Hydrogen Bonding and Charge Transfer in Trifluoromethylated β-Aminoenones: A Combined Experimental and Theoretical Study

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Supporting Information

Table S1. Crystal data and structure refinement results for **1** and **2**.

Compound	**1**	**2**
Chemical formula	$C_{14}H_{15}F_3N_2O_4$	$C_{10}H_7F_3N_2O_4$
Formula weight	332.28	276.18
Temperature (K)	297(2)	295(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	*P*-1	*P* 21/*n*
Unit cell dimensions: *a*(Å) *b*(Å) *c*(Å) α(º) *β*(º) γ(º)	 5.3850(6) 10.5397(9) 14.075(1) 87.615(7) 82.484(9) 78.659(8)	 5.0511(3) 15.3395(7) 14.747(1) 90.00 94.606(7) 90.00
Volume (Å^3)	776.4(1)	1139.0(1)
Z	2	4
Density (calculated, Mg/m^3)	1.421	1.611
Absorption coeff. (mm^{-1})	0.127	0.156
F(000)	344	560
Crystal color	Yellow	Yellow
Crystal size (mm^3)	0.779 x 0.430 x 0.037	0.758 x 0.465 x 0.138
ϑ-range (º) for data collection	3.497 to 28.987	3.073 to 28.652
Index ranges	-7≤h≤7, -14≤k≤10, -16≤l≤18	-6≤h≤6, -20≤k≤18, -19≤l≤19
Reflections collected	5974	5157
Independent reflections	3328 [*R*(int) = 0.0314]	2475 [R(int) = 0.0262]
Observed reflections [$I>2\sigma(I)$]	1489	1600
Completeness (%)	99.8 (to ϑ = 25.242°)	99.8 (to ϑ = 25.242°)
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	3328 / 3 / 210	2475 / 0 / 200
Goodness-of-fit on F^2	1.034	1.035
Final *R* indices[a] [$I > 2\sigma(I)$]	R1 = 0.0780, wR2 = 0.2149	R1 = 0.0607, wR2 = 0.1612
R indices (all data)	R1 = 0.1587, wR2 = 0.2887	R1 = 0.0938, wR2 = 0.1884
Larg. diff. peak / hole (e.Å^{-3})	0.390 / -0.250	0.392 and -0.311

[a] $R1=\Sigma||F_o|-|F_c||/\Sigma|F_o|$, $wR2=[\Sigma w(|F_o|^2-|F_c|^2)^2/\Sigma w(|F_o|^2)^2]^{1/2}$

Table S2a. Experimental and calculated bond distances and angles for **1**.

Parameters	Exp.	Calc.[a]	Parameters	Exp.	Calc.[a]
Bond lengths (Å)					
C7=O2	1.252(4)	1.266	C4-C3	1.360(5)	1.377
C2-O1	1.338(4)	1.331	C3-C2	1.398(5)	1.407
C1-C7	1.495(5)	1485	C2-C1	1.409(5)	1.427
C7-C8	1.415(5)	1.432	C6-C1	1.393(4)	1.400
C8=C9	1.378(5)	1.381	N1-C11	1.459(5)	1.465
C9-C10	1.508(6)	1.528	C11-C12	1.457(8)	1.534
C9-N1	1.334(5)	1.339	C12-C13	1.335(8)	1.533
C6-C5	1.367(5)	1.383	C13-C14	1.484(9)	1.531
C5-C4	1.392(5)	1.400	C10-F*	1.327	1.342
C5-N2	1.466(5)	1.468	N2-O*	1.212	1.227
Bond angles (°)					
C1-C2-O1	122.0(3)	122.5	C5-C4-C3	118.5(3)	119.0
C6-C1-C7	121.9(3)	122.5	C4-C3-C2	120.8(4)	120.7
C1-C7-C8	120.1(3)	120.9	C3-C2-C1	120.4(3)	120.1
C7-C8-C9	123.5(3)	122.7	C2-C1-C6	118.0(3)	118.0
C8-C9-C10	118.5(4)	117.8	C5-C6-C1	120.0(3)	120.6
C8-C9-N1	124.3(3)	124.6	C2-C1-C7	120.1(3)	119.5
C10-C9-N1	117.2(3)	117.5	C9-N1-C11	130.6(4)	130.2
C1-C7-O2	118.3(3)	118.3	N1-C11-C12	113.3(4)	112.6
C8-C7-O2	121.6(3)	120.8	C11-C12-C13	125.1(8)	112.2
C6-C5-C4	122.7(3)	121.5	C12-C13-C14	123.4(8)	112.8
O4-N2-O3	122.7(4)	124.4	O3-N2-C5	118.9(3)	118.9
O4-N2-C5	119.3(4)	117.6			

[a]B3LYP/6-311++G(d,p)

Table S2b. Experimental and calculated bond distances and angles for **2**.

