Supporting Information

N-Methylimidazole Promotes the Reaction of Homophthalic Anhydride with Imines

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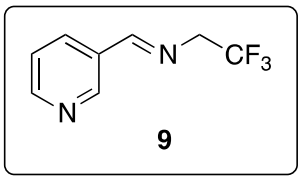
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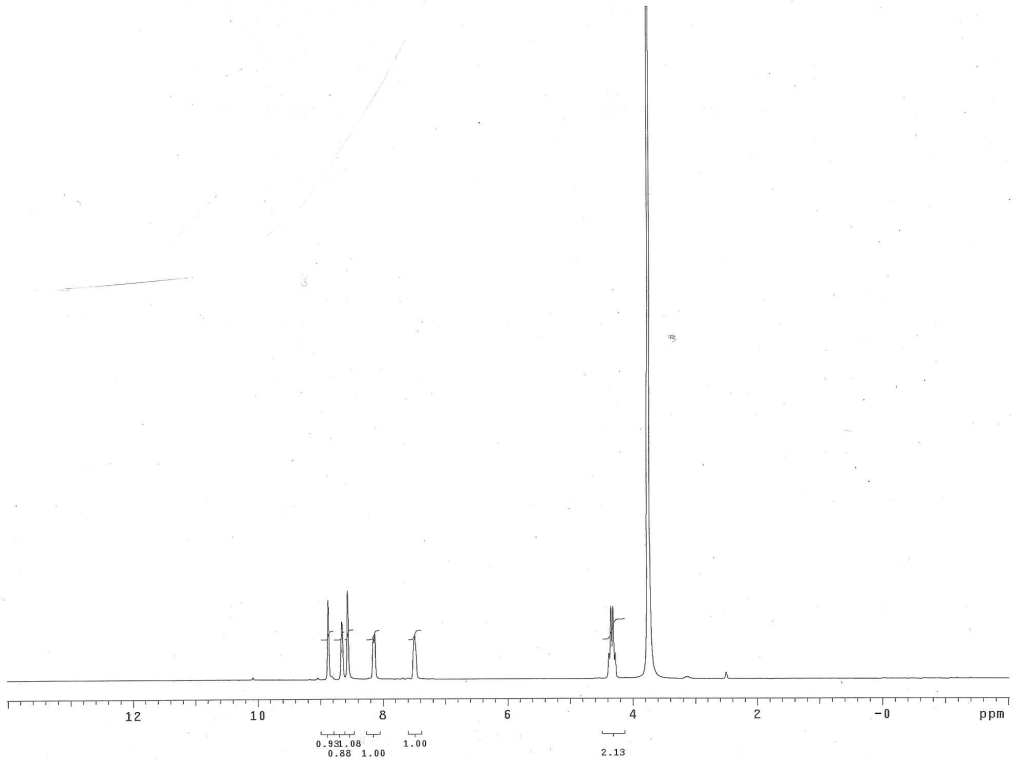
CF3
N
9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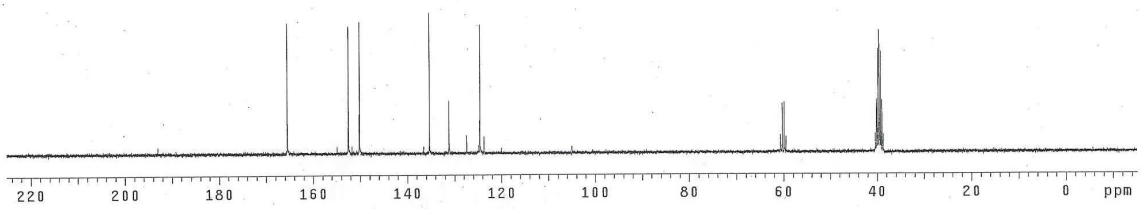
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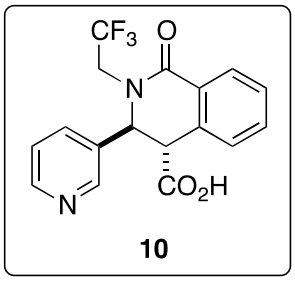
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O
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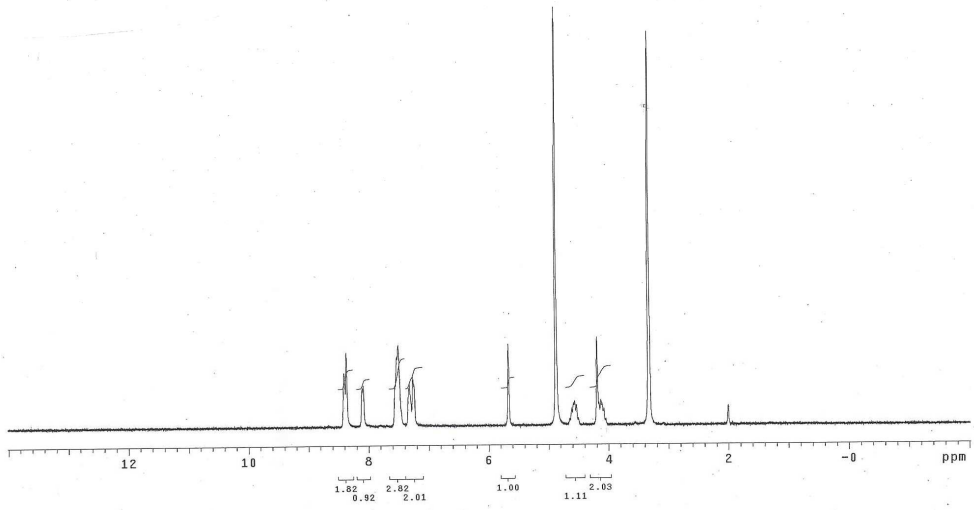
CO2H
N
10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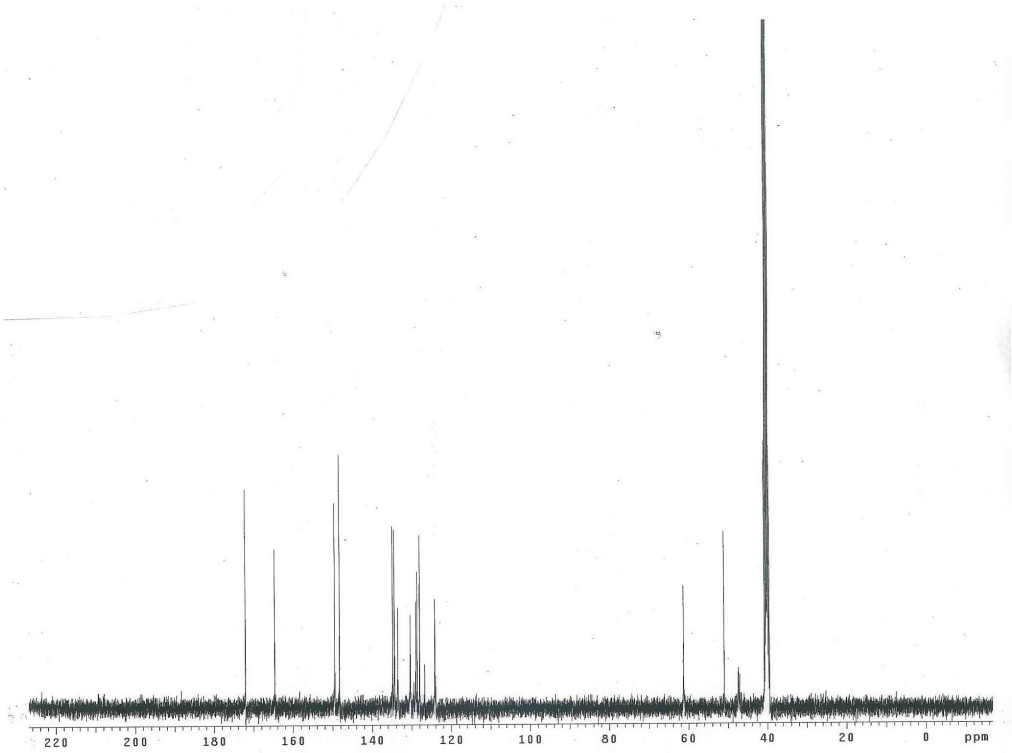
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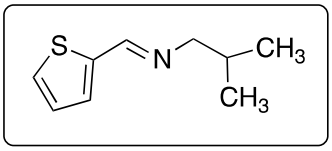
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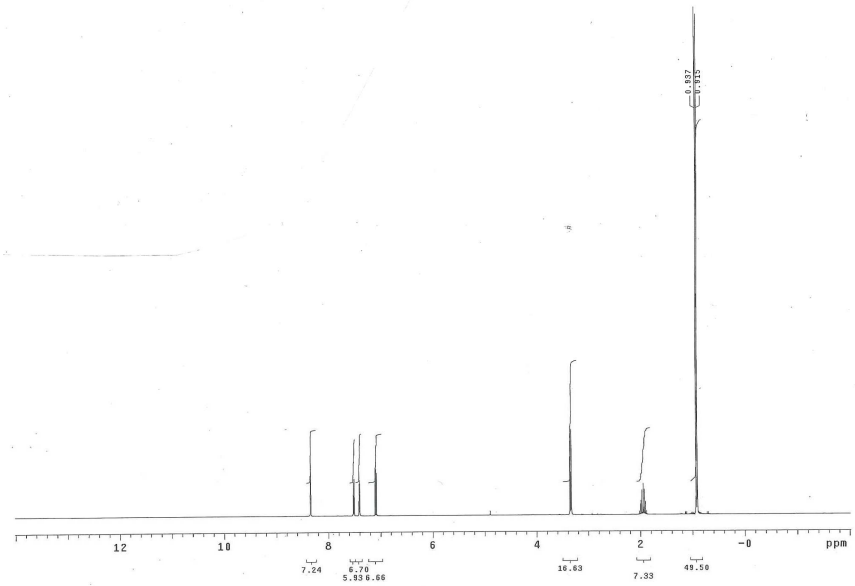
N
CH3
CH3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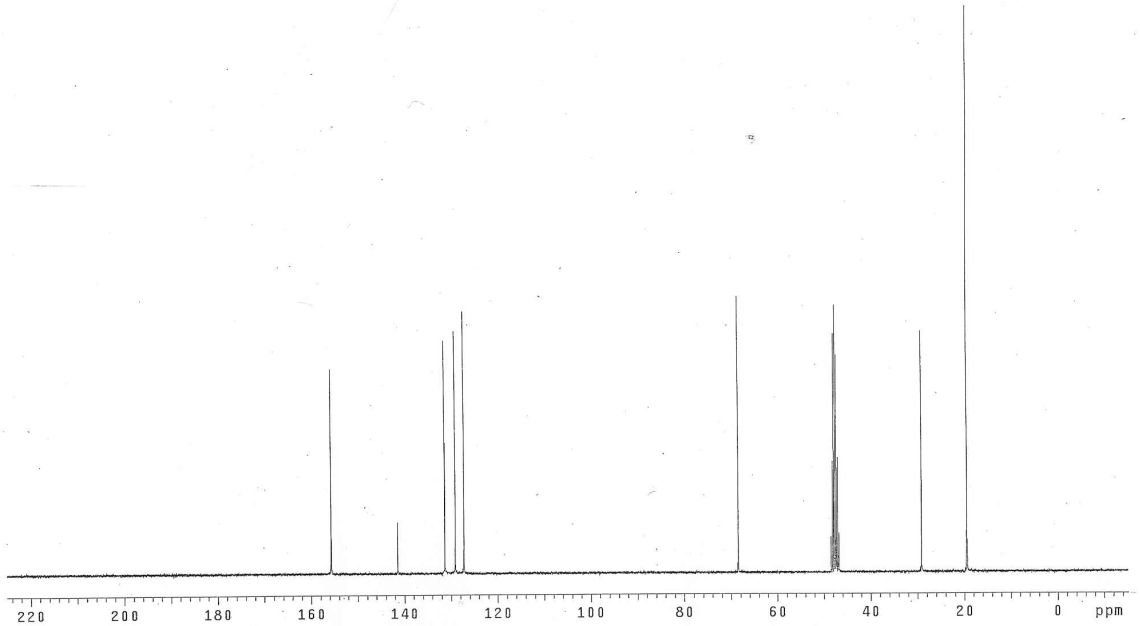
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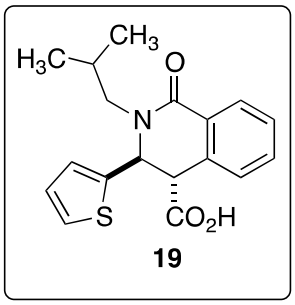
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H3C
O
N
S
CO2H
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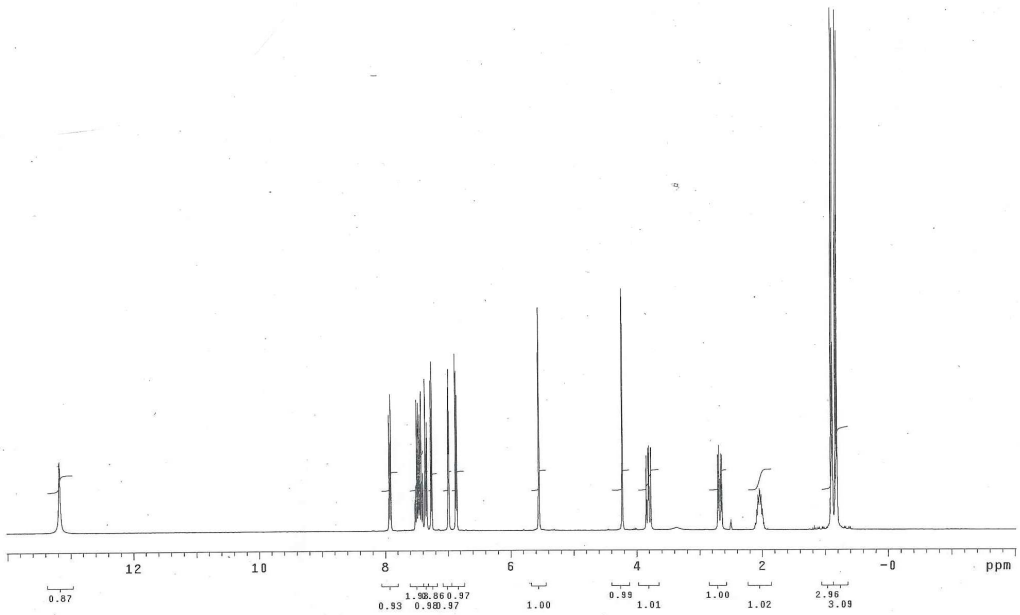
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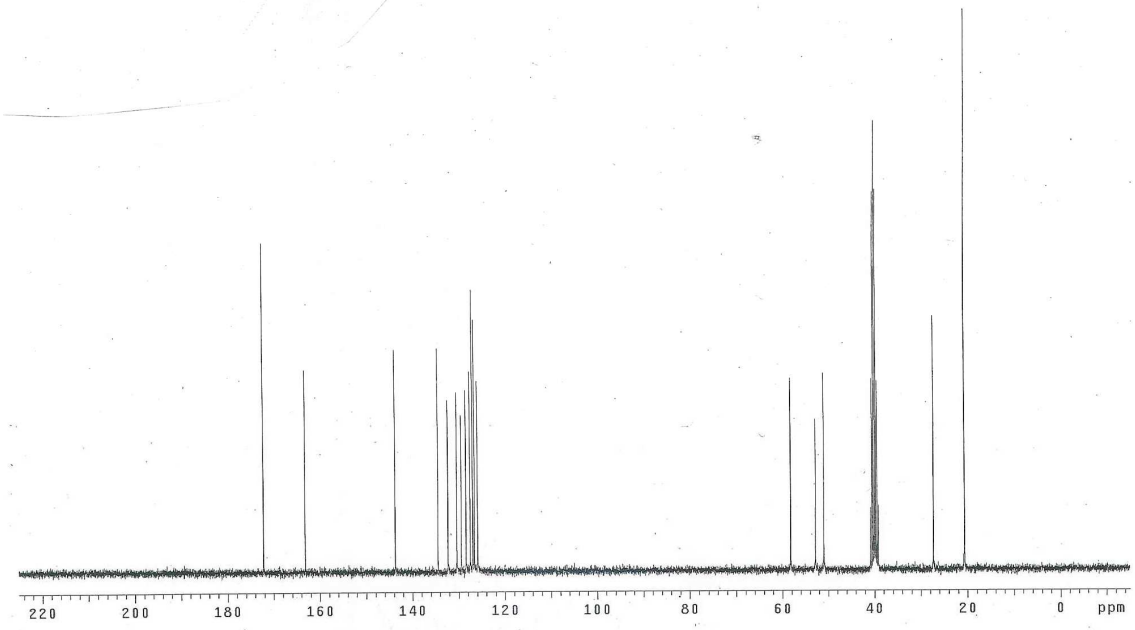
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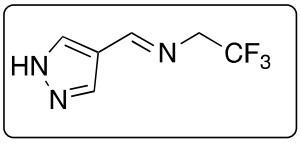
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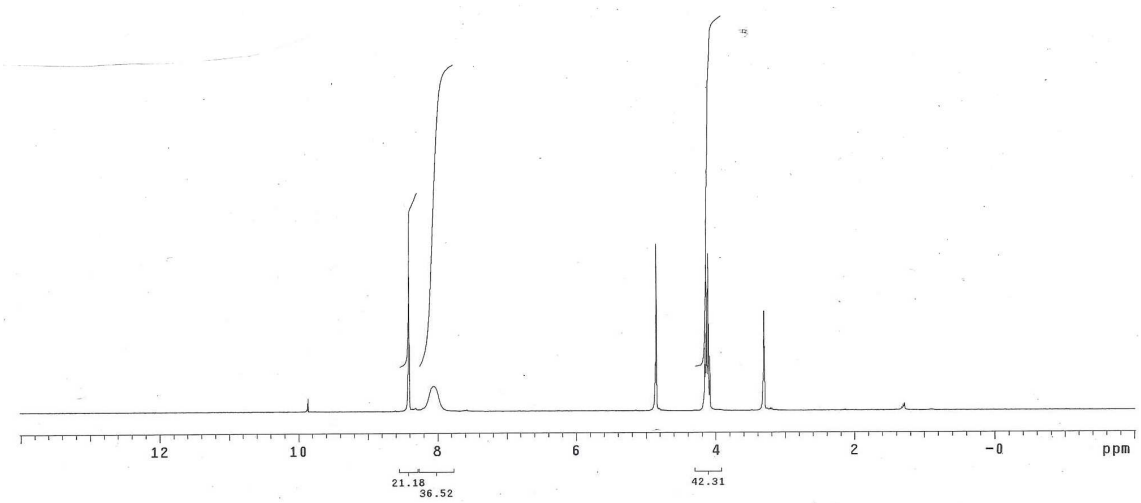
N
N
CF3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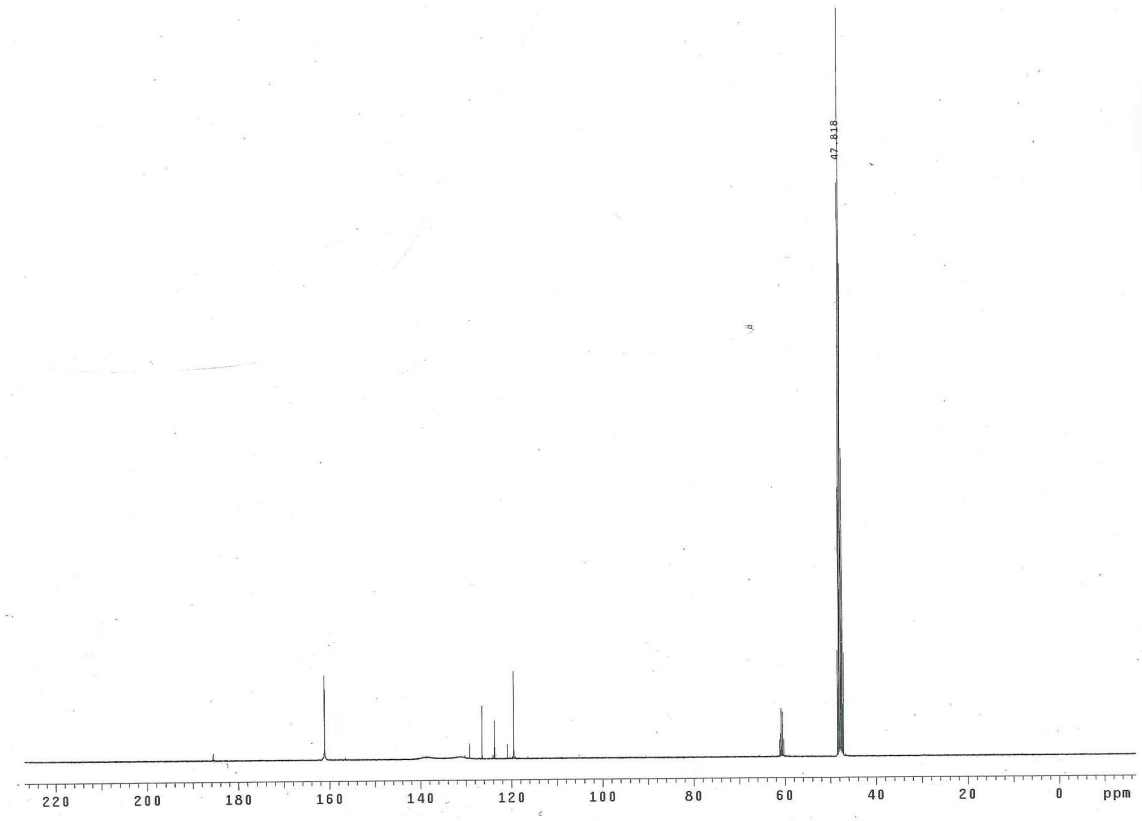
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180
160
140
120
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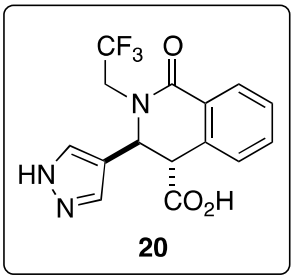
80
60
40
20
0
ppm


