Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

Environmentally Benign and Atom-Economical Protocol for the Regioselective Synthesis of Isoquinolones from *o*-Alkynylaldehydes

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X-Ray Crystallographic Studies

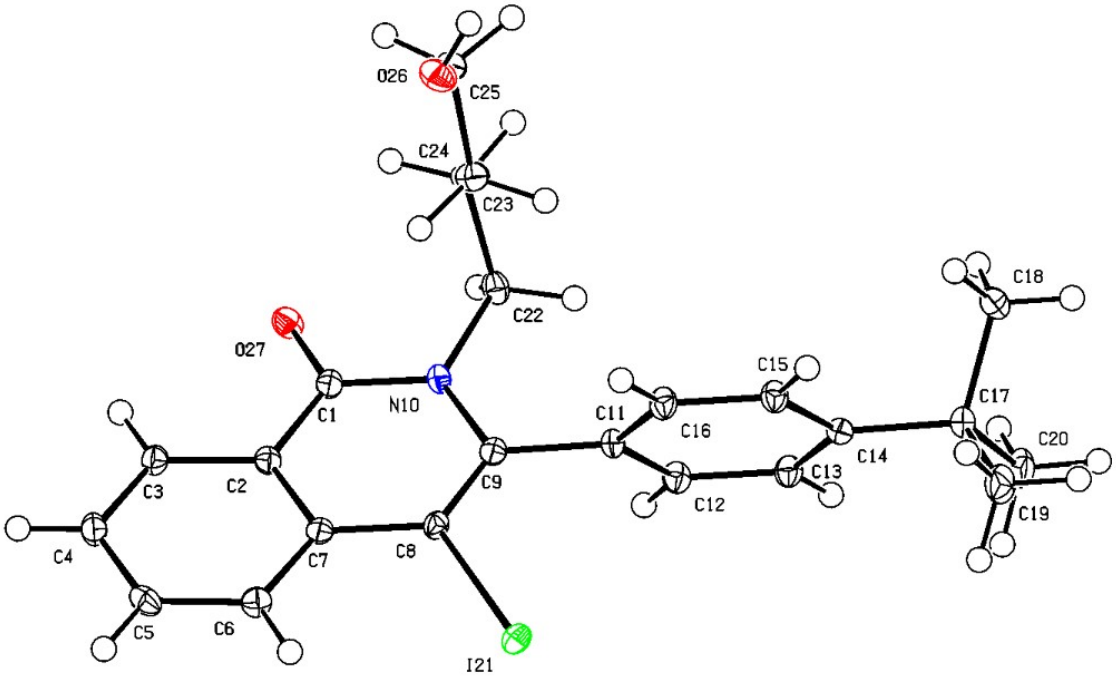


Figure S1. ORTEP diagram for each molecule of the asymmetric unit of compound **3e** drawn with 50% ellipsoid probability.

The crystals of **3e** of suitable quality were obtained from $CHCl_2$/*n*-hexane. The compound **3e** crystallized in Triclinic crystal system with space group P c. The single-crystal X-ray data were collected on an Oxford XCalibur CCD diffractometer using graphite monochromated Mo Kα radiation. The structures was solved using SIR-92 and refined by full matrix least square technique on F^2 using the SHELXL-97[1-4] program within the WinGX v 1.80.05 software package. Atomic coordinates, bond lengths, bond angles, and thermal parameters for compounds **3e** has been deposited at the Cambridge Crystallographic Data Centre. CCDC deposit number for **3e** is 2301423.

Table SI. Crystallographic data and structure refinement for compounds 3e

Identification code	**3e**
Empirical formula	$C_{23}\ H_{26}\ I\ N\ O_2$
Formula weight	475.35
Temperature	293 K
Wavelength	0.71073 Å

Crystal system	Monoclinic	
Space group	P c	
Unit cell dimensions	a = 8.95427(16)	α = 90
	b = 13.54255(18)	β = 113.787(2)
	c = 9.03971(16)	γ = 90
Volume	1003.07 $Å^3$	
Z	2	
Density (calculated)	1.574 g/cm^3	
Absorption coefficient	1.614 mm^{-1}	
F(000)	480.0	
Index ranges	h= 12, k = 18, l = 12	
Reflections collected	0.0207(5248)	
Completeness to theta = 26.37 °	99.7 %	
Final R indices [I>2 sigma(I)][a,b]	R1 = 0.0207, wR2 = 0.0449	

[a] $R = \sum(\|Fo\| - |Fc\|)/\sum|Fo|$; [b] $Rw = \{\sum[w(Fo^2 - Fc^2)^2]/\sum[w(Fo^2)^2]\}^{1/2}$

References:

1. CrysAlisPro, Agilent Technologies, Version 1.171.34.49, 2011.

2. Sheldrick, G. M., *Acta Cryst*. 2008, *A64*, 112.

3. Farrugia, L. *J*. WinGX Version 1.80.05, An integrated system of Windows Programs for the Solution, Refinement and Analysis of Single Crystal X-Ray Diffraction Data*;* Department of Chemistry, University of Glasgow,1997-2009.