Parameters	Exp.	Calc.[a]	Parameters	Exp.	Calc.[a]
Bond lengths (Å)					
C7=O2	1.259(3)	1.266	C4-C3	1.365(4)	1.377
C2-O1	1.337(3)	1.331	C3-C2	1.398(3)	1.407
C1-C7	1.485(3)	1485	C2-C1	1.419(3)	1.427
C7-C8	1.419(3)	1.432	C6-C1	1.390(3)	1.400
C8=C9	1.365(3)	1.381	C10-F*	1.304	1.347
C9-C10	1.510(3)	1.528	N2-O*	1.214	1.227
C9-N1	1.320(3)	1.339			
C6-C5	1.373(4)	1.383			
C5-C4	1.384(4)	1.400			
C5-N2	1.456(3)	1.468			
Bond angles (°)					
C1-C2-O1	122.1(2)	122.5	C5-C4-C3	118.6(2)	119.1
C6-C1-C7	122.0(2)	122.5	C4-C3-C2	120.9(3)	120.7
C1-C7-C8	120.0(2)	120.9	C3-C2-C1	120.1(2)	120.1
C7-C8-C9	123.3(2)	122.0	C2-C1-C6	117.9(2)	118.1
C8-C9-C10	118.0(2)	119.0	C5-C6-C1	120.3(2)	120.5
C8-C9-N1	127.6(2)	126.4	C2-C1-C7	120.0(2)	119.5
C10-C9-N1	114.3(2)	114.5	C6-C5-N2	118.4(2)	119.1
C1-C7-O2	118.7(2)	118.7	C4-C5-N2	119.5(2)	119.4
C8-C7-O2	121.2(2)	120.4	C3-C2-O1	117.8(2)	117.4
C6-C5-C4	122.1(2)	121.5	F-C10-F	106.4	107.5
O4-N2-O3	122.7(2)	124.5	F-C10-C9	112.4	111.4
O4-N2-C5	118.0(2)	117.6	O3-N2-C5	119.4(2)	117.9

[a]B3LYP/6-311++G(d,p)

Table S3a. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **1**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
C(1)	1899(6)	2897(3)	4082(2)	48(1)
C(2)	949(6)	3125(3)	5053(3)	53(1)
C(3)	-939(7)	2490(4)	5507(3)	60(1)
C(4)	-1878(7)	1634(4)	5025(3)	61(1)
C(5)	-910(7)	1402(3)	4068(3)	58(1)
C(6)	927(6)	2012(3)	3600(3)	54(1)

C(7)	3848(6)	3616(3)	3590(3)	51(1)
C(8)	4950(6)	3315(4)	2638(3)	57(1)
C(9)	6688(7)	3965(4)	2126(3)	60(1)
C(10)	7809(9)	3516(5)	1130(3)	85(1)
C(11)	9214(9)	5774(5)	1996(4)	84(1)
C(12)	7977(13)	7130(7)	1942(7)	156(3)
C(13)	5862(15)	7577(8)	1536(8)	188(4)
C(14)	4874(16)	8974(8)	1367(6)	176(3)
N(1)	7527(6)	4946(3)	2464(2)	68(1)
N(2)	-1918(7)	495(3)	3533(3)	74(1)
O(1)	1798(5)	3946(3)	5574(2)	66(1)
O(2)	4479(5)	4474(2)	4045(2)	62(1)
O(3)	-949(7)	258(4)	2714(3)	118(2)
O(4)	-3659(6)	13(3)	3917(3)	96(1)
F(1)	7352(6)	4433(4)	465(2)	118(1)
F(2)	10339(5)	3140(3)	1055(2)	114(1)
F(3)	6899(6)	2526(3)	870(2)	119(1)

Table S3b. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for **2**.

Atom	x	y	z	U(eq)
F(1)	12747(5)	8869(1)	5637(2)	125(1)
F(2)	10024(6)	9087(1)	4523(2)	111(1)
F(3)	8758(5)	9062(1)	5843(2)	100(1)
O(1)	6838(4)	4566(1)	5875(1)	55(1)
O(2)	8975(4)	5906(1)	5244(1)	50(1)
O(3)	1340(5)	7512(1)	7940(2)	86(1)
O(4)	-478(5)	6389(2)	8473(2)	85(1)
N(1)	11466(5)	7343(2)	4633(2)	55(1)
N(2)	1058(5)	6730(2)	7983(2)	55(1)
C(1)	5815(5)	6027(2)	6332(2)	37(1)
C(2)	5467(5)	5113(2)	6366(2)	42(1)
C(3)	3663(6)	4752(2)	6932(2)	51(1)
C(4)	2228(6)	5271(2)	7463(2)	51(1)
C(5)	2580(5)	6165(2)	7426(2)	44(1)
C(6)	4323(5)	6542(2)	6876(2)	42(1)
C(7)	7730(5)	6408(2)	5729(2)	40(1)
C(8)	8182(5)	7321(2)	5730(2)	44(1)
C(9)	9945(5)	7716(2)	5208(2)	43(1)
C(10)	10345(6)	8687(2)	5310(2)	56(1)

Table S4a. Anisotropic displacement parameters (Å^2 x 10^3) for **1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2\,hka^*b^*\,U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	49(2)	45(2)	50(2)	4(2)	-11(2)	-9(2)
C(2)	52(2)	48(2)	59(2)	5(2)	-11(2)	-11(2)
C(3)	56(2)	62(2)	62(2)	1(2)	-3(2)	-14(2)
C(4)	55(2)	53(2)	76(3)	14(2)	-7(2)	-15(2)
C(5)	54(2)	41(2)	81(3)	5(2)	-18(2)	-10(2)