CF3
O
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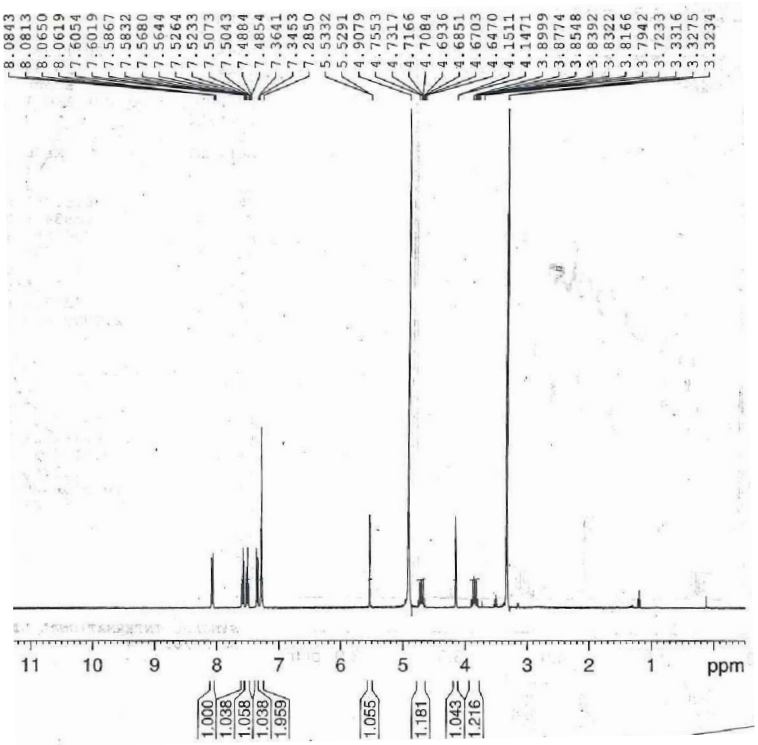
HN
N
CO2H
20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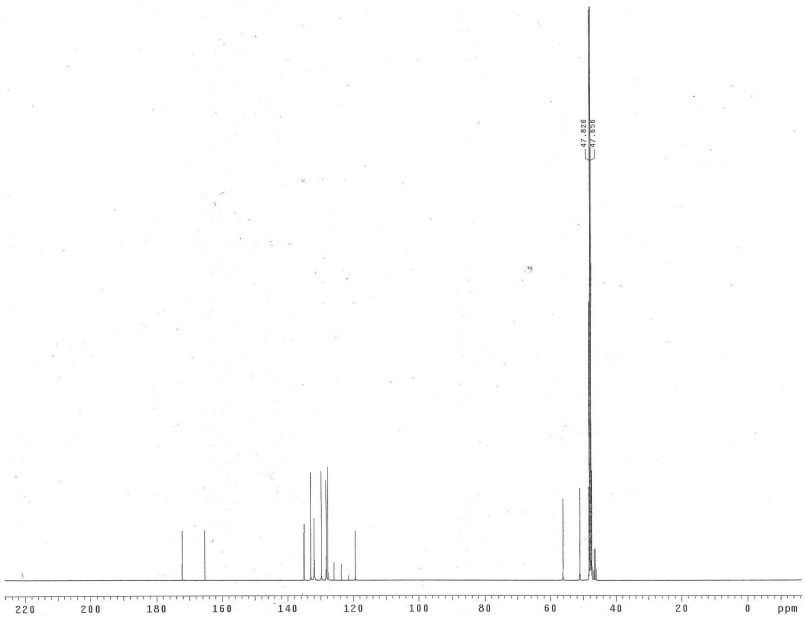
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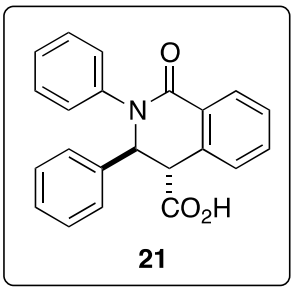
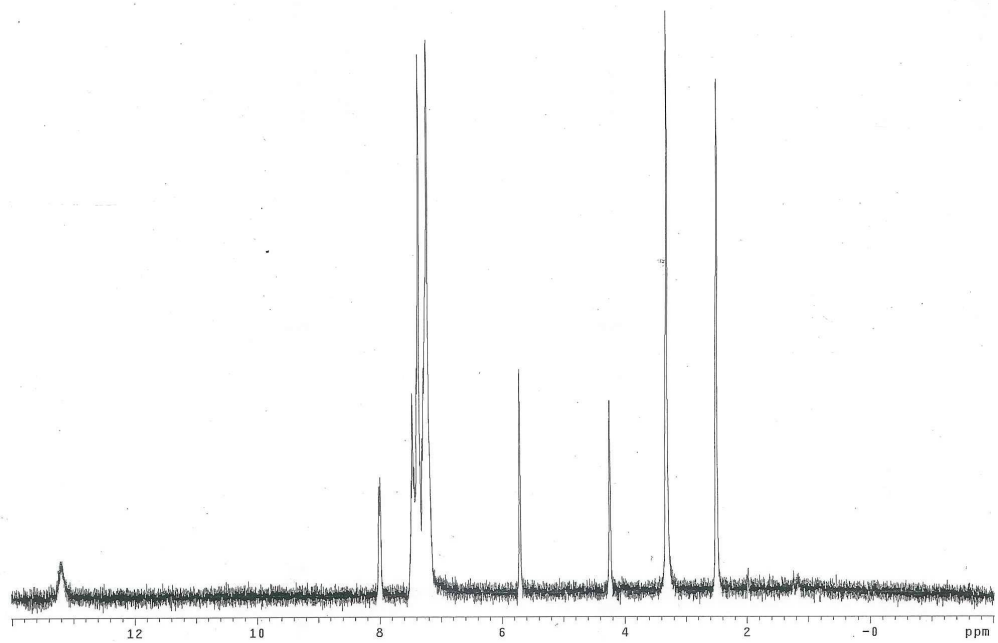