4. (a) Foresman, *J*. B.; Frisch, A. E. *Exploring Chemistry with Electronic Structure Methods*; Gaussian, Inc.: Pittsburgh, PA. 1995. (b) Hehre, W. *J*., Radom, L., Schleyer, P. V. R.; Pople, *J*. A. *Ab Initio Molecular Orbital Theory*; Wiley: New York, 1985.

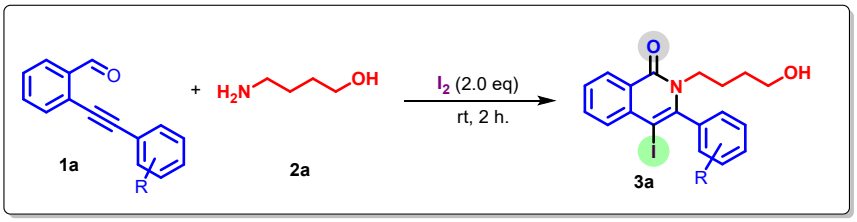
General Experimental

General Method. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$/$(CD_3)_2SO$. Chemical shifts for protons and carbons are reported in ppm from tetramethylsilane and are referenced to the carbon resonance of the solvent. Data are reported as follows: chemical shift, multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet), coupling constants in Hertz and integration. High-resolution mass spectra were recorded on electrospray mass spectrometer. Crystal structure analysis was accomplished on single needles X-ray diffractometer. TLC analysis was performed on commercially prepared 60 F_{254} silica gel plates. All purchased chemicals were used as received. All melting points are uncorrected. The starting materials were synthesised according to the literature reporting method.[1]

References:

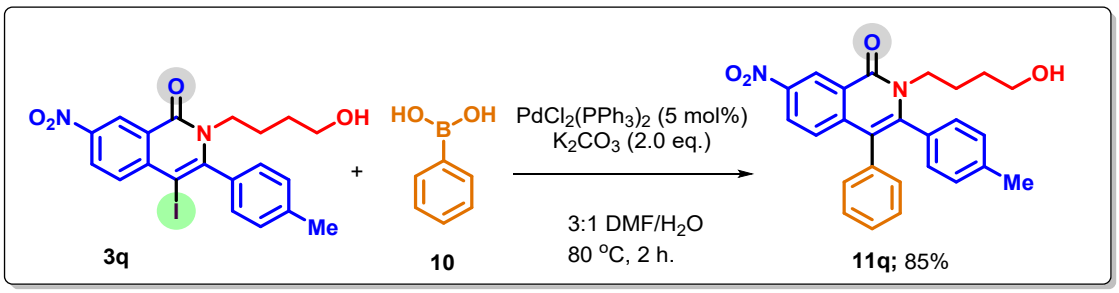
1.(a) R. Kumar, A. Chandra, B. A. Mir, and G. Shukla, *J. Org. Chem*. **2019**, *84*, 10710−10723. (b) K. Mishra, *J*. B. Singh, T. Gupta and R. M. Singh, *Org. Chem. Front*. **2017**, *4*, 1926–1930. (c) A. K. Verma, D. Choudhary, R. K. Saunthwal, V. Rustagi, M. Patel, and R. K. Tiwari, *J. Org. Chem.* **2013**, *78*, 6657−6669. (d) A. K. Verma, V. Rustagi, T. Aggarwal, and A. P. Singh, *J. Org. Chem.*, **2010**, *75*, 7691–7703. (e) N. Sharma, M. Asthana, D. Nandini, R. P. Singh, R. M. Singh, *Tetrahedron,* **2013**, *69*, 1822–1829. (f) V. Rustagi, R. Tiwari, and A. K. Verma, *Eur. J. Org. Chem.* **2012**, 4590–4602. (g) S., T. Aggarwal, N. Shibata, and A. K. Verma, *Chem. Eur. J.* **2019**, *25*, 16063 –16067. (h) P. Kumar, T. Aggarwal, and A. K. Verma, *J. Org. Chem.*,**2017**, *82*, 6388−6397. (h) M. Asthana, *J*. B. Singh, R. M. Singh, *Tetrahedron Lett.* **2016**, *57*,615–618. (i) S. Kumar, C. Cruz, S. Pal, R. K. Saunthwal, R. K. Tiwari, E. *J*uaristi, and A. K. Verma, *J. Org. Chem.* **2015**, *80*, 10548–10560.

