Supporting Information

Synthesis and in vitro biological evaluation of carbonyl group-containing analogues for σ_1 receptors

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Contents of Supporting Information:

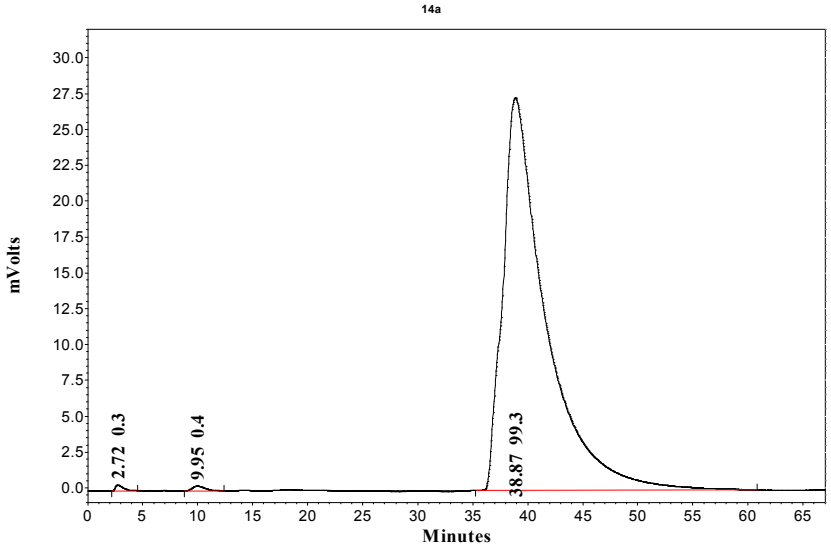
Table 3. Elemental Analysis of Analogues

Compounds	Molecular Formulas	Calculated			Found		
		C	H	N	C	H	N
14a	$C_{24}H_{28}F_2N_2O_2 \bullet H_2C_2O_4 \bullet 0.25H_2O$	61.35	6.04	5.50	61.43	5.91	5.54
14b	$C_{25}H_{31}FN_2O_3 \bullet H_2C_2O_4 \bullet 0.\ 5H_2O$	61.70	6.52	5.33	62.09	6.41	5.30
14c	$C_{24}H_{29}FN_2O_2 \bullet H_2C_2O_4 \bullet 0.25H_2O$	63.60	6.47	5.70	63.52	6.47	5.67
14d	$C_{23}H_{28}FN_3O_2 \bullet 1.5H_2C_2O_4 \bullet H_2O$	56.72	6.04	7.63	56.88	5.93	7.31
14e	$C_{25}H_{31}FN_2O_3 \bullet H_2C_2O_4 \bullet 0.5H_2O$	61.70	6.52	5.33	61.56	6.28	5.24
14f	$C_{25}H_{31}FN_2O_3 \bullet H_2C_2O_4 \bullet 0.\ 5H_2O$	62.56	6.94	5.21	62.42	6.78	5.19
14g	$C_{25}H_{32}N_2O_3 \bullet 2H_2C_2O_4$	65.04	6.87	5.62	64.58	6.72	5.56
14h	$C_{24}H_{31}N_3O_3 \bullet 2H_2C_2O_4 \bullet 0.5H_2O$	56.18	6.06	7.02	56.37	5.98	6.82
15a	$C_{24}H_{26}F_2N_2O_5 \bullet H_2C_2O_4$	60.23	5.44	5.40	59.96	5.43	5.37
15b	$C_{24}H_{27}FN_2O_3 \bullet H_2C_2O_4 \bullet 0.5H_2O$	60.10	5.98	5.19	59.99	6.07	5.14
15c	$C_{24}H_{27}FN_2O_3 \bullet H_2C_2O_4 \bullet H_2O$	60.22	6.03	5.40	60.72	5.98	5.50
15d	$C_{23}H_{26}FN_3O_3 \bullet H_2C_2O_4 \bullet 0.25H_2O$	59.34	5.61	8.30	59.25	5.55	8.26
15e	$C_{25}H_{29}FN_2O_4 \bullet H_2C_2O_4$	61.12	5.89	5.28	61.03	5.92	5.26
15f	$C_{25}H_{30}N_2O_8 \bullet H_2C_2O_4 \bullet 1.5H_2O$	60.10	6.54	5.19	60.47	6.39	5.41
15g	$C_{24}H_{29}N_3O_4 \bullet H_2C_2O_4$	60.81	6.08	8.18	60.57	6.06	7.96
16a	$C_{24}H_{28}F_2N_2O_2 \bullet H_2C_2O_4$	61.90	5.99	5.55	61.78	5.97	5.52
16b	$C_{25}H_{31}FN_2O_3 \bullet H_2C_2O_4$	62.78	6.44	5.42	62.61	6.32	5.40
16c	$C_{24}H_{29}FN_2O_2 \bullet H_2C_2O_4$	64.18	6.42	5.76	63.95	6.40	5.71
16d	$C_{23}H_{28}FN_3O_2 \bullet H_2C_2O_4 \bullet 0.5H_2O$	60.47	6.29	8.46	60.59	6.29	8.41
16e	$C_{25}H_{31}FN_2O_3 \bullet H_2C_2O_4$	52.78	6.44	5.42	62.55	6.49	5.46
16f	$C_{26}H_{34}N_2O_4 \bullet H_2C_2O_4$	63.62	6.86	5.30	63.33	6.87	5.27
16g	$C_{25}H_{32}N_2O_3 \bullet H_2C_2O_4 \bullet 0.5H_2O$	63.89	6.95	5.52	63.93	6.85	5.51
16h	$C_{24}H_{31}N_3O_3 \bullet H_2C_2O_4$	62.51	6.66	8.41	62.14	6.72	8.27

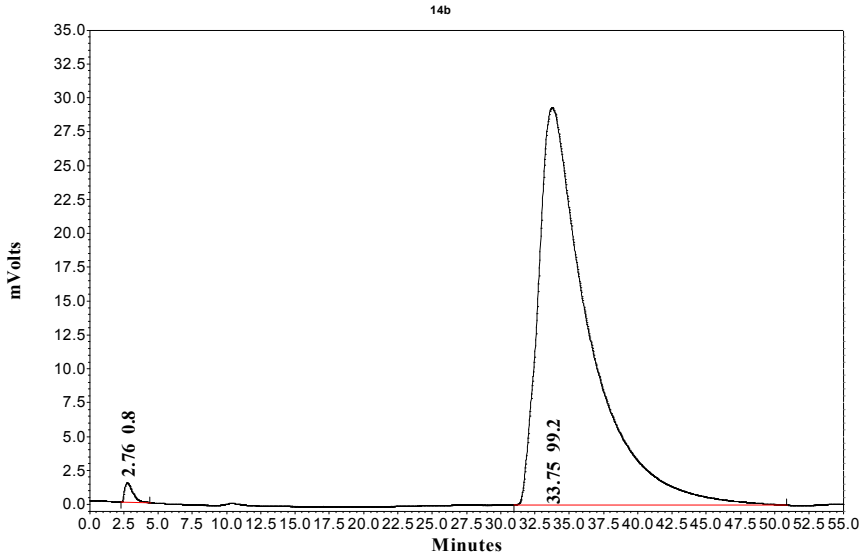
II. HPLC chromatographs for compounds14a-h, 15a-g and 16a-h.

HPLC conditions to verify the purity of final compounds

The HPLC analysis was performed on a Alltech Econosil reversed phase C18 column (250 × 4.6 mm, 10 uA). The mobile phase used was 35% acetonitrile and 65% 0.1M ammonium formate buffer (pH = 4.5) at 1.0 mL/min flow rate. The UV detection was at 254 nM wavelength. The retention time and the percent area of the peaks were recorded near the peaks on the chromatograms. The purity of all the final compounds was >97.5%.