O
N
CO2H

21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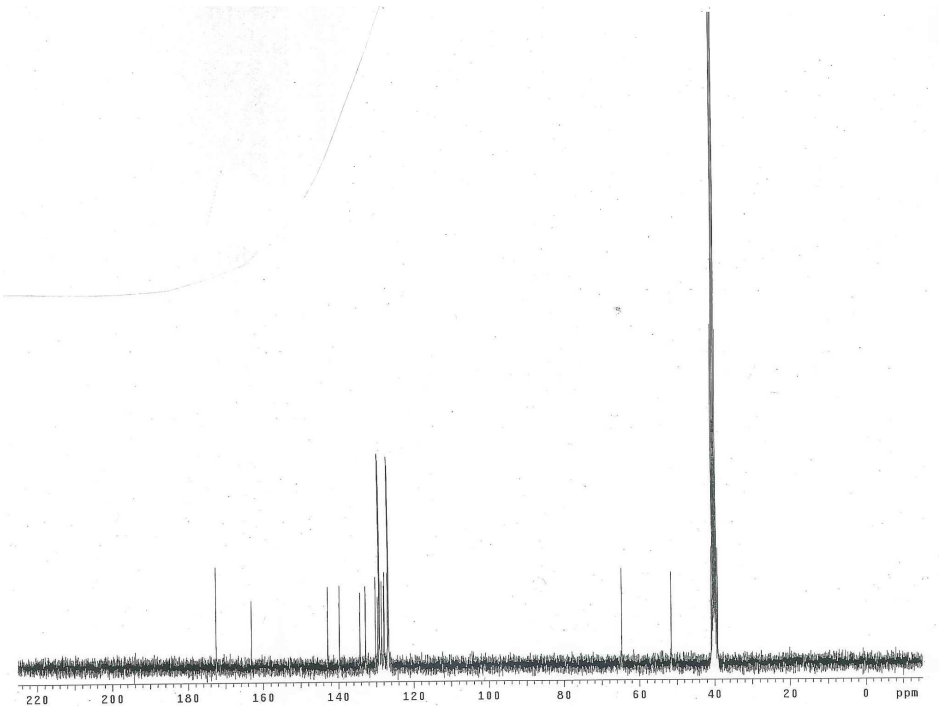
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20
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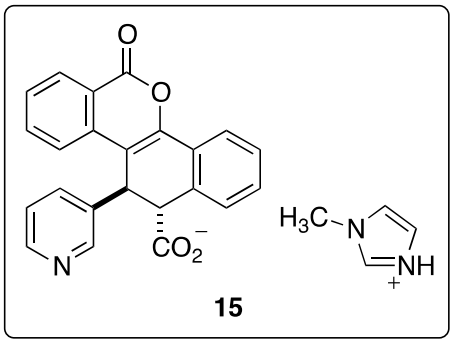
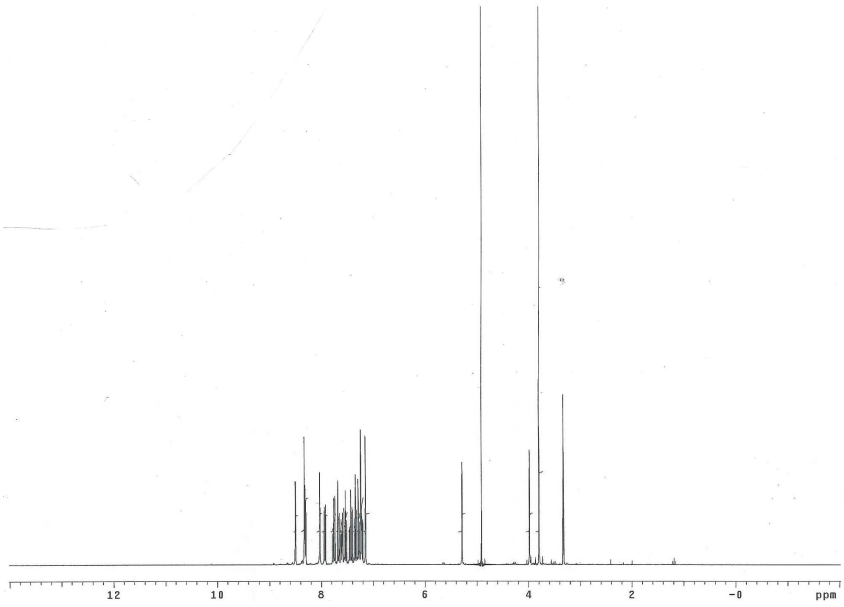