General Procedure for the Synthesis of iodo-substituted isoquinolones(3a-v)



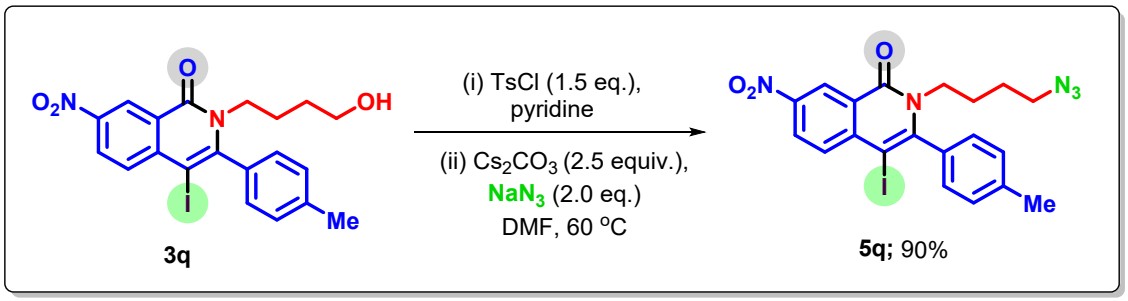
In a 10 ml. oven-dried round bottom flask, o-alkynyl aldehyde **1** (0.25 mmol, 1.0 equiv.), 4-amino-1-butanol **2** (0.75 mmol, 3.0 equiv.) and I_2 (0.40 mmol, 2.0 equiv) was taken and stirred at room temperature for 2 h. The progression of the reaction was monitored by TLC analysis. The reaction mixture was then quenched with satd. aq. $Na_2S_2O_3$ (5.0 mL) and water (5.0 mL). The resulting solution was extracted using ethyl acetate. The combined organic extracts were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The crude material obtained was purified by column chromatography on silica gel (100−200) (hexane:ethyl acetate; 75/25).

Transformation of 3q *via* Suzuki coupling reaction :



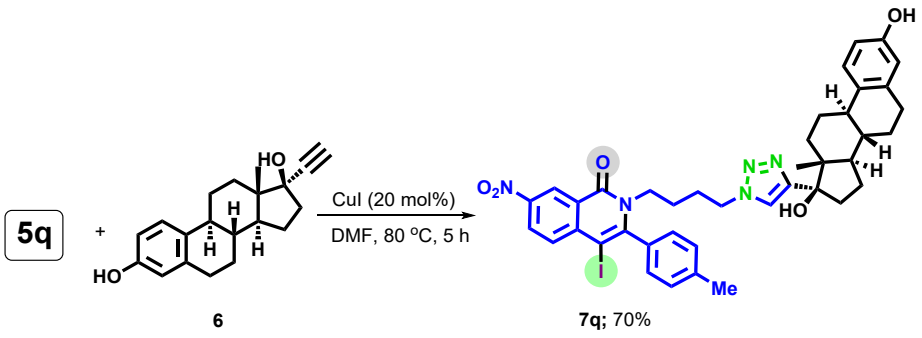
Representative procedure: An oven-dried vial was charged with the obtained product **3q** (0.10 mmol, 1.0 equiv), phenylboronic acid (0.13 mmol, 1.3 equiv.), $Pd(PPh_3)_2Cl_2$ (5 mol %), K_2CO_3 (0.20 mmol, 2.0 equiv.) in 3:1 DMF/H_2O. The reactants were degassed and filled with N_2 for three times. Then the reaction mixture was stirred at 80 °C for 2 h. When the reaction was completed, the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate. Then organic layer was washed with aqueous saturated brine solution and dried over Na_2SO_4 and concentrated under reduced pressure. The crude material obtained was purified by column chromatography on silica gel (100−200) (hexane:ethyl acetate; 70/30) to afford the desired product **11q** in 85% yield.

