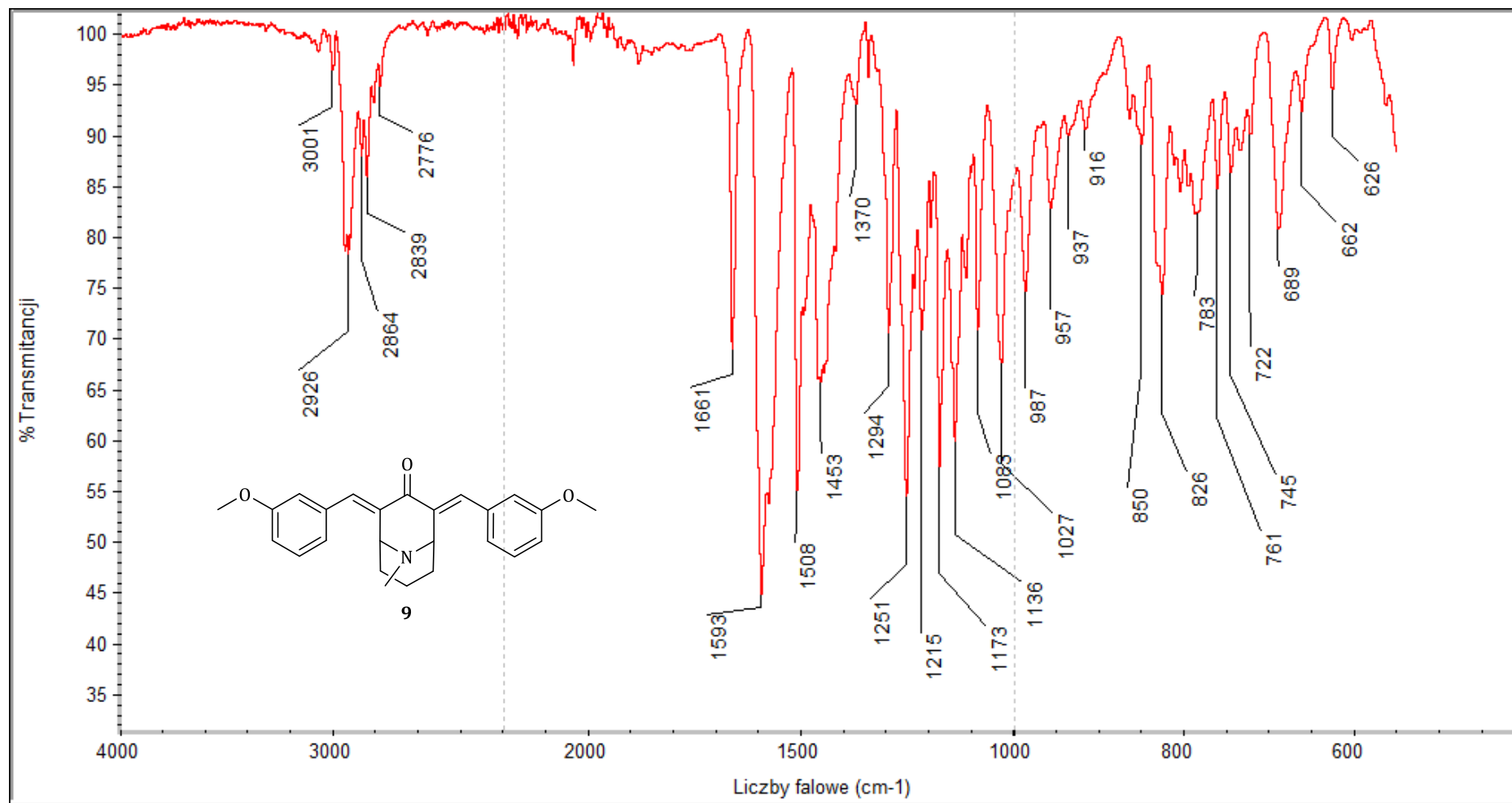


Figure S27. FTIR (ATR): 4-Bis((*E*)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**9**)

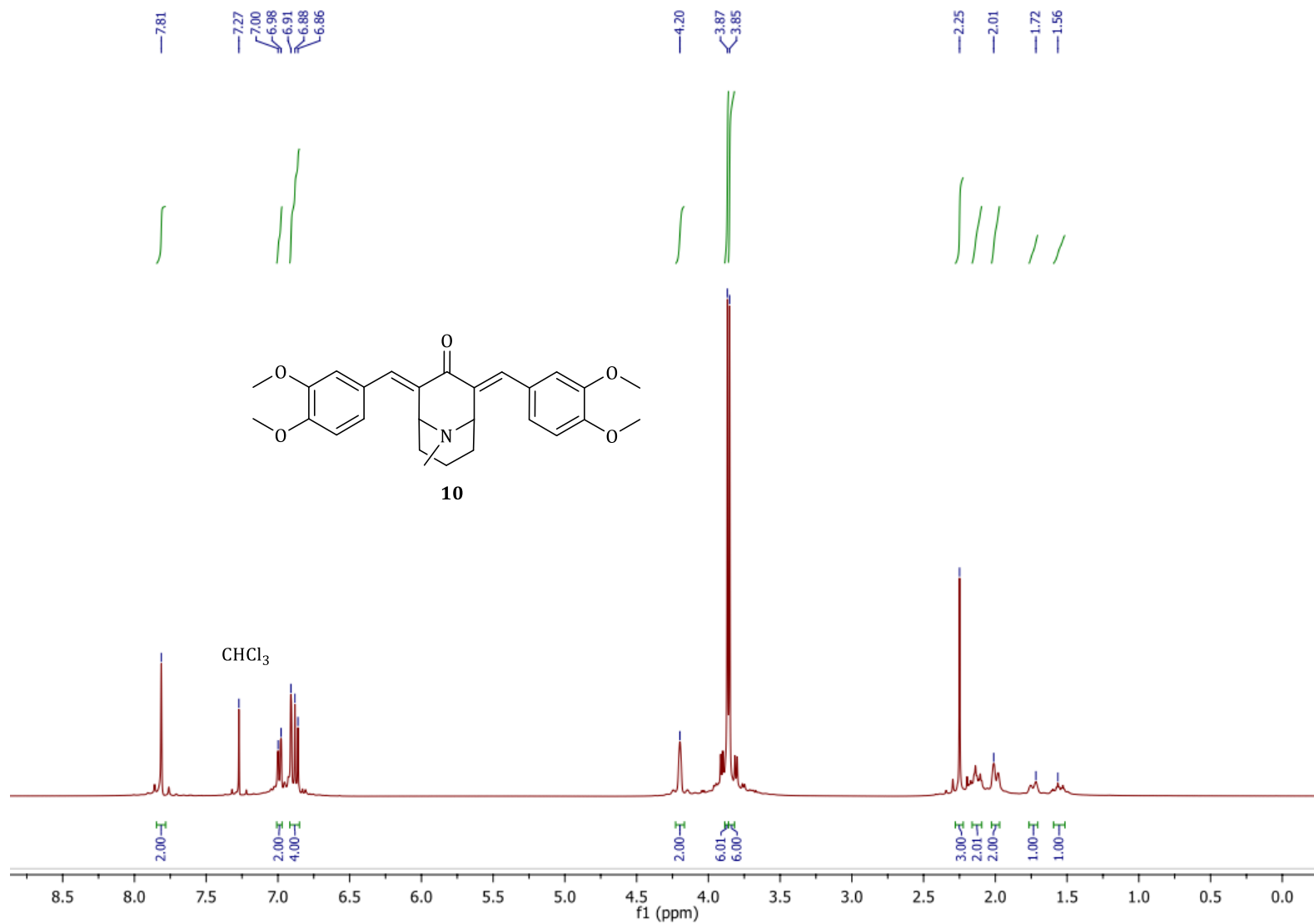


Figure S28. ¹H NMR (400 MHz, CDCl₃): 2,4-Bis((E)-3,4-dimethoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**10**)