HPLC of 14a



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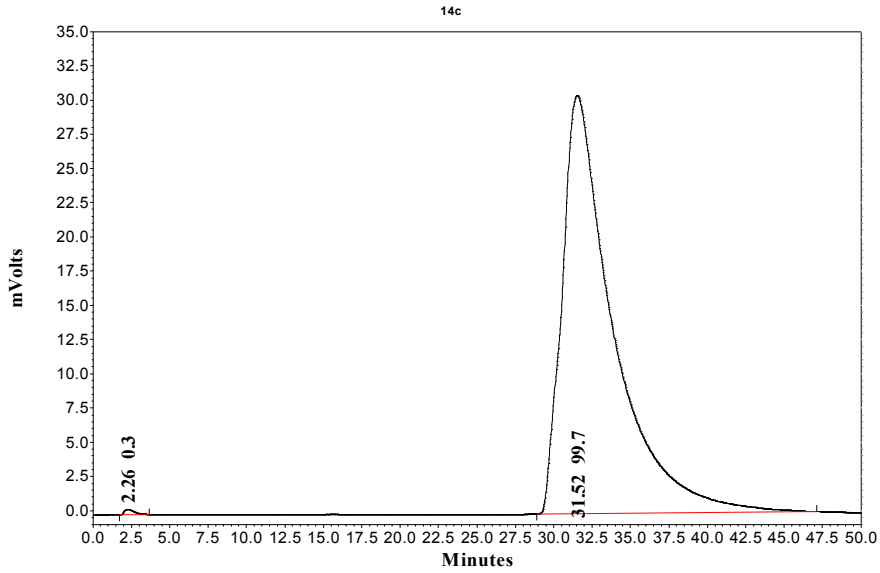

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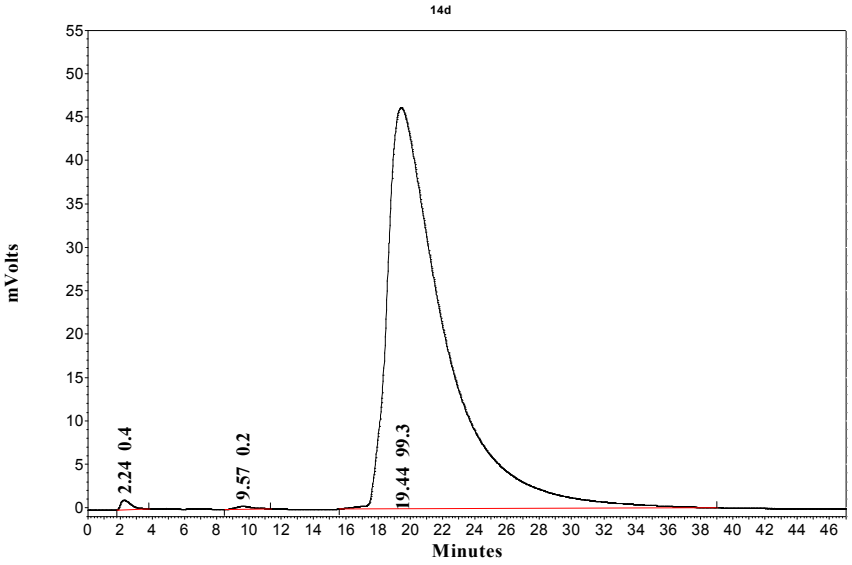
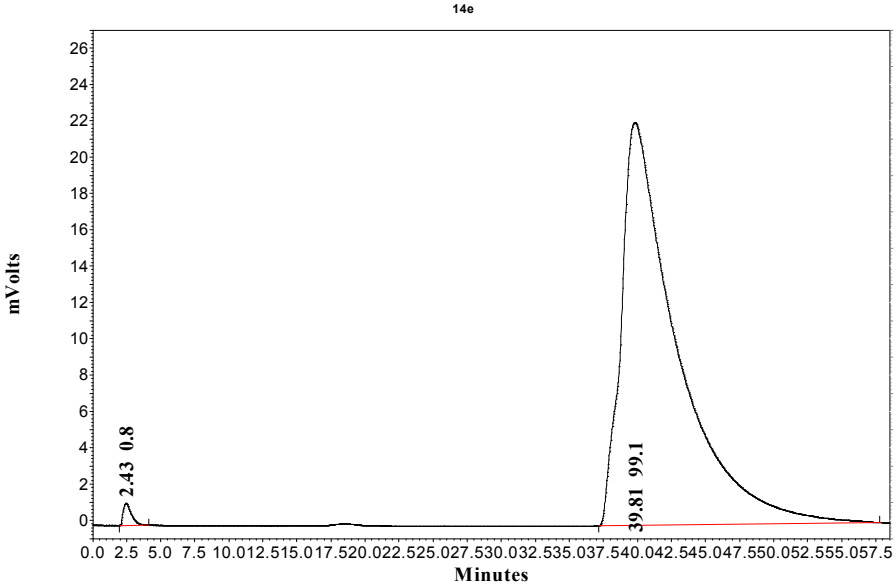
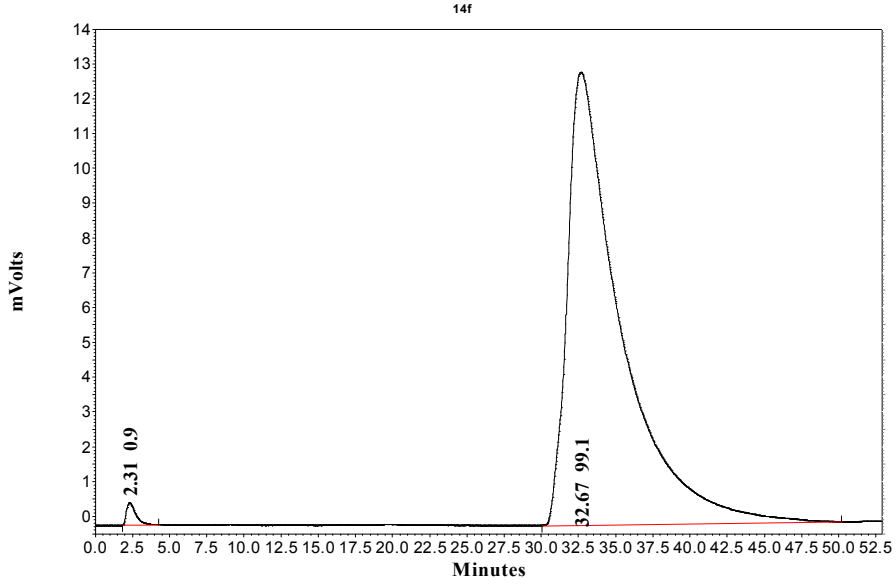