C(6)	54(2)	49(2)	59(2)	-1(2)	-10(2)	-9(2)
C(7)	55(2)	46(2)	52(2)	4(2)	-11(2)	-11(2)
C(8)	56(2)	63(2)	54(2)	-2(2)	-9(2)	-18(2)
C(9)	60(2)	68(3)	53(2)	4(2)	-12(2)	-14(2)
C(10)	89(3)	116(4)	55(3)	-10(3)	-2(2)	-38(3)
C(11)	85(3)	86(3)	85(3)	12(3)	-3(2)	-32(3)
C(12)	118(5)	136(7)	215(8)	77(6)	-24(5)	-39(5)
C(13)	125(6)	136(7)	301(13)	38(7)	-41(7)	-19(5)
C(14)	197(8)	135(7)	191(9)	12(6)	-72(7)	9(6)
N(1)	74(2)	82(2)	53(2)	0(2)	2(2)	-33(2)
N(2)	74(2)	58(2)	94(3)	-4(2)	-13(2)	-23(2)
O(1)	78(2)	72(2)	56(2)	-5(1)	-6(1)	-34(1)
O(2)	72(2)	60(2)	60(2)	-6(1)	-1(1)	-28(1)
O(3)	143(3)	119(3)	108(3)	-42(3)	12(3)	-77(3)
O(4)	84(2)	89(2)	128(3)	-7(2)	-10(2)	-48(2)
F(1)	144(3)	159(3)	55(2)	20(2)	-14(2)	-47(2)
F(2)	86(2)	160(3)	90(2)	-27(2)	14(2)	-18(2)
F(3)	149(3)	154(3)	71(2)	-36(2)	14(2)	-78(2)

Table S4b. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2**.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(1)	84(2)	63(1)	221(3)	-41(2)	-19(2)	-17(1)
F(2)	199(3)	43(1)	93(2)	17(1)	27(2)	-3(1)
F(3)	127(2)	40(1)	145(2)	-16(1)	77(2)	-7(1)
O(1)	65(1)	33(1)	71(1)	-6(1)	35(1)	2(1)
O(2)	58(1)	38(1)	60(1)	-5(1)	30(1)	-1(1)
O(3)	115(2)	48(1)	102(2)	-15(1)	54(2)	12(1)
O(4)	92(2)	83(2)	90(2)	-1(1)	61(2)	14(1)
N(1)	63(2)	40(1)	64(2)	1(1)	27(1)	-7(1)
N(2)	59(2)	58(2)	50(1)	-2(1)	19(1)	14(1)
C(1)	41(1)	33(1)	38(1)	0(1)	10(1)	2(1)
C(2)	47(1)	34(1)	46(1)	-2(1)	13(1)	3(1)
C(3)	60(2)	35(1)	62(2)	6(1)	25(1)	1(1)
C(4)	55(2)	49(2)	53(2)	9(1)	23(1)	4(1)
C(5)	47(1)	45(1)	40(1)	0(1)	12(1)	10(1)
C(6)	48(1)	34(1)	44(1)	-1(1)	10(1)	4(1)
C(7)	43(1)	36(1)	43(1)	-1(1)	13(1)	1(1)
C(8)	47(1)	35(1)	52(2)	-2(1)	14(1)	2(1)
C(9)	45(1)	34(1)	50(2)	1(1)	5(1)	1(1)
C(10)	62(2)	39(1)	69(2)	2(1)	19(2)	-5(1)

Table S5a. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for **1**.

Atom	x	y	z	U(eq)
H(3)	-15612654	614772		
H(4)	-31391214	532773		
H(6)	15271836	296065		
H(8)	44772642	234268		

H(11A)	106775702	2344101
H(11B)	98375466	1352101
H(12A)	76167430	2597188
H(12B)	92667583	1624188
H(13A)	44947244	1921226
H(13B)	61067167	917226
H(14A)	44239405	1970264
H(14B)	33939074	1038264
H(14C)	61689346	984264
H(1)	69835119	305581
H(1A)	29134252	524299

Table S5b. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2**.

Atom	x	y	z	U(eq)
H(1O)	7960(70)	4900(20)	5550(20)	78(11)
H(1N)	11300(60)	6750(20)	4560(20)	68(9)
H(2N)	12470(60)	7650(20)	4340(20)	66(10)
H(3)	3500(60)	4130(20)	6960(20)	63(9)
H(4)	1080(60)	5050(20)	7910(20)	68(9)
H(6)	4530(50)	7146(19)	6879(18)	49(7)
H(8)	7300(50)	7696(18)	6109(19)	51(8)

Table S6a. Torsion angles [°] in **1**.