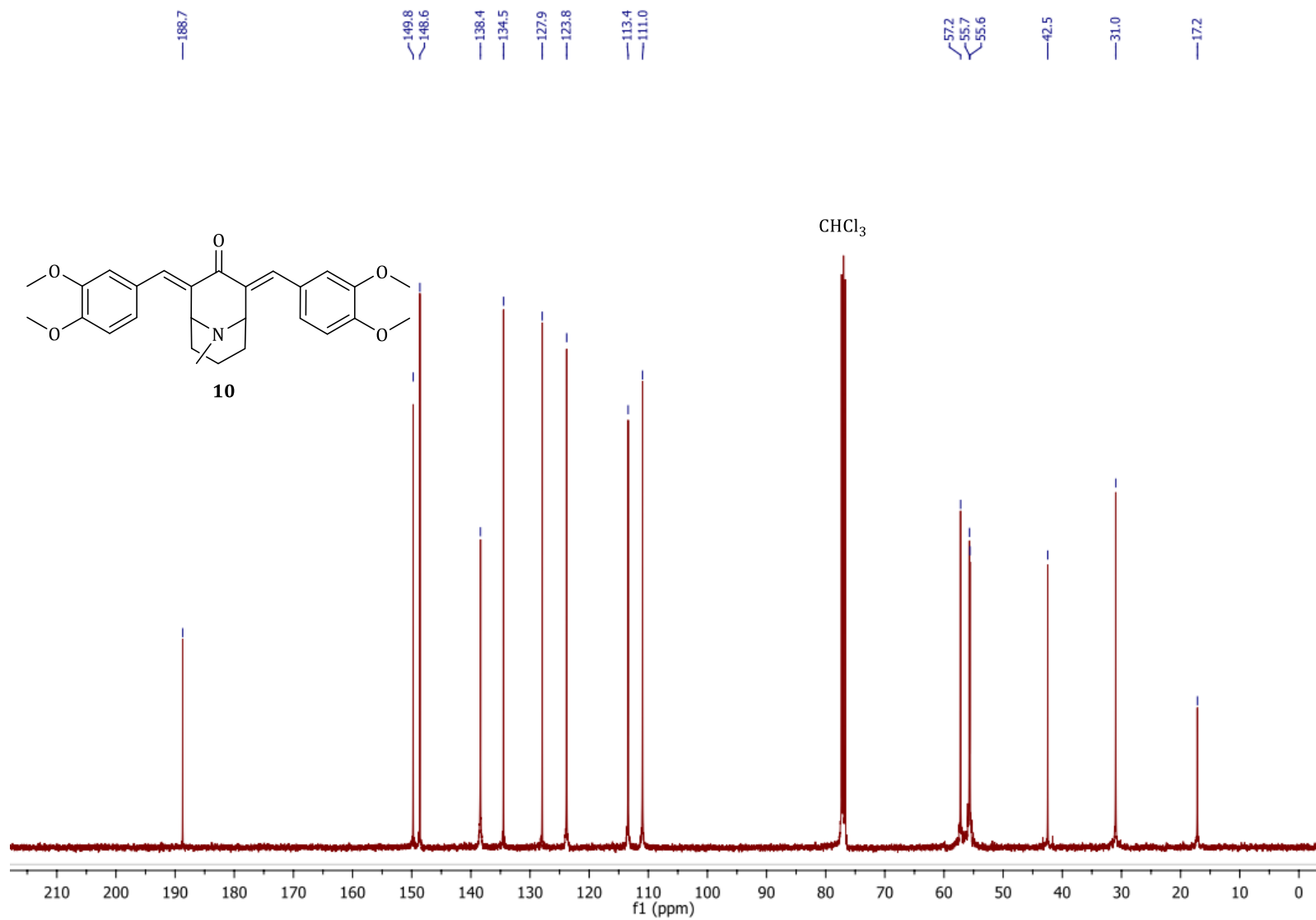


Figure S29. ¹³C NMR (101 MHz, CDCl₃): 2,4-bis((*E*)-3,4-dimethoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**10**)

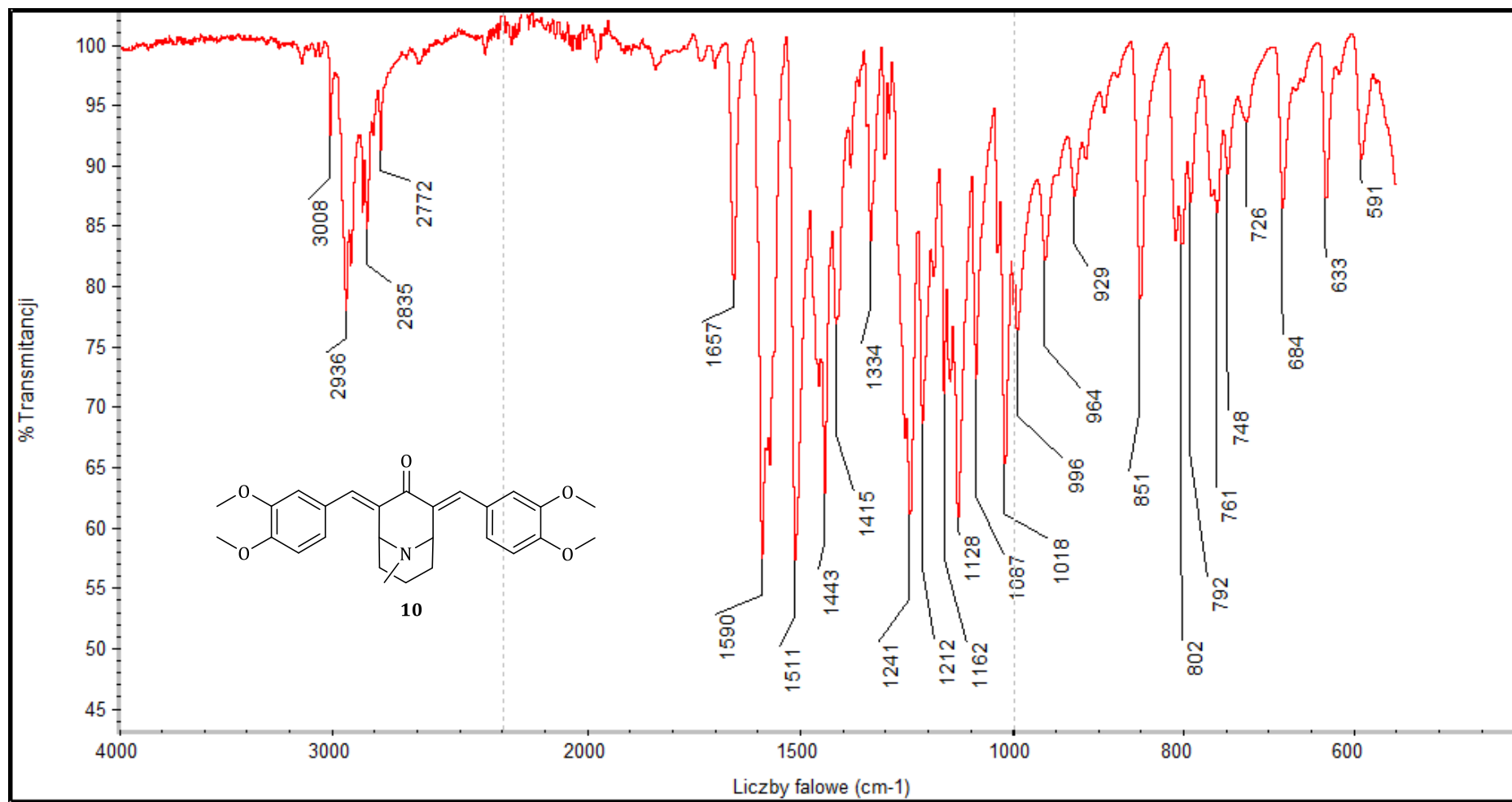


Figure S30. FTIR (ATR): 2,4-bis((*E*)-3,4-dimethoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**10**)