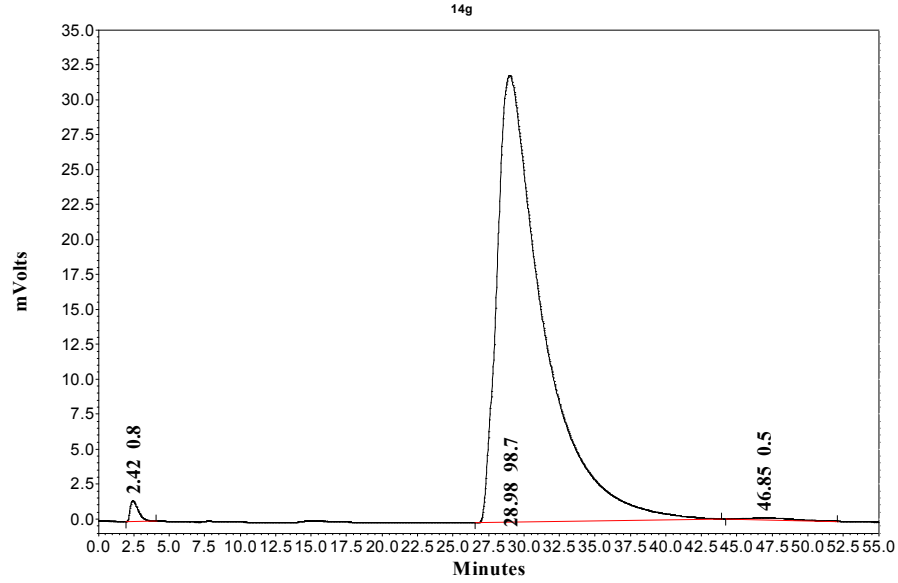
HPLC of 14d



HPLC of 14e



HPLC of 14f



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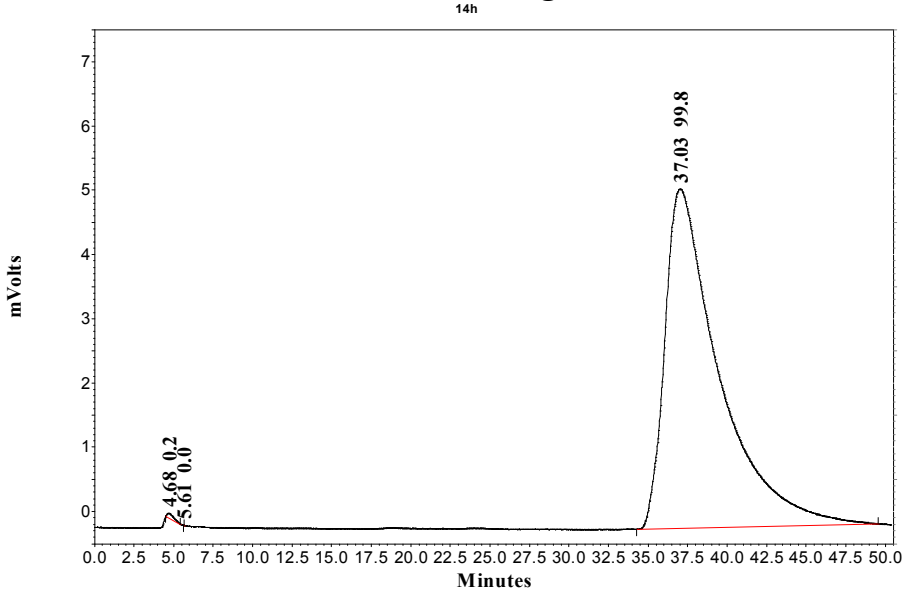
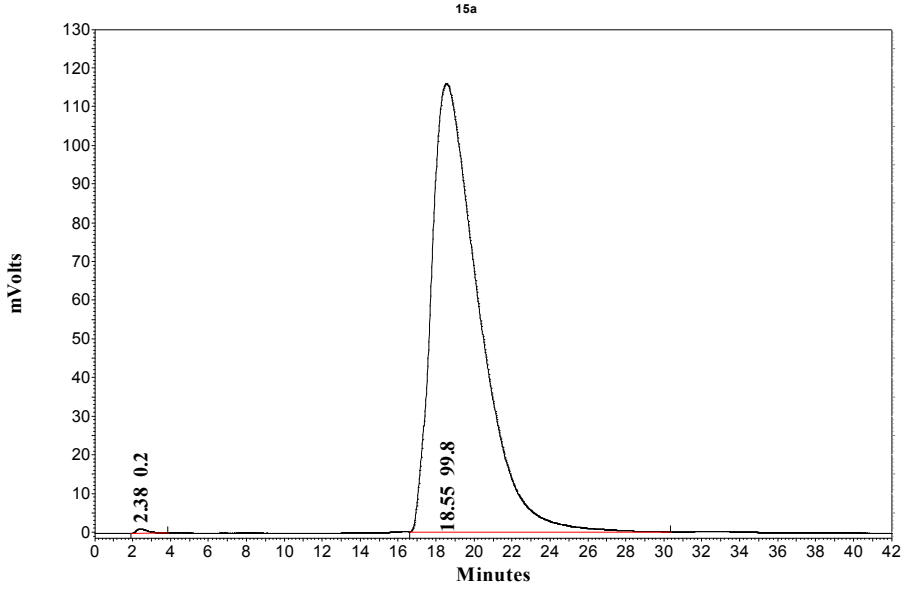
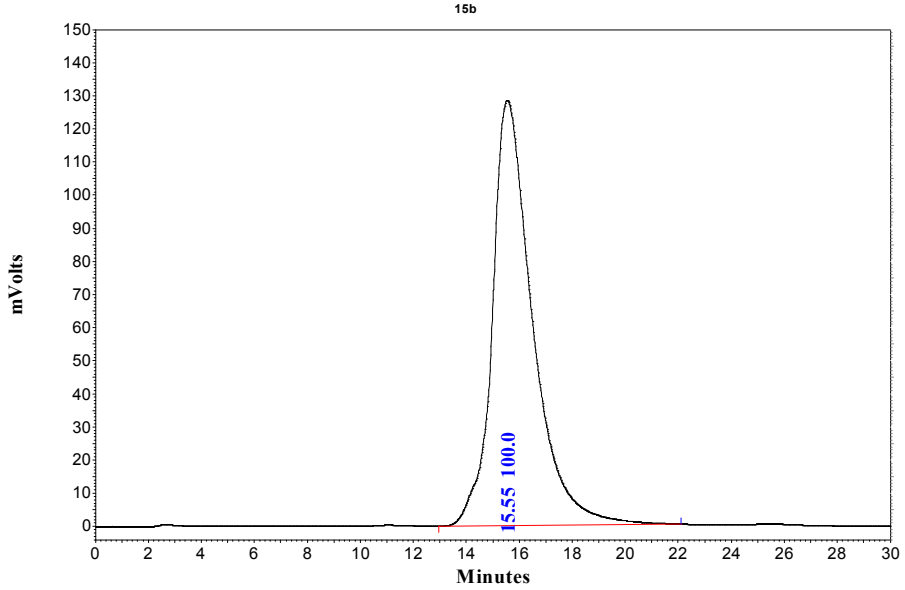


HPLC of 14h



HPLC of 15a



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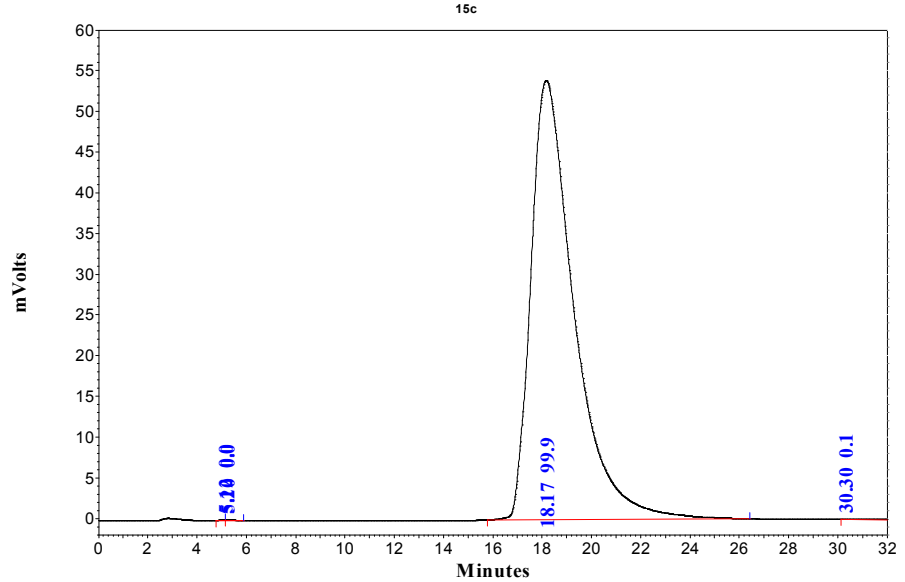
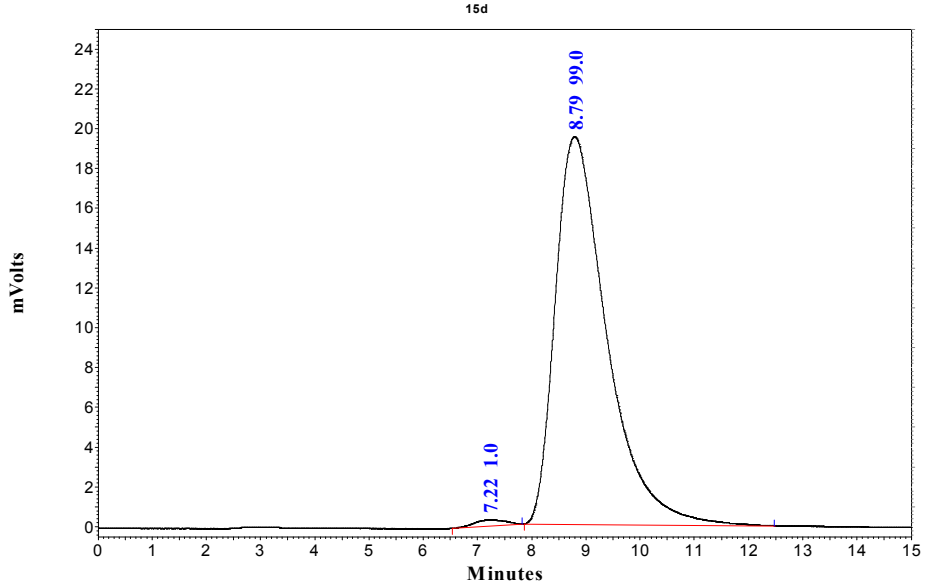
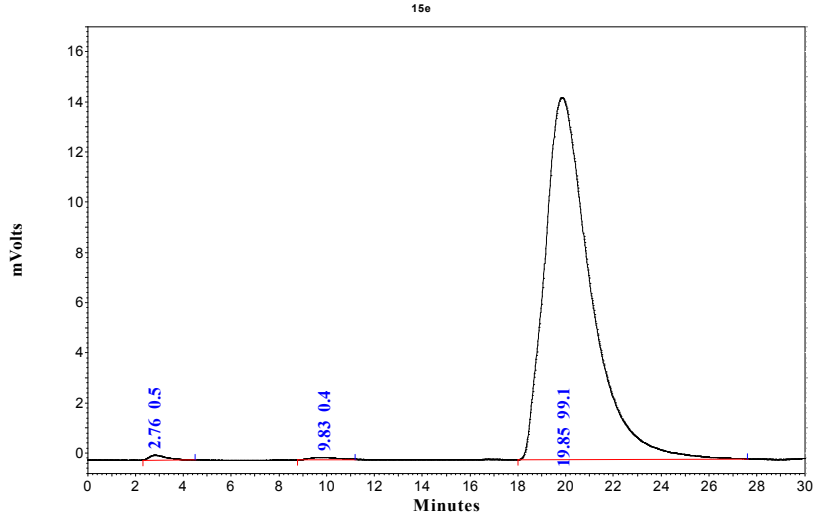


