

One-Pot Aldol Cascade for the Preparation of Isospiropyrans, Flavylum Salts and *bis*-Spiropyrans

Taylor A. Neal,^[a] Allyson B. Eippert,^[a] Austin Chivington,^[b] Andrew Jamison,^[b] Chukwunalu Chukwuma,^[b] Curtis E. Moore,^[a] Jennifer F. Neal,^[a] Heather C. Allen,^[a] Laura M. Wysocki,^[b] Noel M. Paul,^[a] Jovica D. Badjić*^[a]

^[a]Department of Chemistry and Biochemistry, The Ohio State University, 100 West 18th Avenue, Columbus Ohio 43210 (USA)

E-mail: badjic.1@osu.edu

^[b]Department of Chemistry, Wabash College, 301 W. Wabash Avenue, Crawfordsville, IN 47933.

SUPPORTING INFORMATION

Table of Contents

1. Spectroscopic data.....	S1-S28
2. UV-Vis spectroscopy.....	S29
3. Crystallographic Data	S30-S49

Spectroscopic Data

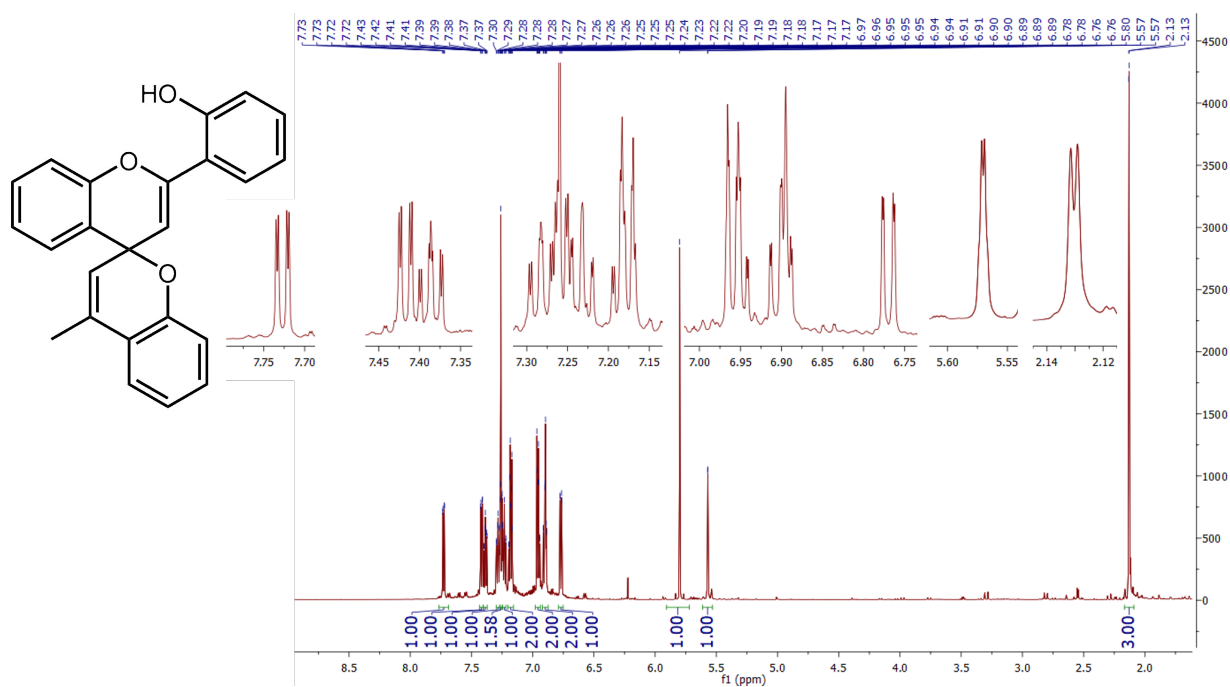


Figure S1. ¹H NMR spectrum (600 MHz, 298 K) of isospiropyran **11** in CDCl₃.

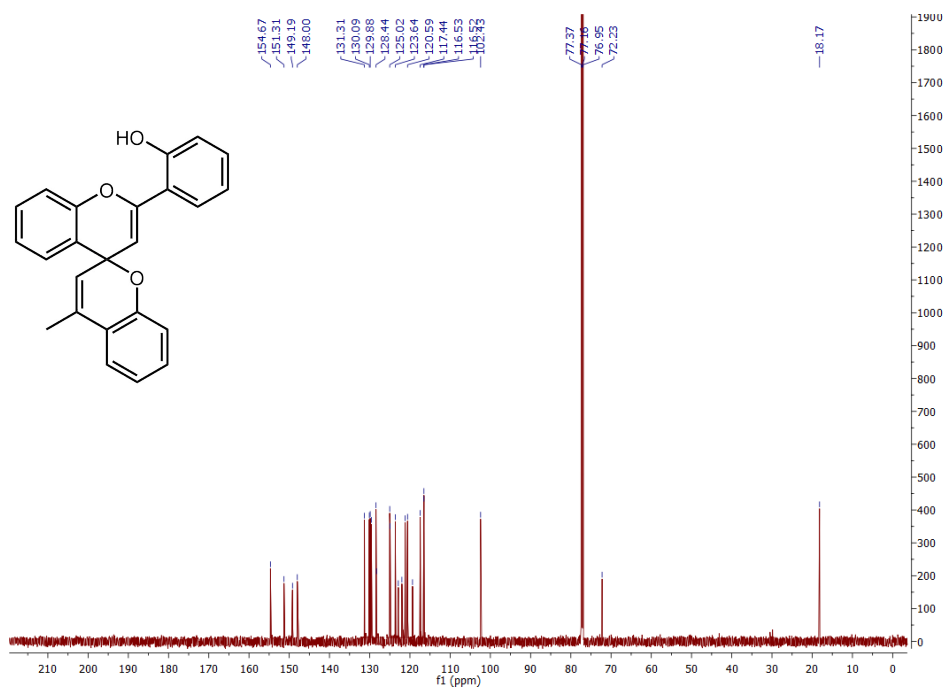


Figure S2. ¹³C NMR spectrum (150 MHz, 298 K) of isospiropyran **11** in CDCl₃.

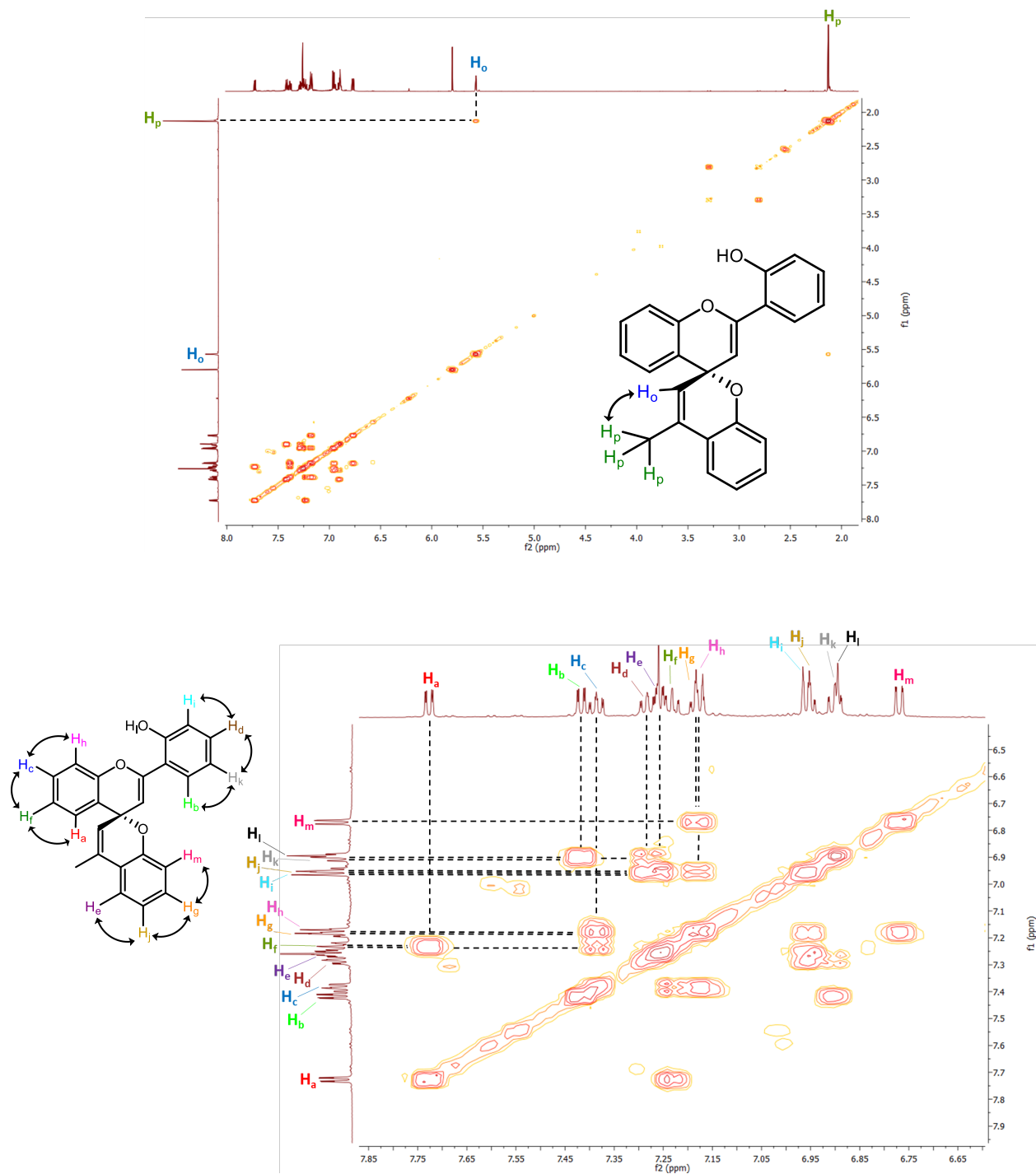


Figure S3. Two different regions of ^1H - ^1H COSY NMR (600 MHz, 298 K) spectrum of isospiropyran **11** in CDCl_3 .

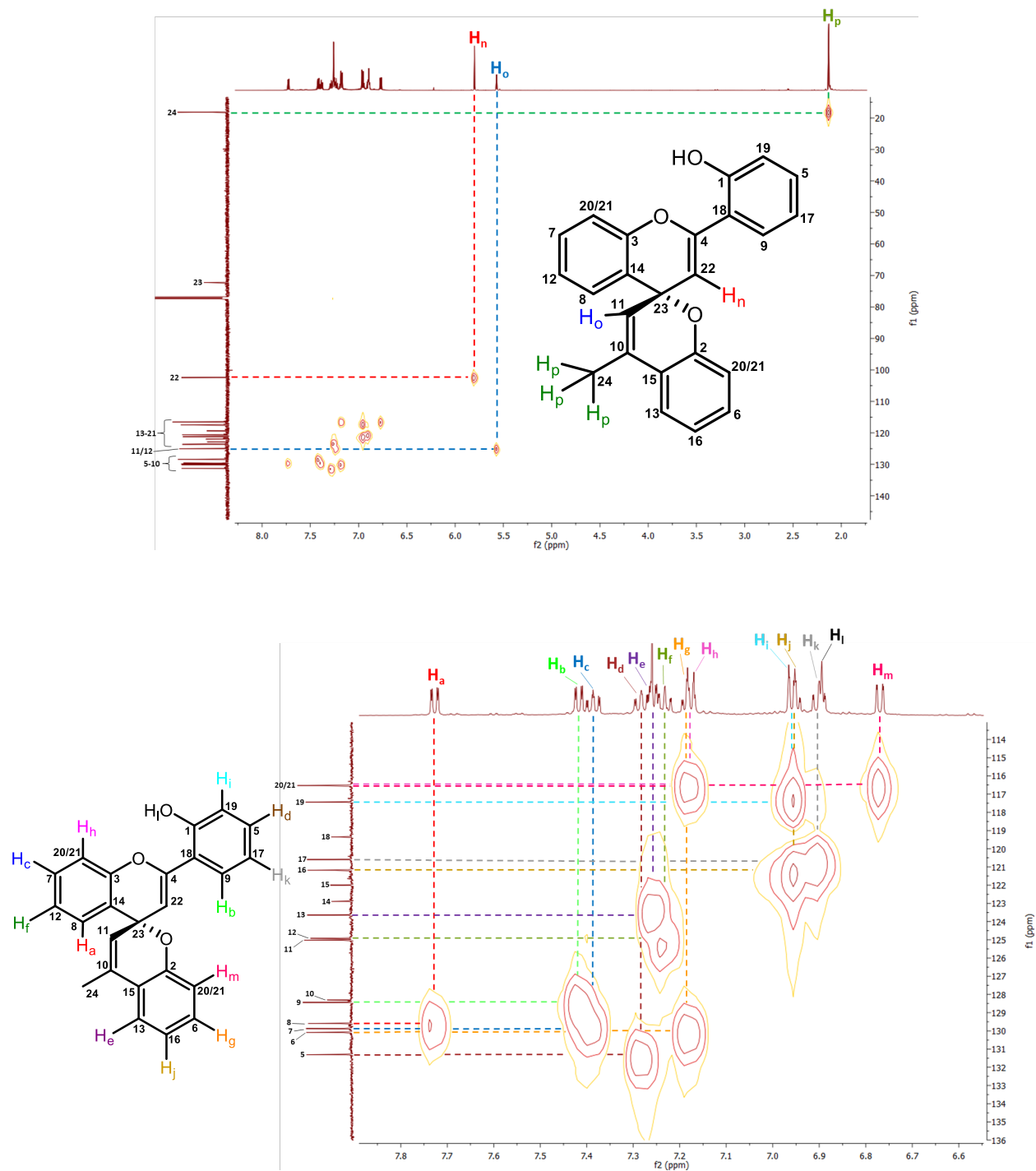


Figure S4. Two different regions of ^1H - ^{13}C HSQC NMR (600 MHz, 298 K) spectrum of isospiropyran **11** in CDCl_3 .

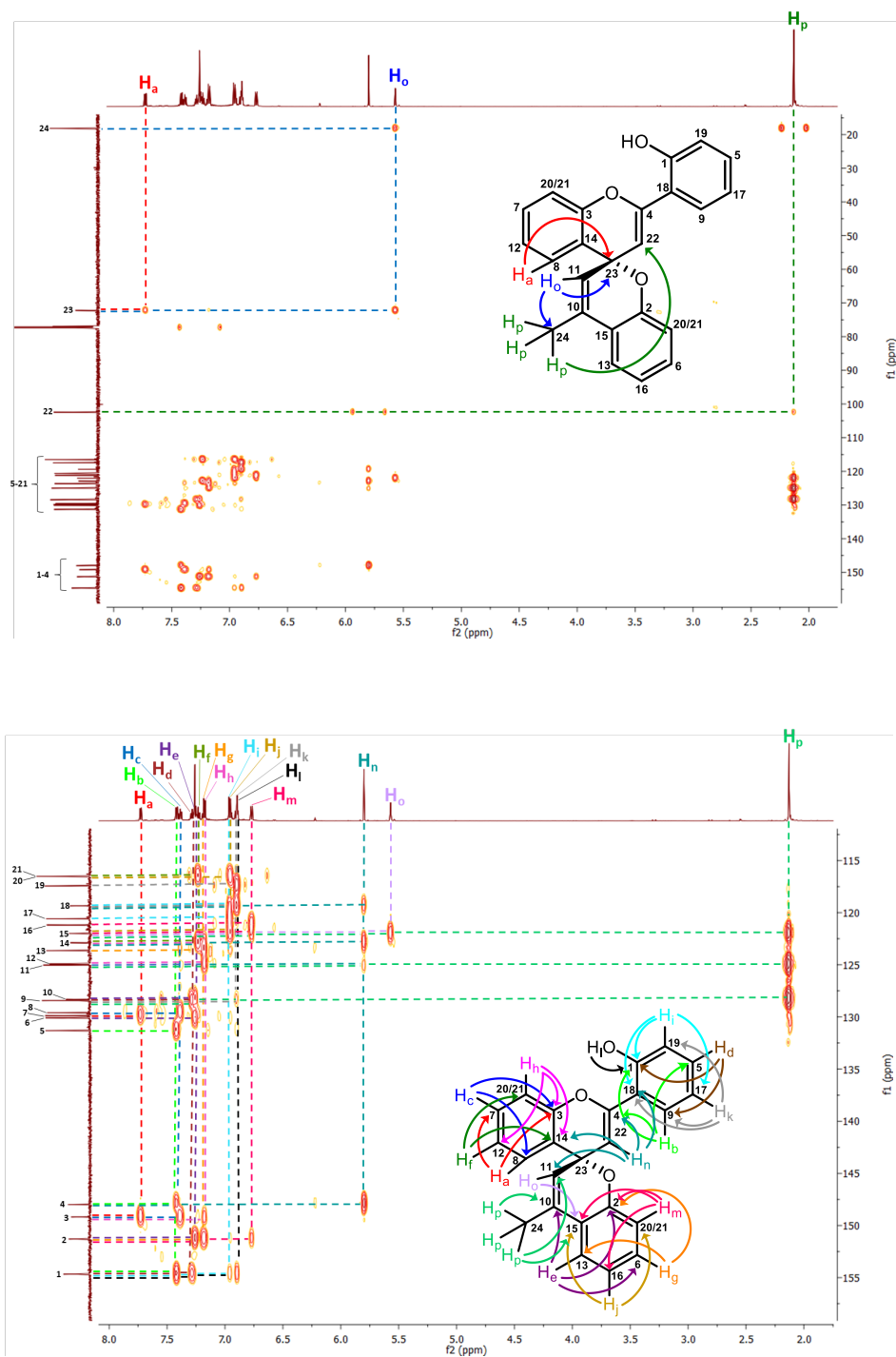


Figure S5. Two different regions of ^1H - ^{13}C HMBC NMR (600 MHz, 298 K) spectrum of isopiripyrans **11** in CDCl_3 .