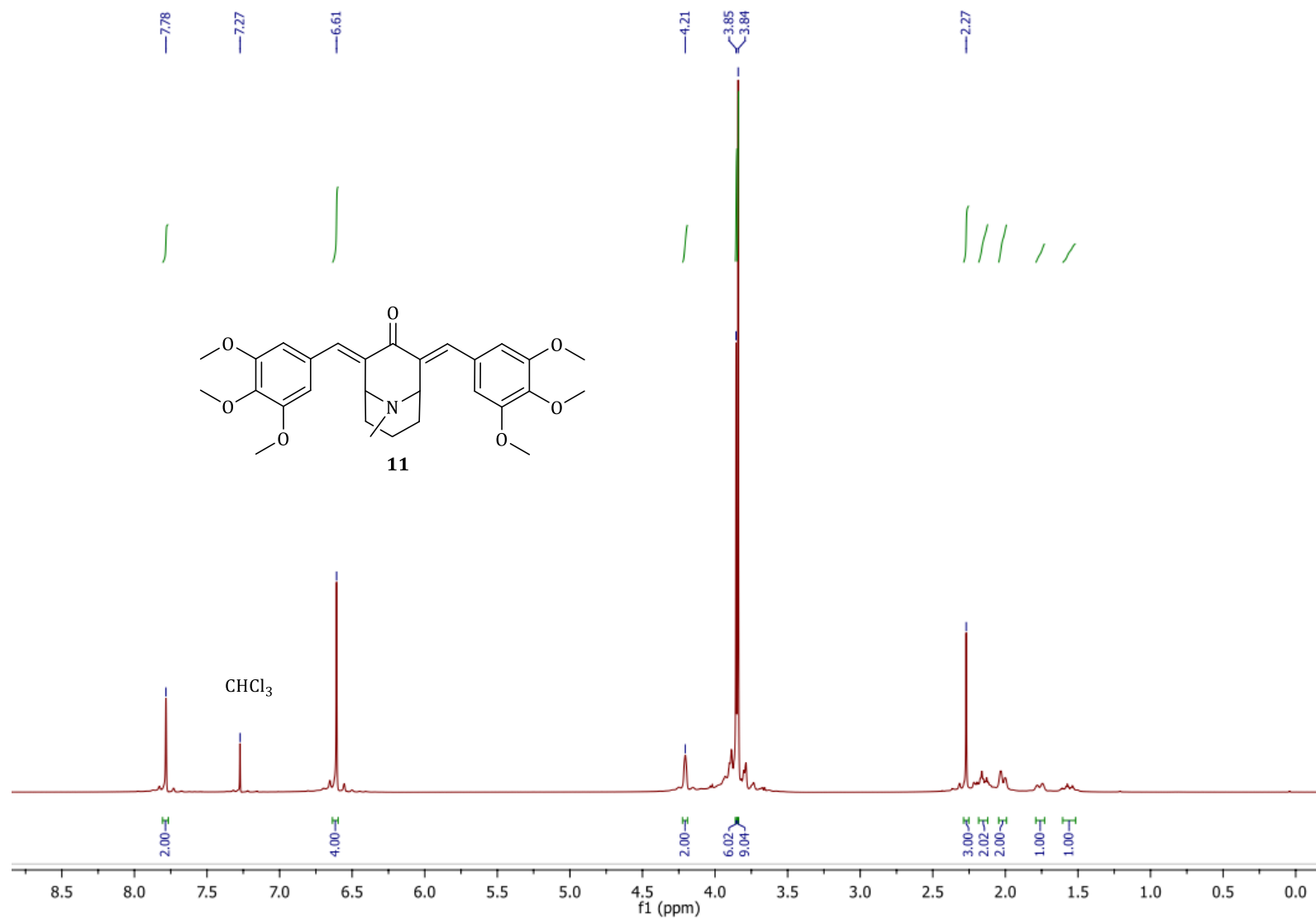


Figure S31. ¹H NMR (400 MHz, CDCl₃): 9-Methyl-2,4-bis((*E*)-3,4,5-trimethoxybenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (**11**)

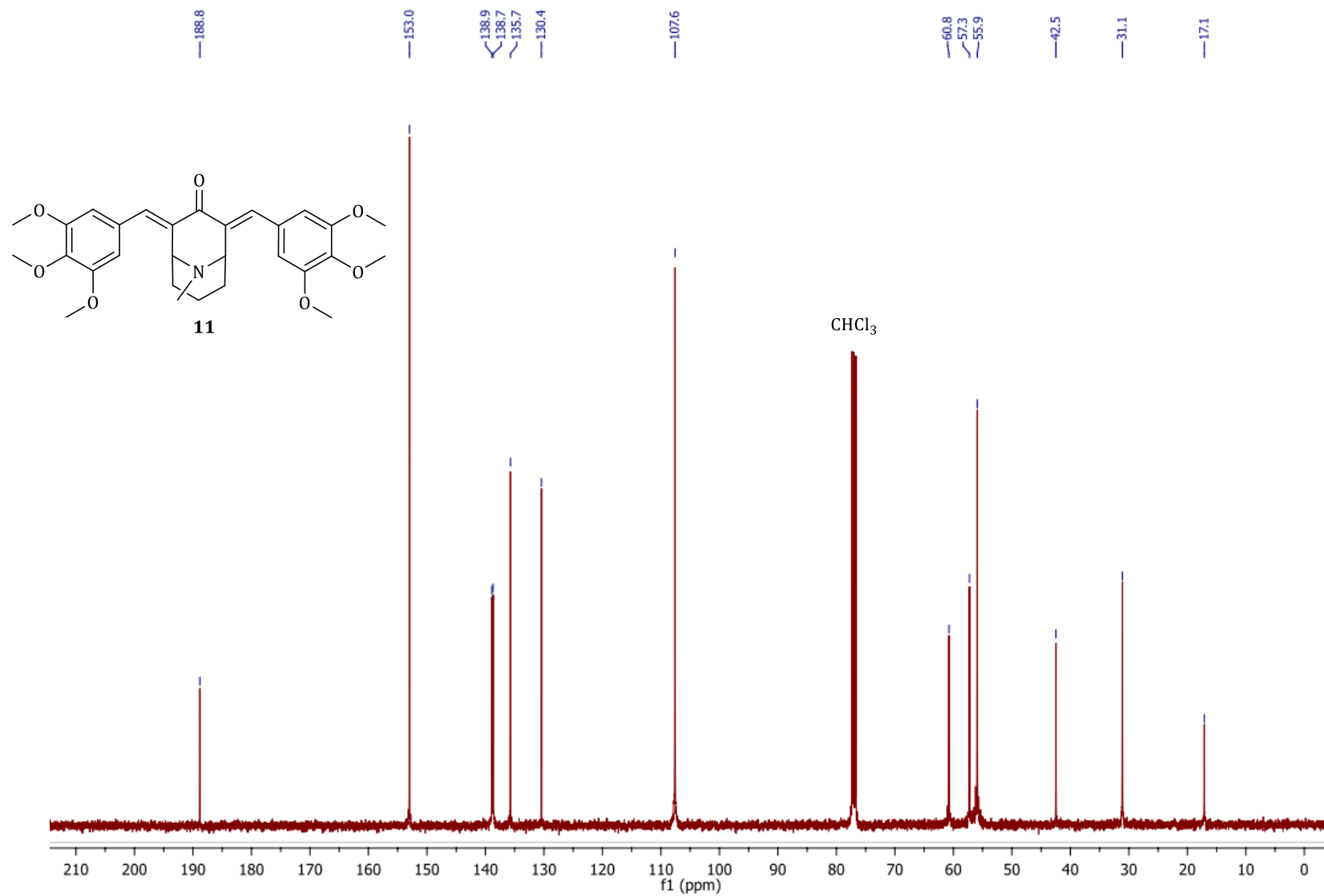


Figure S32. ¹³C NMR (101 MHz, CDCl₃): 9-Methyl-2,4-bis((*E*)-3,4,5-trimethoxybenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (**11**)