HPLC of 15c



HPLC of 15d



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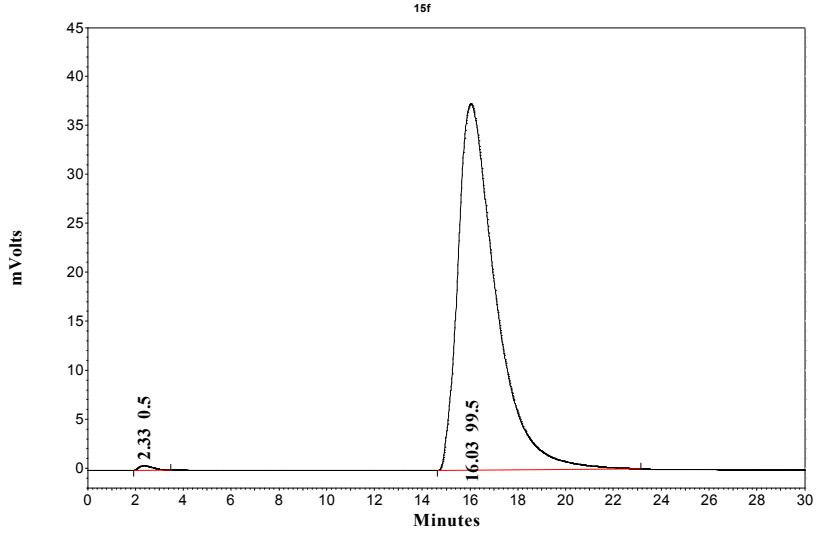

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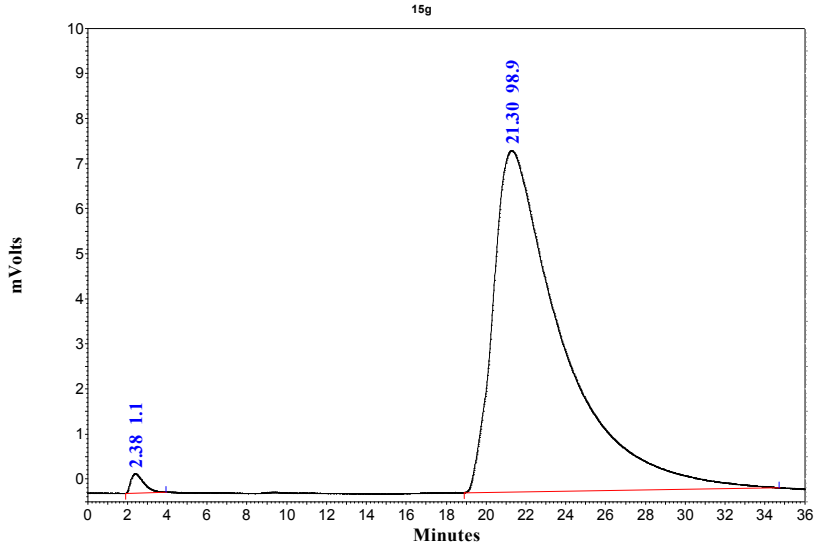

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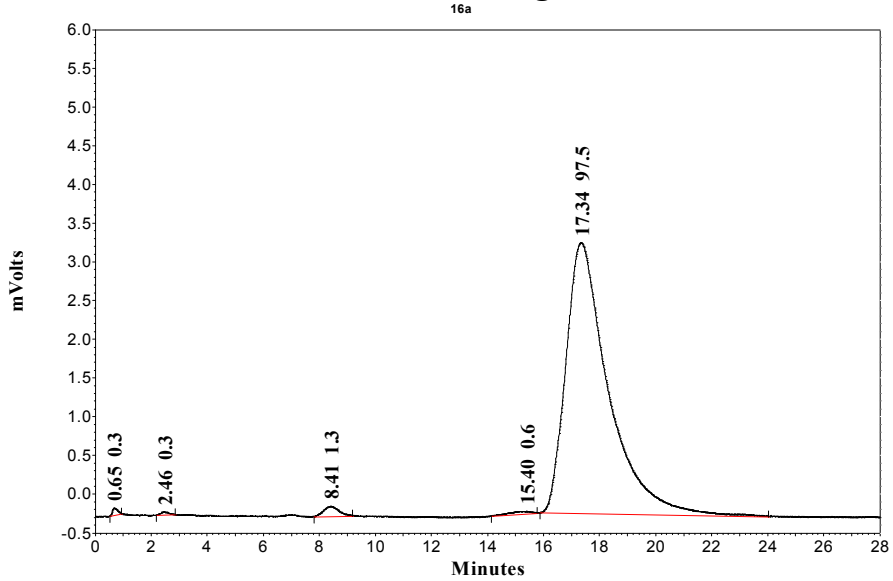
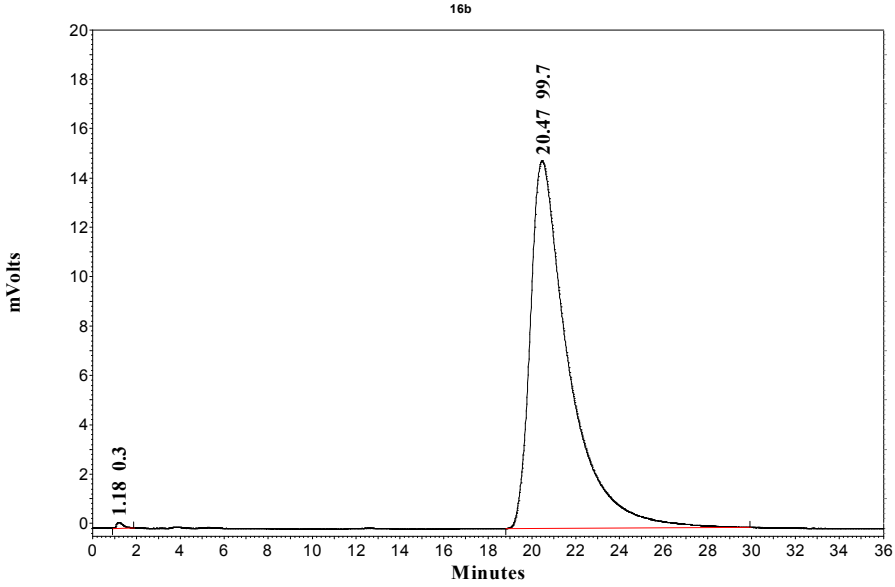
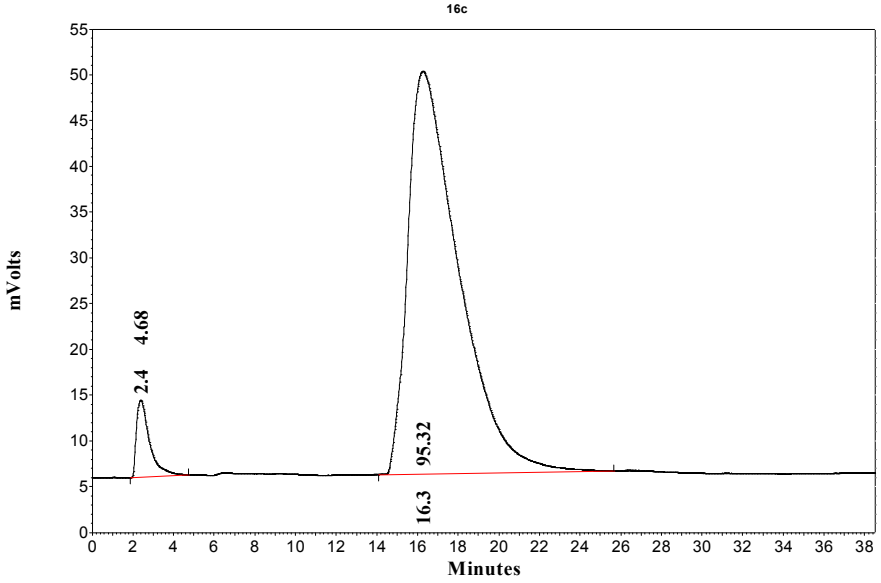