C(6)-C(1)-C(2)-O(1)	179.1(3)	C(1)-C(7)-C(8)-C(9)	177.6(3)
C(7)-C(1)-C(2)-O(1)	-2.3(5)	C(7)-C(8)-C(9)-N(1)	0.0(6)
C(6)-C(1)-C(2)-C(3)	-0.9(5)	C(7)-C(8)-C(9)-C(10)	177.5(4)
C(7)-C(1)-C(2)-C(3)	177.7(3)	N(1)-C(9)-C(10)-F(2)	58.5(6)
O(1)-C(2)-C(3)-C(4)	-179.5(3)	C(8)-C(9)-C(10)-F(2)	-119.2(4)
C(1)-C(2)-C(3)-C(4)	0.5(5)	N(1)-C(9)-C(10)-F(3)	178.7(4)
C(2)-C(3)-C(4)-C(5)	0.2(6)	C(8)-C(9)-C(10)-F(3)	1.0(6)
C(3)-C(4)-C(5)-C(6)	-0.6(6)	N(1)-C(9)-C(10)-F(1)	-61.2(5)
C(3)-C(4)-C(5)-N(2)	-179.3(3)	C(8)-C(9)-C(10)-F(1)	121.2(4)
C(2)-C(1)-C(6)-C(5)	0.5(5)	N(1)-C(11)-C(12)-C(13)	-56.7(11)
C(7)-C(1)-C(6)-C(5)	-178.1(3)	C(11)-C(12)-C(13)-C(14)	-170.6(8)
C(4)-C(5)-C(6)-C(1)	0.2(5)	C(8)-C(9)-N(1)-C(11)	-175.6(4)
N(2)-C(5)-C(6)-C(1)	178.9(3)	C(10)-C(9)-N(1)-C(11)	6.9(6)
C(6)-C(1)-C(7)-O(2)	174.2(3)	C(12)-C(11)-N(1)-C(9)	116.9(6)
C(2)-C(1)-C(7)-O(2)	-4.4(5)	C(4)-C(5)-N(2)-O(4)	3.7(5)
C(6)-C(1)-C(7)-C(8)	-6.4(5)	C(6)-C(5)-N(2)-O(4)	-175.1(3)
C(2)-C(1)-C(7)-C(8)	175.0(3)	C(4)-C(5)-N(2)-O(3)	-176.3(4)
O(2)-C(7)-C(8)-C(9)	-3.0(6)	C(6)-C(5)-N(2)-O(3)	4.9(6)

Table S6b. Torsion angles [°] in **2**.

C(6)-C(1)-C(2)-O(1)	-179.2(2)	C(7)-C(1)-C(6)-C(5)	-179.8(2)
C(7)-C(1)-C(2)-O(1)	0.7(4)	C(6)-C(1)-C(7)-O(2)	-178.7(2)
C(6)-C(1)-C(2)-C(3)	0.2(4)	C(2)-C(1)-C(7)-O(2)	1.4(3)
C(7)-C(1)-C(2)-C(3)	-179.9(2)	C(6)-C(1)-C(7)-C(8)	3.2(4)
O(1)-C(2)-C(3)-C(4)	178.9(3)	C(2)-C(1)-C(7)-C(8)	-176.7(2)
C(1)-C(2)-C(3)-C(4)	-0.5(4)	O(2)-C(7)-C(8)-C(9)	0.7(4)
C(2)-C(3)-C(4)-C(5)	0.5(4)	C(1)-C(7)-C(8)-C(9)	178.8(2)
C(3)-C(4)-C(5)-C(6)	-0.2(4)	C(7)-C(8)-C(9)-N(1)	0.4(4)
C(3)-C(4)-C(5)-N(2)	179.6(2)	C(7)-C(8)-C(9)-C(10)	-176.8(2)
O(4)-N(2)-C(5)-C(6)	-179.7(3)	N(1)-C(9)-C(10)-F(1)	-62.5(4)
O(3)-N(2)-C(5)-C(6)	0.9(4)	C(8)-C(9)-C(10)-F(1)	115.0(3)
O(4)-N(2)-C(5)-C(4)	0.5(4)	N(1)-C(9)-C(10)-F(3)	176.8(3)
O(3)-N(2)-C(5)-C(4)	-178.9(3)	C(8)-C(9)-C(10)-F(3)	-5.7(4)
C(4)-C(5)-C(6)-C(1)	-0.1(4)	N(1)-C(9)-C(10)-F(2)	55.5(3)
N(2)-C(5)-C(6)-C(1)	-179.9(2)	C(8)-C(9)-C(10)-F(2)	-127.0(3)
C(2)-C(1)-C(6)-C(5)	0.1(4)		

Table S7. Interatomic D-H···A distances (Å) and angles (°) of related non-conjugated β-hydroxy- and β-aminoalkyl-β-trifluoromethyl ketones Data taken from Cambridge Structural Database (CSD).