O
O
N
CO_2^-
H_3C
N
NH
+
15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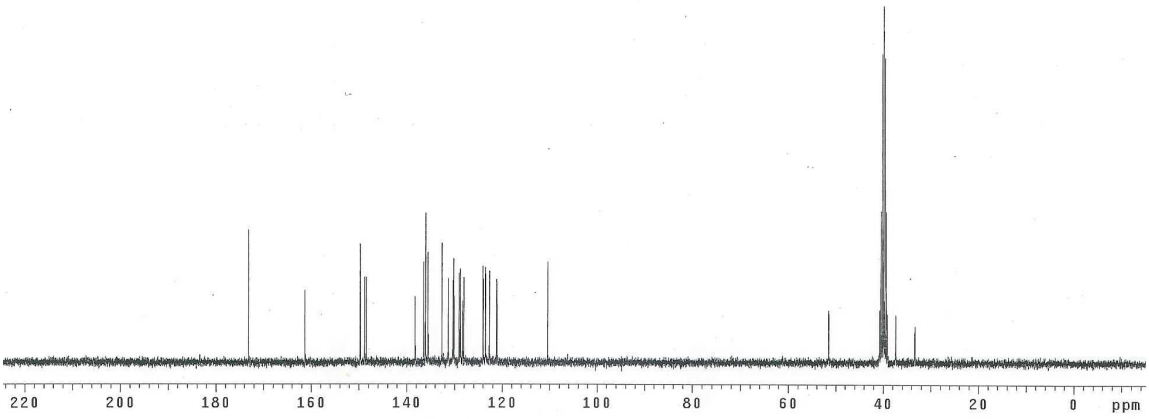
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60
40
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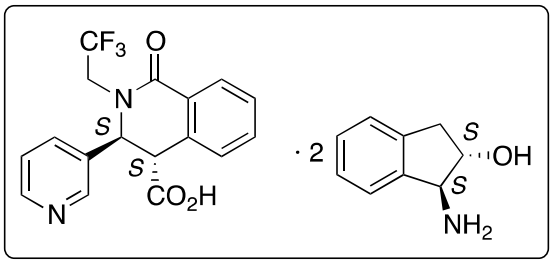
ppm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O
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S
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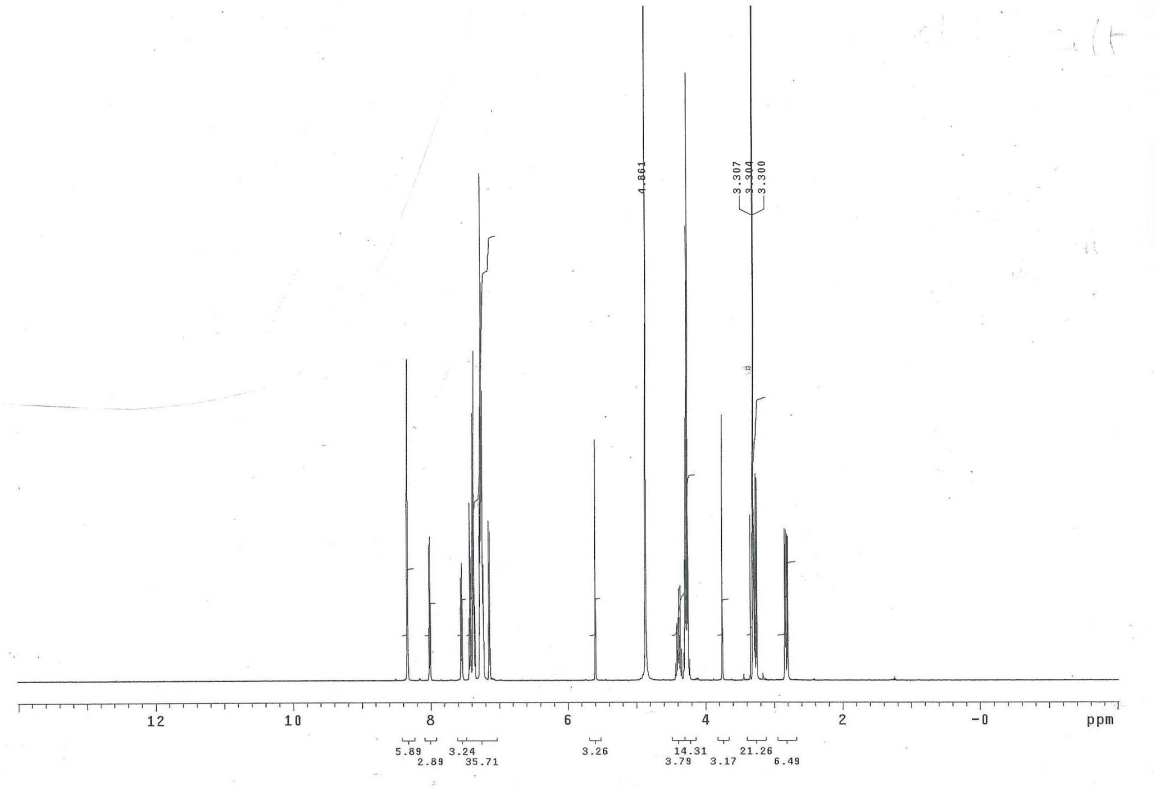
CO2H
N
· 2
S
OH
S
NH2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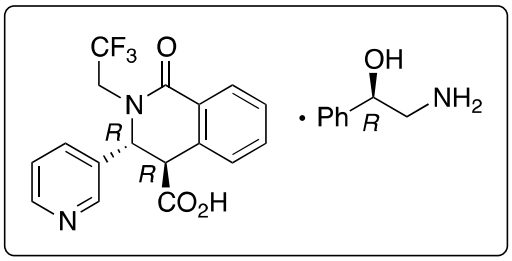
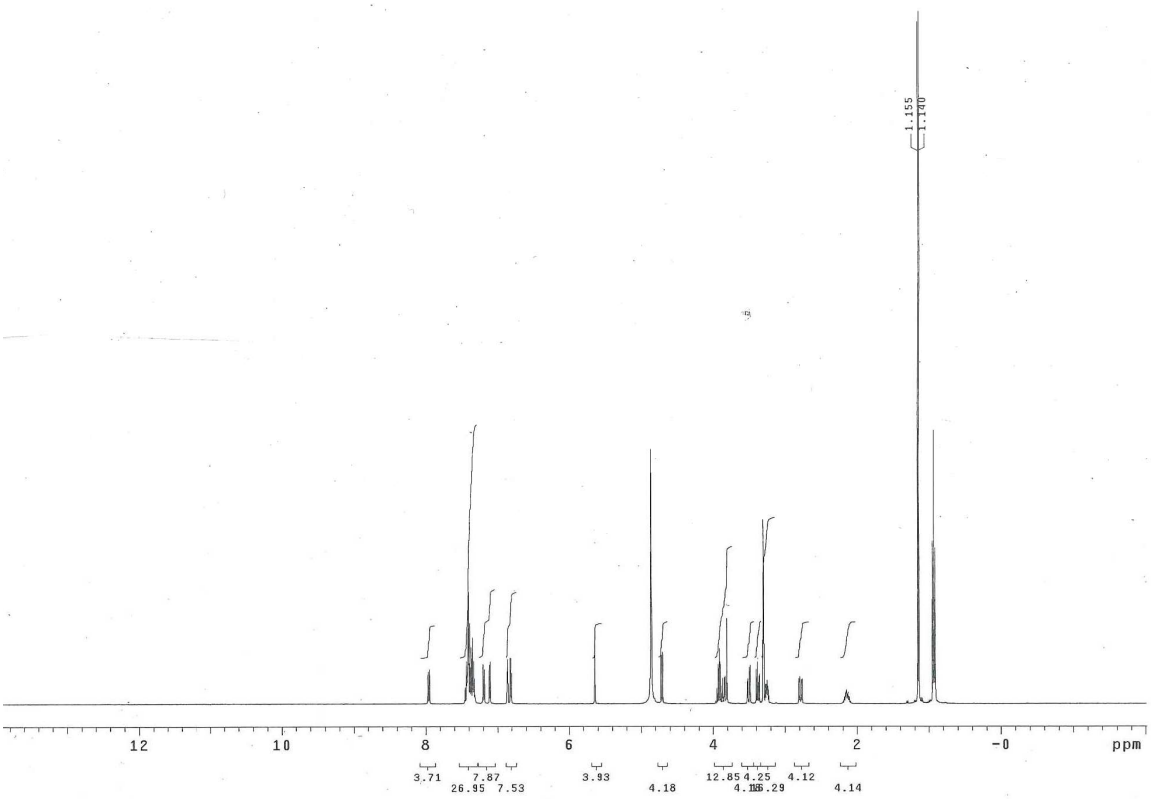

CF3
O
N
R
R
CO2H
N
• Ph
OH
R
NH2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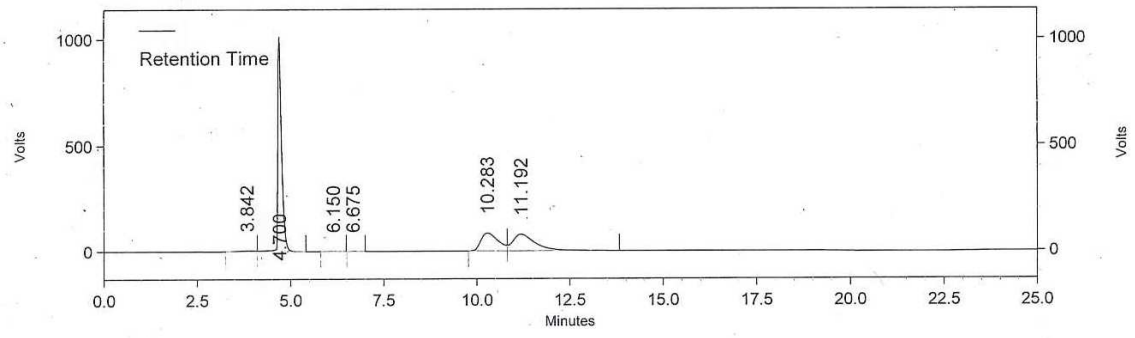
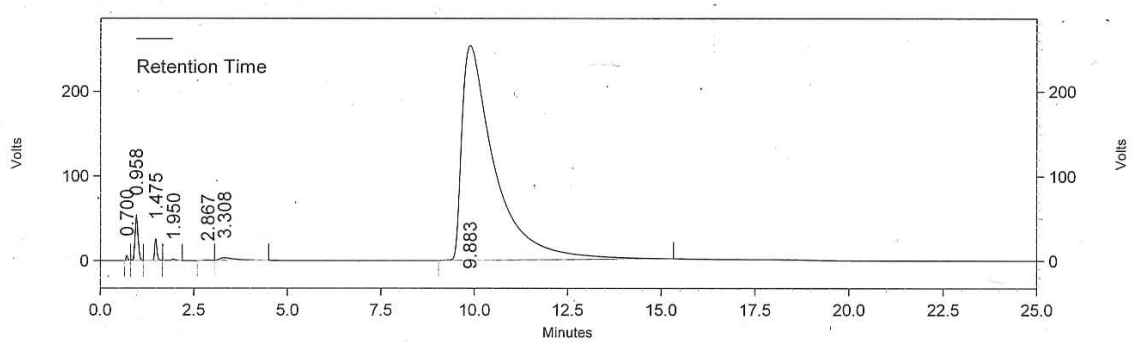
4.14

Chiral HPLC traces of *rac*-**10** (top) and (+)-**10** (bottom)





General Methods. All reactions were run in small, capped vials under a nitrogen atmosphere. Flash chromatography was performed by using disposable silica columns. Silica gel 60 F_{254} pre-coated plates were used for thin layer chromatography, and visualization was accomplished with UV light (254 nm) and iodine stain. Melting points are uncorrected. High-resolution ESI mass spectra were detected by using a quadrupole time-of-flight mass analyzer. ^{1}H and ^{13}C NMR spectra were obtained on a 300, 400, or 500 MHz instruments. Chemical shifts (δ) are reported in parts per million (ppm) and are referenced to the residual solvent peak. Coupling constants (*J*) are reported in hertz (Hz). The usual abbreviations are used to designate multiplicities: s=singlet; d=doublet; t=triplet; q=quartet. Commercial NMR solvents were used as received: chloroform-d (99.8% D), methanol-d_4 (99.8% D), dichloromethane-d_2 (99.9% D), and dimethyl sulfoxide-d_6 (99.9% D). All other commercially available reagents were used as received and without any further purification.

Calculational Methods

Full Gaussian 09 reference:

Gaussian 09, Revision A.02, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2009.