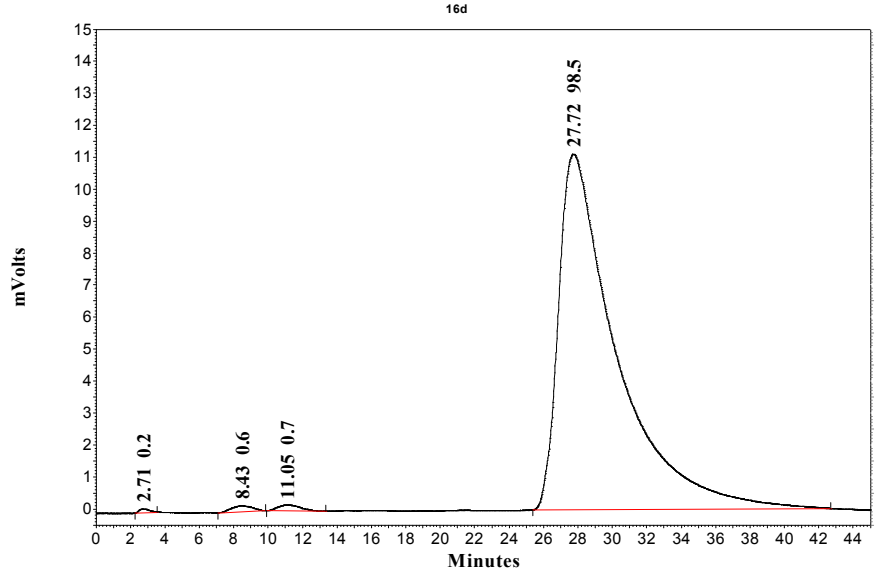
HPLC of 16a



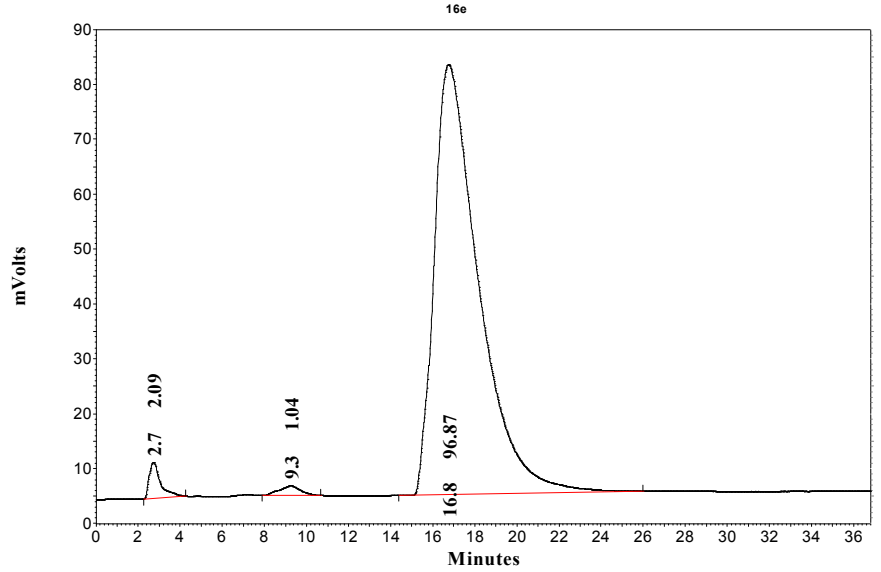
HPLC of 16b



HPLC of 16c



HPLC of 16d



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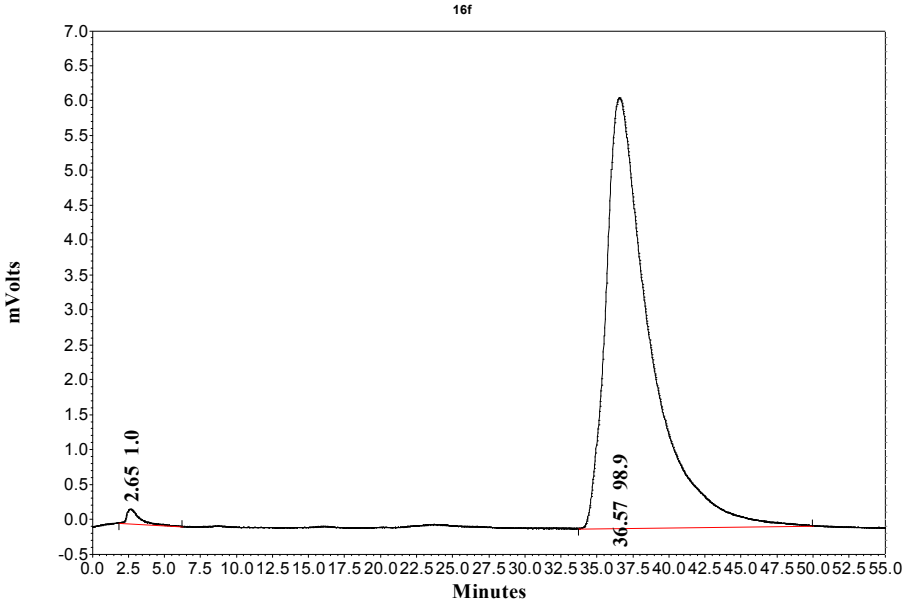

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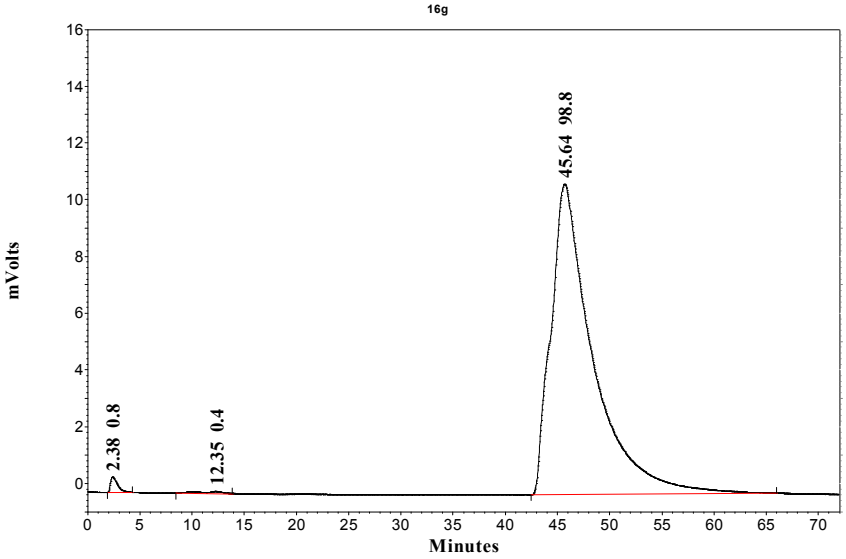
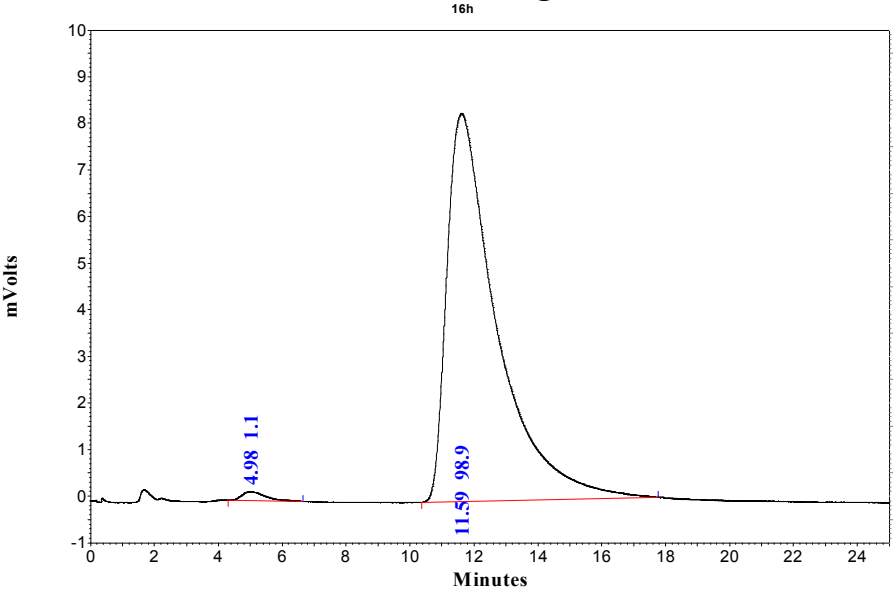