	D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(D–H···A)	Ref.
3	O–H···O=C intra	0.81	2.41	2.867	117	[24]
	O-H···O^{i}=C inter	0.81	2.15	2.937	165	
4	O–H···O=C intra	0.82	2.57	2.924	108	[24]
	O-H···O^{i}=C inter	0.82	2.09	2.890	164	
5	O–H···O=C intra	0.82	2.81	3.167	108	[24]
	O-H···O^{i}=C inter	0.82	1.98	2.793	170	
6	O–H···O=C intra	0.83	2.33	2.912	128	[25]
	O-H···O^{i}=C inter	0.83	2.12	2.887	153	
7	O–H···O=C intra	0.87	3.42	3.017	55	[25]
	O-H···O^{i}=C inter	0.87	1.96	2.821	175	
8	N–H···O=C intra	0.879	2.82	2.825	81	[26]
	N-H···O^{i}=C inter	–	–	–	–	
9	N–H···O=C intra	0.860	3.62	3.253	58	[27]
	N-H···O^{i}=C inter	–	–	–	–	
10	N–H···O=C intra	0.880	3.42	2.972	53	[28]
	N-H···O^{i}=C inter	0.880	2.06	2.909	161	
11	N–H···O=C intra	0.894	3.58	3.037	46	[29]
	N-H···O^{i}=C inter	0.894	2.12	3.001	166	
12	N–H···O=C intra	0.692	3.06	3.008	23	[30]

	N-H···O^{i}=C inter	0.692	2.17	2.843	163	
13	N–H···O=C intra	0.860	3.14	2.977	71	[31]
	N-H···O^{i}=C inter	0.860	1.94	2.786	170	
14	N–H···O=C intra	0.893	3.19	3.118	77	[32]
	N-H···O^{i}=C inter	0.893	1.88	3.580	173	

The new references added are:

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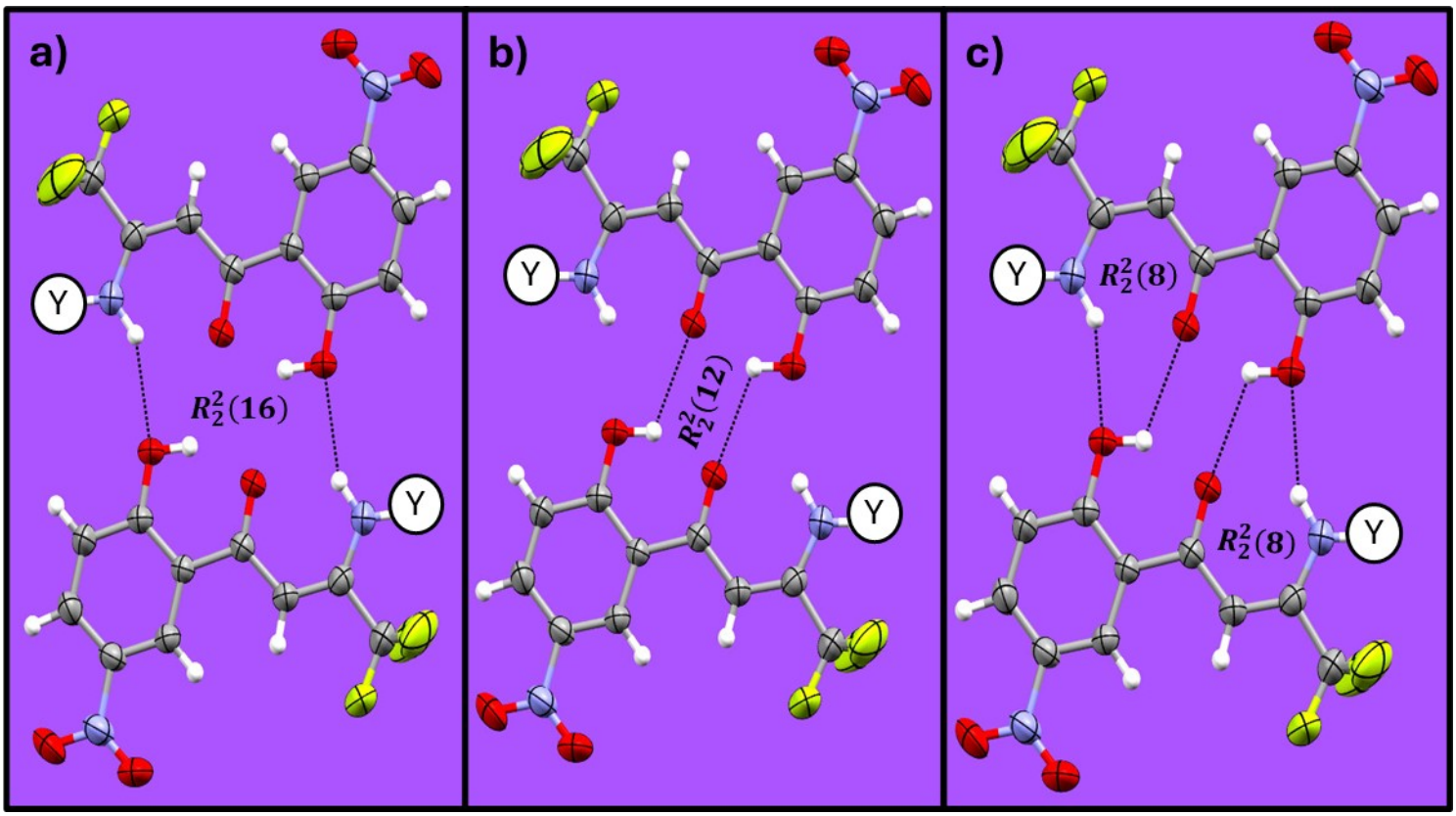

Figure S1. Representation of different graph-set motifs **a)** , **b)** and **c)** . **Y**: H / butyl.