Structure 14 (2S,3S,4R) at B3LYP/6-31G(d):

NImag=0
E(RB3LYP) = -1429.71159863
Thermal correction to Gibbs Free Energy= 0.288172
Sum of electronic and thermal Free Energies= -1429.423427

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	6	0	2.763977	-1.131103	1.530595
2	8	0	1.601345	-0.833542	2.248974
3	6	0	0.347132	-0.717448	1.721570
4	6	0	0.139274	-0.819144	0.206974
5	6	0	1.371011	-1.293533	-0.547079
6	6	0	1.296724	-1.591566	-1.913155
7	6	0	2.431199	-1.981616	-2.619411
8	6	0	3.669143	-2.080790	-1.974613
9	6	0	3.757299	-1.795235	-0.619946
10	6	0	2.611033	-1.405610	0.091730
11	8	0	3.794119	-1.154152	2.149516
12	8	0	-0.566284	-0.504161	2.480491
13	6	0	-0.314651	0.584435	-0.350705
14	6	0	-1.697793	1.034596	0.177181
15	6	0	-2.762581	-0.052844	0.075768
16	6	0	-4.122064	0.283416	0.085660
17	6	0	-5.104978	-0.700756	0.009070
18	6	0	-4.749506	-2.050425	-0.076043
19	6	0	-3.407271	-2.400251	-0.072384
20	6	0	-2.408742	-1.413068	0.007405
21	6	0	-1.002193	-1.877507	0.045108
22	6	0	0.744752	1.663564	-0.169975
23	6	0	1.485709	2.105367	-1.276417
24	7	0	2.438445	3.040142	-1.228726
25	6	0	2.697187	3.591998	-0.036928
26	6	0	2.019496	3.243795	1.131144
27	6	0	1.026776	2.267975	1.062661
28	6	0	-2.122328	2.253965	-0.642174
29	8	0	-2.010040	2.362104	-1.842018
30	8	0	-2.664096	3.221381	0.134774
31	8	0	-0.703054	-3.056545	-0.017134
32	1	0	0.343032	-1.533075	-2.429848
33	1	0	2.349212	-2.212023	-3.677888
34	1	0	4.552887	-2.382563	-2.528501
35	1	0	4.697892	-1.866123	-0.084623
36	1	0	-0.443230	0.437858	-1.428064
37	1	0	-1.625225	1.336274	1.225666
38	1	0	-4.416803	1.325716	0.160237

39	1	0	-6.152493	-0.412001	0.016552
40	1	0	-5.515576	-2.817840	-0.136228
41	1	0	-3.093335	-3.437431	-0.121880
42	1	0	1.288562	1.677139	-2.258203
43	1	0	3.479321	4.349024	-0.018234
44	1	0	2.262001	3.724814	2.073817
45	1	0	0.485902	1.987818	1.962205
46	1	0	-2.908816	3.951454	-0.466962

Proton Chemical Shift Values:
Actual command: #n BLYP/cc-pVDZ nmr=GIAO SCRF(solvent=chloroform) iop(3/76=1000001189,3/77=0961409999,3/78=0000109999)
Standard command: #n BLYP/cc-pVDZ nmr=GIAO SCRF(solvent=chloroform) iop(3/76=1000001189,3/77=0961409999,3/78=0000109999)
RMS deviation on test set: before scaling = 0.14 after scaling = 0.12
(scaled chemical shift) = (31.8440 - (unscaled shielding)) / 1.0205

Atom	Shielding	Chem. Shift (scaled)
H32	24.5060	7.1906
H33	24.0867	7.6015
H34	24.3460	7.3474
H35	23.8506	7.8328
H36	27.5635	4.1945
H37	26.9328	4.8125
H38	24.2963	7.3961
H39	24.1525	7.5370
H40	24.3143	7.3784
H41	23.5606	8.1170
H42	24.0439	7.6434
H43	23.3877	8.2864
H44	24.6253	7.0737
H45	24.0874	7.6008
H46	25.3063	6.4064

Proton Coupling Constants:
Actual command: #n B3LYP/gen nmr=(FCOnly,ReadAtoms) int=nobasistransform geom=check
Standard command: #n B3LYP/gen nmr=(FCOnly,ReadAtoms) int=nobasistransform
RMS deviation on test set: before scaling = 0.93 after scaling = 0.51
(scaled J-value) = 0.9155 * (unscaled J-value)) + 0.0000

Unscaled coupling constants:

	H32	H33	H34	H35	H36	H37	H38	H39	H40	H41	H42	H43	H44	H45	H46
H32															
H33	8.79														
H34	1.01	8.11													
H35	0.60	1.40	8.81												
H36	-0.35	-0.02	0.03	-0.04											
H37	0.08	-0.01	0.01	-0.01	12.52										
H38	-0.00	0.00	0.00	-0.00	0.05	-1.42									
H39	0.00	-0.00	-0.00	-0.00	-0.06	0.56	8.73								
H40	-0.00	0.00	-0.00	0.00	0.04	-1.14	1.09	8.04							
H41	0.00	-0.00	0.00	0.00	-0.06	0.47	0.59	1.46	8.84						
H42	0.06	-0.01	0.00	-0.01	-0.39	0.02	-0.01	-0.00	-0.00	0.00					
H43	-0.00	0.00	-0.00	-0.00	-0.06	0.02	-0.00	-0.00	-0.00	0.00	-0.58				
H44	0.01	-0.00	0.00	-0.00	0.51	-0.01	0.00	0.00	0.00	-0.00	0.92	5.27			
H45	-0.00	0.00	-0.00	-0.00	-0.46	-0.31	-0.01	0.00	-0.01	0.00	2.50	1.57	8.84		

H46 -0.01 0.00 -0.01 0.01 -0.06 1.07 0.08 -0.00 -0.00
0.03 -0.00 -0.00 0.00 -0.00

Scaled coupling constants:

	H32	H33	H34	H35	H36	H37	H38	H39	H40	H41	H42	H43	H44	H45	H46
H32															
H33	8.05														
H34	0.93	7.43													
H35	0.55	1.28	8.06												
H36	-0.32	-0.02	0.02	-0.04											
H37	0.07	-0.01	0.01	-0.01	11.46										
H38	-0.00	0.00	0.00	-0.00	0.05	-1.30									
H39	0.00	-0.00	-0.00	-0.00	-0.05	0.51	7.99								
H40	-0.00	0.00	-0.00	0.00	0.04	-1.04	1.00	7.36							
H41	0.00	-0.00	0.00	0.00	-0.05	0.43	0.54	1.34	8.09						
H42	0.05	-0.01	0.00	-0.01	-0.35	0.02	-0.00	-0.00	-0.00	0.00					
H43	-0.00	0.00	-0.00	-0.00	-0.05	0.02	-0.00	-0.00	-0.00	0.00	-0.53				
H44	0.01	-0.00	0.00	-0.00	0.47	-0.01	0.00	0.00	0.00	-0.00	0.84	4.82			
H45	-0.00	0.00	-0.00	-0.00	-0.42	-0.28	-0.01	0.00	-0.01	0.00	2.29	1.44	8.09		
H46	-0.00	0.00	-0.01	0.00	-0.05	0.98	0.08	-0.00	-0.00	0.03	-0.00	-0.00	0.00	-0.00	

Structure 14 (2R,3S,4R) at B3LYP/6-31G(d):
NImag=0
E(RB3LYP) = -1429.70656987
Thermal correction to Gibbs Free Energy= 0.288339
Sum of electronic and thermal Free Energies= -1429.418231

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	6	0	2.964163	-1.537305	-0.721736
2	8	0	2.307066	-1.110324	-1.866142
3	6	0	0.981721	-0.768565	-1.938309
4	6	0	0.123939	-0.736318	-0.661174
5	6	0	0.789771	-1.392674	0.542063
6	6	0	0.078340	-1.679044	1.717370
7	6	0	0.712173	-2.258978	2.815188
8	6	0	2.073133	-2.572826	2.765140
9	6	0	2.786480	-2.317754	1.601664
10	6	0	2.148507	-1.738715	0.494575
11	8	0	4.147170	-1.741288	-0.802721
12	8	0	0.540582	-0.430505	-3.002759
13	6	0	-0.234340	0.789029	-0.472657
14	6	0	-1.391779	1.014370	0.524532
15	6	0	-2.601543	0.131155	0.222996
16	6	0	-3.871942	0.479960	0.700603
17	6	0	-4.984116	-0.311962	0.423913
18	6	0	-4.850251	-1.479452	-0.334405
19	6	0	-3.595837	-1.850295	-0.796485
20	6	0	-2.467138	-1.057912	-0.521441
21	6	0	-1.152963	-1.559420	-1.001341
22	6	0	0.977086	1.658373	-0.171519
23	6	0	1.507188	2.477210	-1.181817
24	7	0	2.560050	3.284996	-1.026981
25	6	0	3.144331	3.309826	0.176240
26	6	0	2.704877	2.545968	1.257658
27	6	0	1.604763	1.709594	1.080340
28	6	0	-1.795946	2.488539	0.446651
29	8	0	-1.936490	3.121802	-0.572224
30	8	0	-2.000675	3.016478	1.678537
31	8	0	-1.036356	-2.615363	-1.595182
32	1	0	-0.984399	-1.471369	1.776647
33	1	0	0.135909	-2.472945	3.711066
34	1	0	2.565265	-3.024467	3.621195
35	1	0	3.839422	-2.563334	1.515755
36	1	0	-0.620576	1.099188	-1.448540
37	1	0	-1.059139	0.824877	1.550763
38	1	0	-3.993575	1.382281	1.292355
39	1	0	-5.959332	-0.014437	0.799450
40	1	0	-5.718452	-2.093682	-0.554222
41	1	0	-3.449463	-2.756798	-1.374183
42	1	0	1.046468	2.477382	-2.168476
43	1	0	4.001003	3.973652	0.280086
44	1	0	3.212135	2.605086	2.216043
45	1	0	1.245629	1.107051	1.909542
46	1	0	-2.255323	3.949324	1.537045

Proton Chemical Shift Values:
Actual command: #n BLYP/cc-pVDZ nmr=GIAO SCRF(solvent=chloroform) iop(3/76=1000001189,3/77=0961409999,3/78=0000109999)
Standard command: #n BLYP/cc-pVDZ nmr=GIAO SCRF(solvent=chloroform) iop(3/76=1000001189,3/77=0961409999,3/78=0000109999)
RMS deviation on test set: before scaling = 0.14 after scaling = 0.12
(scaled chemical shift) = (31.8440 - (unscaled shielding)) / 1.0205

Atom	Shielding	Chem. Shift (scaled)
H32	24.8697	6.8342
H33	24.2362	7.4550
H34	24.2672	7.4246
H35	23.6000	8.0784
H36	27.1193	4.6298
H37	27.4588	4.2971
H38	24.2879	7.4043
H39	24.1084	7.5802
H40	24.2255	7.4655
H41	23.4652	8.2105
H42	23.3693	8.3045
H43	23.3057	8.3668
H44	25.0171	6.6898
H45	25.4165	6.2984
H46	25.3629	6.3509

Proton Coupling Constants:
Actual command: #n B3LYP/gen nmr=(FCOnly,ReadAtoms) int=nobasistransform geom=check
Standard command: #n B3LYP/gen nmr=(FCOnly,ReadAtoms) int=nobasistransform
RMS deviation on test set: before scaling = 0.93 after scaling = 0.51
(scaled J-value) = 0.9155 * (unscaled J-value)) + 0.0000

Unscaled coupling constants:

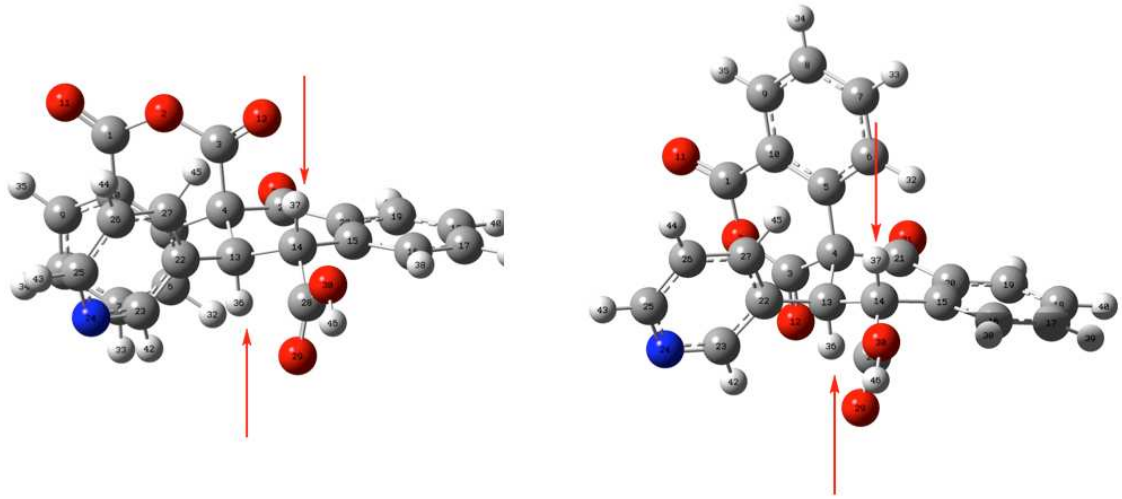
	H32	H33	H34	H35	H36	H37	H38	H39	H40	H41	H42	H43	H44	H45	H46
H32															
H33	8.78														
H34	1.03	8.10													
H35	0.60	1.48	8.77												
H36	0.12	0.05	-0.05	0.12											
H37	-0.19	-0.02	0.01	-0.01	13.51										
H38	0.01	-0.00	0.00	-0.00	0.05	-1.40									
H39	-0.01	-0.00	-0.00	0.00	-0.06	0.56	8.72								
H40	0.01	0.00	-0.00	0.00	0.04	-1.13	1.09	8.05							
H41	-0.02	-0.00	-0.00	0.00	-0.06	0.47	0.59	1.46	8.84	0.00					
H42	-0.00	0.00	-0.00	0.00	-0.38	0.03	-0.01	-0.00	0.00	0.00	-0.59				
H43	0.00	-0.00	-0.00	-0.00	-0.07	0.02	-0.00	-0.00	-0.00	0.00	0.92	5.26			
H44	0.00	-0.00	0.00	-0.00	0.51	-0.00	0.00	0.00	0.00	-0.00	2.44	1.63	8.74		
H45	0.01	0.01	-0.02	0.01	-0.45	-0.30	-0.01	0.00	-0.01	0.03	-0.00	-0.00	0.00	-0.00	
H46	-0.00	0.00	-0.00	0.00	-0.06	1.13	0.08	-0.00	-0.00	0.03	-0.00	-0.00	0.00	-0.00	

Scaled coupling constants:

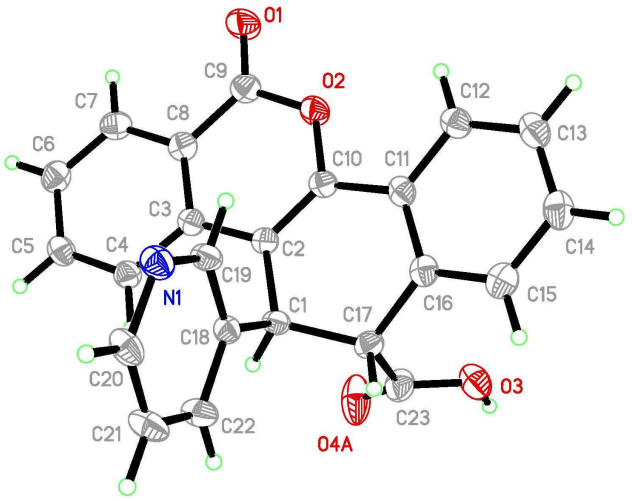
	H32	H33	H34	H35	H36	H37	H38	H39	H40	H41	H42	H43	H44	H45	H46
H32															

H33	8.04											
H34	0.94	7.42										
H35	0.55	1.35	8.03									
H36	0.11	0.04	-0.04	0.11								
H37	-0.17	-0.02	0.01	-0.01	12.37							
H38	0.00	-0.00	0.00	-0.00	0.05	-1.29						
H39	-0.01	-0.00	-0.00	0.00	-0.05	0.51	7.98					
H40	0.01	0.00	-0.00	0.00	0.04	-1.04	1.00	7.37				
H41	-0.02	-0.00	-0.00	0.00	-0.05	0.43	0.54	1.34	8.09			
H42	-0.00	0.00	-0.00	0.00	-0.35	0.03	-0.00	-0.00	0.00	0.00		
H43	0.00	-0.00	-0.00	-0.00	-0.06	0.02	-0.00	-0.00	-0.00	0.00	-0.54	
H44	0.00	-0.00	0.00	-0.00	0.47	-0.00	0.00	0.00	0.00	-0.00	0.84	4.81
H45	0.01	0.01	-0.02	0.01	-0.41	-0.27	-0.01	0.00	-0.01	0.00	2.24	1.49	8.00
H46	-0.00	0.00	-0.00	0.00	-0.05	1.04	0.07	-0.00	-0.00	0.03	-0.00	-0.00	0.00	-0.00

Calculated ball-and-stick structures for (2*S**,3*S**,4*R**)-**14** (major isomer, left) and (2*R**,3*S**,4*R**)-**14** (minor isomer, right). The red arrows indicate the respective pseudo-*trans*-diaxial vicinal ring protons that together give rise to four wide doublets in the H-1 NMR spectrum (see text, Figure 2, for line structures and arbitrary 1-tetralone ring carbon numbering).



ORTEP drawings for the dibenzodihydroisocoumarin **15**, top, and (–)-**10** as the (R)-2-amino-1-phenylethanol salt, bottom. Isopropanols of crystallization are not shown.



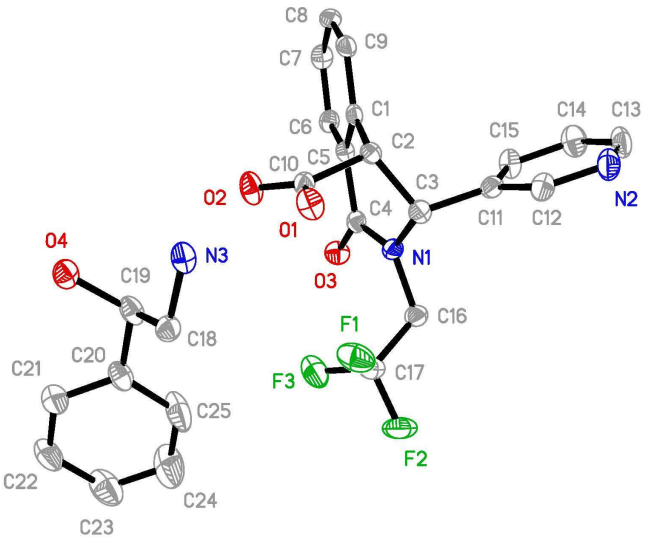


Table S1. Crystal data and structure refinement for **15**.

Identification code	**15**	
Empirical formula	C24.50 H19 N O4.50	
Formula weight	399.41	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.2269(6) Å	α= 90°.
	b = 15.8840(9) Å	β= 98.281(1)°.
	c = 12.1416(7) Å	γ = 90°.
Volume	1951.77(19) $Å^3$	
Z	4	
Density (calculated)	1.359 Mg/m^3	
Absorption coefficient	0.094 mm^{-1}	
F(000)	836	
Crystal size	0.22 x 0.18 x 0.14 mm^3	
Theta range for data collection	2.13 to 28.28°.	
Index ranges	-13<=h<=13, -21<=k<=21, -16<=l<=15	
Reflections collected	18279	
Independent reflections	4845 [R(int) = 0.0485]	
Completeness to theta = 28.28°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7458 and 0.6865	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4845 / 42 / 292	
Goodness-of-fit on F^2	1.000	
Final R indices [I>2sigma(I)]	R1 = 0.0725, wR2 = 0.1644	
R indices (all data)	R1 = 0.1040, wR2 = 0.1824	
Largest diff. peak and hole	0.614 and -0.484 e.$Å^{-3}$	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **15**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	6325(2)	9430(1)	-464(2)	36(1)
O(2)	5519(2)	8930(1)	990(2)	26(1)
O(3)	4042(2)	6530(1)	3550(2)	34(1)
O(4A)	3163(6)	6283(5)	1832(6)	47(2)
O(4B)	2559(6)	6234(5)	2047(6)	47(2)
N(1)	330(2)	9977(1)	1390(2)	25(1)
C(1)	2291(2)	7946(1)	1458(2)	20(1)
C(2)	3392(2)	8275(1)	870(2)	19(1)
C(3)	3242(2)	8332(2)	-334(2)	21(1)
C(4)	2122(2)	8046(2)	-1041(2)	23(1)
C(5)	2013(3)	8165(2)	-2176(2)	26(1)
C(6)	3006(3)	8568(2)	-2649(2)	28(1)
C(7)	4129(3)	8846(2)	-1974(2)	27(1)
C(8)	4243(2)	8730(2)	-830(2)	23(1)
C(9)	5431(2)	9059(2)	-137(2)	26(1)
C(10)	4497(2)	8584(2)	1470(2)	21(1)
C(11)	4718(2)	8642(2)	2684(2)	22(1)
C(12)	5766(2)	9110(2)	3258(2)	25(1)
C(13)	5909(3)	9173(2)	4407(2)	28(1)
C(14)	5011(3)	8782(2)	4986(2)	29(1)
C(15)	3980(3)	8313(2)	4423(2)	26(1)
C(16)	3830(2)	8225(2)	3272(2)	21(1)
C(17)	2791(2)	7655(2)	2657(2)	22(1)
C(18)	1169(2)	8570(1)	1444(2)	19(1)
C(19)	1337(2)	9431(2)	1395(2)	22(1)
C(20)	-887(2)	9683(2)	1431(2)	26(1)
C(21)	-1139(2)	8836(2)	1500(2)	30(1)
C(22)	-103(2)	8279(2)	1510(2)	26(1)
C(23)	3260(3)	6730(2)	2673(2)	29(1)
O(5)	3807(5)	4716(4)	931(5)	66(2)
C(24)	5220(8)	3778(6)	166(8)	70(2)

C(25)	5020(60)	4632(17)	550(30)	92(5)
C(26)	5030(50)	5219(18)	-350(30)	92(5)

Table S3. Bond lengths [Å] and angles [°] for **15**.