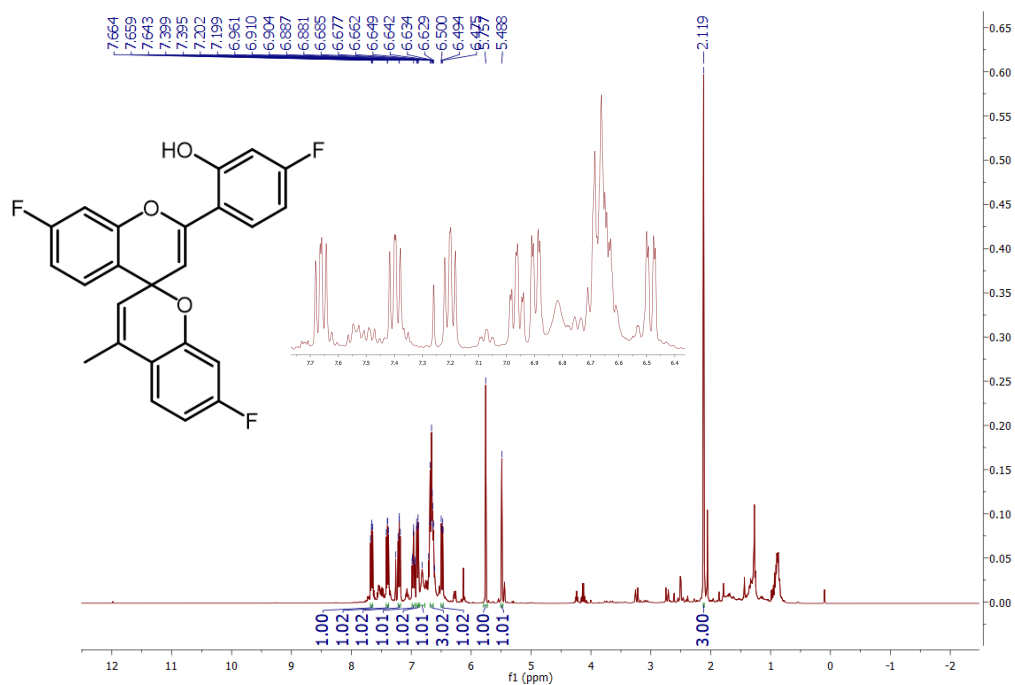


Figure S6. ^1H NMR spectrum (400 MHz, 298 K) of isospiropyran **12** in CDCl_3 .

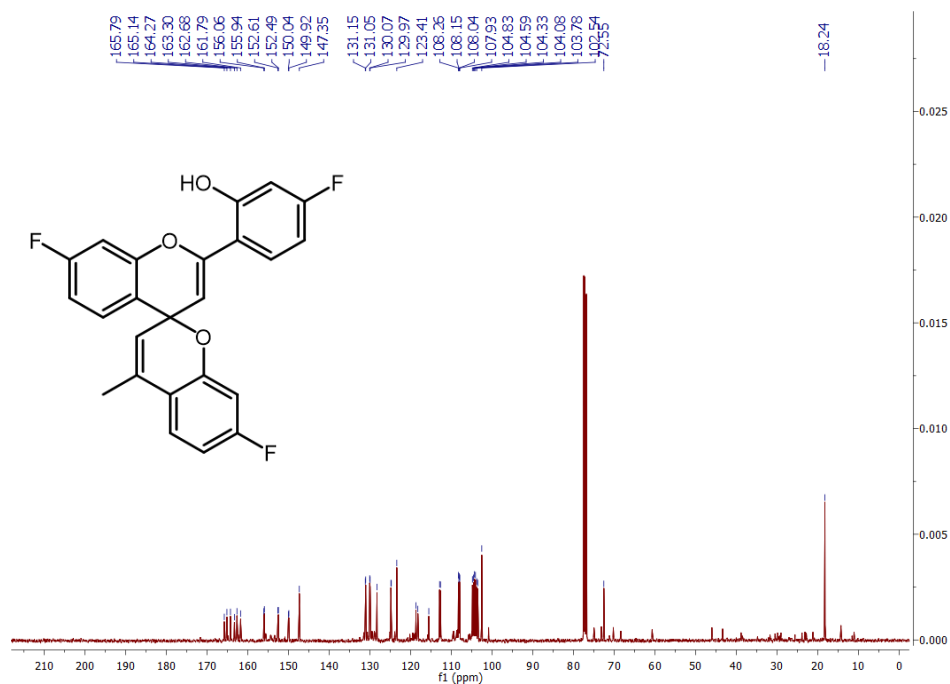


Figure S7. ^{13}C NMR spectrum (100 MHz, 298 K) of isospiropyran **12** in CDCl_3 .

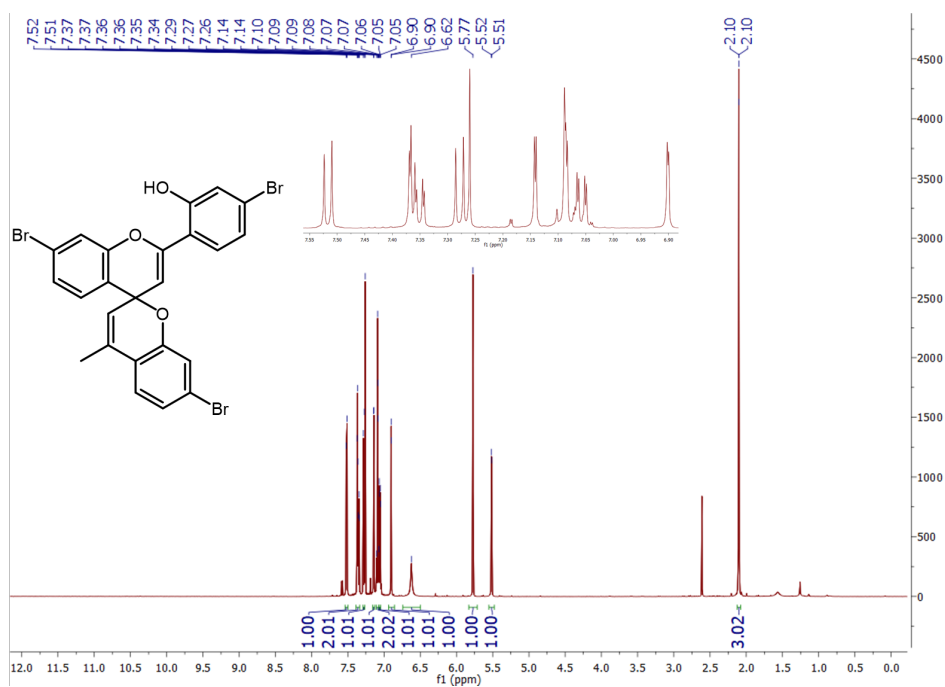


Figure S8. ¹H NMR spectrum (600 MHz, 298 K) of isospiropyran **13** in CDCl₃.

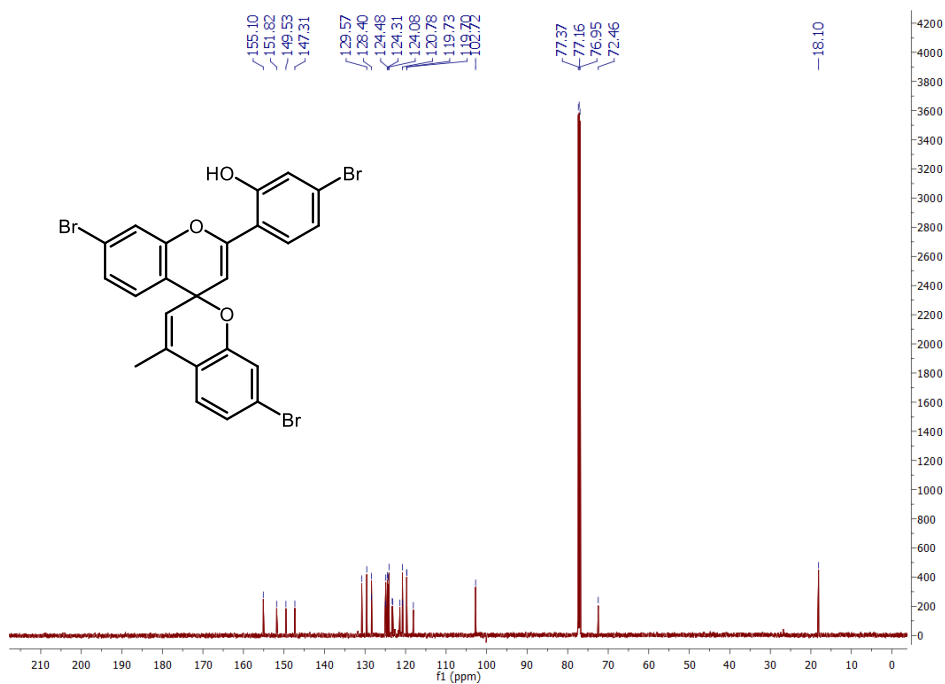


Figure S9. ¹³C NMR spectrum (150 MHz, 298 K) of isospiropyran **13** in CDCl₃.

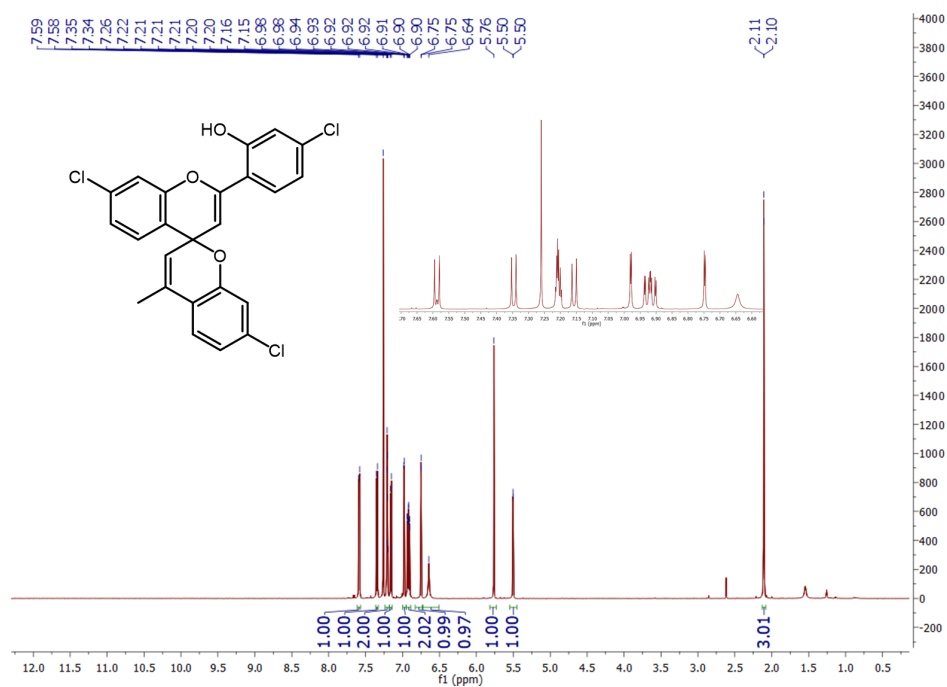


Figure S10. ^1H NMR spectrum (600 MHz, 298 K) of isospiropyran **14** in CDCl_3 .

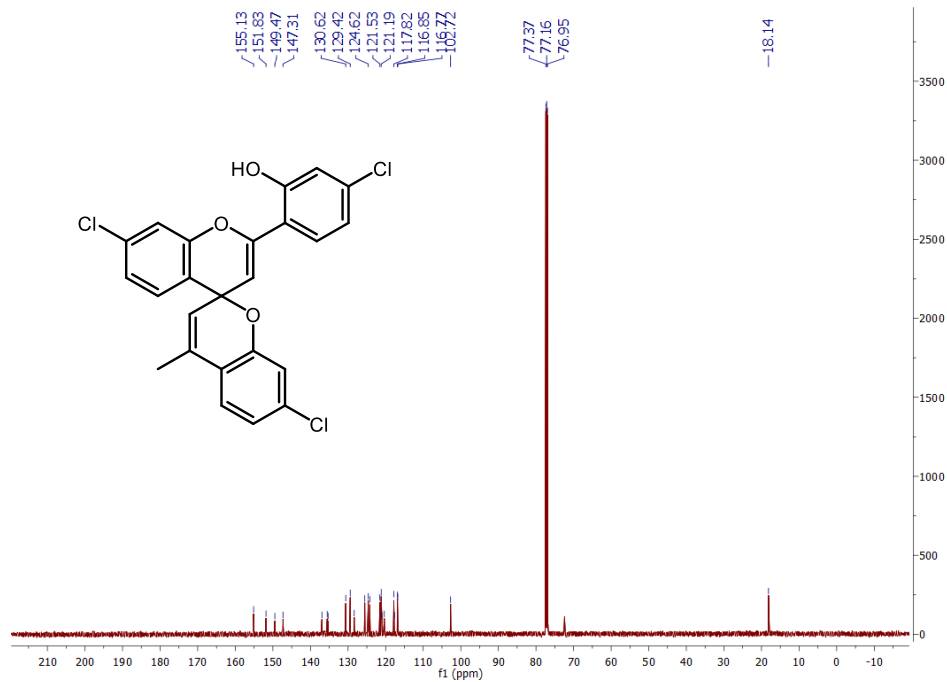
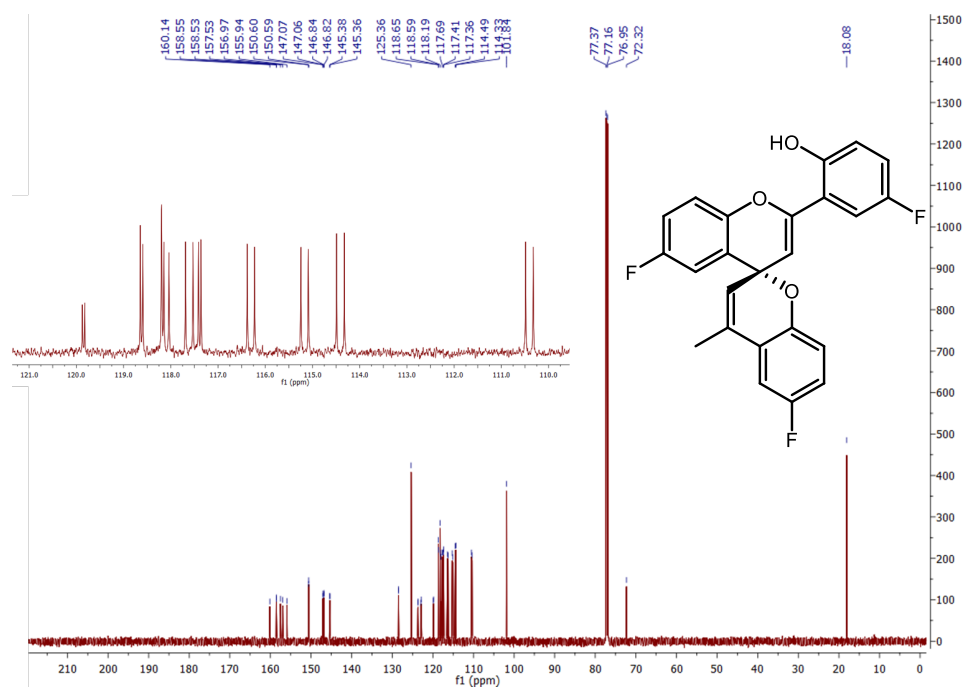
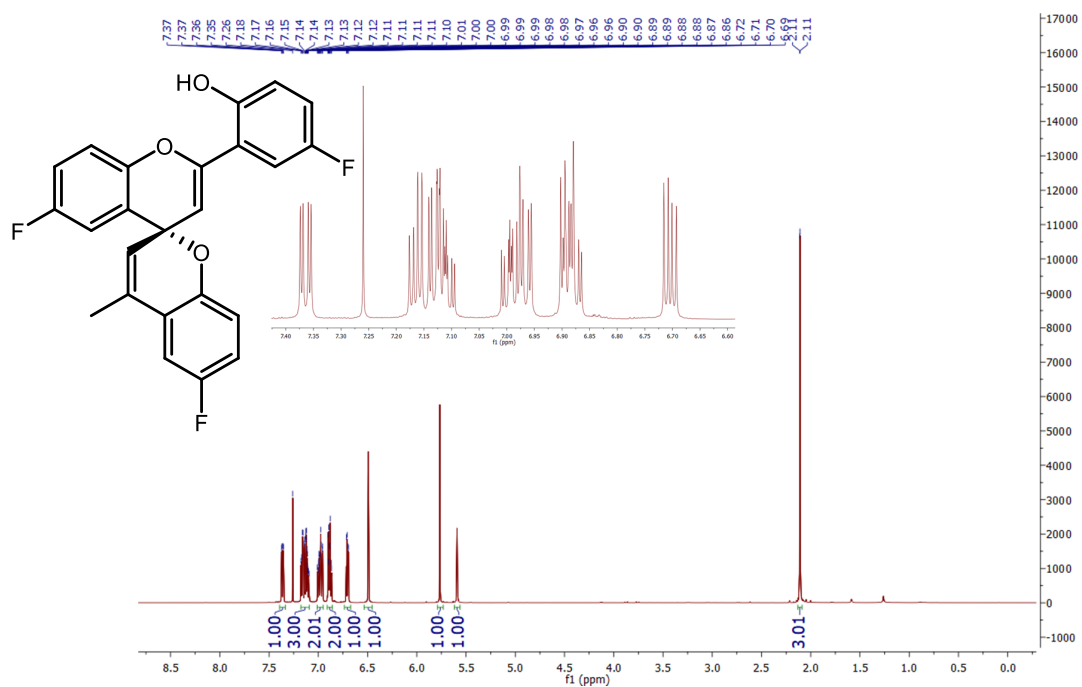


Figure S11. ^{13}C NMR spectrum (150 MHz, 298 K) of isospiropyran **14** in CDCl_3 .