HPLC of 16g



HPLC of 16h

III. X-Ray Diffraction study of 14c.

Crystals of appropriate dimension were obtained by slow evaporation of methanol solutions at 8°C. A crystal with approximate dimensions 0.22 x 0.17 x 0.11 mm^3 was mounted on glass fibers in a random orientation. Preliminary examination and data collection were performed using a Bruker Kappa Apex II Charge Coupled Device (CCD) Detector system single crystal X-Ray diffractometer equipped with an Oxford Cryostream LT device. All data were collected using graphite monochromated Mo Kα radiation (λ= 0.71073 Å) from a fine focus sealed tube X-Ray source. Preliminary unit cell constants were determined with a set of 36 narrow frame scans. Intensity data set included combinations of ϖ and ϕ scan frames with scan width of 0.5° and counting time of 20 seconds/frame at a crystal to detector distance of 4.0 cm. The collected frames were integrated using an orientation matrix determined from the narrow frame scans. Apex II and SAINT software packages (Bruker Analytical X-Ray, Madison, WI, 2006) were used for data collection and data integration. Analysis of the integrated data did not show any decay. Final cell constants were determined by global refinement of xyz centroids of 1393 reflections from the complete data set. Collected data were corrected for systematic errors using SADABS (Blessing, R. H., Acta Cryst. (1995), A51, 33-38) based on the Laue symmetry using equivalent reflections.

Crystal data and intensity data collection parameters are listed in supporting material (Table 4).

Structure solution and refinement were carried out using the SHELXTL- PLUS software package (Sheldrick, G. M., Bruker Analytical X-Ray Division, Madison, WI, 2006). The structure was solved by direct methods and refined successfully in the space group, $P2_1$. Full matrix least-squares refinement was carried out by minimizing $\Sigma w(F_o^2-F_c^2)^2$. The non-hydrogen atoms were refined anisotropically to convergence. All hydrogen atoms were treated using appropriate riding model (AFIX m3). The final residual values and structure refinement parameters are available via the Internet with the supporting information (Table 4).

Complete listings of positional and isotropic displacement coefficients for hydrogen atoms, anisotropic displacement coefficients for the non-hydrogen atoms are available via the Internet with the supporting information (Tables 5-9).

Table 4. Crystal data and structure refinement for **14c**.

Identification code	z7711t5/lt/smart/TZ3-106	
Empirical formula	C_{24} H_{29} F N_2 O_2	
Formula weight	396.49	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 20.1709(13) Å	α= 90°.
	b = 6.1129(3) Å	β= 112.862(3)°.
	c = 18.1330(11) Å	γ = 90°.
Volume	2060.2(2) $Å^3$	
Z	4	
Density (calculated)	1.278 Mg/m^3	
Absorption coefficient	0.087 mm^{-1}	
F(000)	848	
Crystal size	0.22 x 0.17 x 0.11 mm^3	
Theta range for data collection	2.19 to 25.39°.	
Index ranges	$0\leq h\leq 24$, $-7\leq k\leq 0$, $-21\leq l\leq 20$	
Reflections collected	59997	
Independent reflections	3784 [R(int) = 0.059]	
Completeness to theta = 25.00°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9906 and 0.9811	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3784 / 3 / 274
Goodness-of-fit on F^2	1.077
Final R indices [I>2sigma(I)]	R1 = 0.0357, wR2 = 0.0778
R indices (all data)	R1 = 0.0485, wR2 = 0.0836
Largest diff. peak and hole	0.153 and -0.192 e.$Å^{-3}$

Table 5. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **14c**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
F(1)	1888(1)	-618(2)	3824(1)	34(1)
O(1)	4382(1)	5798(2)	4056(1)	28(1)
O(2)	7676(1)	4204(3)	4673(1)	34(1)
O(2')	6673(8)	-2142(16)	3177(11)	45(6)
N(1)	6409(1)	1945(2)	3940(1)	18(1)
N(2)	8251(1)	881(2)	3337(1)	21(1)
C(1)	2477(1)	452(3)	3821(1)	23(1)
C(2)	2910(1)	-596(3)	3505(1)	23(1)
C(3)	3524(1)	486(3)	3528(1)	21(1)
C(4)	3691(1)	2592(3)	3849(1)	18(1)
C(5)	3222(1)	3597(3)	4146(1)	22(1)
C(6)	2609(1)	2538(3)	4132(1)	25(1)
C(7)	4328(1)	3862(3)	3863(1)	20(1)
C(8)	4896(1)	2791(3)	3626(1)	18(1)
C(9)	5345(1)	1091(3)	4246(1)	19(1)
C(10)	5942(1)	196(3)	4014(1)	20(1)
C(11)	5993(1)	3482(3)	3311(1)	22(1)
C(12)	5402(1)	4513(3)	3519(1)	22(1)
C(13)	7070(1)	1100(3)	3875(1)	19(1)
C(14)	6999(1)	-150(3)	3118(1)	22(1)

C(15)	7740(1)	-944(3)	3190(1)	24(1)
C(16)	8330(1)	2017(3)	4078(1)	23(1)
C(17)	7609(1)	2962(3)	3992(1)	24(1)
C(18)	8943(1)	36(3)	3372(1)	26(1)
C(19)	9504(1)	1797(3)	3504(1)	23(1)
C(20)	10178(1)	1616(4)	4114(1)	31(1)
C(21)	10696(1)	3209(4)	4231(1)	38(1)
C(22)	10550(1)	5021(4)	3739(2)	39(1)
C(23)	9874(1)	5213(4)	3116(2)	36(1)
C(24)	9358(1)	3627(3)	3003(1)	30(1)

Table 6. Bond lengths [Å] and angles [°] for **14c**.

F(1)-C(1)	1.357(2)
O(1)-C(7)	1.227(2)
O(2)-C(17)	1.410(2)
O(2)-H(2)	0.8400
O(2')-C(14)	1.408(5)
O(2')-H(2')	0.8400
N(1)-C(11)	1.465(2)
N(1)-C(10)	1.465(2)
N(1)-C(13)	1.476(2)
N(2)-C(16)	1.466(2)
N(2)-C(18)	1.467(2)
N(2)-C(15)	1.471(2)
C(1)-C(2)	1.376(3)
C(1)-C(6)	1.378(3)
C(2)-C(3)	1.390(3)
C(2)-H(2A)	0.9500
C(3)-C(4)	1.398(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.400(3)
C(4)-C(7)	1.493(3)
C(5)-C(6)	1.388(3)
C(5)-H(5)	0.9500
C(6)-H(6)	0.9500

C(7)-C(8)	1.521(3)
C(8)-C(12)	1.529(2)
C(8)-C(9)	1.541(2)
C(8)-H(8)	1.0000
C(9)-C(10)	1.523(3)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.519(3)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-C(14)	1.528(2)
C(13)-C(17)	1.530(2)
C(13)-H(13)	1.0000
C(14)-C(15)	1.529(3)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(14)-H(14C)	1.0000
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-C(17)	1.515(3)
C(16)-H(16A)	0.9900

C(16)-H(16B)	0.9900
C(17)-H(17)	1.0000
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900
C(18)-C(19)	1.512(3)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.384(3)
C(19)-C(24)	1.398(3)
C(20)-C(21)	1.383(3)
C(20)-H(20)	0.9500
C(21)-C(22)	1.380(3)
C(21)-H(21)	0.9500
C(22)-C(23)	1.396(3)
C(22)-H(22)	0.9500
C(23)-C(24)	1.379(3)
C(23)-H(23)	0.9500
C(24)-H(24)	0.9500