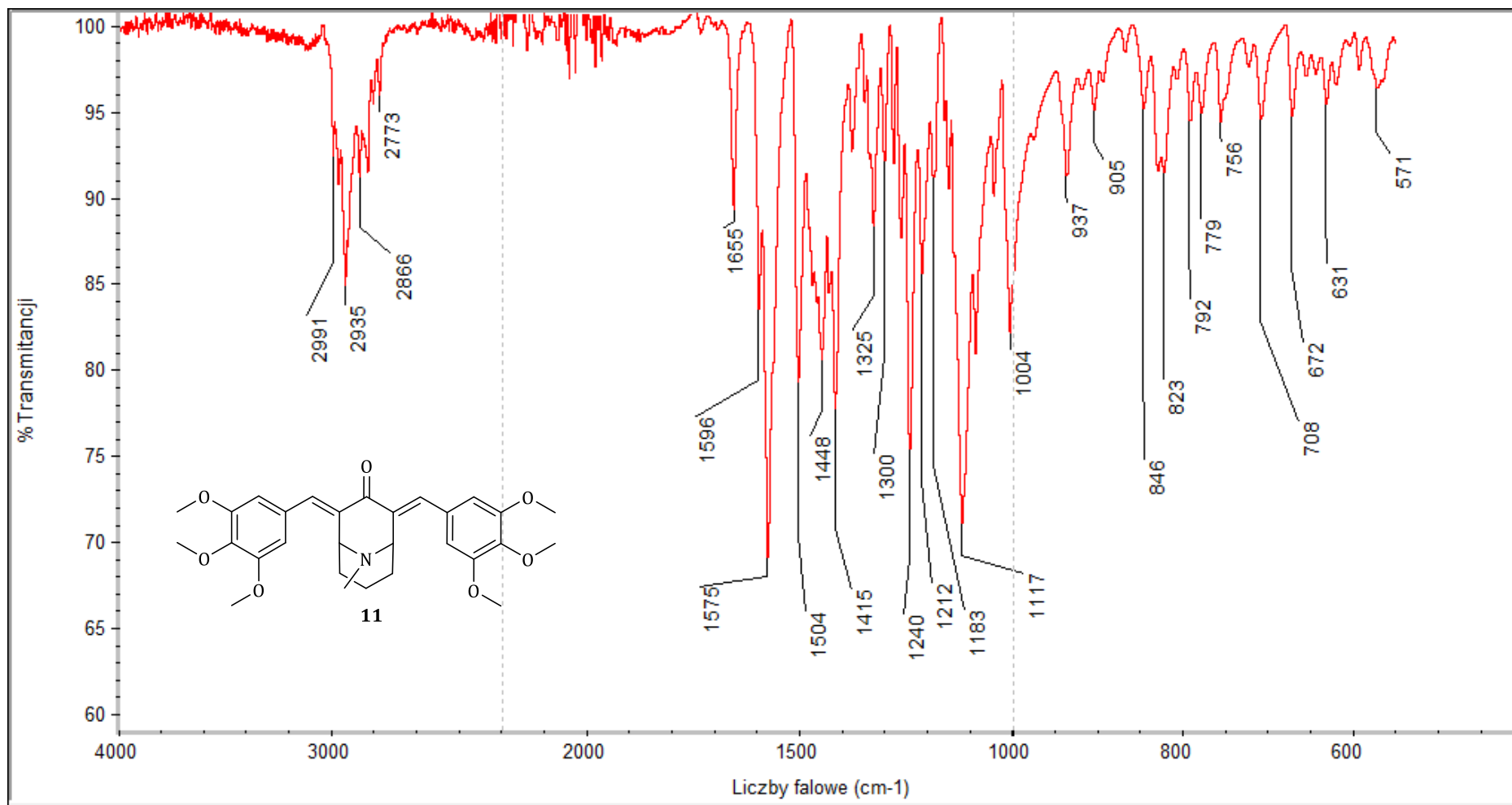


Figure S33. FTIR (ATR): 9-Methyl-2,4-bis((E)-3,4,5-trimethoxybenzylidene)-9-azabicyclo[3.3.1]nonan-3-one (**11**)

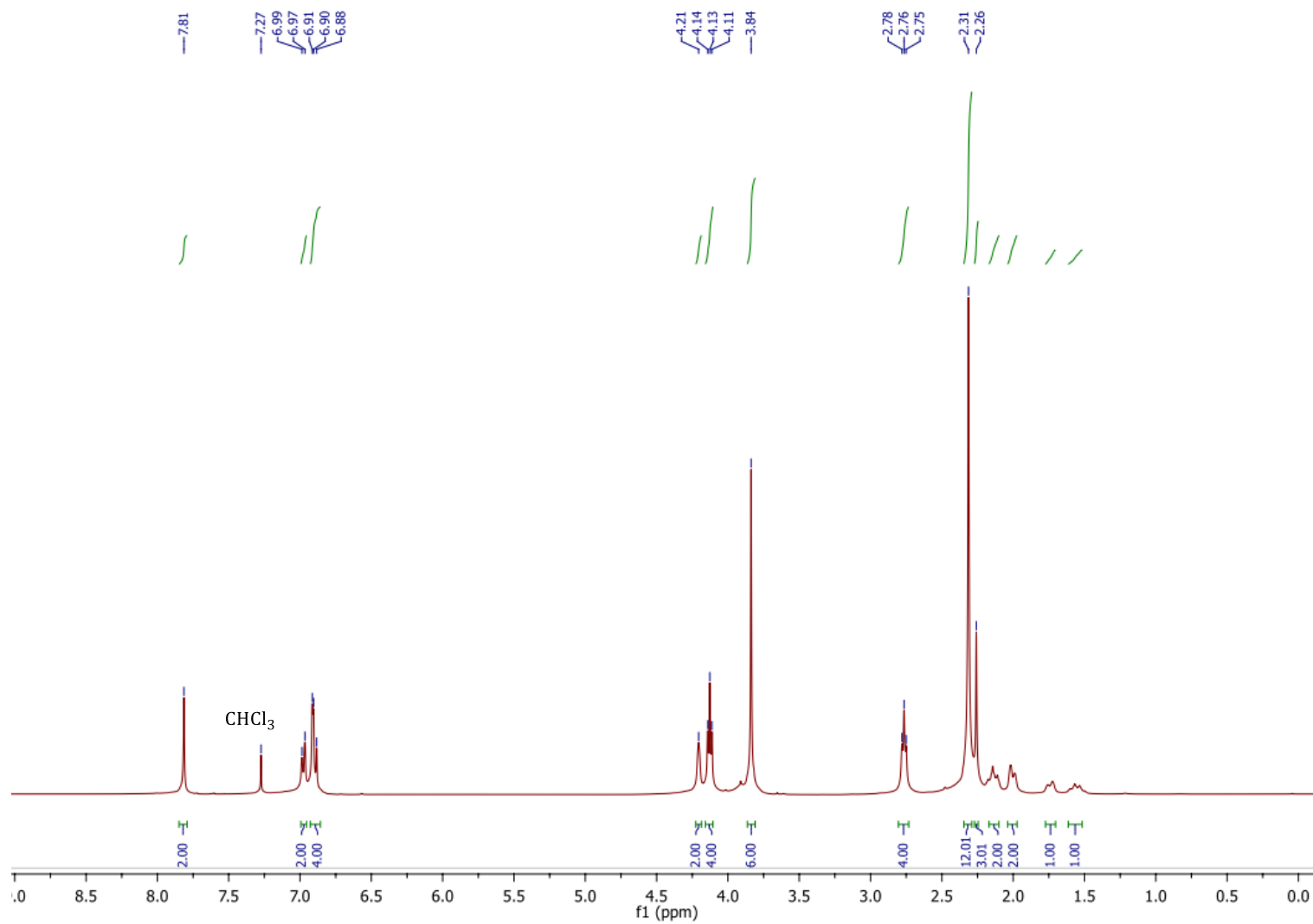


Figure S34. ¹H NMR (400 MHz, CDCl₃): 2,4-Bis((*E*)-4-(2-(dimethylamino)ethoxy)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**12**)