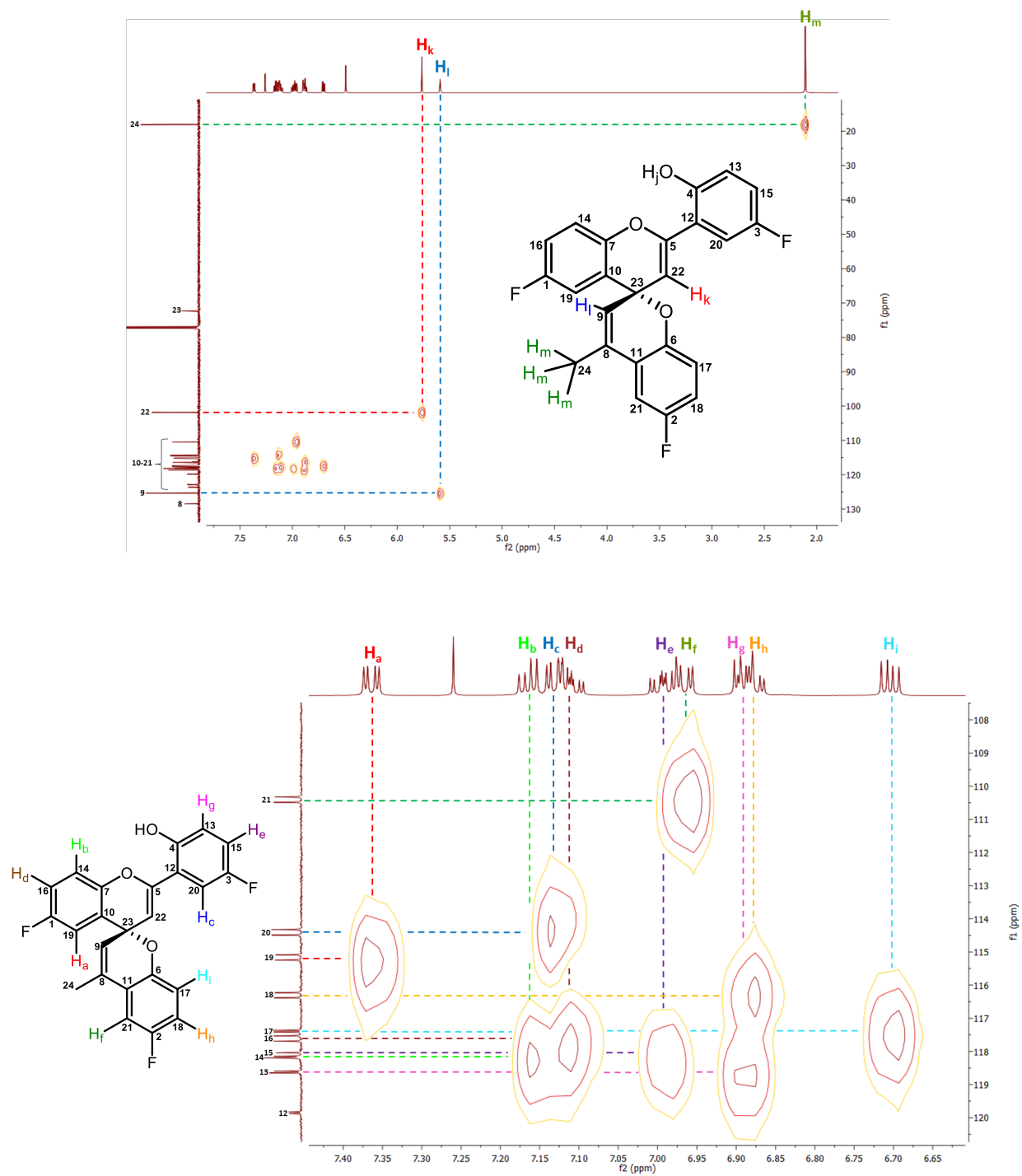
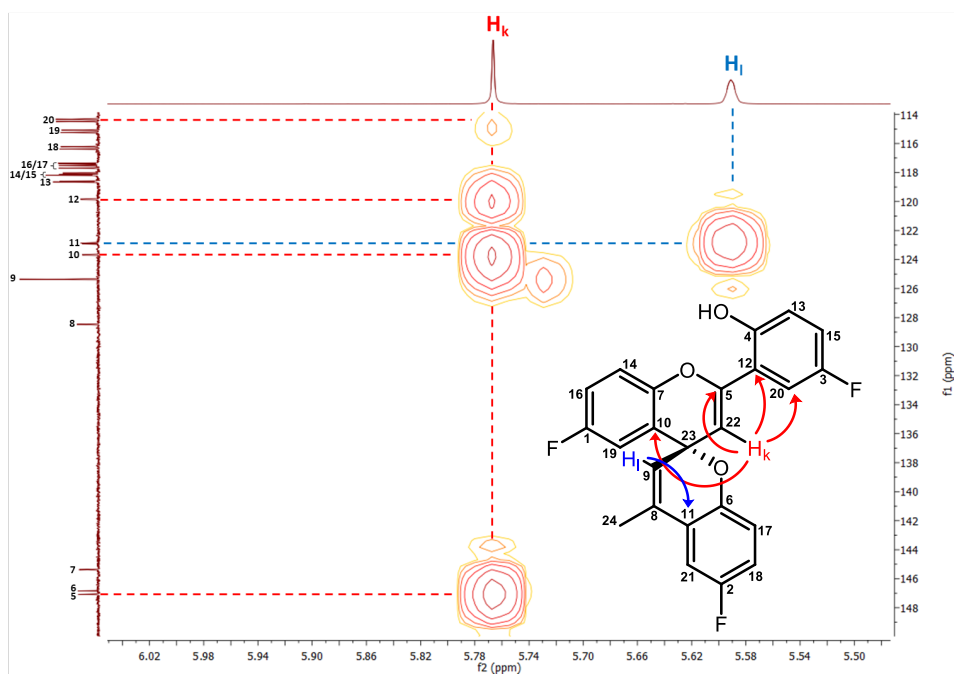
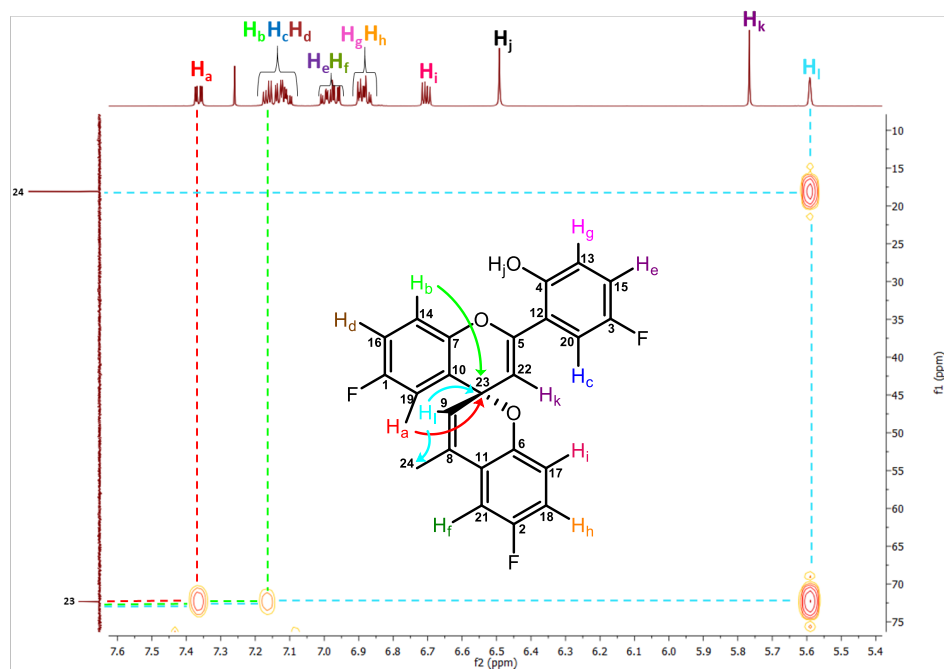


Figure S14. Two different regions of ^1H - ^{13}C HSQC NMR (600 MHz, 298 K) spectrum of isospiropyran **15** in CDCl_3 .



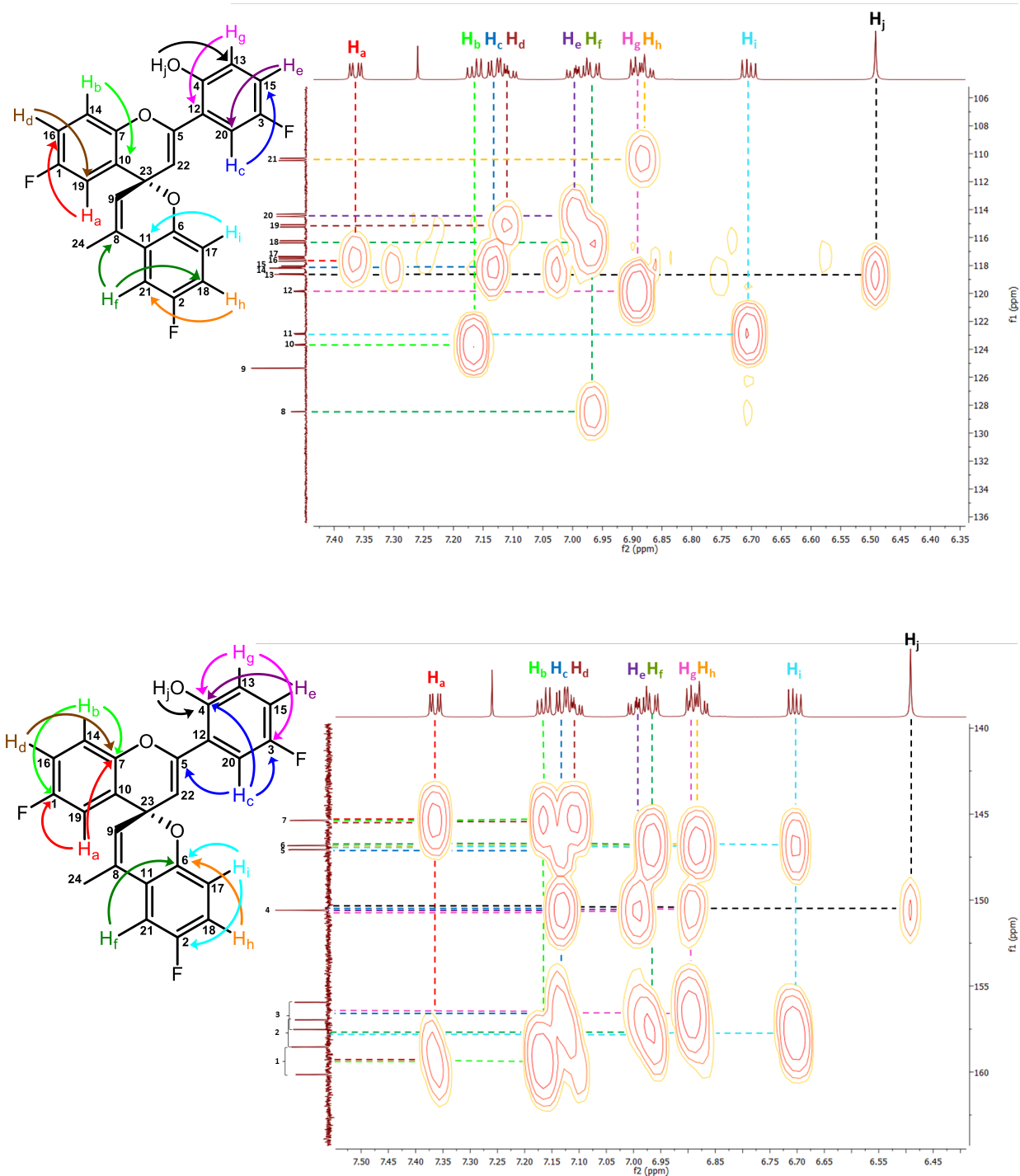


Figure S15. Four different regions of ^1H - ^{13}C HMBC NMR (600 MHz, 298 K) spectrum of isopiripyrane **15** in CDCl_3 .

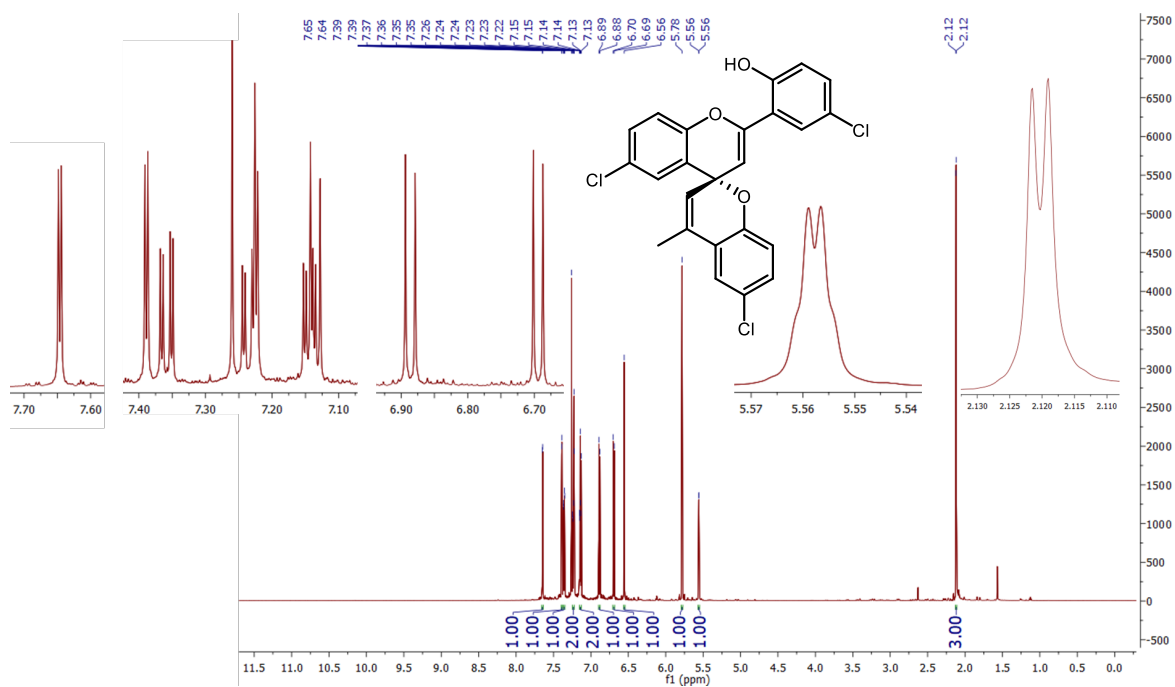


Figure S16. ¹H NMR spectrum (600 MHz, 298 K) of isospiropyran **16** in CDCl₃.

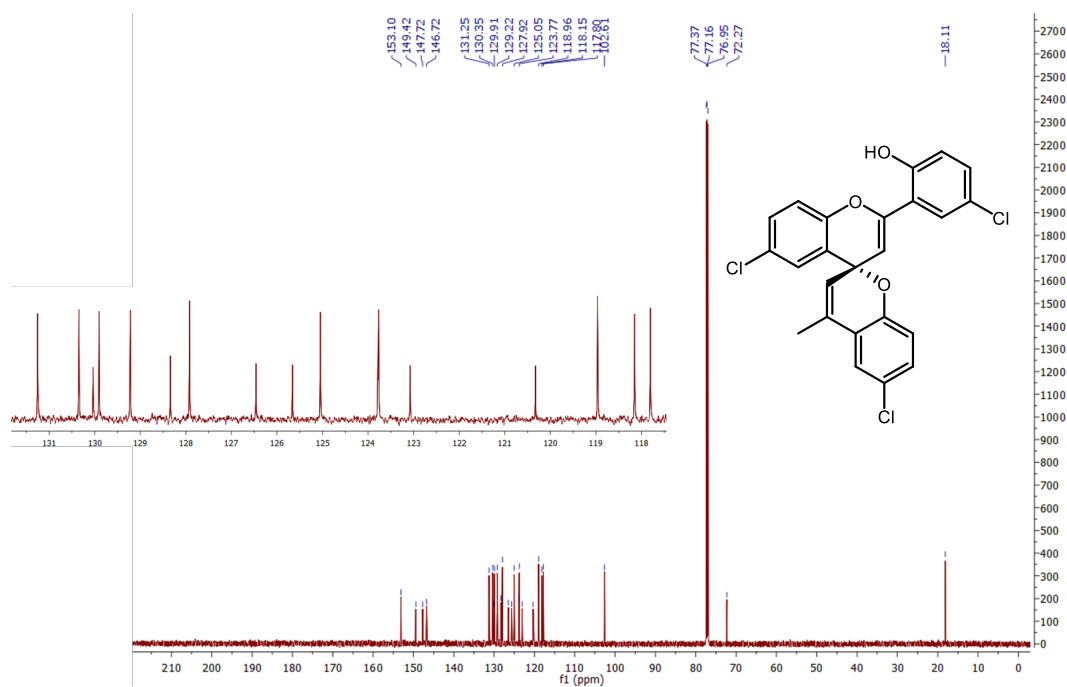


Figure S17. ¹³C NMR spectrum (150 MHz, 298 K) of isospiropyran **16** in CDCl₃.

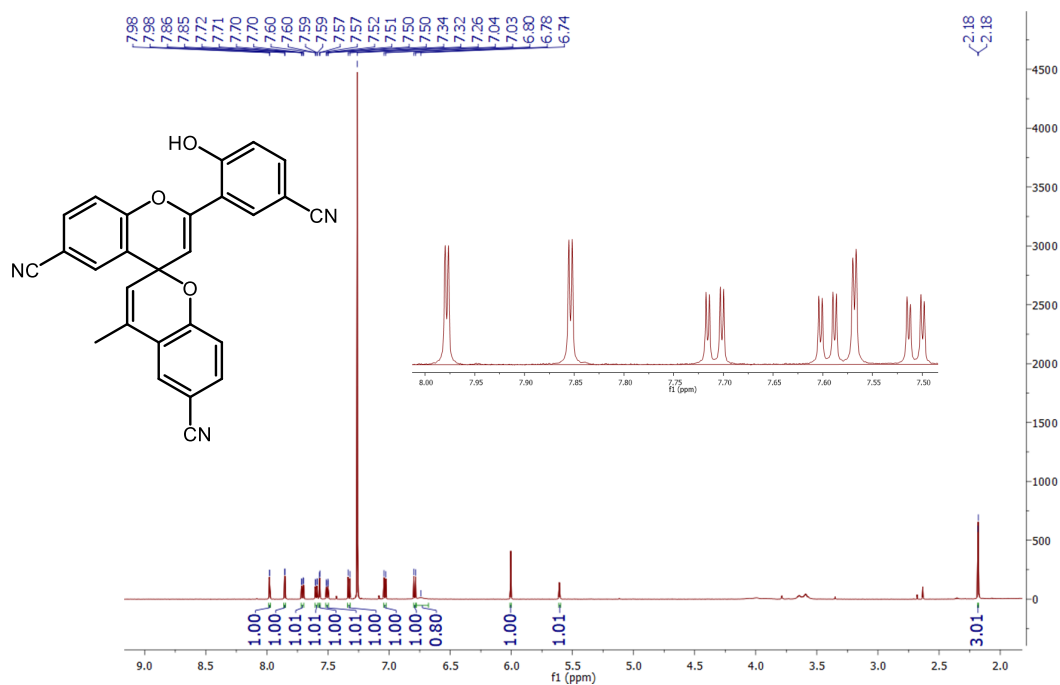


Figure S18. ^1H NMR spectrum (600 MHz, 298 K) of isospiropyran **18** in CDCl_3 .

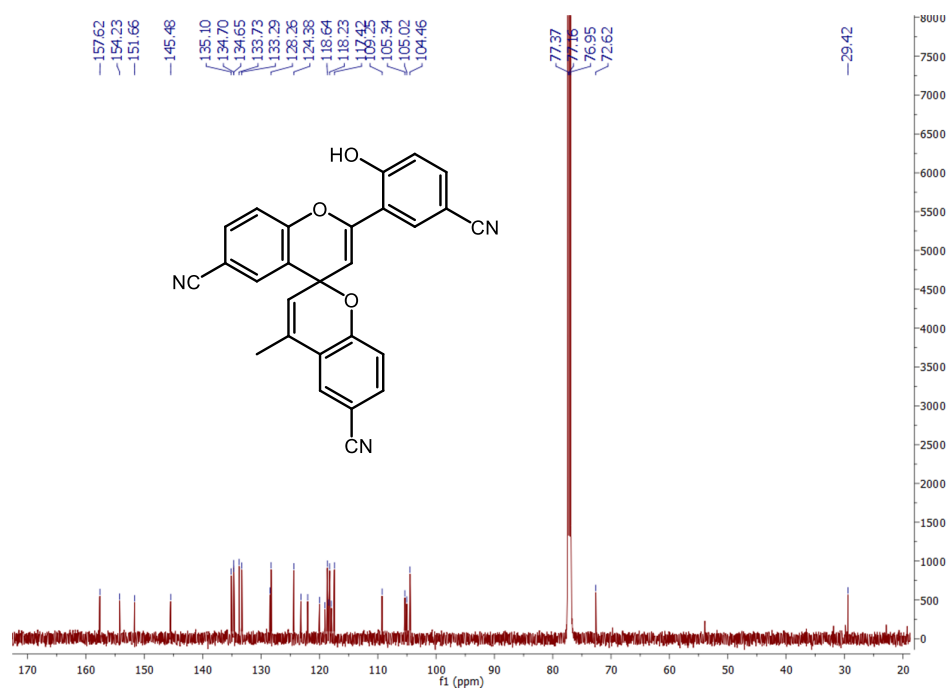


Figure S19. ^{13}C NMR spectrum (150 MHz, 298 K) of isospiropyran **18** in CDCl_3 .