C(17)-O(2)-H(2)	109.5
C(14)-O(2')-H(2')	109.5
C(11)-N(1)-C(10)	110.16(13)
C(11)-N(1)-C(13)	115.15(14)
C(10)-N(1)-C(13)	112.62(14)
C(16)-N(2)-C(18)	111.41(14)

C(16)-N(2)-C(15)	110.15(15)
C(18)-N(2)-C(15)	109.17(14)
F(1)-C(1)-C(2)	118.40(16)
F(1)-C(1)-C(6)	118.34(17)
C(2)-C(1)-C(6)	123.25(17)
C(1)-C(2)-C(3)	117.94(17)
C(1)-C(2)-H(2A)	121.0
C(3)-C(2)-H(2A)	121.0
C(2)-C(3)-C(4)	121.19(17)
C(2)-C(3)-H(3)	119.4
C(4)-C(3)-H(3)	119.4
C(3)-C(4)-C(5)	118.37(17)
C(3)-C(4)-C(7)	123.50(16)
C(5)-C(4)-C(7)	118.08(16)
C(6)-C(5)-C(4)	121.25(18)
C(6)-C(5)-H(5)	119.4
C(4)-C(5)-H(5)	119.4
C(1)-C(6)-C(5)	117.95(18)
C(1)-C(6)-H(6)	121.0
C(5)-C(6)-H(6)	121.0
O(1)-C(7)-C(4)	119.23(16)
O(1)-C(7)-C(8)	120.13(16)
C(4)-C(7)-C(8)	120.64(15)
C(7)-C(8)-C(12)	110.65(15)
C(7)-C(8)-C(9)	112.18(15)

C(12)-C(8)-C(9)	108.97(14)
C(7)-C(8)-H(8)	108.3
C(12)-C(8)-H(8)	108.3
C(9)-C(8)-H(8)	108.3
C(10)-C(9)-C(8)	110.13(15)
C(10)-C(9)-H(9A)	109.6
C(8)-C(9)-H(9A)	109.6
C(10)-C(9)-H(9B)	109.6
C(8)-C(9)-H(9B)	109.6
H(9A)-C(9)-H(9B)	108.1
N(1)-C(10)-C(9)	111.62(15)
N(1)-C(10)-H(10A)	109.3
C(9)-C(10)-H(10A)	109.3
N(1)-C(10)-H(10B)	109.3
C(9)-C(10)-H(10B)	109.3
H(10A)-C(10)-H(10B)	108.0
N(1)-C(11)-C(12)	109.98(15)
N(1)-C(11)-H(11A)	109.7
C(12)-C(11)-H(11A)	109.7
N(1)-C(11)-H(11B)	109.7
C(12)-C(11)-H(11B)	109.7
H(11A)-C(11)-H(11B)	108.2
C(11)-C(12)-C(8)	111.81(15)
C(11)-C(12)-H(12A)	109.3
C(8)-C(12)-H(12A)	109.3

C(11)-C(12)-H(12B)	109.3
C(8)-C(12)-H(12B)	109.3
H(12A)-C(12)-H(12B)	107.9
N(1)-C(13)-C(14)	118.29(15)
N(1)-C(13)-C(17)	110.07(14)
C(14)-C(13)-C(17)	108.09(15)
N(1)-C(13)-H(13)	106.6
C(14)-C(13)-H(13)	106.6
C(17)-C(13)-H(13)	106.6
O(2')-C(14)-C(13)	104.3(8)
O(2')-C(14)-C(15)	100.9(7)
C(13)-C(14)-C(15)	109.56(15)
O(2')-C(14)-H(14A)	121.8
C(13)-C(14)-H(14A)	109.8
C(15)-C(14)-H(14A)	109.8
C(13)-C(14)-H(14B)	109.8
C(15)-C(14)-H(14B)	109.8
H(14A)-C(14)-H(14B)	108.2
O(2')-C(14)-H(14C)	113.6
C(13)-C(14)-H(14C)	113.6
C(15)-C(14)-H(14C)	113.6
H(14B)-C(14)-H(14C)	100.1
N(2)-C(15)-C(14)	111.56(15)
N(2)-C(15)-H(15A)	109.3
C(14)-C(15)-H(15A)	109.3

N(2)-C(15)-H(15B)	109.3
C(14)-C(15)-H(15B)	109.3
H(15A)-C(15)-H(15B)	108.0
N(2)-C(16)-C(17)	109.17(15)
N(2)-C(16)-H(16A)	109.8
C(17)-C(16)-H(16A)	109.8
N(2)-C(16)-H(16B)	109.8
C(17)-C(16)-H(16B)	109.8
H(16A)-C(16)-H(16B)	108.3
O(2)-C(17)-C(16)	110.64(16)
O(2)-C(17)-C(13)	110.11(16)
C(16)-C(17)-C(13)	109.40(15)
O(2)-C(17)-H(17)	108.9
C(16)-C(17)-H(17)	108.9
C(13)-C(17)-H(17)	108.9
O(2)-C(17)-H(17A)	107.1
C(16)-C(17)-H(17A)	109.8
C(13)-C(17)-H(17A)	109.8
C(16)-C(17)-H(17B)	109.8
C(13)-C(17)-H(17B)	109.8
H(17)-C(17)-H(17B)	110.1
H(17A)-C(17)-H(17B)	108.2
N(2)-C(18)-C(19)	113.41(15)
N(2)-C(18)-H(18A)	108.9
C(19)-C(18)-H(18A)	108.9

N(2)-C(18)-H(18B)	108.9
C(19)-C(18)-H(18B)	108.9
H(18A)-C(18)-H(18B)	107.7
C(20)-C(19)-C(24)	118.53(19)
C(20)-C(19)-C(18)	120.77(18)
C(24)-C(19)-C(18)	120.69(17)
C(21)-C(20)-C(19)	121.0(2)
C(21)-C(20)-H(20)	119.5
C(19)-C(20)-H(20)	119.5
C(22)-C(21)-C(20)	120.5(2)
C(22)-C(21)-H(21)	119.8
C(20)-C(21)-H(21)	119.8
C(21)-C(22)-C(23)	119.1(2)
C(21)-C(22)-H(22)	120.4
C(23)-C(22)-H(22)	120.4
C(24)-C(23)-C(22)	120.3(2)
C(24)-C(23)-H(23)	119.8
C(22)-C(23)-H(23)	119.8
C(23)-C(24)-C(19)	120.6(2)
C(23)-C(24)-H(24)	119.7
C(19)-C(24)-H(24)	119.7

Table 7. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **14c**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(1)	29(1)	42(1)	37(1)	-5(1)	18(1)	-13(1)
O(1)	28(1)	20(1)	40(1)	-6(1)	16(1)	-3(1)
O(2)	28(1)	41(1)	42(1)	-28(1)	23(1)	-15(1)
O(2')	29(9)	40(11)	59(13)	-7(8)	10(8)	-10(8)
N(1)	17(1)	17(1)	21(1)	2(1)	9(1)	-1(1)
N(2)	17(1)	22(1)	25(1)	-3(1)	10(1)	0(1)
C(1)	18(1)	29(1)	20(1)	4(1)	6(1)	-4(1)
C(2)	25(1)	20(1)	24(1)	0(1)	9(1)	-3(1)
C(3)	21(1)	20(1)	22(1)	1(1)	10(1)	3(1)
C(4)	19(1)	19(1)	15(1)	3(1)	5(1)	2(1)
C(5)	24(1)	21(1)	20(1)	0(1)	8(1)	1(1)
C(6)	23(1)	32(1)	21(1)	-2(1)	10(1)	2(1)
C(7)	20(1)	21(1)	17(1)	2(1)	5(1)	2(1)
C(8)	18(1)	19(1)	18(1)	0(1)	7(1)	-1(1)
C(9)	19(1)	20(1)	19(1)	2(1)	8(1)	-2(1)
C(10)	20(1)	17(1)	22(1)	3(1)	9(1)	0(1)
C(11)	22(1)	21(1)	26(1)	6(1)	11(1)	0(1)
C(12)	20(1)	20(1)	26(1)	5(1)	10(1)	0(1)
C(13)	17(1)	18(1)	24(1)	-1(1)	8(1)	0(1)
C(14)	23(1)	20(1)	27(1)	-5(1)	12(1)	-6(1)