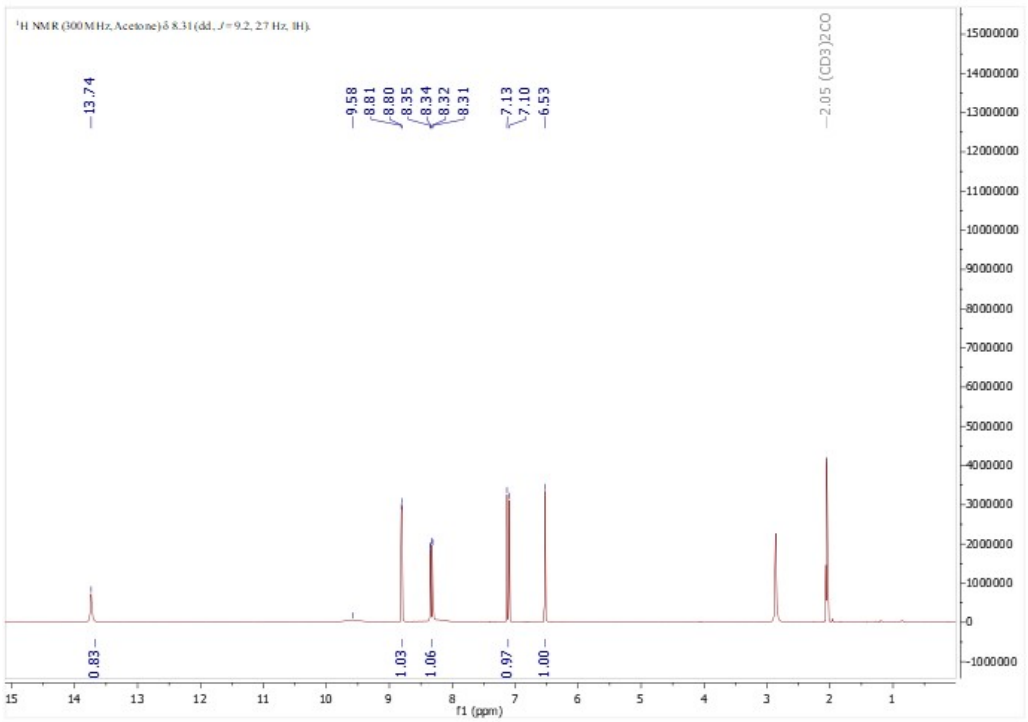

Figure S2a. ^{1}H NMR spectrum of **1** in $CDCl_3$.

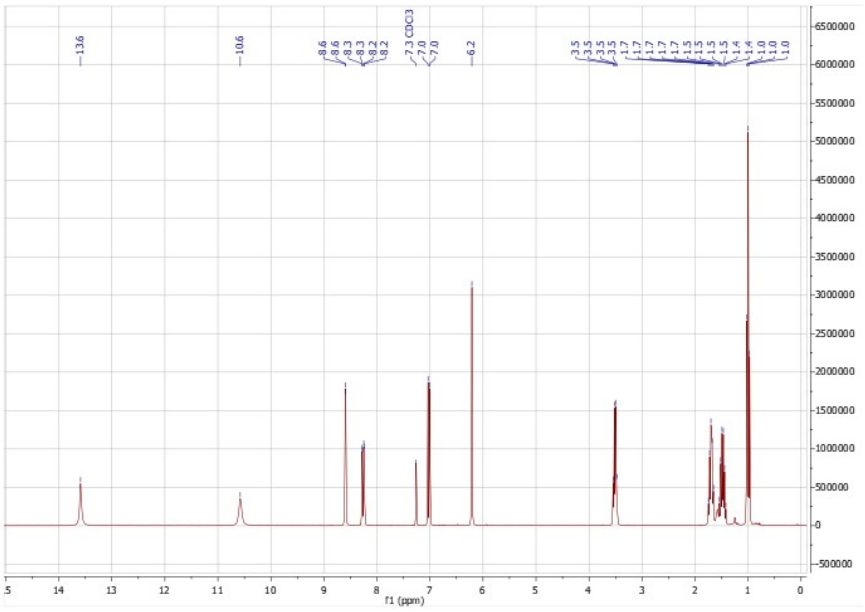

Figure S2b. ^{1}H NMR spectrum of **2** in $(CD_3)_2CO$.