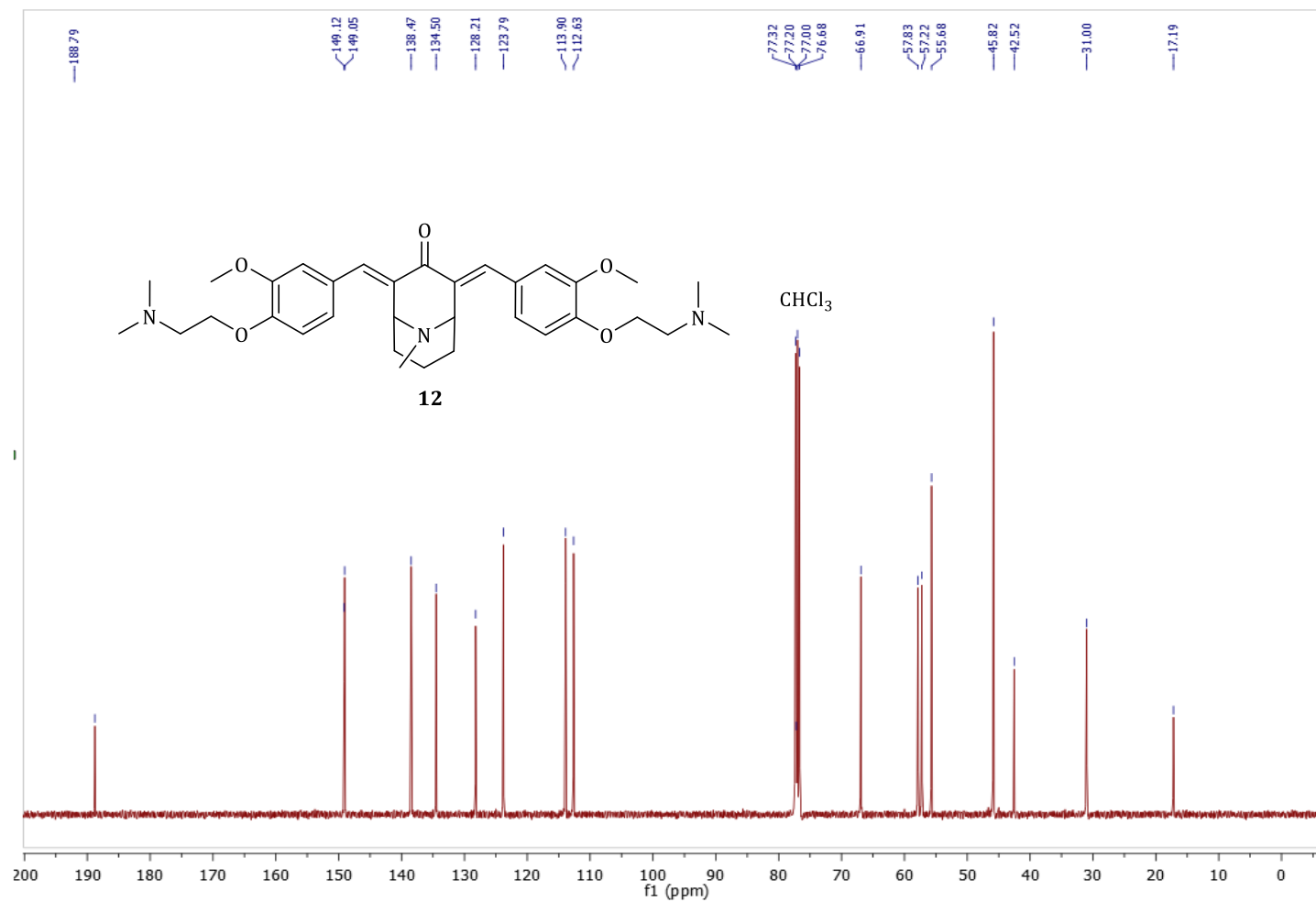


Figure S35. ¹³C NMR (101 MHz, CDCl₃): 2,4-Bis((*E*)-4-(2-(dimethylamino)ethoxy)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**12**)

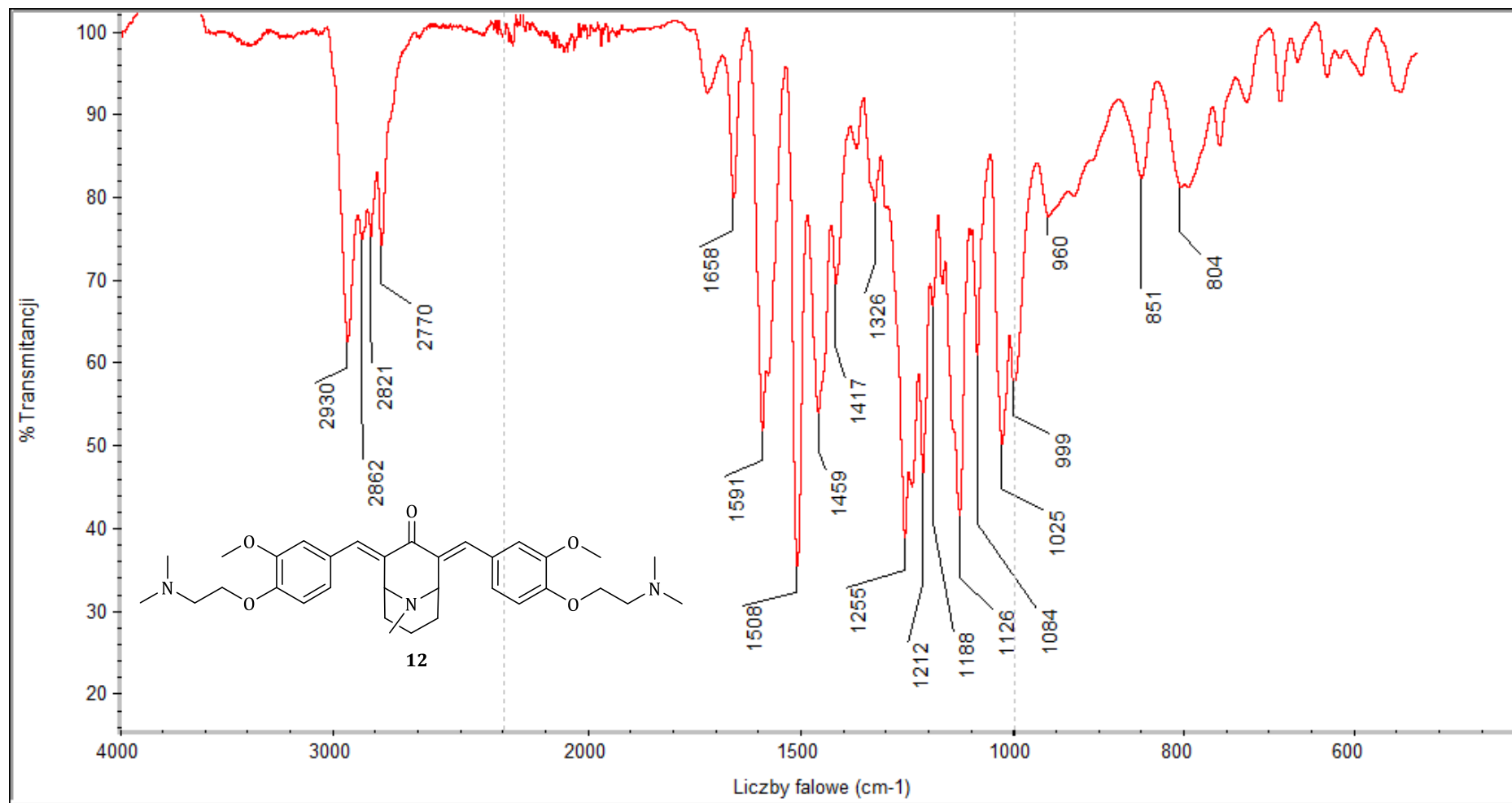


Figure S36. FTIR (ATR): 2,4-Bis((*E*)-4-(2-(dimethylamino)ethoxy)-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**12**)