O(1)-C(9)	1.202(3)	C(12)-H(12)	0.9500
O(2)-C(9)	1.374(3)	C(13)-C(14)	1.382(4)
O(2)-C(10)	1.381(3)	C(13)-H(13)	0.9500
O(3)-C(23)	1.277(3)	C(14)-C(15)	1.388(4)
O(3)-H(3O)	0.912(10)	C(14)-H(14)	0.9500
O(4A)-C(23)	1.236(7)	C(15)-C(16)	1.391(3)
O(4B)-C(23)	1.248(8)	C(15)-H(15)	0.9500
N(1)-C(20)	1.337(3)	C(16)-C(17)	1.509(3)
N(1)-C(19)	1.346(3)	C(17)-C(23)	1.545(3)
C(1)-C(2)	1.511(3)	C(17)-H(17)	1.0000
C(1)-C(18)	1.515(3)	C(18)-C(19)	1.381(3)
C(1)-C(17)	1.542(3)	C(18)-C(22)	1.394(3)
C(1)-H(1)	1.0000	C(19)-H(19)	0.9500
C(2)-C(10)	1.346(3)	C(20)-C(21)	1.375(4)
C(2)-C(3)	1.450(3)	C(20)-H(20)	0.9500
C(3)-C(4)	1.405(3)	C(21)-C(22)	1.379(4)
C(3)-C(8)	1.410(3)	C(21)-H(21)	0.9500
C(4)-C(5)	1.379(3)	C(22)-H(22)	0.9500
C(4)-H(4)	0.9500	O(5)-C(25)	1.39(5)
C(5)-C(6)	1.393(4)	O(5)-H(5O)	0.843(10)
C(5)-H(5)	0.9500	C(24)-C(25)	1.46(3)
C(6)-C(7)	1.383(4)	C(24)-H(24A)	0.9800
C(6)-H(6)	0.9500	C(24)-H(24B)	0.9800
C(7)-C(8)	1.390(4)	C(24)-H(24C)	0.9800
C(7)-H(7)	0.9500	C(25)-C(26)	1.436(10)
C(8)-C(9)	1.470(4)	C(25)-H(25)	1.0000
C(10)-C(11)	1.462(3)	C(26)-H(26A)	0.9800
C(11)-C(16)	1.400(3)	C(26)-H(26B)	0.9800
C(11)-C(12)	1.405(3)	C(26)-H(26C)	0.9800
C(12)-C(13)	1.385(4)		

C(9)-O(2)-C(10)	122.31(19)
C(23)-O(3)-H(3O)	112(2)
C(20)-N(1)-C(19)	119.4(2)

C(2)-C(1)-C(18)	112.54(19)
C(2)-C(1)-C(17)	112.42(19)
C(18)-C(1)-C(17)	110.72(19)
C(2)-C(1)-H(1)	106.9
C(18)-C(1)-H(1)	106.9
C(17)-C(1)-H(1)	106.9
C(10)-C(2)-C(3)	118.8(2)
C(10)-C(2)-C(1)	119.7(2)
C(3)-C(2)-C(1)	121.3(2)
C(4)-C(3)-C(8)	117.5(2)
C(4)-C(3)-C(2)	123.7(2)
C(8)-C(3)-C(2)	118.7(2)
C(5)-C(4)-C(3)	120.5(2)
C(5)-C(4)-H(4)	119.8
C(3)-C(4)-H(4)	119.8
C(4)-C(5)-C(6)	121.1(2)
C(4)-C(5)-H(5)	119.4
C(6)-C(5)-H(5)	119.4
C(7)-C(6)-C(5)	119.6(2)
C(7)-C(6)-H(6)	120.2
C(5)-C(6)-H(6)	120.2
C(6)-C(7)-C(8)	119.5(2)
C(6)-C(7)-H(7)	120.2
C(8)-C(7)-H(7)	120.2
C(7)-C(8)-C(3)	121.7(2)
C(7)-C(8)-C(9)	117.9(2)
C(3)-C(8)-C(9)	120.4(2)
O(1)-C(9)-O(2)	117.2(2)
O(1)-C(9)-C(8)	126.2(2)
O(2)-C(9)-C(8)	116.6(2)
C(2)-C(10)-O(2)	122.9(2)
C(2)-C(10)-C(11)	124.3(2)
O(2)-C(10)-C(11)	112.6(2)
C(16)-C(11)-C(12)	120.1(2)
C(16)-C(11)-C(10)	117.9(2)
C(12)-C(11)-C(10)	121.9(2)

C(13)-C(12)-C(11)	120.0(2)
C(13)-C(12)-H(12)	120.0
C(11)-C(12)-H(12)	120.0
C(14)-C(13)-C(12)	119.9(2)
C(14)-C(13)-H(13)	120.0
C(12)-C(13)-H(13)	120.0
C(13)-C(14)-C(15)	120.3(3)
C(13)-C(14)-H(14)	119.9
C(15)-C(14)-H(14)	119.9
C(14)-C(15)-C(16)	121.0(3)
C(14)-C(15)-H(15)	119.5
C(16)-C(15)-H(15)	119.5
C(15)-C(16)-C(11)	118.6(2)
C(15)-C(16)-C(17)	121.7(2)
C(11)-C(16)-C(17)	119.5(2)
C(16)-C(17)-C(1)	113.69(19)
C(16)-C(17)-C(23)	111.6(2)
C(1)-C(17)-C(23)	110.9(2)
C(16)-C(17)-H(17)	106.7
C(1)-C(17)-H(17)	106.7
C(23)-C(17)-H(17)	106.7
C(19)-C(18)-C(22)	117.1(2)
C(19)-C(18)-C(1)	123.3(2)
C(22)-C(18)-C(1)	119.6(2)
N(1)-C(19)-C(18)	122.5(2)
N(1)-C(19)-H(19)	118.7
C(18)-C(19)-H(19)	118.7
N(1)-C(20)-C(21)	121.8(2)
N(1)-C(20)-H(20)	119.1
C(21)-C(20)-H(20)	119.1
C(20)-C(21)-C(22)	118.6(2)
C(20)-C(21)-H(21)	120.7
C(22)-C(21)-H(21)	120.7
C(21)-C(22)-C(18)	120.5(2)
C(21)-C(22)-H(22)	119.7
C(18)-C(22)-H(22)	119.7

O(4A)-C(23)-O(4B)	33.2(4)
O(4A)-C(23)-O(3)	120.8(4)
O(4B)-C(23)-O(3)	125.9(4)
O(4A)-C(23)-C(17)	123.2(4)
O(4B)-C(23)-C(17)	116.3(4)
O(3)-C(23)-C(17)	113.9(2)
C(25)-O(5)-H(5O)	114(6)
C(25)-C(24)-H(24A)	109.5
C(25)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
C(25)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
O(5)-C(25)-C(26)	106.8(15)
O(5)-C(25)-C(24)	111(3)
C(26)-C(25)-C(24)	110.1(16)
O(5)-C(25)-H(25)	109.5
C(26)-C(25)-H(25)	109.5
C(24)-C(25)-H(25)	109.5
C(25)-C(26)-H(26A)	109.5
C(25)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(25)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5

Table S4. Hydrogen bonds for **15** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3)-H(3O)...N(1)#1	0.912(10)	1.636(11)	2.547(3)	177(4)
O(5)-H(5O)...O(4A)	0.843(10)	2.01(2)	2.835(9)	168(8)
O(5)-H(5O)...O(4B)	0.843(10)	2.35(3)	3.127(8)	154(7)

Symmetry transformations used to generate equivalent atoms: #1 -x+1/2,y-1/2,-z+1/2

Table S5. Crystal data and structure refinement for (–)-**10**.

Identification code	(–)-**10**
Empirical formula	C28 H32 F3 N3 O5
Formula weight	547.57
Temperature	100(2) K
Wavelength	1.54178 Å
Crystal system	Monoclinic
Space group	P2(1)
Unit cell dimensions	a = 12.6288(6) Å □= 90°.
	b = 9.4968(5) Å □= 118.149(2)°.
	c = 13.1535(7) Å □ = 90°.
Volume	1390.95(12) Å3
Z	2
Density (calculated)	1.307 Mg/m3
Absorption coefficient	0.873 mm-1
F(000)	576
Crystal size	0.49 x 0.11 x 0.04 mm3
Theta range for data collection	3.81 to 64.37°.
Index ranges	-14<=h<=14, -9<=k<=10, -15<=l<=13
Reflections collected	7917
Independent reflections	3686 [R(int) = 0.0222]
Completeness to theta = 64.37°	95.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.752 and 0.637
Refinement method	Full-matrix least-squares on F2
Data / restraints / parameters	3686 / 163 / 391
Goodness-of-fit on F2	1.005
Final R indices [I>2sigma(I)]	R1 = 0.0343, wR2 = 0.0763
R indices (all data)	R1 = 0.0361, wR2 = 0.0773
Absolute structure parameter	0.02(14)
Largest diff. peak and hole	0.195 and -0.187 e.Å-3

Table S6. Atomic coordinates (x 104) and equivalent isotropic displacement parameters (Å2x 103) for (–)-**10**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
F(1)	3704(1)	6431(2)	1478(1)	41(1)
F(2)	2907(1)	6549(2)	-371(1)	47(1)
F(3)	3191(1)	8431(2)	624(1)	44(1)
O(1)	5656(1)	6288(2)	4423(1)	26(1)
O(2)	5628(2)	8615(2)	4228(2)	29(1)
O(3)	5457(1)	10083(2)	1264(1)	24(1)
N(1)	5814(2)	7796(2)	1806(2)	19(1)
N(2)	8529(2)	3939(2)	2358(2)	31(1)
C(1)	7693(2)	8631(2)	3978(2)	18(1)
C(2)	7056(2)	7267(2)	3880(2)	17(1)
C(3)	6523(2)	6694(2)	2639(2)	20(1)
C(4)	6074(2)	9193(3)	1986(2)	19(1)
C(5)	7175(2)	9587(2)	3067(2)	18(1)
C(6)	7702(2)	10891(2)	3126(2)	20(1)
C(7)	8760(2)	11246(3)	4104(2)	24(1)
C(8)	9287(2)	10303(3)	5009(2)	23(1)
C(9)	8753(2)	9010(3)	4956(2)	21(1)
C(10)	6028(2)	7420(3)	4215(2)	19(1)
C(11)	7477(2)	6058(3)	2374(2)	21(1)
C(12)	7698(2)	4619(3)	2532(2)	26(1)
C(13)	9173(2)	4721(3)	2007(2)	33(1)
C(14)	9033(2)	6152(3)	1831(2)	33(1)
C(15)	8159(2)	6838(3)	2008(2)	28(1)
C(16)	4900(2)	7326(3)	673(2)	22(1)
C(17)	3676(2)	7186(3)	603(2)	28(1)
O(4)	2914(2)	9013(2)	5072(2)	26(1)
N(3)	3934(2)	6396(2)	5149(2)	24(1)
C(18)	2809(2)	6866(3)	4136(2)	23(1)
C(19)	2821(2)	8454(3)	4029(2)	26(1)
C(20)	1713(2)	8964(3)	2964(2)	27(1)
C(21)	857(2)	9835(3)	3025(2)	33(1)

C(22)	-124(2)	10321(3)	2032(2)	40(1)
C(23)	-276(3)	9925(4)	957(3)	50(1)
C(24)	560(3)	9062(4)	889(3)	59(1)
C(25)	1544(3)	8582(4)	1873(2)	47(1)
O(5A)	4977(9)	2280(60)	2450(50)	35(2)
C(26A)	3718(8)	2801(11)	1828(9)	35(3)
C(27A)	3550(20)	3660(20)	2708(19)	48(2)
C(28A)	2933(10)	1504(11)	1488(12)	51(3)
O(5B)	4957(4)	2420(20)	2430(20)	35(2)
C(26B)	3712(4)	2126(6)	2208(4)	39(1)
C(27B)	3399(10)	3301(8)	2788(8)	48(2)
C(28B)	2928(5)	2124(7)	920(4)	57(2)

Table S7. Bond lengths [Å] and angles [°] for (–)-**10**.