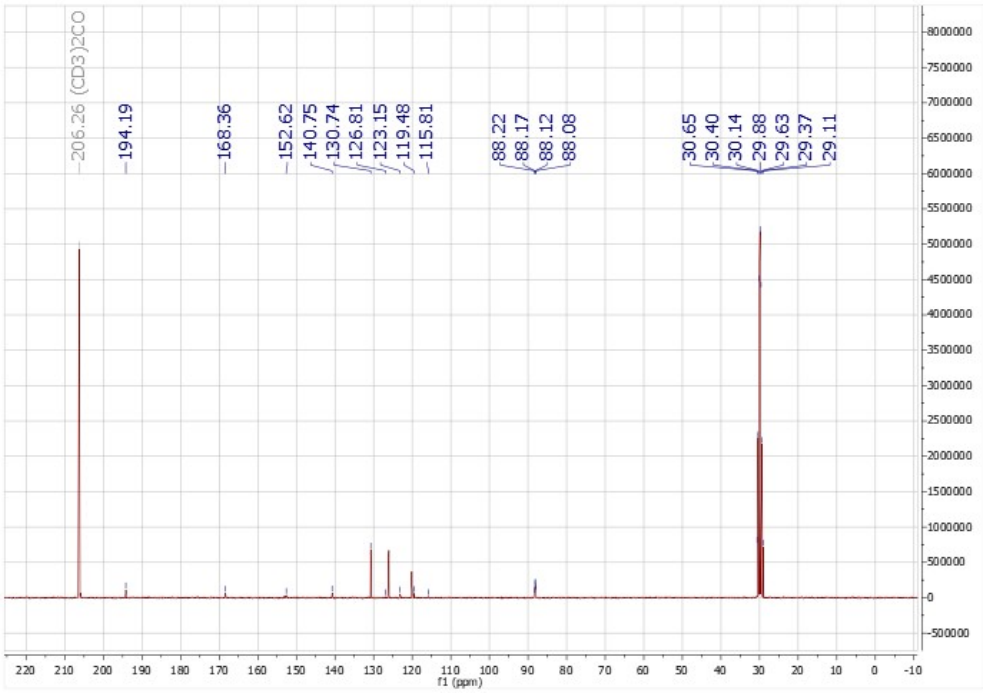

Figure S3a. ^{13}C NMR spectrum of **1** in $CDCl_3$.

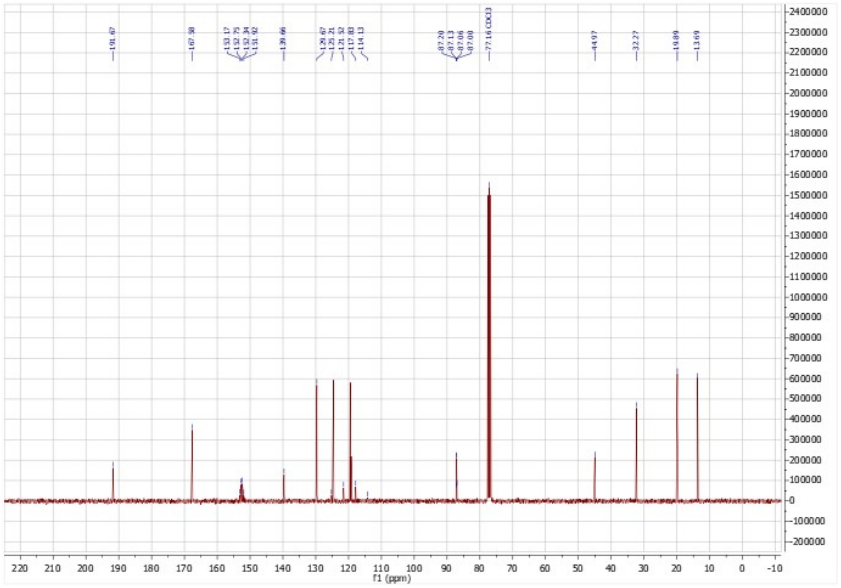

Figure S3b. ^{13}C NMR spectrum of **2** in $(CD_3)_2CO$.