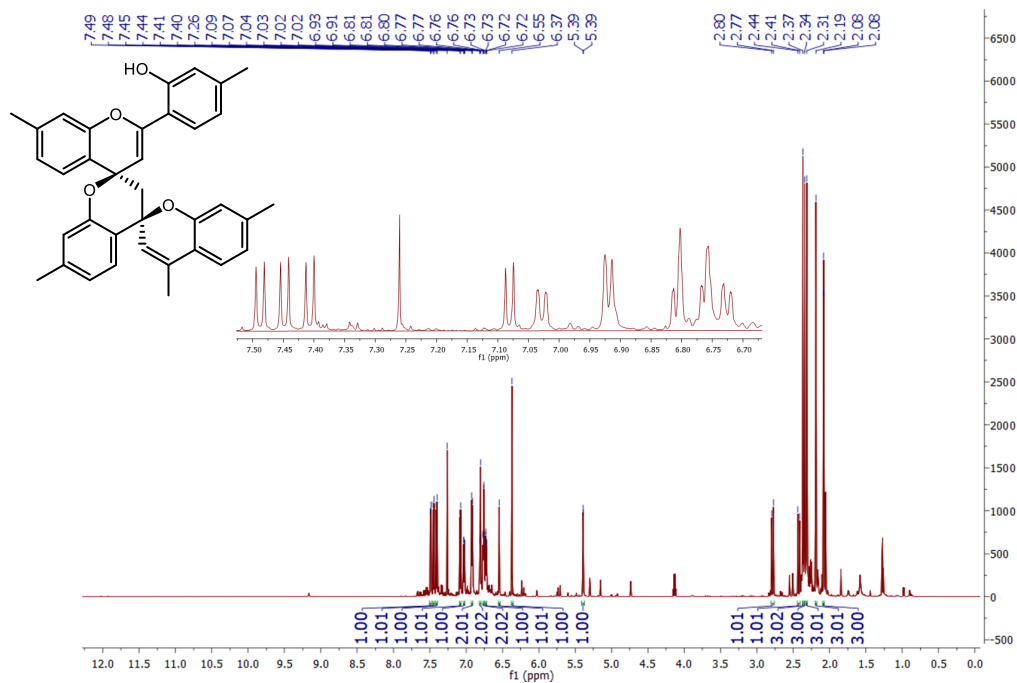


Figure S20. ^1H NMR spectrum (600 MHz, 298 K) of compound **30** in CDCl_3 .

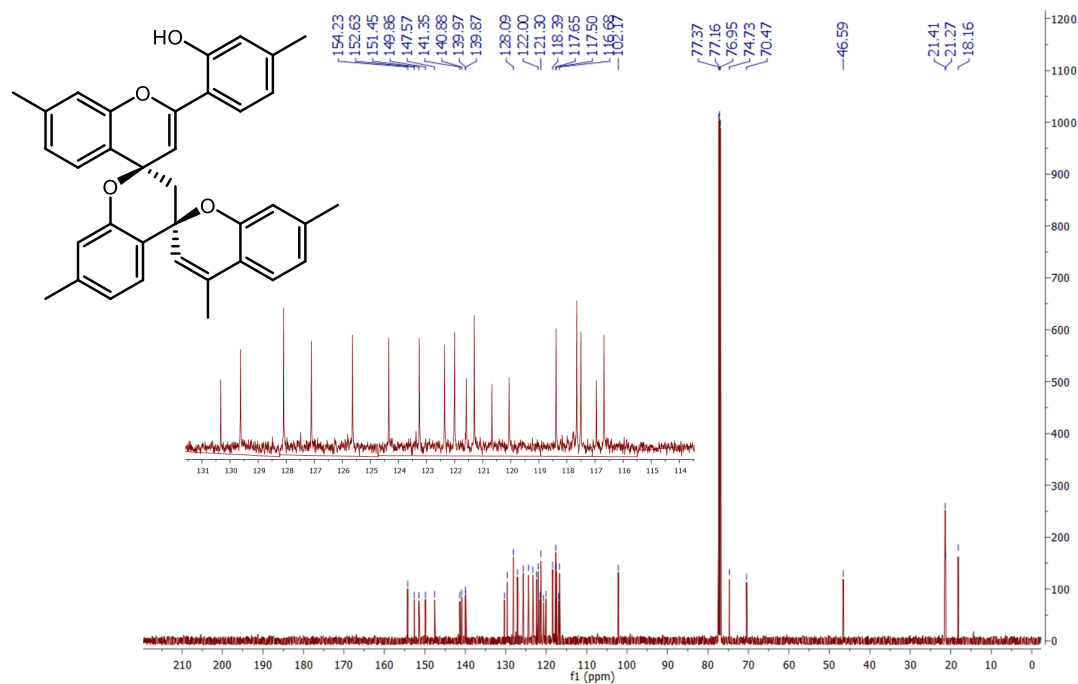


Figure S21. ^{13}C NMR spectrum (150 MHz, 298 K) of compound **30** in CDCl_3 .

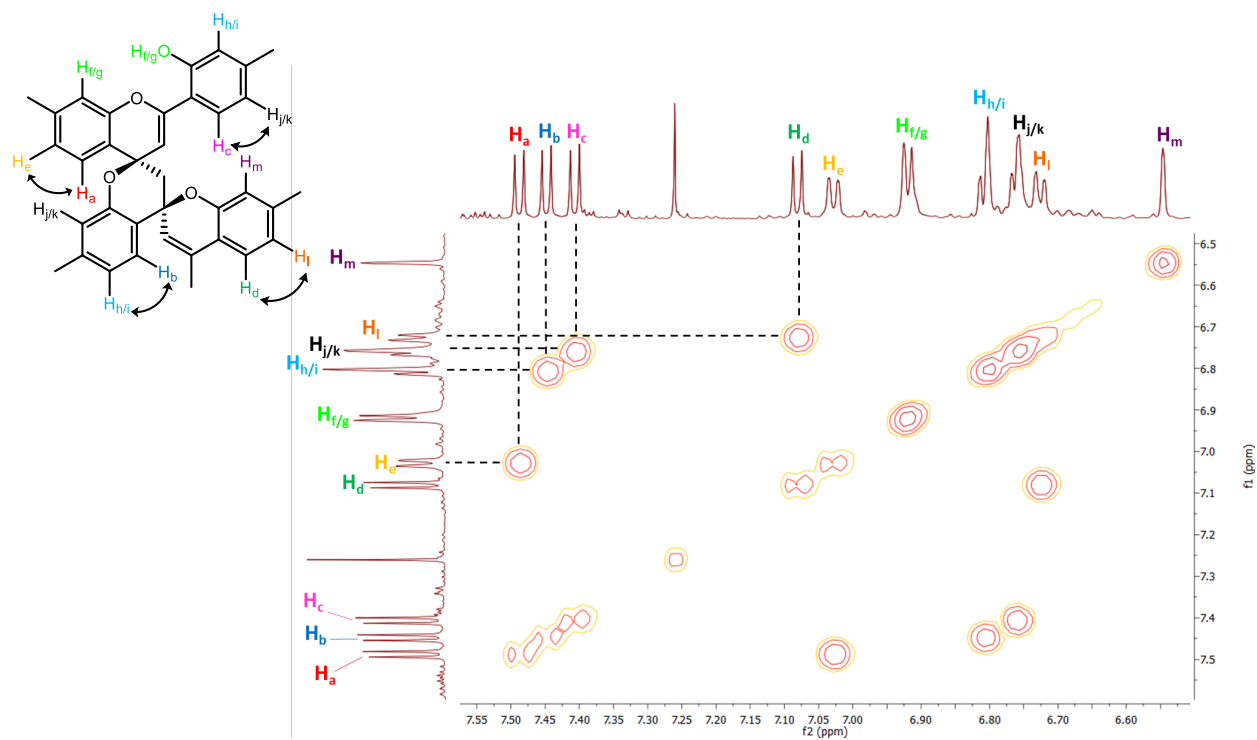
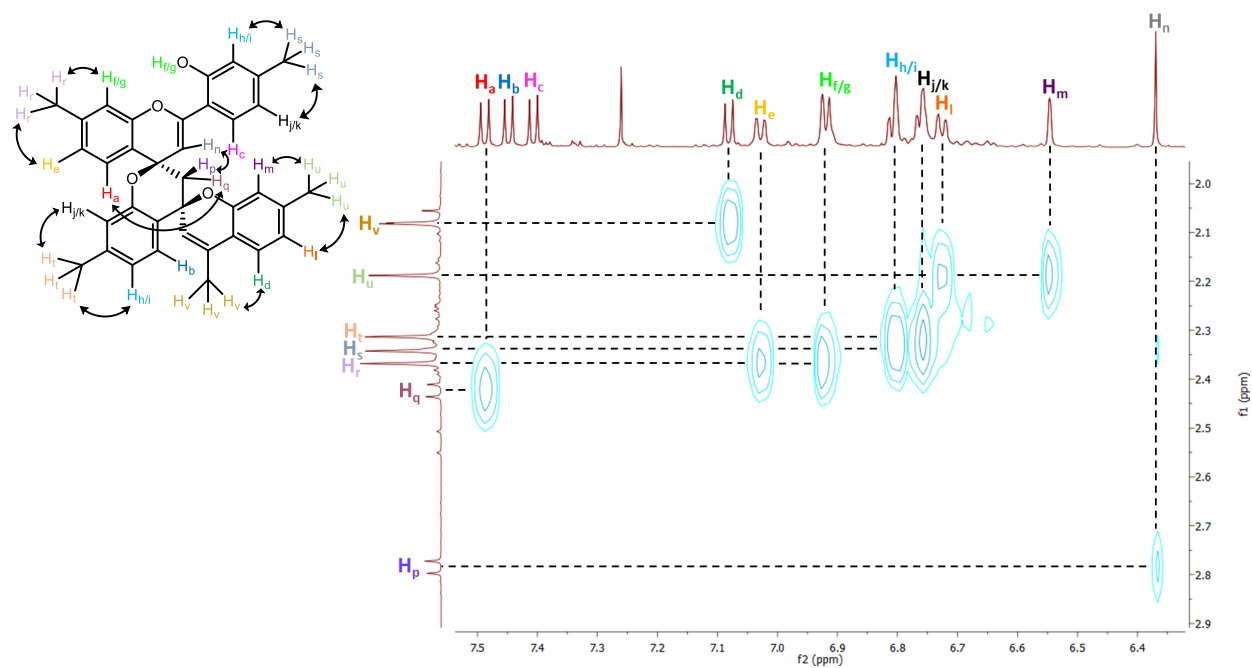


Figure S22. ^1H - ^1H COSY NMR (600 MHz, 298 K) spectrum of compound **30** in CDCl_3 .



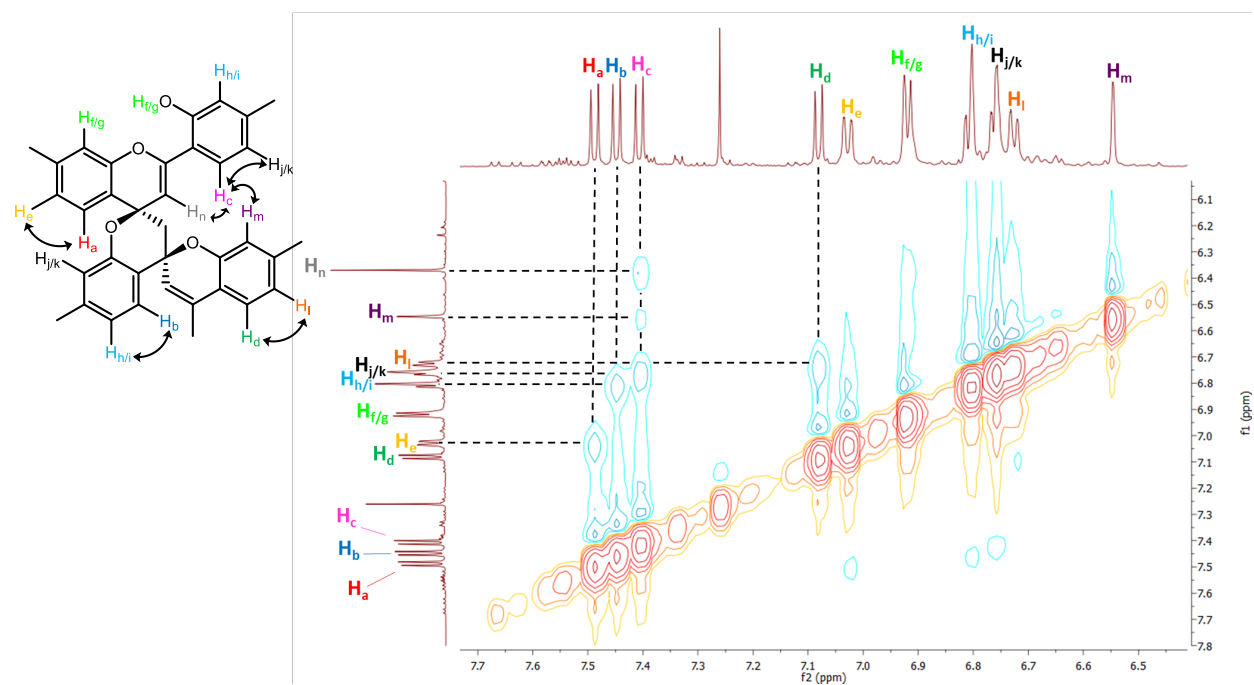
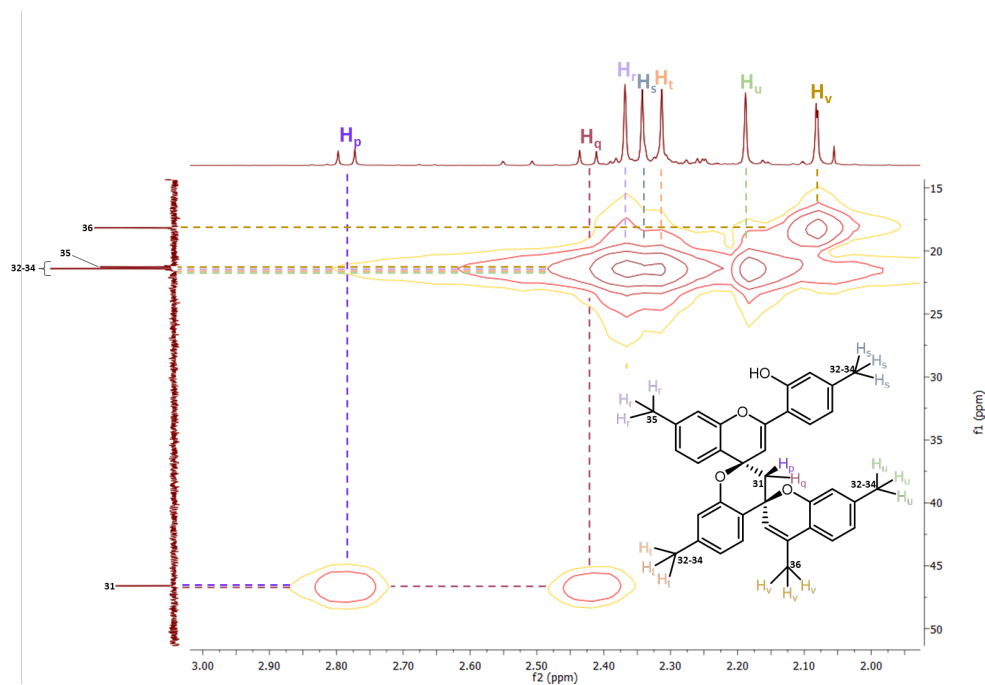


Figure S23. Two different regions of ^1H - ^1H NOESY NMR (600 MHz, 298 K) spectrum of tetramer **30** in CDCl_3 .