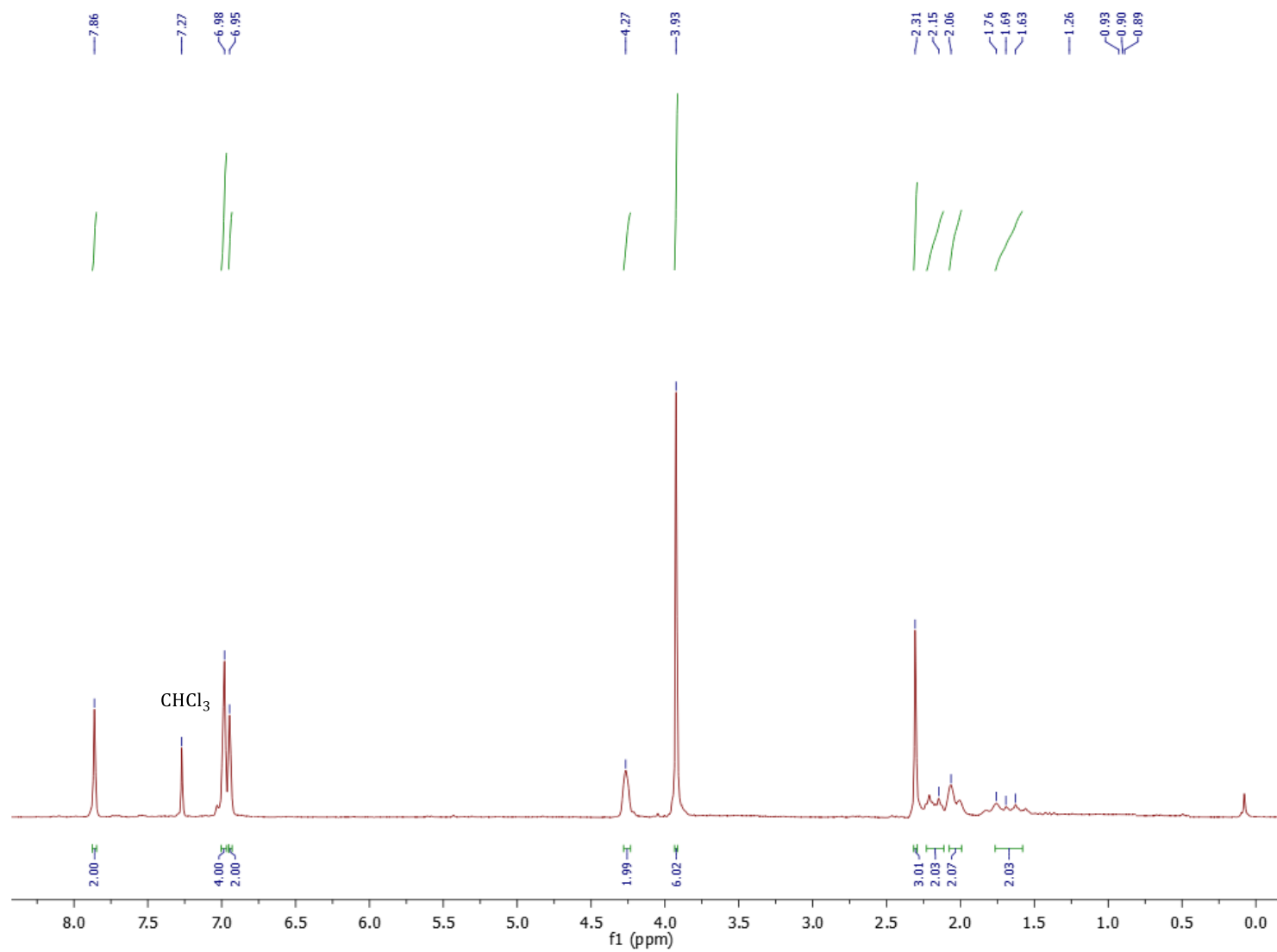


Figure S37. ¹H NMR (200 MHz, CDCl₃): 2,4-Bis((*E*)-4-hydroxy-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**13**)

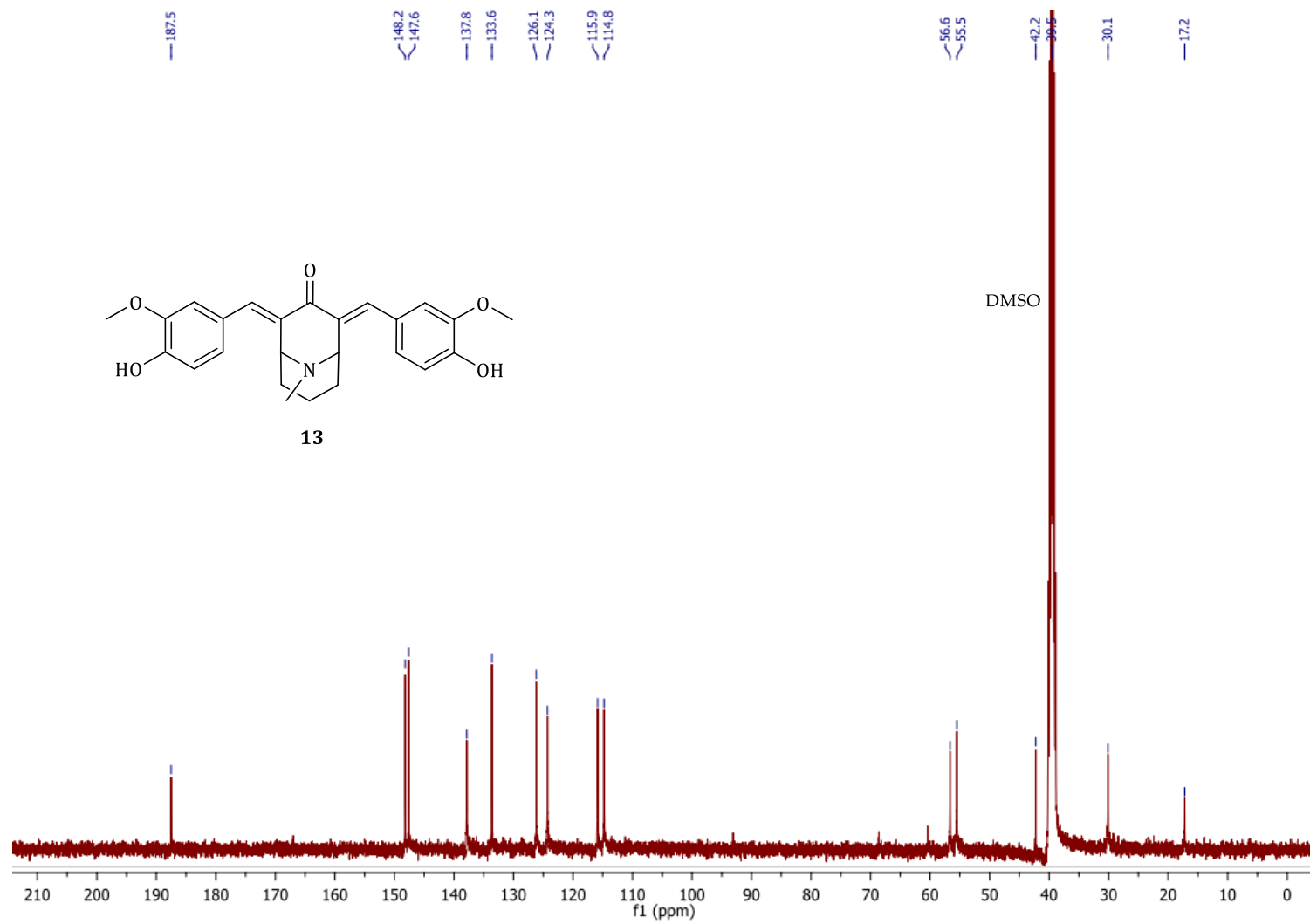


Figure S38. ¹³C NMR (101 MHz, DMSO-*d*₆): 2,4-Bis((*E*)-4-hydroxy-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**13**)