C(15)	25(1)	21(1)	28(1)	-6(1)	12(1)	-2(1)
C(16)	20(1)	26(1)	25(1)	-5(1)	10(1)	-3(1)
C(17)	24(1)	23(1)	28(1)	-6(1)	13(1)	-6(1)
C(18)	22(1)	27(1)	31(1)	-3(1)	13(1)	2(1)
C(19)	20(1)	27(1)	26(1)	-2(1)	14(1)	3(1)
C(20)	24(1)	40(1)	32(1)	6(1)	13(1)	3(1)
C(21)	21(1)	53(2)	39(1)	-4(1)	11(1)	-5(1)
C(22)	33(1)	37(1)	58(2)	-10(1)	30(1)	-8(1)
C(23)	40(1)	28(1)	51(2)	5(1)	31(1)	5(1)
C(24)	26(1)	34(1)	34(1)	4(1)	15(1)	7(1)

Table 8. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **14c**.

	x	y	z	U(eq)
H(2)	7302	4064	4767	52
H(2')	6937	-2806	3593	67
H(2A)	2794	-2016	3278	28
H(3)	3836	-216	3323	25
H(5)	3326	5032	4362	26
H(6)	2289	3229	4330	30
H(8)	4648	2027	3102	22
H(9A)	5558	1782	4781	23
H(9B)	5031	-122	4274	23
H(10A)	6234	-863	4425	23
H(10B)	5726	-590	3498	23
H(11A)	5777	2701	2794	27
H(11B)	6314	4638	3256	27
H(12A)	5121	5536	3087	26
H(12B)	5622	5365	4020	26
H(13)	7295	69	4333	23
H(14A)	6789	817	2646	27
H(14B)	6674	-1417	3045	27
H(14C)	6730	671	2612	27

H(15A)	7929	-2008	3635	29
H(15B)	7695	-1702	2690	29
H(16A)	8689	3206	4187	28
H(16B)	8501	981	4533	28
H(17)	7429	3933	3511	29
H(17A)	7431	3963	3527	29
H(17B)	7662	3808	4478	29
H(18A)	8866	-735	2865	31
H(18B)	9128	-1045	3812	31
H(20)	10287	378	4457	37
H(21)	11156	3056	4653	46
H(22)	10904	6123	3822	46
H(23)	9770	6443	2769	43
H(24)	8898	3780	2582	36

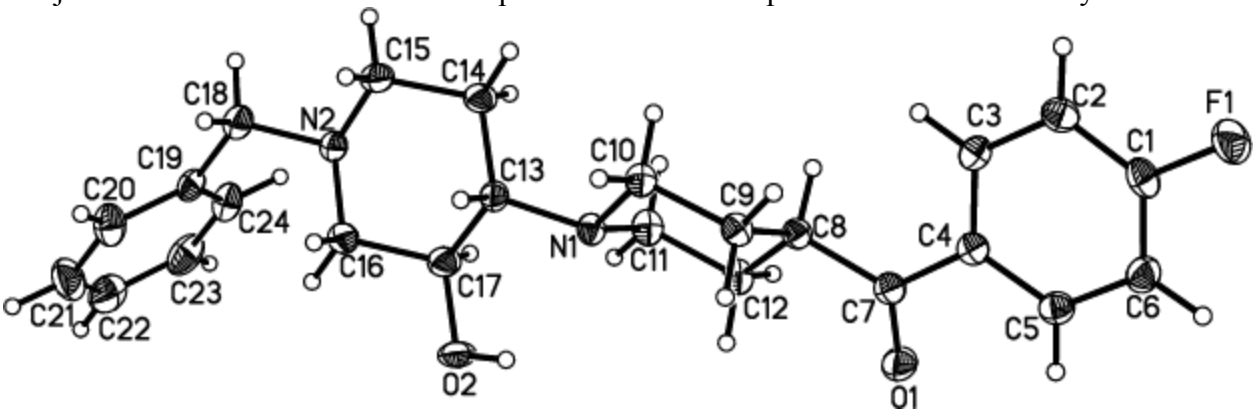
Table 9. Torsion angles [°] for **14c**.

F(1)-C(1)-C(2)-C(3)	-177.94(17)
C(6)-C(1)-C(2)-C(3)	2.2(3)
C(1)-C(2)-C(3)-C(4)	-1.1(3)
C(2)-C(3)-C(4)-C(5)	-0.2(3)
C(2)-C(3)-C(4)-C(7)	-177.74(17)
C(3)-C(4)-C(5)-C(6)	0.5(3)
C(7)-C(4)-C(5)-C(6)	178.19(18)
F(1)-C(1)-C(6)-C(5)	178.24(17)
C(2)-C(1)-C(6)-C(5)	-1.9(3)
C(4)-C(5)-C(6)-C(1)	0.5(3)
C(3)-C(4)-C(7)-O(1)	170.22(18)
C(5)-C(4)-C(7)-O(1)	-7.3(3)
C(3)-C(4)-C(7)-C(8)	-8.5(3)
C(5)-C(4)-C(7)-C(8)	173.97(16)
O(1)-C(7)-C(8)-C(12)	-10.6(2)
C(4)-C(7)-C(8)-C(12)	168.11(16)
O(1)-C(7)-C(8)-C(9)	111.33(19)
C(4)-C(7)-C(8)-C(9)	-70.0(2)
C(7)-C(8)-C(9)-C(10)	-175.66(14)
C(12)-C(8)-C(9)-C(10)	-52.8(2)
C(11)-N(1)-C(10)-C(9)	-61.34(19)
C(13)-N(1)-C(10)-C(9)	168.62(15)
C(8)-C(9)-C(10)-N(1)	57.5(2)

C(10)-N(1)-C(11)-C(12)	60.70(19)
C(13)-N(1)-C(11)-C(12)	-170.62(14)
N(1)-C(11)-C(12)-C(8)	-58.3(2)
C(7)-C(8)-C(12)-C(11)	177.88(15)
C(9)-C(8)-C(12)-C(11)	54.1(2)
C(11)-N(1)-C(13)-C(14)	-59.4(2)
C(10)-N(1)-C(13)-C(14)	68.0(2)
C(11)-N(1)-C(13)-C(17)	65.46(19)
C(10)-N(1)-C(13)-C(17)	-167.09(16)
N(1)-C(13)-C(14)-O(2')	-70.5(7)
C(17)-C(13)-C(14)-O(2')	163.6(7)
N(1)-C(13)-C(14)-C(15)	-177.91(16)
C(17)-C(13)-C(14)-C(15)	56.2(2)
C(16)-N(2)-C(15)-C(14)	59.3(2)
C(18)-N(2)-C(15)-C(14)	-178.02(16)
O(2')-C(14)-C(15)-N(2)	-166.4(8)
C(13)-C(14)-C(15)-N(2)	-56.8(2)
C(18)-N(2)-C(16)-C(17)	176.90(16)
C(15)-N(2)-C(16)-C(17)	-61.79(19)
N(2)-C(16)-C(17)-O(2)	-175.69(16)
N(2)-C(16)-C(17)-C(13)	62.8(2)
N(1)-C(13)-C(17)-O(2)	47.7(2)
C(14)-C(13)-C(17)-O(2)	178.29(16)
N(1)-C(13)-C(17)-C(16)	169.53(15)
C(14)-C(13)-C(17)-C(16)	-59.9(2)

C(16)-N(2)-C(18)-C(19)	-59.4(2)
C(15)-N(2)-C(18)-C(19)	178.74(16)
N(2)-C(18)-C(19)-C(20)	127.7(2)
N(2)-C(18)-C(19)-C(24)	-53.4(3)
C(24)-C(19)-C(20)-C(21)	0.3(3)
C(18)-C(19)-C(20)-C(21)	179.23(19)
C(19)-C(20)-C(21)-C(22)	0.0(3)
C(20)-C(21)-C(22)-C(23)	-0.6(3)
C(21)-C(22)-C(23)-C(24)	0.9(3)
C(22)-C(23)-C(24)-C(19)	-0.7(3)
C(20)-C(19)-C(24)-C(23)	0.0(3)
C(18)-C(19)-C(24)-C(23)	-178.90(18)

Projection view with 50% thermal ellipsoids- disorder component omitted for clarity:



Notes:

The structure is twinned (2-component twin). Twin refinement was carried out with HKLF 5 data and BASF refined to 0.5178.

The OH group is disordered over two equivalent C's C17 and C14 (refined to 90.5:9.5%).

The checkcif shows alert level B due to short interaction between H11b…..H2' = 1.95A. The H2' is the calculated H on disorder O2'. Both H calculation using HFIX 147 and HFIX 83 used and HFIX 83 results in best geometry.