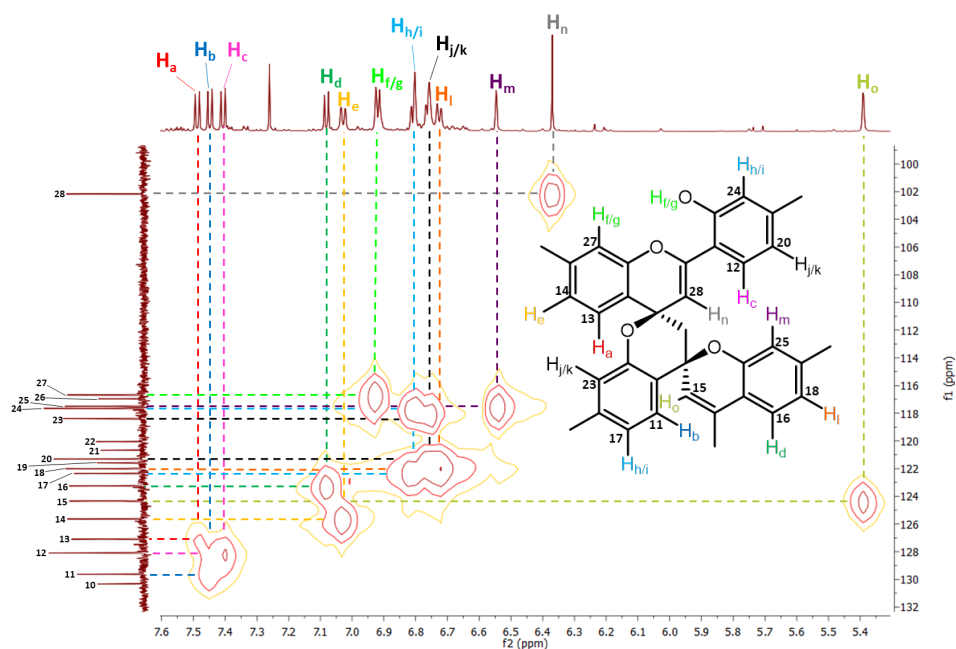
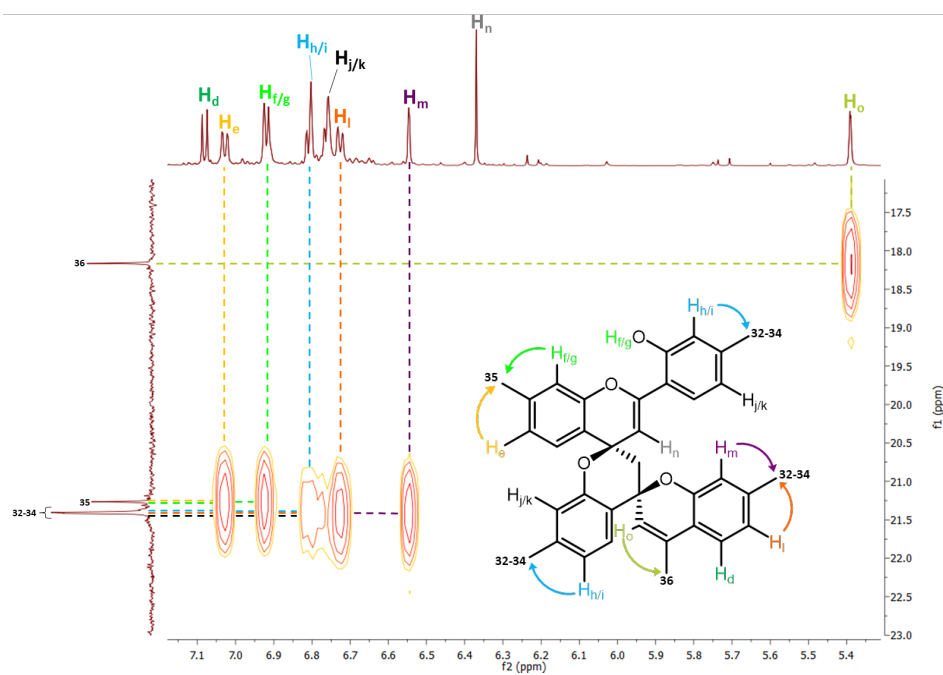
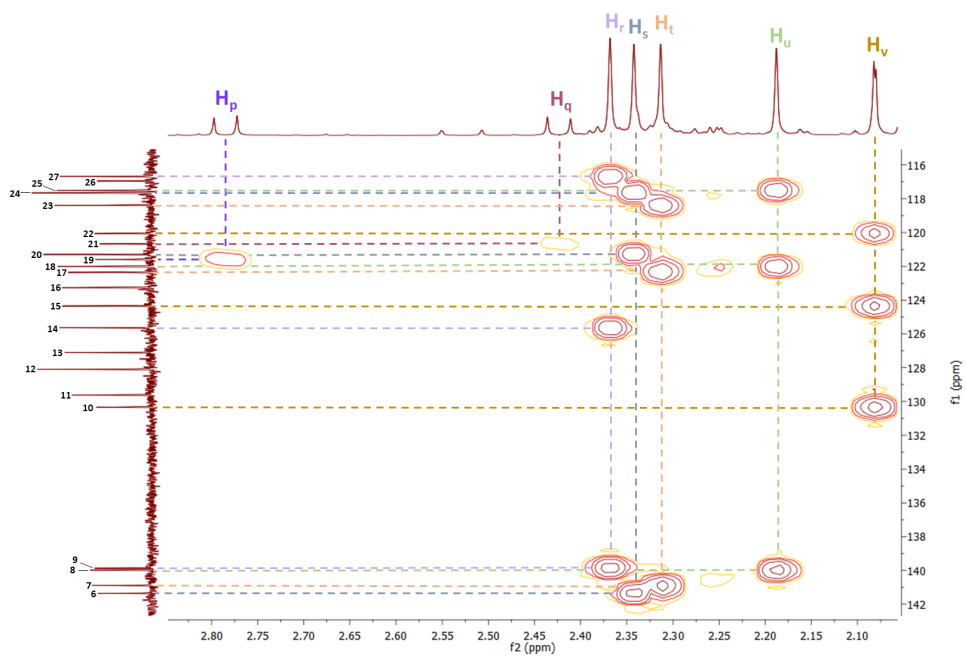
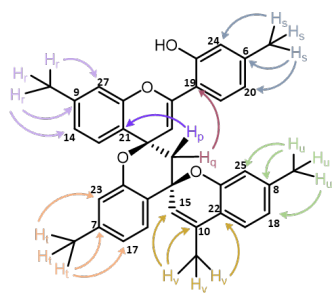
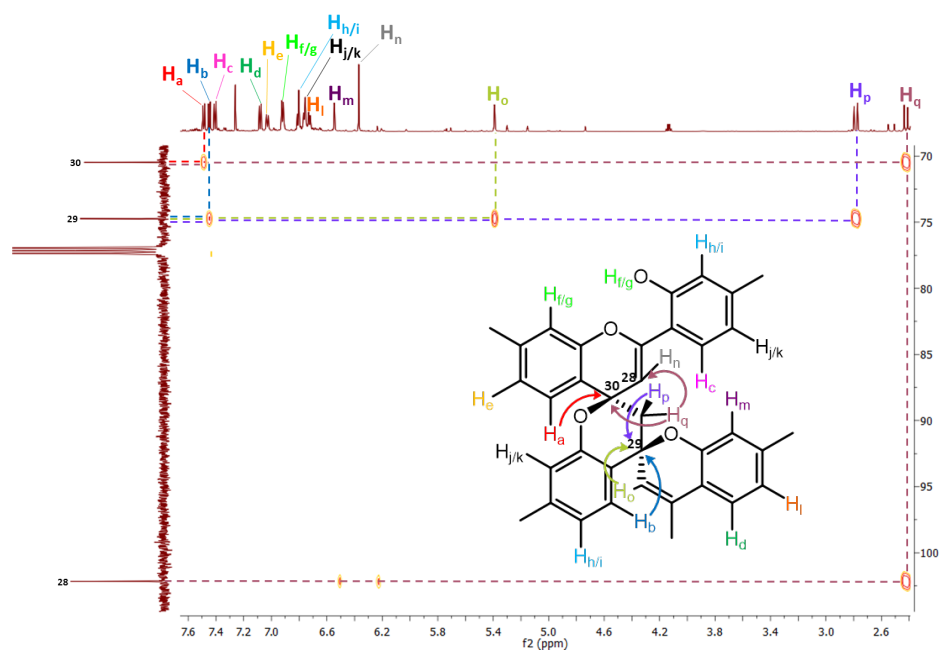


Figure S24. Two different regions of ^1H - ^{13}C HSQC NMR (600 MHz, 298 K) spectrum of tetramer **30** in CDCl_3 .





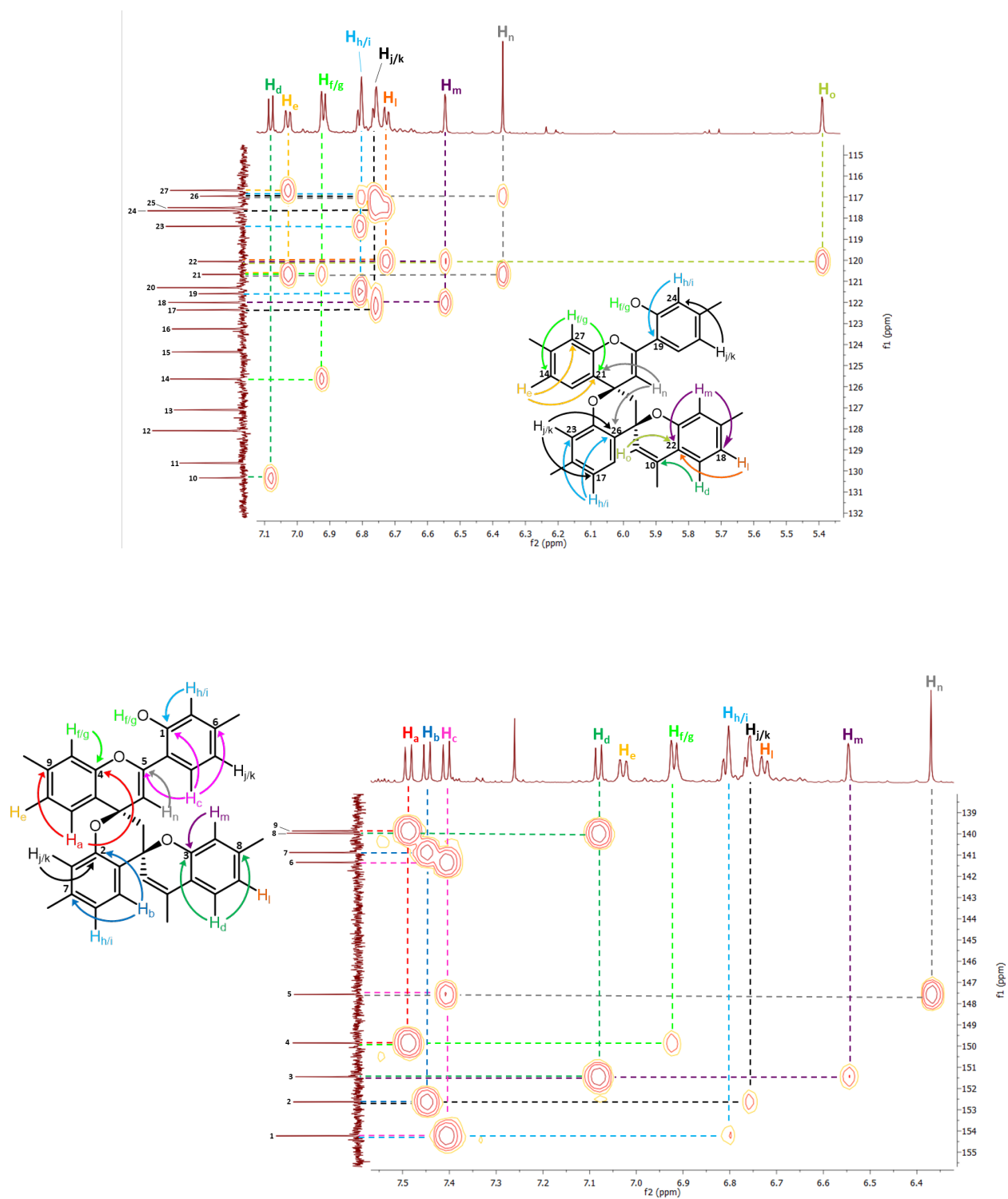


Figure S25. Five different regions of ^1H - ^{13}C HMBC NMR (600 MHz, 298 K) spectrum of tetramer **30** in CDCl_3 .

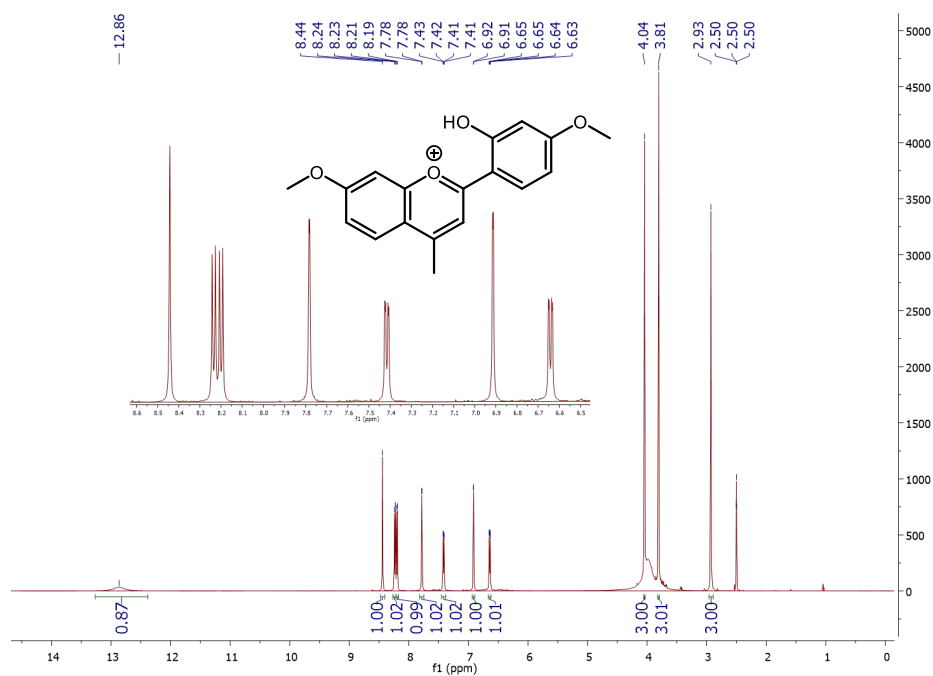


Figure S26. ¹H NMR spectrum (600 MHz, 298 K) of anthocyanidin **31** in *d*₆-DMSO.

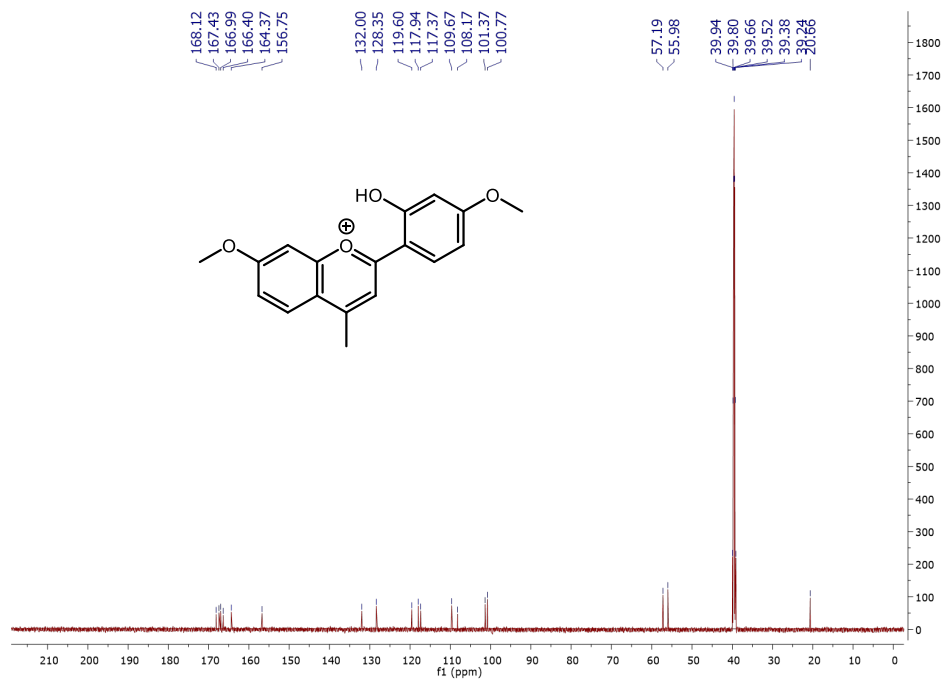


Figure S27. ¹³C NMR spectrum (150 MHz, 298 K) of anthocyanidin **31** in *d*₆-DMSO.

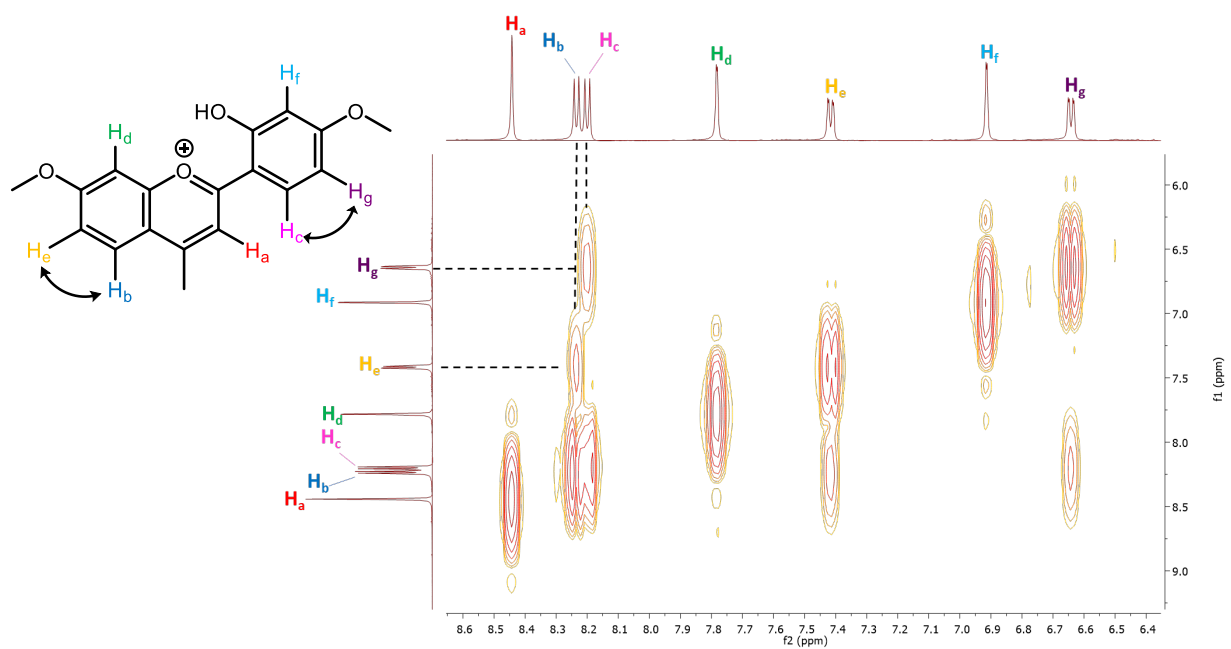


Figure S28. ^1H - ^1H COSY NMR (600 MHz, 298 K) spectrum of anthocyanidin **31** in d_6 -DMSO.