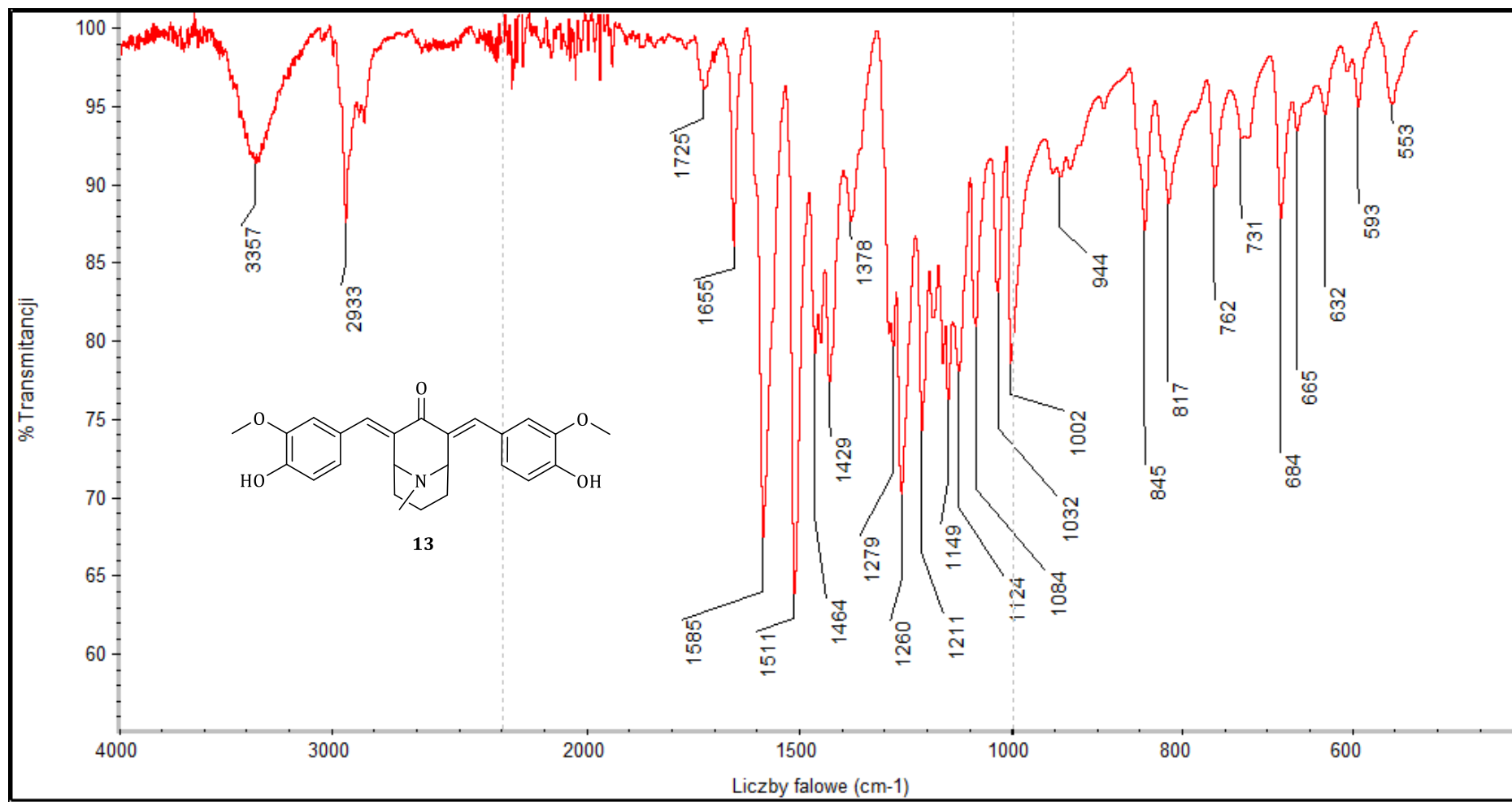


Figure S39. FTIR (ATR): 2,4-Bis((E)-4-hydroxy-3-methoxybenzylidene)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (**13**)

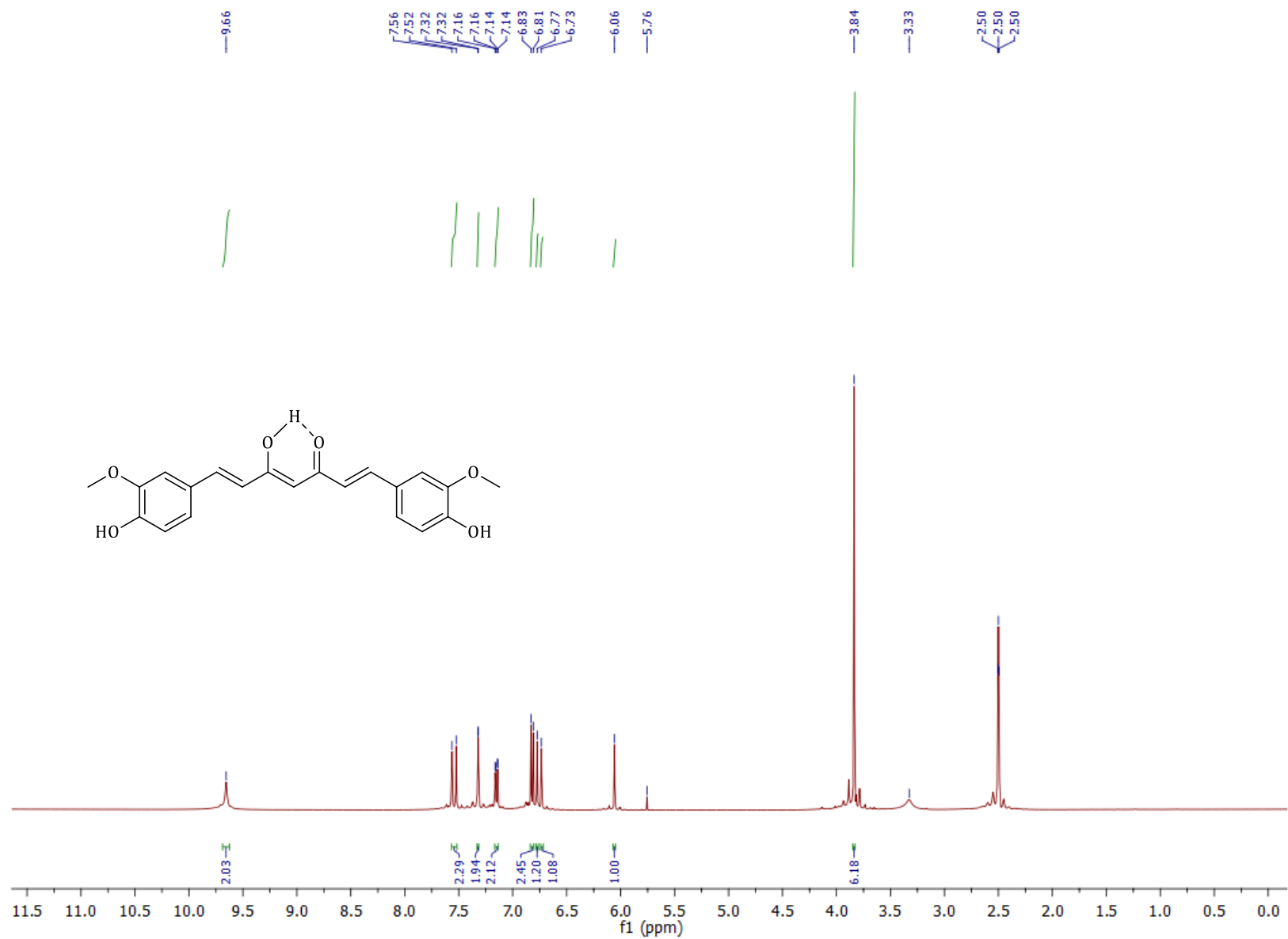


Figure S40. ¹H NMR (200 MHz, DMSO): CUR.