F(1)-C(17)	1.342(3)	C(14)-H(14)	0.9500
F(2)-C(17)	1.333(3)	C(15)-H(15)	0.9500
F(3)-C(17)	1.338(3)	C(16)-C(17)	1.510(3)
O(1)-C(10)	1.254(3)	C(16)-H(16A)	0.9900
O(2)-C(10)	1.246(3)	C(16)-H(16B)	0.9900
O(3)-C(4)	1.235(3)	O(4)-C(19)	1.424(3)
N(1)-C(4)	1.360(3)	O(4)-H(4O)	0.86(3)
N(1)-C(16)	1.460(3)	N(3)-C(18)	1.485(3)
N(1)-C(3)	1.473(3)	N(3)-H(3A)	0.85(3)
N(2)-C(13)	1.334(4)	N(3)-H(3B)	0.97(3)
N(2)-C(12)	1.341(3)	N(3)-H(3C)	0.94(3)
C(1)-C(5)	1.396(3)	C(18)-C(19)	1.516(4)
C(1)-C(9)	1.397(3)	C(18)-H(18A)	0.9900
C(1)-C(2)	1.498(3)	C(18)-H(18B)	0.9900
C(2)-C(3)	1.542(3)	C(19)-C(20)	1.518(3)
C(2)-C(10)	1.561(3)	C(19)-H(19)	1.0000
C(2)-H(2)	1.0000	C(20)-C(21)	1.392(4)
C(3)-C(11)	1.528(3)	C(20)-C(25)	1.396(4)
C(3)-H(3)	1.0000	C(21)-C(22)	1.389(4)
C(4)-C(5)	1.494(3)	C(21)-H(21)	0.9500
C(5)-C(6)	1.390(3)	C(22)-C(23)	1.385(4)
C(6)-C(7)	1.390(3)	C(22)-H(22)	0.9500
C(6)-H(6)	0.9500	C(23)-C(24)	1.373(4)
C(7)-C(8)	1.384(3)	C(23)-H(23)	0.9500
C(7)-H(7)	0.9500	C(24)-C(25)	1.382(4)
C(8)-C(9)	1.387(3)	C(24)-H(24)	0.9500
C(8)-H(8)	0.9500	C(25)-H(25)	0.9500
C(9)-H(9)	0.9500	O(5A)-C(26A)	1.487(18)
C(11)-C(15)	1.384(3)	O(5A)-H(5A)	0.8400
C(11)-C(12)	1.391(3)	C(26A)-C(27A)	1.510(9)
C(12)-H(12)	0.9500	C(26A)-C(28A)	1.510(9)
C(13)-C(14)	1.377(4)	C(26A)-H(26A)	1.0000
C(13)-H(13)	0.9500	C(27A)-H(27A)	0.9800
C(14)-C(15)	1.393(3)	C(27A)-H(27B)	0.9800

C(27A)-H(27C)	0.9800	C(26B)-H(26B)	1.0000
C(28A)-H(28A)	0.9800	C(27B)-H(27D)	0.9800
C(28A)-H(28B)	0.9800	C(27B)-H(27E)	0.9800
C(28A)-H(28C)	0.9800	C(27B)-H(27F)	0.9800
O(5B)-C(26B)	1.483(15)	C(28B)-H(28D)	0.9800
O(5B)-H(5B)	0.8400	C(28B)-H(28E)	0.9800
C(26B)-C(27B)	1.506(6)	C(28B)-H(28F)	0.9800
C(26B)-C(28B)	1.507(6)		

C(4)-N(1)-C(16)	119.05(19)	C(5)-C(6)-H(6)	120.2
C(4)-N(1)-C(3)	123.47(19)	C(8)-C(7)-C(6)	120.1(2)
C(16)-N(1)-C(3)	116.89(19)	C(8)-C(7)-H(7)	119.9
C(13)-N(2)-C(12)	116.4(2)	C(6)-C(7)-H(7)	119.9
C(5)-C(1)-C(9)	118.7(2)	C(7)-C(8)-C(9)	120.3(2)
C(5)-C(1)-C(2)	118.25(19)	C(7)-C(8)-H(8)	119.9
C(9)-C(1)-C(2)	123.0(2)	C(9)-C(8)-H(8)	119.9
C(1)-C(2)-C(3)	110.15(18)	C(8)-C(9)-C(1)	120.4(2)
C(1)-C(2)-C(10)	112.32(18)	C(8)-C(9)-H(9)	119.8
C(3)-C(2)-C(10)	109.00(17)	C(1)-C(9)-H(9)	119.8
C(1)-C(2)-H(2)	108.4	O(2)-C(10)-O(1)	125.6(2)
C(3)-C(2)-H(2)	108.4	O(2)-C(10)-C(2)	119.0(2)
C(10)-C(2)-H(2)	108.4	O(1)-C(10)-C(2)	115.4(2)
N(1)-C(3)-C(11)	112.29(18)	C(15)-C(11)-C(12)	117.6(2)
N(1)-C(3)-C(2)	110.20(18)	C(15)-C(11)-C(3)	123.7(2)
C(11)-C(3)-C(2)	112.54(18)	C(12)-C(11)-C(3)	118.6(2)
N(1)-C(3)-H(3)	107.2	N(2)-C(12)-C(11)	124.5(2)
C(11)-C(3)-H(3)	107.2	N(2)-C(12)-H(12)	117.7
C(2)-C(3)-H(3)	107.2	C(11)-C(12)-H(12)	117.7
O(3)-C(4)-N(1)	121.4(2)	N(2)-C(13)-C(14)	123.9(2)
O(3)-C(4)-C(5)	121.8(2)	N(2)-C(13)-H(13)	118.1
N(1)-C(4)-C(5)	116.7(2)	C(14)-C(13)-H(13)	118.1
C(6)-C(5)-C(1)	120.9(2)	C(13)-C(14)-C(15)	118.8(2)
C(6)-C(5)-C(4)	118.8(2)	C(13)-C(14)-H(14)	120.6
C(1)-C(5)-C(4)	120.2(2)	C(15)-C(14)-H(14)	120.6
C(7)-C(6)-C(5)	119.6(2)	C(11)-C(15)-C(14)	118.7(2)
C(7)-C(6)-H(6)	120.2	C(11)-C(15)-H(15)	120.6

C(14)-C(15)-H(15)	120.6	C(22)-C(21)-H(21)	119.5
N(1)-C(16)-C(17)	112.48(19)	C(20)-C(21)-H(21)	119.5
N(1)-C(16)-H(16A)	109.1	C(23)-C(22)-C(21)	120.2(3)
C(17)-C(16)-H(16A)	109.1	C(23)-C(22)-H(22)	119.9
N(1)-C(16)-H(16B)	109.1	C(21)-C(22)-H(22)	119.9
C(17)-C(16)-H(16B)	109.1	C(24)-C(23)-C(22)	119.2(3)
H(16A)-C(16)-H(16B)	107.8	C(24)-C(23)-H(23)	120.4
F(2)-C(17)-F(3)	106.70(19)	C(22)-C(23)-H(23)	120.4
F(2)-C(17)-F(1)	107.1(2)	C(23)-C(24)-C(25)	121.1(3)
F(3)-C(17)-F(1)	106.3(2)	C(23)-C(24)-H(24)	119.5
F(2)-C(17)-C(16)	111.08(19)	C(25)-C(24)-H(24)	119.5
F(3)-C(17)-C(16)	112.8(2)	C(24)-C(25)-C(20)	120.7(3)
F(1)-C(17)-C(16)	112.49(19)	C(24)-C(25)-H(25)	119.7
C(19)-O(4)-H(4O)	110(2)	C(20)-C(25)-H(25)	119.7
C(18)-N(3)-H(3A)	108.7(18)	O(5A)-C(26A)-C(27A)	104.9(12)
C(18)-N(3)-H(3B)	110.3(16)	O(5A)-C(26A)-C(28A)	106.1(19)
H(3A)-N(3)-H(3B)	108(3)	C(27A)-C(26A)-C(28A)	110.7(10)
C(18)-N(3)-H(3C)	110.7(17)	O(5A)-C(26A)-H(26A)	111.6
H(3A)-N(3)-H(3C)	108(2)	C(27A)-C(26A)-H(26A)	111.6
H(3B)-N(3)-H(3C)	111(2)	C(28A)-C(26A)-H(26A)	111.6
N(3)-C(18)-C(19)	109.6(2)	C(26A)-C(27A)-H(27A)	109.5
N(3)-C(18)-H(18A)	109.7	C(26A)-C(27A)-H(27B)	109.5
C(19)-C(18)-H(18A)	109.7	H(27A)-C(27A)-H(27B)	109.5
N(3)-C(18)-H(18B)	109.7	C(26A)-C(27A)-H(27C)	109.5
C(19)-C(18)-H(18B)	109.7	H(27A)-C(27A)-H(27C)	109.5
H(18A)-C(18)-H(18B)	108.2	H(27B)-C(27A)-H(27C)	109.5
O(4)-C(19)-C(18)	106.34(19)	C(26A)-C(28A)-H(28A)	109.5
O(4)-C(19)-C(20)	113.1(2)	C(26A)-C(28A)-H(28B)	109.5
C(18)-C(19)-C(20)	111.0(2)	H(28A)-C(28A)-H(28B)	109.5
O(4)-C(19)-H(19)	108.8	C(26A)-C(28A)-H(28C)	109.5
C(18)-C(19)-H(19)	108.8	H(28A)-C(28A)-H(28C)	109.5
C(20)-C(19)-H(19)	108.8	H(28B)-C(28A)-H(28C)	109.5
C(21)-C(20)-C(25)	117.9(2)	C(26B)-O(5B)-H(5B)	109.5
C(21)-C(20)-C(19)	122.6(2)	O(5B)-C(26B)-C(27B)	105.4(5)
C(25)-C(20)-C(19)	119.5(2)	O(5B)-C(26B)-C(28B)	107.2(12)
C(22)-C(21)-C(20)	121.0(3)	C(27B)-C(26B)-C(28B)	112.4(6)

O(5B)-C(26B)-H(26B)	110.6
C(27B)-C(26B)-H(26B)	110.6
C(28B)-C(26B)-H(26B)	110.6
C(26B)-C(27B)-H(27D)	109.5
C(26B)-C(27B)-H(27E)	109.5
H(27D)-C(27B)-H(27E)	109.5
C(26B)-C(27B)-H(27F)	109.5
H(27D)-C(27B)-H(27F)	109.5
H(27E)-C(27B)-H(27F)	109.5
C(26B)-C(28B)-H(28D)	109.5
C(26B)-C(28B)-H(28E)	109.5
H(28D)-C(28B)-H(28E)	109.5
C(26B)-C(28B)-H(28F)	109.5
H(28D)-C(28B)-H(28F)	109.5
H(28E)-C(28B)-H(28F)	109.5

Table S8. Hydrogen bonds for (–)-**10** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(4)-H(4O)...O(1)#1	0.86(3)	1.83(3)	2.690(2)	173(3)
N(3)-H(3A)...O(1)	0.85(3)	1.92(3)	2.759(2)	166(3)
N(3)-H(3B)...O(2)#2	0.97(3)	1.82(3)	2.743(3)	158(2)
N(3)-H(3C)...O(5A)#1	0.94(3)	2.05(7)	2.91(7)	150(3)
O(5A)-H(5A)...O(3)#3	0.84	2.01	2.84(3)	169.8
N(3)-H(3C)...O(5B)#1	0.94(3)	2.10(4)	2.97(3)	153(3)
O(5B)-H(5B)...O(3)#3	0.84	2.24	2.934(12)	140.0

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y+1/2,-z+1 #2 -x+1,y-1/2,-z+1 #3 x,y-1,z