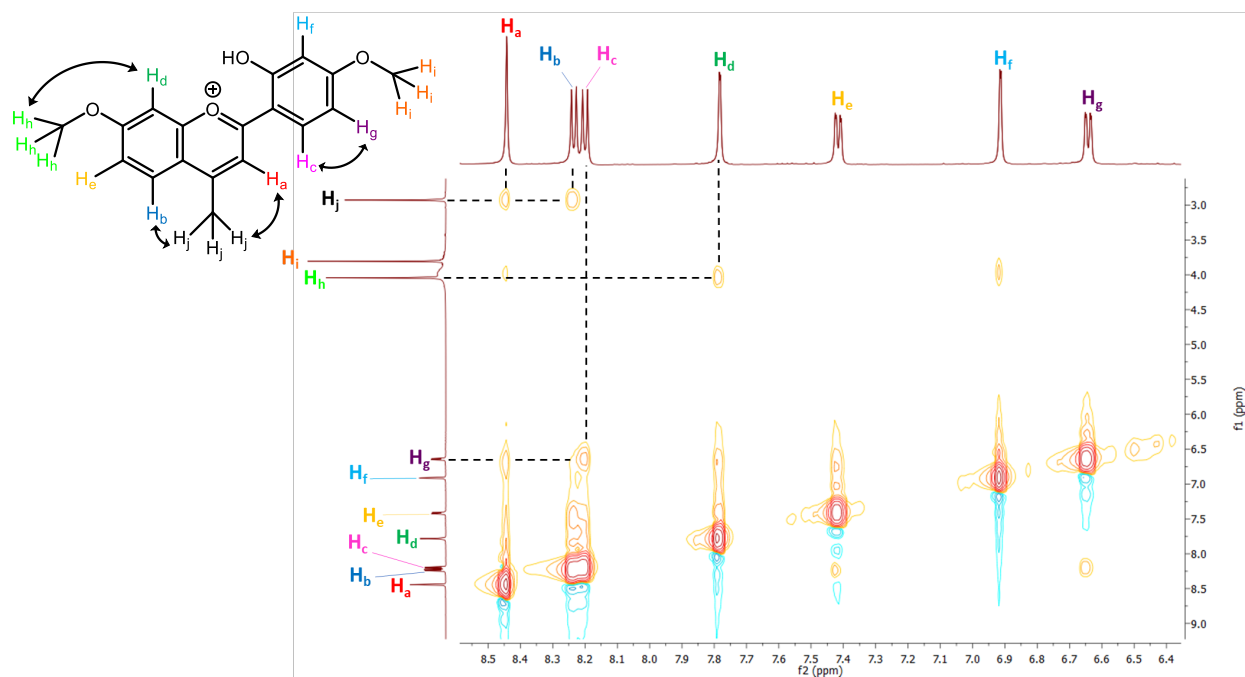


Figure S29. ^1H - ^1H NOESY NMR (600 MHz, 298 K) spectrum of anthocyanidin **31** in d_6 -DMSO.

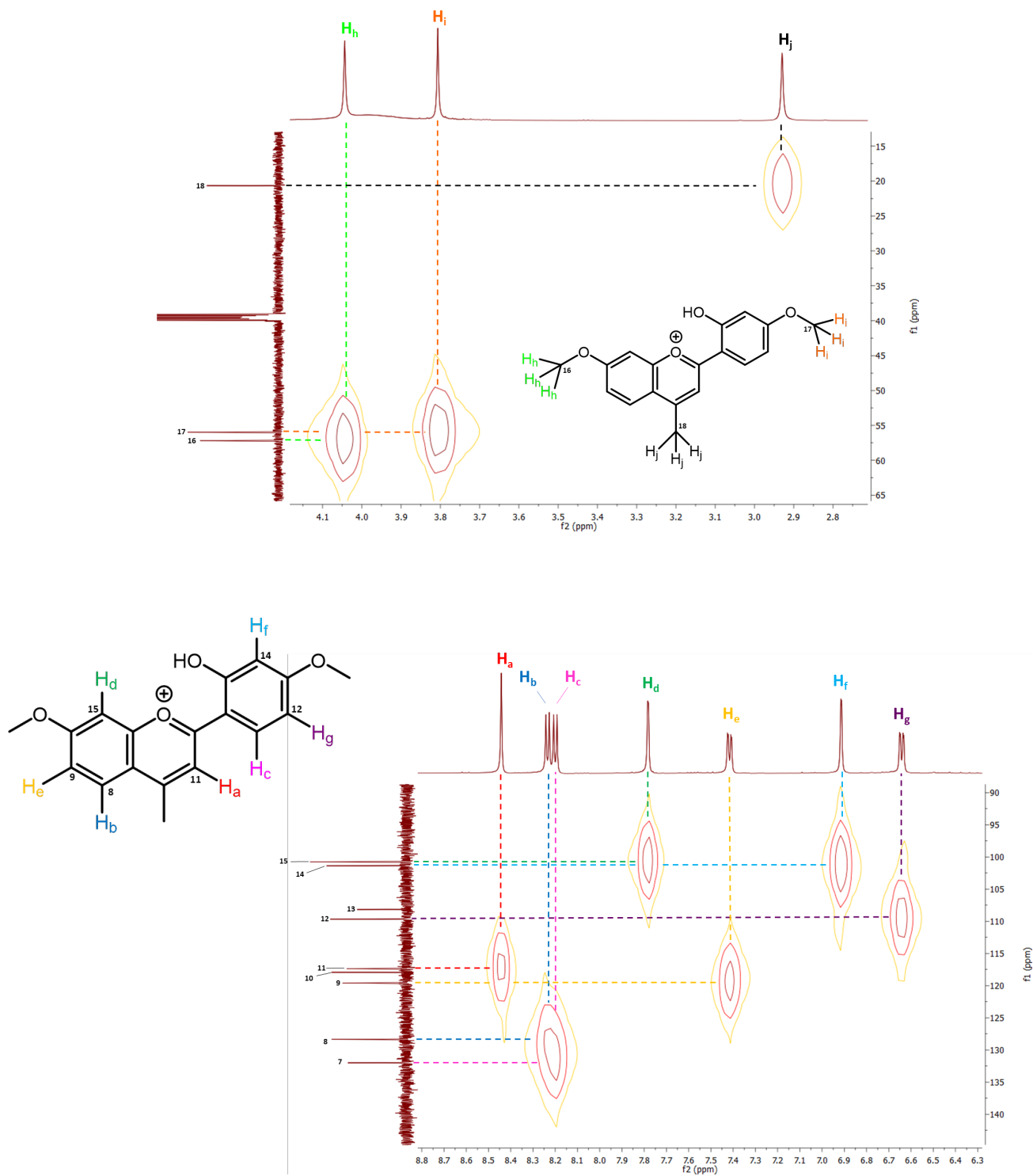


Figure S30. Two different regions of ^1H - ^{13}C HSQC NMR (600 MHz, 298 K) spectrum of anthocyanidin **31** in d_6 -DMSO.

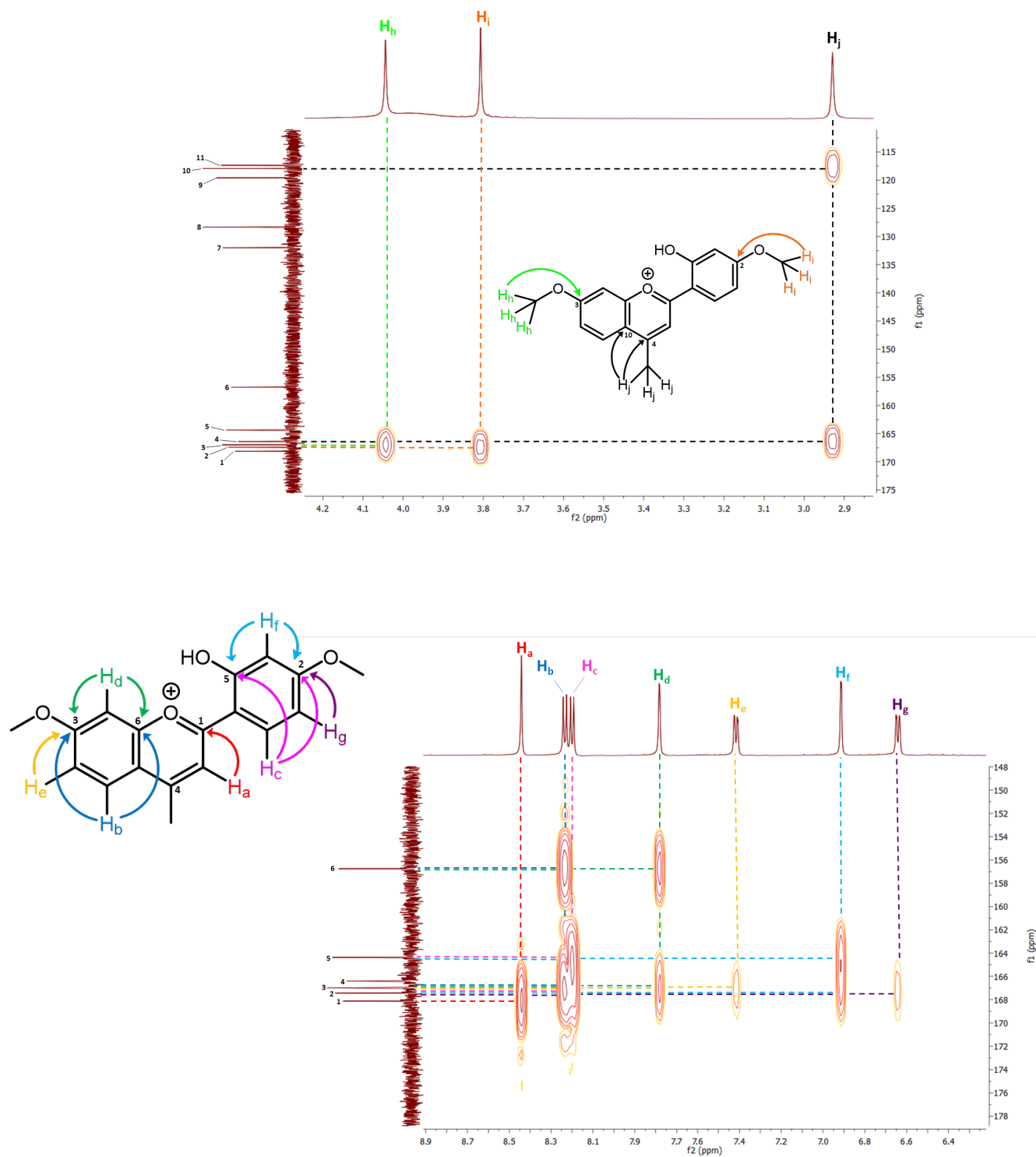


Figure S31. Two different regions of ^1H - ^{13}C HMBC NMR (600 MHz, 298 K) spectrum of anthocyanidin **31** in d_6 -DMSO.

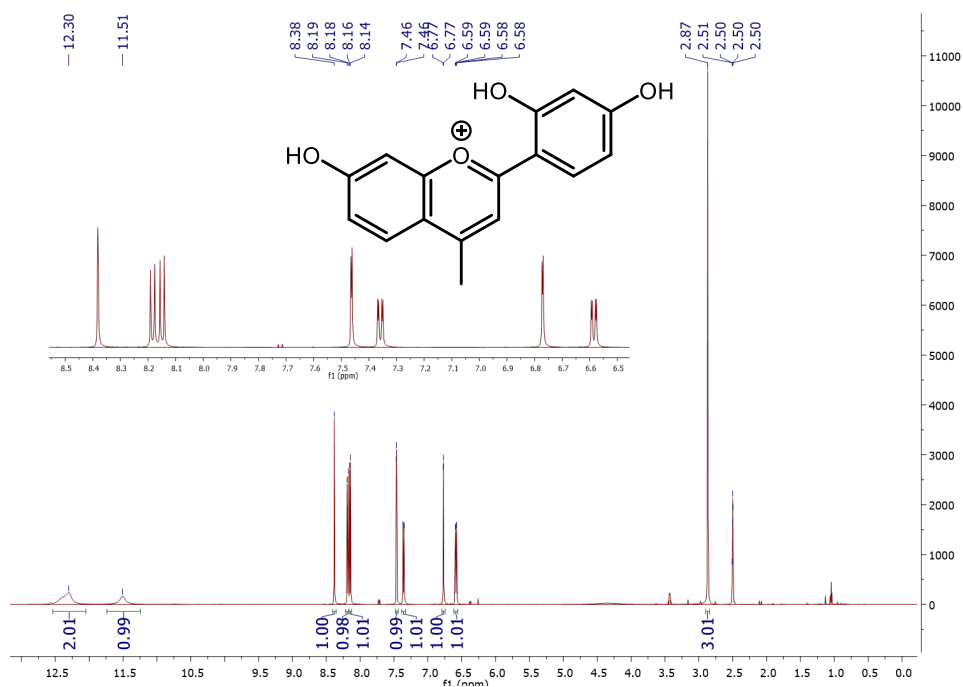


Figure S32. ^1H NMR spectrum (600 MHz, 298 K) of anthocyanidin **32** in d_6 -DMSO.

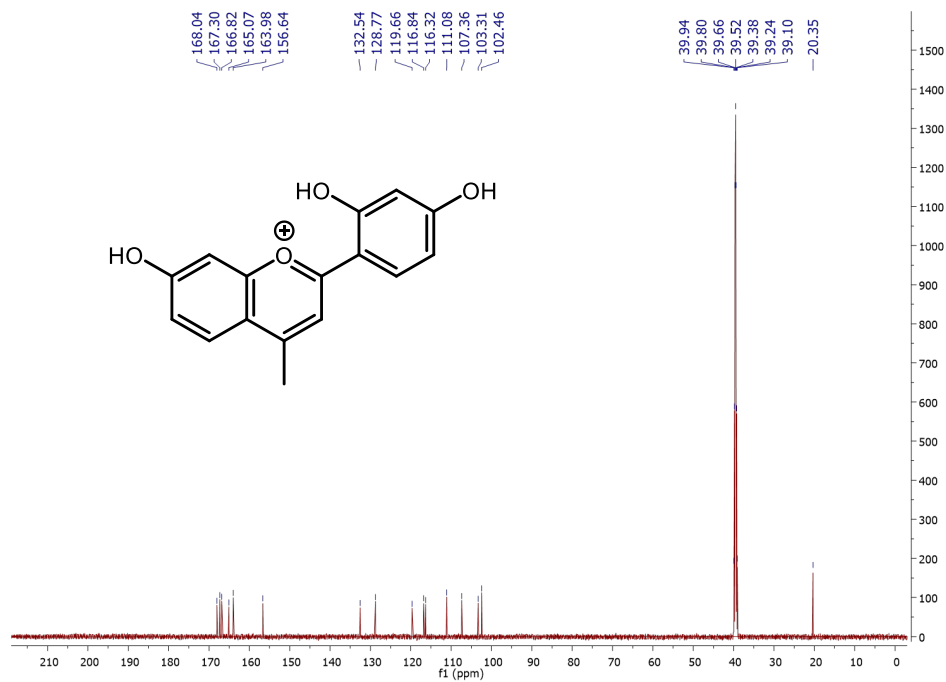


Figure S33. ^{13}C NMR spectrum (150 MHz, 298 K) of anthocyanidin **32** in d_6 -DMSO.

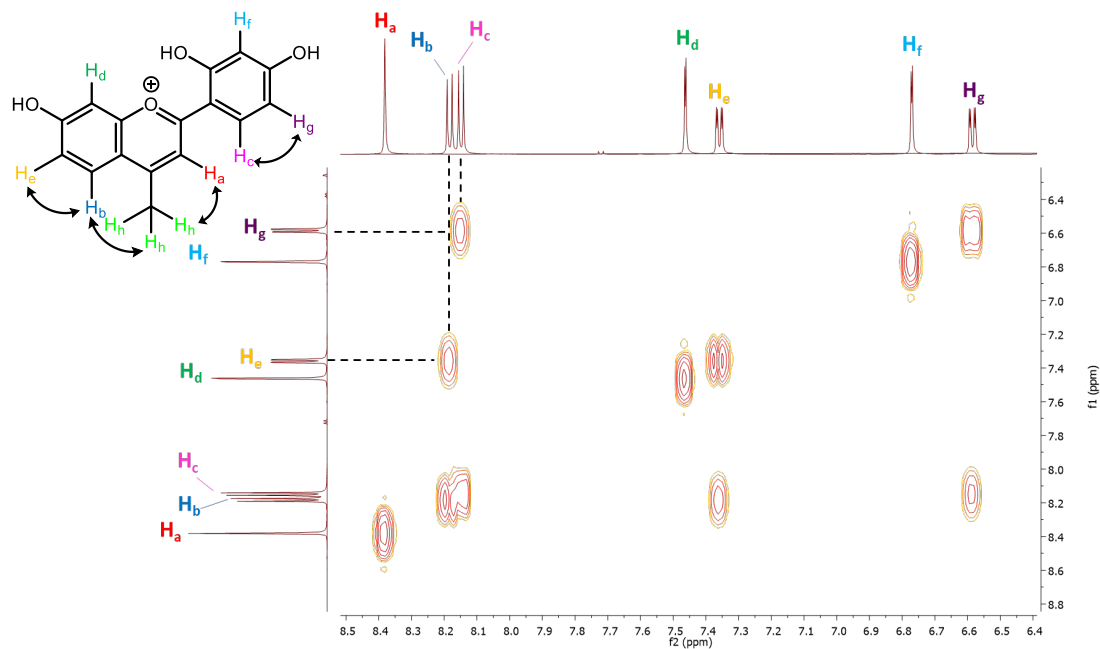


Figure S34. ^1H - ^1H COSY NMR (600 MHz, 298 K) spectrum of anthocyanidin **32** in d_6 -DMSO.

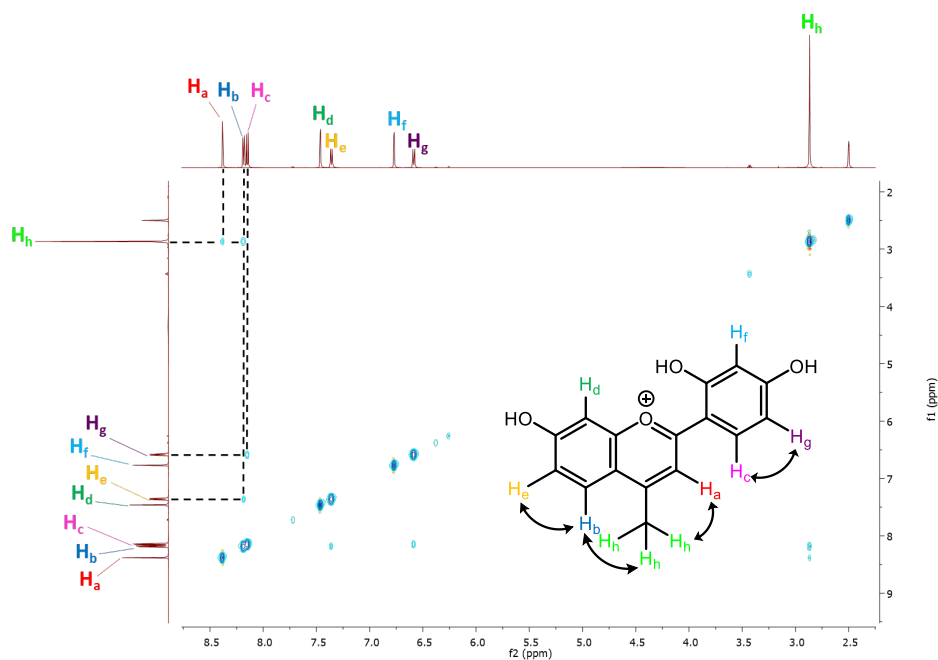


Figure S35. ^1H - ^1H NOESY NMR (600 MHz, 298 K) spectrum of anthocyanidin **32** in d_6 -DMSO.