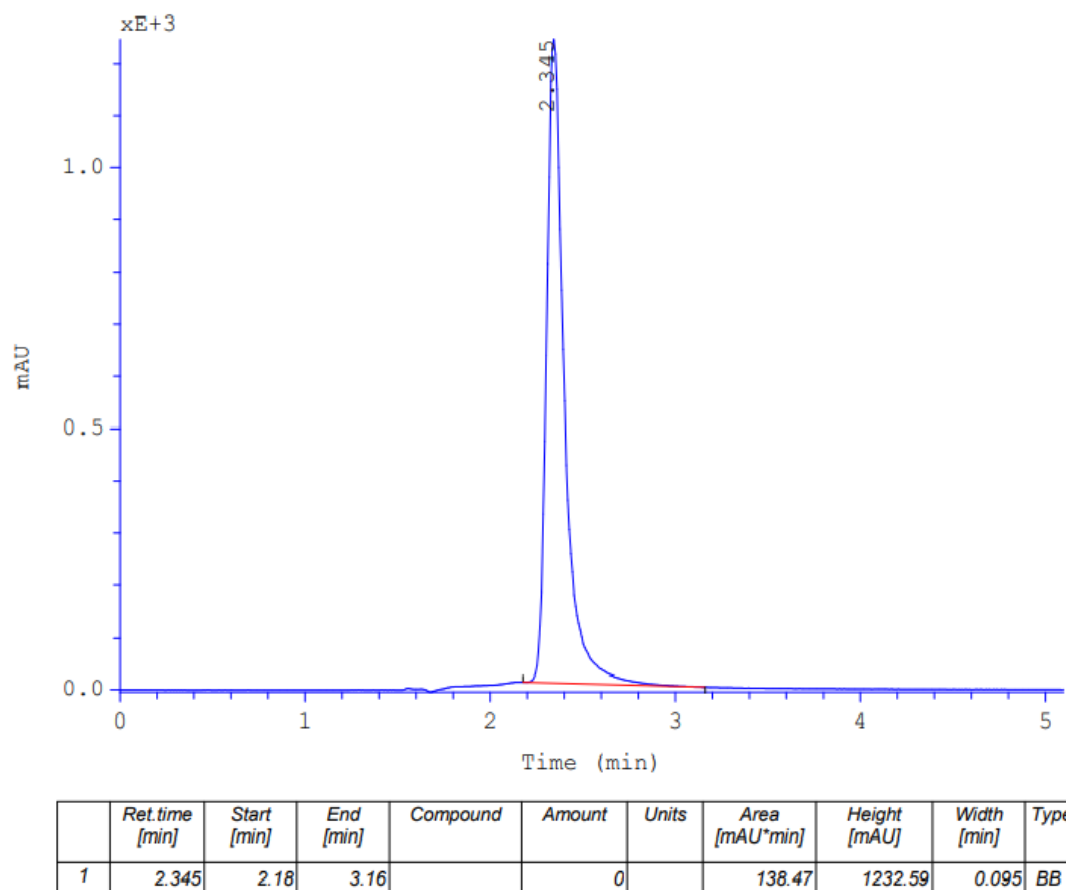


Figure S41. Analysis of the purity of CUR by HPLC. Parameters: Column: Zorbax Eclipse Plus C18, 4.6 x 150 mm, 5 μ m; Eluent: MeOH/H₂O (85/15; v/v), Flow rate: 1 mL/min, 427 nm, Injection volume: 10 μ L; Detector: Knauer DAD K-2800; Pump: Knauer Maxi-Star K-1000; Program: EuroChrom, Version 3.05 P5.

Table S1. Crystal data and structure refinement details for compounds: **3**, **4**, **5**, **6**, **7**, **8**, **10** and **11**.

Dataset	3	4	5	6	7	8	10	11
Formula	C ₂₃ H ₂₁ F ₂ NO	C ₂₃ H ₂₁ Cl ₂ NO	2(C ₂₃ H ₂₁ Br ₂ NO)	C ₂₅ H ₂₁ F ₆ NO	C ₂₃ H ₂₁ N ₃ O ₅	C ₂₅ H ₂₇ NO ₃	C ₂₇ H ₃₁ NO ₅	C ₂₉ H ₃₅ NO ₇
Molecular weight	365.41	398.31	974.45	465.43	419.43	389.48	449.53	509.58
Temperature [K]	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)
Wavelength[Å]	1.54184	1.54184	1.54184	1.54184	1.54184	1.54184	1.54184	1.54184
Crystal system	monoclinic	triclinic	triclinic	monoclinic	triclinic	monoclinic	monoclinic	triclinic
Space group	<i>C</i> 2/c	<i>P</i> -1	<i>P</i> -1	<i>P</i> 2 ₁ / <i>n</i>	<i>P</i> -1	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> -1
Unit cell parameters [Å, °]								
<i>a</i>	33.1187 (6)	6.9779 (2)	11.1114 (2)	16.3392 (2)	7.1824 (3)	15.9091 (2)	25.7000 (3)	10.1179 (5)
<i>b</i>	8.3246 (2)	7.0132 (2)	12.2568 (2)	6.9282 (1)	8.0268 (3)	18.1459 (3)	6.7437 (1)	10.8629 (6)
<i>c</i>	13.4451 (2)	21.6817 (5)	17.2501 (2)	18.8687 (2)	19.3231 (9)	6.9504 (1)	13.7240 (2)	11.9565 (9)
<i>α</i>	90	83.390 (2)	97.579 (1)	90	97.229 (4)	90	90	87.177 (5)
<i>β</i>	95.679 (2)	85.489 (2)	105.099 (1)	98.624 (1)	93.509 (4)	92.436 (1)	102.310 (1)	80.806 (5)
<i>γ</i>	90	63.120 (2)	115.166 (2)	90	116.419 (4)	90	90	78.120 (4)
Volume [Å ³]	3688.61 (11)	939.69 (5)	1972.14 (6)	2111.82 (4)	981.09 (8)	2004.66 (5)	2323.88 (5)	1269.29 (13)
Z	8	2	2	4	2	4	4	2
Absorption coefficient [mm ⁻¹]	0.77	3.20	5.31	1.09	0.84	0.67	0.71	0.78
F(000)	1536	416	976	960	440	832	960	544
Crystal size [mm ³]	0.18×0.16×0.06	0.11×0.11×0.03	0.16×0.11×0.06	0.54×0.38×0.07	0.11×0.02×0.02	0.58×0.30×0.04	0.52×0.36×0.05	0.16×0.14×0.02
θ range for data collection [°]	2.7 to 78.5	2.1 to 78.1	2.8 to 78.5	3.3 to 78.2	2.3 to 75.8	2.8 to 75.8	3.5 to 78.5	3.8 to 75.9
	−42<=h<=42	−8<=h<=7	−14<=h<=14	−20<=h<=20	−8<=h<=8	−16<=h<=17	−16<=h<=17	−9<=h<=12
Index ranges	−10<=k<=1	−8<=k<=8	−15<=k<=15	−8<=k<=8	−10<=k<=10	−18<=k<=18	−18<=k<=18	−13<=k<=13
	−8<=l<=16	−27<=l<=27	−21<=l<=21	−23<=l<=23	−7<=l<=23	−20<=l<=20	−20<=l<=20	−15<=l<=14
Reflections collected	20228	19911	31519	23095	11176	19392	24279	14723
Independent reflections, <i>R</i> _{int}	3923, 0.025	3970, 0.076	8277, 0.062	4492, 0.058	3893, 0.057*	4062, 0.065	4831, 0.041	5076, 0.043
Data / restraints / parameters	3923 / 0 / 246	3970 / 0 / 245	8277 / 0 / 490	4492 / 36 / 356	3893 / 0 / 282*	4062 / 0 / 266	4831 / 0 / 304	5076 / 0 / 342
Goodness-of-fit on <i>F</i> ²	1.06	1.04	1.07	1.05	1.25*	1.04	1.04	1.06
Final <i>R</i> indices (all data)	<i>R</i> 1 = 0.037 w <i>R</i> 2 = 0.099	<i>R</i> 1 = 0.050 w <i>R</i> 2 = 0.139	<i>R</i> 1 = 0.054 w <i>R</i> 2 = 0.149	<i>R</i> 1 = 0.045 w <i>R</i> 2 = 0.120	<i>R</i> 1 = 0.085* w <i>R</i> 2 = 0.254*	<i>R</i> 1 = 0.048 w <i>R</i> 2 = 0.130	<i>R</i> 1 = 0.041 w <i>R</i> 2 = 0.110	<i>R</i> 1 = 0.052 w <i>R</i> 2 = 0.143
Largest diff. peak and hole [e·Å ⁻³]	0.27 and -0.20	0.65 and -0.49	1.43 and -1.03	0.47 and -0.34	0.40 and -0.38*	0.39 and -0.32	0.39 and -0.22	0.74 and -0.34
CCDC number	2096435	2097003	2097005	2096436	2097179	2097004	2097006	2096434

* Values correspond to an analysis and refinement based on crystallographic data in HKLF 5 format generated by *TwinRotMat* program.

Table S2. Solubility (S) in distilled water at 21 ± 1°C

Compounds	S (g/L)	S (mmol/L)
3	0.026	0.070
4	0.024	0.060
5	0.032	0.065
6	0.048	0.103
7	0.012	0.029
8	0.293	0.751
9	0.034	0.088
10	0.095	0.212
11	0,108	0.211
12	1.500	2.661
12A (12 x 3 HCl-salt)	448.0	665.9
13	1.901	4.508