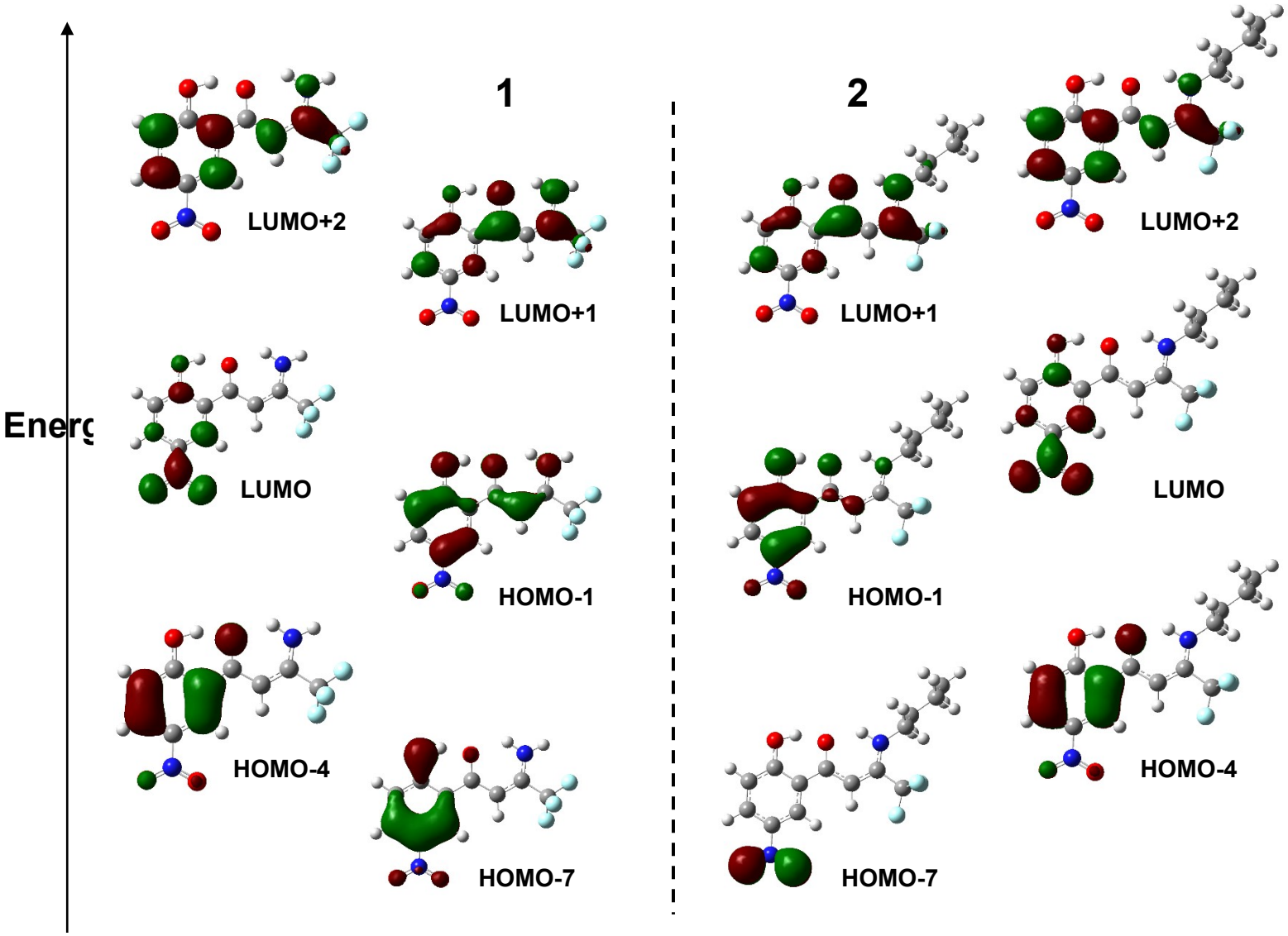

Figure S4. Molecular orbitals involved in the electronic transitions of **1** and **2** with solvent. The energy scale is only qualitative and does not represent the actual energy of the molecular orbitals.

Synthesis of (2Z)-3-butylamino-4,4,4-trifluoro-1-(2-hydroxy-5-nitrophenyl)but-2-en-1-one (1) and (2Z)-3-amino-4,4,4-trifluoro-1-(2-hydroxy-5-nitrophenyl)but-2-en-1-one (2)

6-nitro-2-trifluoromethylchromone (0.8 mmol, 0.21 g for **1** and 0.1 mmol, 0.03 g for **2**) was first dissolved in ethanol inside a round-bottom flask. Subsequently, the corresponding amine in excess (n-butylamine for **1** or ammonium hydroxide solution for **2**) was added to the solution at room temperature. Thin layer chromatography (TCL) was used to follow the reaction. To isolate the pure compounds, the crude products were re-crystallized from hot hexane. By slowly evaporating hexane solutions, appropriate yellow crystals for X-ray

diffraction measurements were obtained. More information is included in the Supplementary Material.

1: mp: 104-105 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 13.59 (s, OH); 10.58 (s, NH); 8.60 (d, H3, *J* = 3 Hz); 8.26 (dd, H4, *J* = 9 and 3 Hz); 7.02 (d, H6, *J* = 9 Hz); 6.21 (s, H8); 3.51 (q, CH_2, *J* = 7 Hz); 1.70 (p, CH_2, *J* = 7 Hz); 1.48 (h, CH_2, *J* = 7 Hz); 1.00 ppm (t, CH_3, *J* = 7 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.7 (C=O); 167.6 (C2); 152.5 (q, C9, *J* = 32); 139.7 (C5); 129.6 (C4); 124.6 (C3); 119.7 (q, CF_3, *J* = 279); 119.4 (C6); 119.2 (C1); 87.1 (q, C8, *J* = 5); 45.0 (C11); 32.3 (C12); 20.0 (C13); 13.7 ppm (CH_3).

2: mp: 182-183 °C; ^{1}H NMR (300 MHz, $(CD_3)_2CO$) δ 13.74 (s, OH); 9.58 (br.s, NH); 8.80 (d, H2, *J* = 3 Hz); 8.33 (dd, H3, *J* = 9 and 3 Hz); 7.12 (dd, H4, *J* = 8 and 1 Hz); 6.53 (s, H8). ^{13}C NMR (75 MHz, $(CD_3)_2CO$) δ 194.2 (C=O); 168.4 (C2); 152.6 (C9); 140.7 (C5); 130.7 (C4); 126.0 (C3); 121.3 (q, CF_3, *J* = 277); 120.1 (C6); 120.0 (C1); 88.1 ppm (q, C8, *J* = 4).