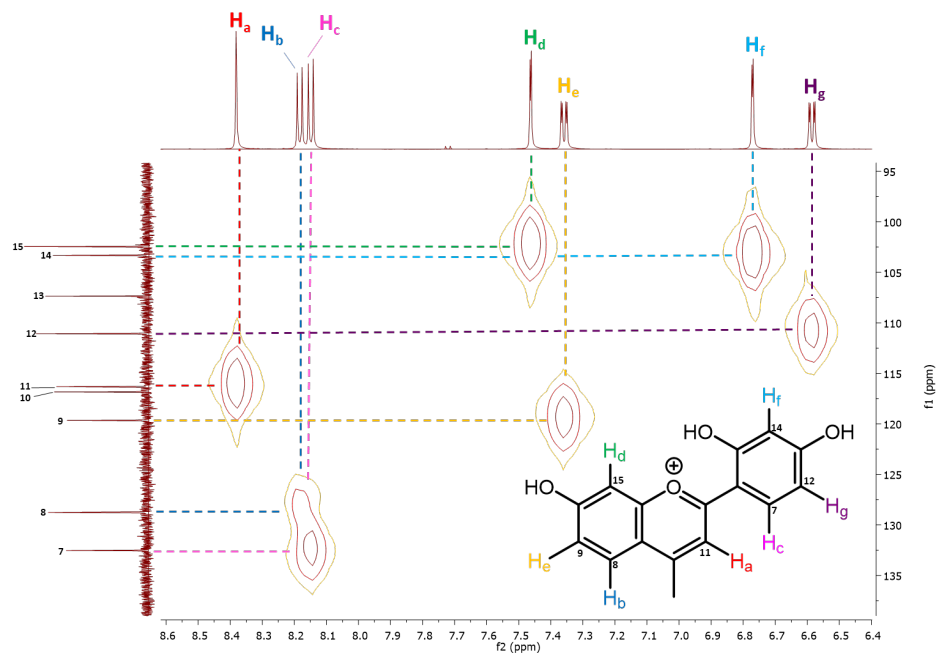


Figure S36. ^1H - ^{13}C HSQC NMR (600 MHz, 298 K) spectrum of anthocyanidin **32** in d_6 -DMSO.

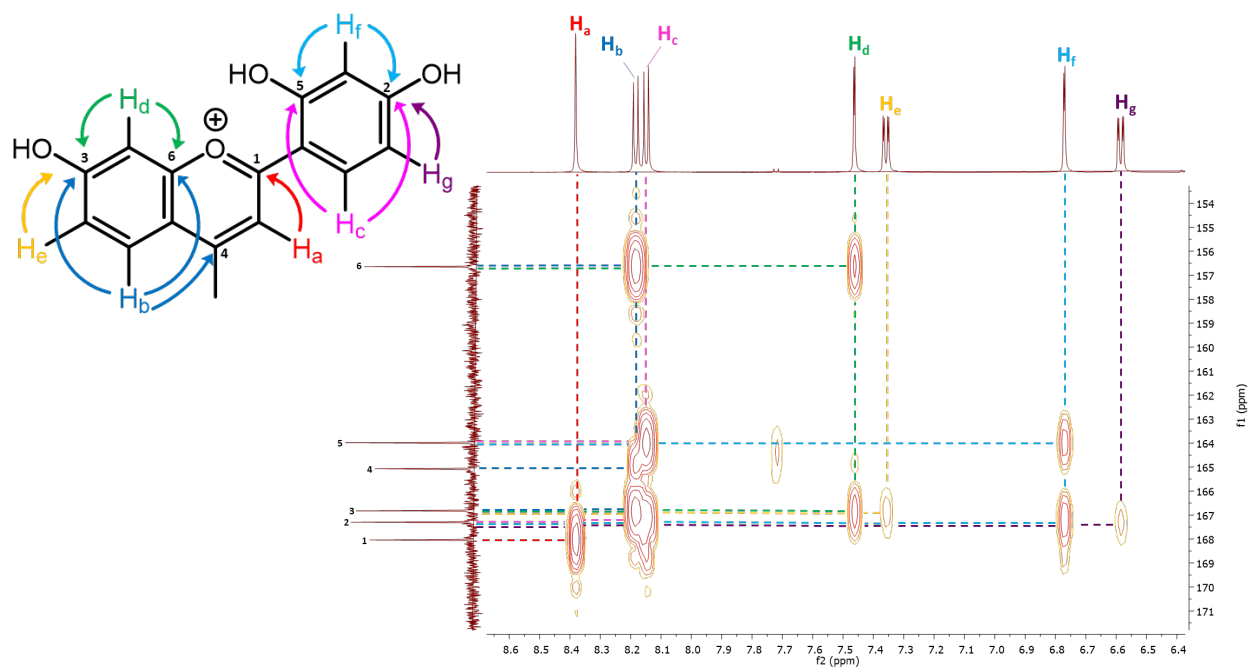


Figure S37. ^1H - ^{13}C HMBC NMR (600 MHz, 298 K) spectrum of anthocyanidin **32** in d_6 -DMSO.

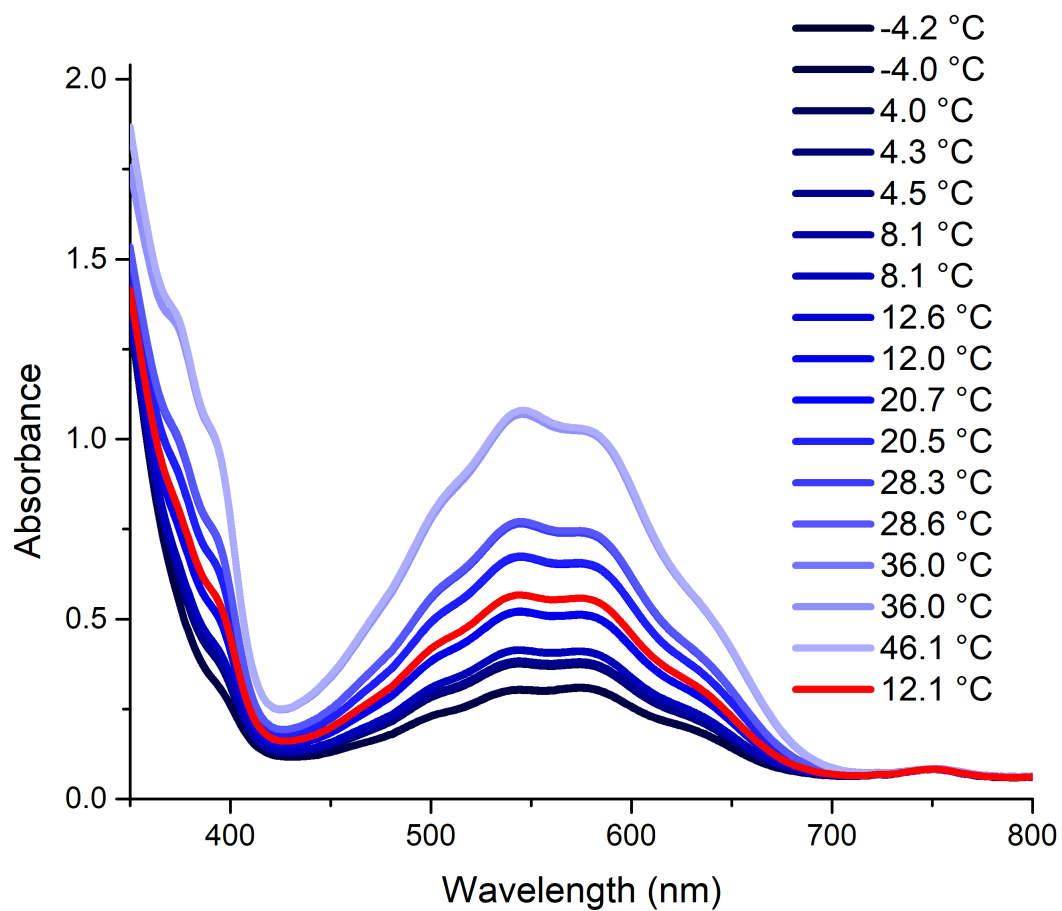
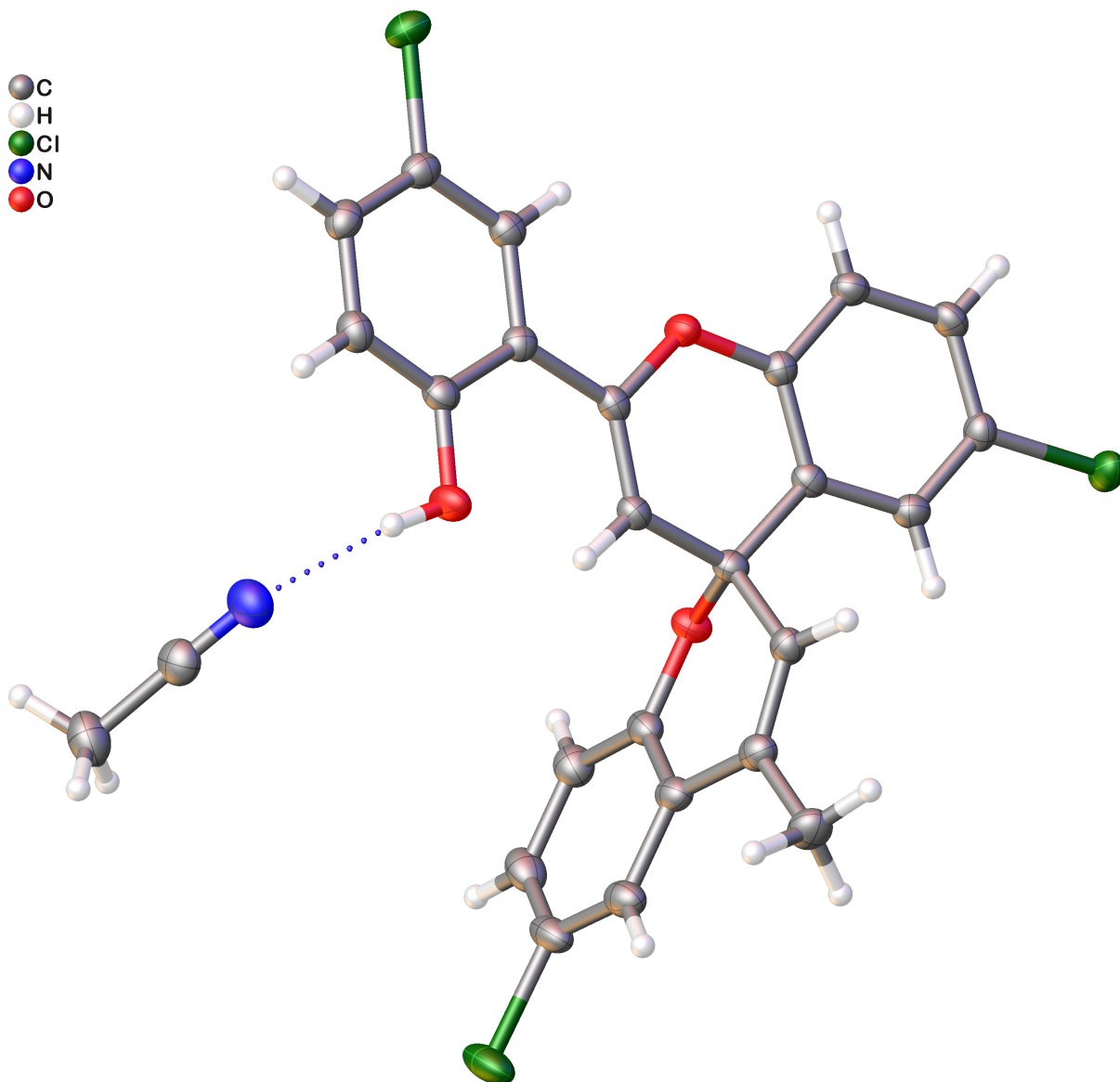


Figure S38. UV-vis spectra of 0.12 mM solution of bis-spiropyran **30** in CH_2Cl_2 from -4.2 to 46.1 °C. After reaching 46.1 °C, we reduced to temperature to 12.1 °C with the spectrum showing that the process was not fully reversible.

Crystallographic Data

Compound 16: The single crystal X-ray diffraction studies were carried out on a Bruker Kappa Photon II CPAD diffractometer equipped with Cu K $_{\alpha}$ radiation ($\lambda = 1.54178$). A 0.197 x 0.153 x 0.118 mm piece of a colorless block was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using ϕ and ω scans. Crystal-to-detector distance was 60 mm using variable exposure time (2s-10s) depending on θ with a scan width of 1.0°. Data collection was 99.6% complete to 68.00° in θ . A total of 20163 reflections were collected covering the indices, $-13 \leq h \leq 13$, $-9 \leq k \leq 12$, $-28 \leq l \leq 27$. 4420 reflections were found to be symmetry independent, with a R_{int} of 0.0269. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be $P2_1/c$. The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SHELXT) produced a complete phasing model for refinement.

All nonhydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2014. Crystallographic data are summarized in Table S1.



ORTEP plot of compound **16** (50% probability), grown by a slow evaporation of its acetonitrile solution.

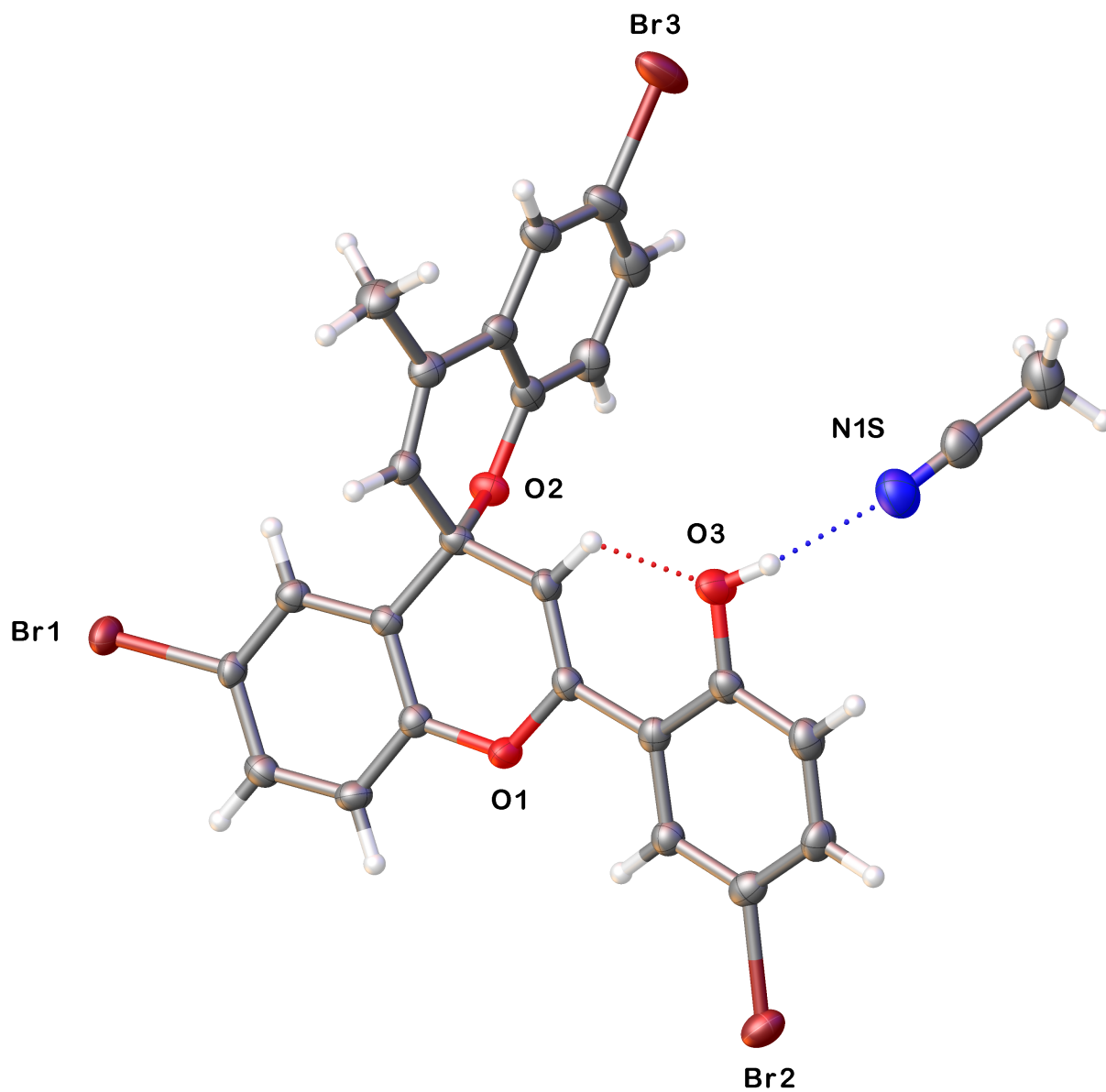
Table S1. Crystal data and structure refinement for compound **16**.

Report date	2020-03-18	
Identification code	TN_ChloroClosed	
Empirical formula	C ₂₆ H ₁₈ Cl ₃ N O ₃	
Molecular formula	C ₂₄ H ₁₅ Cl ₃ O ₃ , C ₂ H ₃ N	
Formula weight	498.76	
Temperature	100 K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 1 2 ₁ /c 1	
Unit cell dimensions	a = 10.7830(2) Å	α = 90°.
	b = 9.0950(2) Å	β = 96.6440(10)°.
	c = 22.4992(4) Å	γ = 90°.
Volume	2191.71(7) Å ³	
Z	4	
Density (calculated)	1.512 Mg/m ³	
Absorption coefficient	4.043 mm ⁻¹	
F(000)	1024	
Crystal size	0.197 x 0.153 x 0.118 mm ³	
Crystal color, habit	Colorless Block	
Theta range for data collection	3.956 to 74.555°.	
Index ranges	-13 ≤ h ≤ 13, -9 ≤ k ≤ 11, -28 ≤ l ≤ 27	
Reflections collected	20163	

Independent reflections	4420 [R(int) = 0.0269, R(sigma) = 0.0196]
Completeness to theta = 68.000°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.3260 and 0.1942
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4420 / 0 / 301
Goodness-of-fit on F ²	1.053
Final R indices [I>2sigma(I)]	R1 = 0.0359, wR2 = 0.0923
R indices (all data)	R1 = 0.0386, wR2 = 0.0946
Extinction coefficient	n/a
Largest diff. peak and hole	1.052 and -0.547 e.Å ⁻³

Compound 17: The single crystal X-ray diffraction studies were carried out on a Bruker Kappa Photon II CPAD diffractometer equipped with Mo K $_{\alpha}$ radiation ($\lambda = 0.71073 \text{ \AA}$). A 0.326 x 0.257 x 0.224 mm piece of a redish orange block was mounted on MiTeGen Micromount with CHRISTO-LUBE MCG 1024 oil. Data were collected in a nitrogen gas stream at 100(2) K using ϕ and ω scans. Crystal-to-detector distance was 60 mm using variable exposure time (2s-10s) depending on θ with a scan width of 1.0°. Data collection was 99.6% complete to 25.00° in θ . A total of 25940 reflections were collected covering the indices, $-14 \leq h \leq 10$, $-12 \leq k \leq 12$, $-30 \leq l \leq 30$. 5440 reflections were found to be symmetry independent, with a R_{int} of 0.0324. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be $P2_1/c$. The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SHELXT) produced a complete phasing model consistent with the proposed structure.

All nonhydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2014. Crystallographic data are summarized in Table S2.



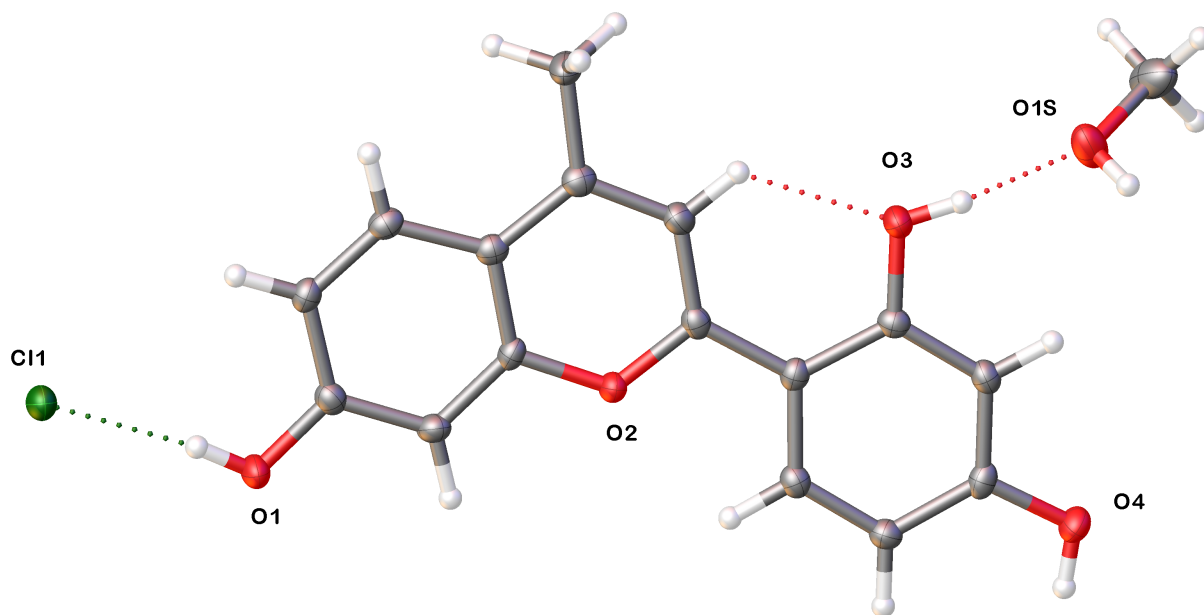
ORTEP plot of compound **17** (50% probability), grown by a slow evaporation of its acetonitrile solution.

Table S2. Crystal data and structure refinement for compound **17**.

Report date	2019-05-13	
Identification code	TN_ClosedCH3CN	
Empirical formula	C ₂₆ H ₁₈ Br ₃ N O ₃	
Molecular formula	C ₂₄ H ₁₅ Br ₃ O ₃ , C ₂ H ₃ N	
Formula weight	632.14	
Temperature	100.0 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 11.0228(6) Å	α = 90°.
	b = 9.1224(6) Å	β = 95.723(2)°.
	c = 22.8268(14) Å	γ = 90°.
Volume	2283.9(2) Å ³	
Z	4	
Density (calculated)	1.838 Mg/m ³	
Absorption coefficient	5.329 mm ⁻¹	
F(000)	1240	
Crystal size	0.326 x 0.257 x 0.224 mm ³	
Crystal color, habit	Redish Orange Block	
Theta range for data collection	2.864 to 28.293°.	
Index ranges	-14 ≤ h ≤ 10, -12 ≤ k ≤ 12, -30 ≤ l ≤ 30	
Reflections collected	25940	
Independent reflections	5440 [R(int) = 0.0324, R(sigma) = 0.0303]	

Completeness to theta = 25.000°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.0962 and 0.0466
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5440 / 0 / 301
Goodness-of-fit on F ²	1.070
Final R indices [I>2sigma(I)]	R1 = 0.0394, wR2 = 0.0856
R indices (all data)	R1 = 0.0548, wR2 = 0.0907
Extinction coefficient	n/a
Largest diff. peak and hole	1.931 and -1.298 e.Å ⁻³

Compound 32 (methanol): The single crystal X-ray diffraction studies were carried out on a Nonius Kappa diffractometer equipped with a Bruker APEX-II CCD and Mo K $_{\alpha}$ radiation ($\lambda = 0.71073 \text{ \AA}$). A 0.135 x 0.126 x 0.084 mm piece of a colorless block was mounted on a MiTeGen Micromount with CHRISTO-LUBE MCG 1024 oil. Data were collected in a nitrogen gas stream at 100(2) K using ϕ and ω scans. Crystal-to-detector distance was 50 mm and exposure time was 10 seconds per frame using a scan width of 1.0°. Data collection was 100% complete to 25.00° in θ . A total of 22284 reflections were collected covering the indices, $-11 \leq h \leq 11$, $-17 \leq k \leq 17$, $-15 \leq l \leq 15$. 3129 reflections were found to be symmetry independent, with a R_{int} of 0.0427. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be $P2_1/n$. The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SHELXT) produced a complete phasing model consistent with the proposed structure. All nonhydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2014. Crystallographic data are summarized in Table S3.



ORTEP plot of compound **32** (50% probability). Compound **32** was dissolved in methanol and then acetonitrile was carefully layered on top. The vial was loosely capped to allow slow evaporation of the solvents. Two polymorphs appeared from this mixture after several days.

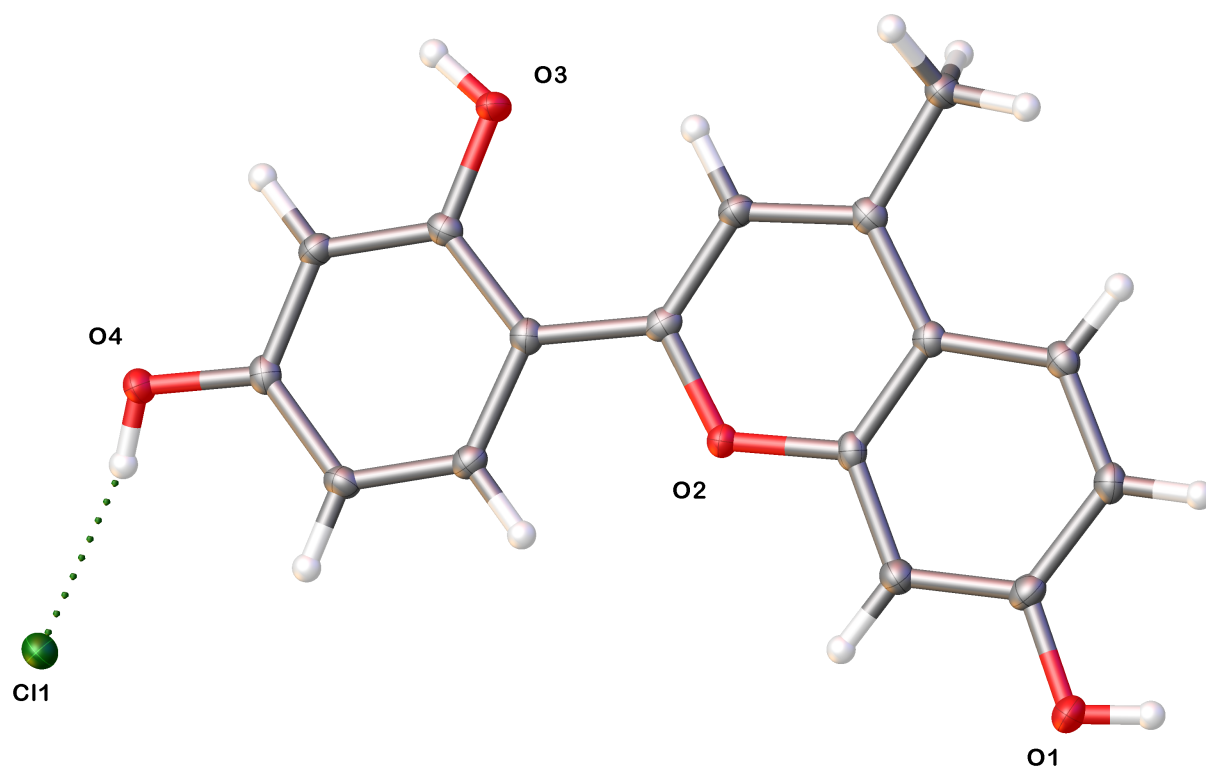
Table S3. Crystal data and structure refinement for **compound 32** (methanol).

Report date	2019-05-13	
Identification code	TN_0301MeOH	
Empirical formula	C17 H17 Cl O5	
Molecular formula	C16 H13 O4, C H4 O, Cl	
Formula weight	336.75	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 9.1565(3) Å	$\alpha = 90^\circ$.
	b = 14.1329(5) Å	$\beta = 106.165(2)^\circ$.
	c = 12.2218(4) Å	$\gamma = 90^\circ$.
Volume	1519.07(9) Å ³	
Z	4	
Density (calculated)	1.472 Mg/m ³	
Absorption coefficient	0.276 mm ⁻¹	
F(000)	704	
Crystal size	0.135 x 0.126 x 0.084 mm ³	
Crystal color, habit	Colorless Block	
Theta range for data collection	2.255 to 26.428°.	
Index ranges	-11 ≤ h ≤ 11, -17 ≤ k ≤ 17, -15 ≤ l ≤ 15	

Reflections collected	22284
Independent reflections	3129 [R(int) = 0.0427, R(sigma) = 0.0300]
Completeness to theta = 25.000°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.2602 and 0.2326
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3129 / 0 / 214
Goodness-of-fit on F ²	1.046
Final R indices [I>2sigma(I)]	R1 = 0.0367, wR2 = 0.0972
R indices (all data)	R1 = 0.0511, wR2 = 0.1043
Extinction coefficient	n/a
Largest diff. peak and hole	0.323 and -0.299 e.Å ⁻³

Compound 32 (neat): The single crystal X-ray diffraction studies were carried out on a Bruker Kappa Photon II CPAD diffractometer equipped with Mo K $_{\alpha}$ radiation ($\lambda = 0.71073 \text{ \AA}$). A 0.164 x 0.157 x 0.148 mm piece of a colorless block was mounted on MiTeGen Micromount with CHRISTO-LUBE MCG 1024 oil. Data were collected in a nitrogen gas stream at 100(2) K using ϕ and ω scans. Crystal-to-detector distance was 60 mm using variable exposure time (4s-10s) depending on θ with a scan width of 1.0° . Data collection was 99.9% complete to 25.00° in θ . A total of 13855 reflections were collected covering the indices, $-7 \leq h \leq 5$, $-19 \leq k \leq 19$, $-17 \leq l \leq 17$. 2718 reflections were found to be symmetry independent, with a R_{int} of 0.0343. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be $P2_1/c$. The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SHELXT) produced a complete phasing model consistent with the proposed structure.

All nonhydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2014. Crystallographic data are summarized in Table S4.



ORTEP plot of compound **32** (50% probability). Compound **32** was dissolved in methanol and then acetonitrile was carefully layered on top. The vial was loosely capped to allow slow evaporation of the solvents. Two polymorphs appeared from this mixture after several days.

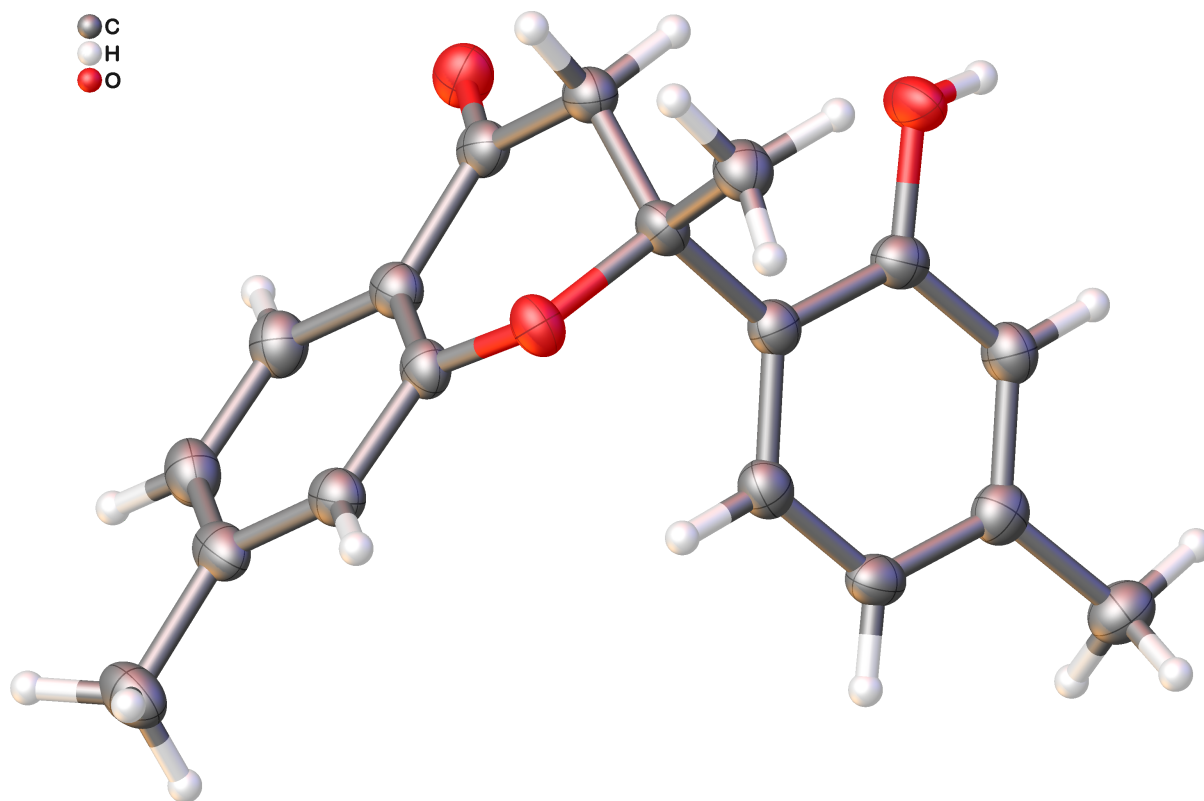
Table S4. Crystal data and structure refinement for **compound 32** (neat).

Report date	2019-05-13	
Identification code	TN_0301Neat	
Empirical formula	C16 H13 Cl O4	
Molecular formula	C16 H13 O4, Cl	
Formula weight	304.71	
Temperature	100.0 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 5.9006(3) Å	$\alpha = 90^\circ$.
	b = 15.9293(9) Å	$\beta = 101.031(2)^\circ$.
	c = 14.4042(9) Å	$\gamma = 90^\circ$.
Volume	1328.87(13) Å ³	
Z	4	
Density (calculated)	1.523 Mg/m ³	
Absorption coefficient	0.301 mm ⁻¹	
F(000)	632	
Crystal size	0.164 x 0.157 x 0.148 mm ³	
Crystal color, habit	Colorless Block	
Theta range for data collection	2.882 to 26.380°.	
Index ranges	-7 ≤ h ≤ 5, -19 ≤ k ≤ 19, -17 ≤ l ≤ 17	
Reflections collected	13855	
Independent reflections	2718 [R(int) = 0.0343, R(sigma) = 0.0269]	

Completeness to $\theta = 25.000^\circ$	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.2602 and 0.2237
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2718 / 0 / 194
Goodness-of-fit on F^2	1.044
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0323$, $wR_2 = 0.0724$
R indices (all data)	$R_1 = 0.0450$, $wR_2 = 0.0809$
Extinction coefficient	n/a
Largest diff. peak and hole	0.266 and -0.263 e. \AA^{-3}

Compound 33: The single crystal X-ray diffraction studies were carried out on a Bruker Kappa Photon II CPAD diffractometer equipped with Mo K $_{\alpha}$ radiation ($\lambda = 0.71073 \text{ \AA}$). A 0.154 x 0.138 x 0.127 mm piece of a colorless block was mounted on a MiTeGen Micromount with CHRISTO-LUBE MCG 1024 oil. Data were collected in a nitrogen gas stream at 100(2) K using ϕ and ω scans. Crystal-to-detector distance was 40 mm and exposure time was 60 seconds per frame using a scan width of 1.0°. Data collection was 99.7% complete to 25.00° in θ . A total of 17236 reflections were collected covering the indices, $-13 \leq h \leq 13$, $-15 \leq k \leq 15$, $-14 \leq l \leq 14$. 2954 reflections were found to be symmetry independent, with a R_{int} of 0.0743. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be $P2_1/n$. The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SHELXT) produced a complete phasing model for refinement.

All nonhydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2014. Crystallographic data are summarized in Table 1.



ORTEP plot of compound **33** (50% probability). Compound **30** was dissolved in a homogenous mixture of water, acetone, acetonitrile, and methanol in a vial. A pentane layer was then carefully added on top and the vial was loosely capped to allow a slow evaporation of solvents. Roughly 1.5-2 months later, the crystals formed from a very small amount of the remaining liquid.

Table S5. Crystal data and structure refinement for **compound 33**.

Report date	2020-03-18	
Identification code	TN_TetramerDecomp	
Empirical formula	C18 H18 O3	
Molecular formula	C18 H18 O3	
Formula weight	282.32	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 10.5278(6) Å	$\alpha = 90^\circ$.
	b = 12.1806(6) Å	$\beta = 99.627(2)^\circ$.
	c = 11.4454(6) Å	$\gamma = 90^\circ$.
Volume	1447.03(13) Å ³	
Z	4	
Density (calculated)	1.296 Mg/m ³	
Absorption coefficient	0.087 mm ⁻¹	
F(000)	600	
Crystal size	0.154 x 0.138 x 0.127 mm ³	
Crystal color, habit	Colorless Block	
Theta range for data collection	2.953 to 26.382°.	
Index ranges	-13 ≤ h ≤ 13, -15 ≤ k ≤ 15, -14 ≤ l ≤ 14	
Reflections collected	17236	

Independent reflections	2954 [R(int) = 0.0743, R(sigma) = 0.0466]
Completeness to theta = 25.000°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.2602 and 0.2161
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2954 / 0 / 195
Goodness-of-fit on F ²	1.066
Final R indices [I>2sigma(I)]	R1 = 0.0457, wR2 = 0.1092
R indices (all data)	R1 = 0.0918, wR2 = 0.1402
Extinction coefficient	0.021(4)
Largest diff. peak and hole	0.217 and -0.235 e.Å ⁻³