Conversion of -OH hydroxy group to azide group:



Representative procedure: An oven-dried vial was charged with the obtained product **3q** (0.10 mmol, 1.0 equiv), tosyl chloride (0.15 mmol, 1.5 equiv.),in 3 mL pyridine.. Then the reaction mixture was stirred at room temperature for 10 h. When the reaction was completed, the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate. Then organic layer was washed with aqueous saturated brine solution and dried over Na_2SO_4 and concentrated under reduced pressure. The crude material thus obtained was treated with NaN_3 (0.25 mmol, 2.5 equiv.), Cs_2CO_3 (0.25 mmol, 2.5 equiv.) in DMF and the reaction mixture was stirred at 60 °C for 4 h. When the reaction was completed, the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate. Then organic layer was washed with aqueous saturated brine solution and dried over Na_2SO_4 and concentrated under reduced pressure. The crude material obtained was purified by column chromatography on silica gel (100−200) (hexane:ethyl acetate; 70/30) to afford the desired product **5q** in 90% yield.

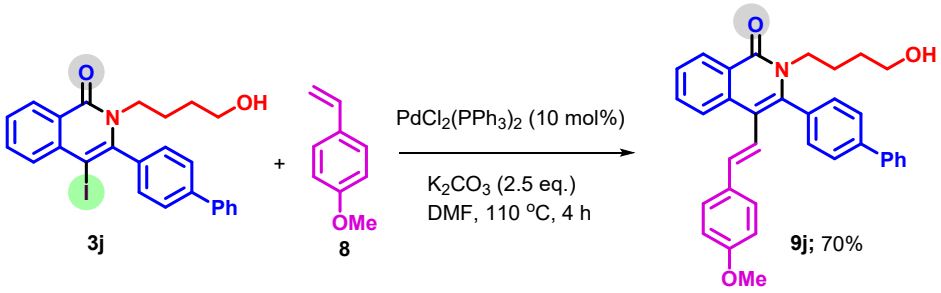
Transformation of 5q *via* Click reaction :



Representative procedure: An oven-dried vial was charged with the obtained product **5q** (0.10 mmol, 1.0 equiv), CuI (0.20 mmol),in 2 mL DMF. Then the reaction mixture was stirred at 80 °C for 5 h. When the reaction was completed, the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate. Then organic layer was washed with aqueous saturated brine solution and dried over Na_2SO_4 and concentrated under reduced pressure. The crude material obtained was

purified by column chromatography on silica gel (100−200) (hexane:ethyl acetate; 70/30) to afford the desired product **7q** in 70% yield.

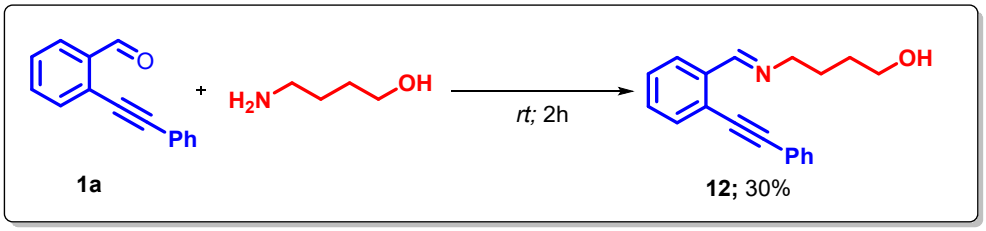
Transformation of 3*j* *via* Heck coupling reaction :



Representative procedure: An oven-dried vial was charged with the obtained product ***3j*** (0.10 mmol, 1.0 equiv), 4-methoxystyrene (0.13 mmol, 1.3 equiv.), $Pd(PPh_3)_2Cl_2$ (10 mol %), K_2CO_3 (0.25 mmol, 2.5 equiv.) in DMF. The reactants were degassed and filled with N_2 for three times. Then the reaction mixture was stirred at 110 °C for 4 h. When the reaction was completed, the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate. Then organic layer was washed with aqueous saturated brine solution and dried over Na_2SO_4 and concentrated under reduced pressure. The crude material obtained was purified by column chromatography on silica gel (100−200) (hexane:ethyl acetate; 80/20) to afford the desired product ***9J*** in 70% yield.

Mechanistic Control Investigations :

When, we treated the *ortho*-alkynyl aldehyde with amino alcohol in the absence of iodine , the aldimine intermediate **12** was obtained in a 30% yield . The starting material was not completely consumed in the reaction mixture even after 24 h as clear from the crude NMR of the reaction mixture.



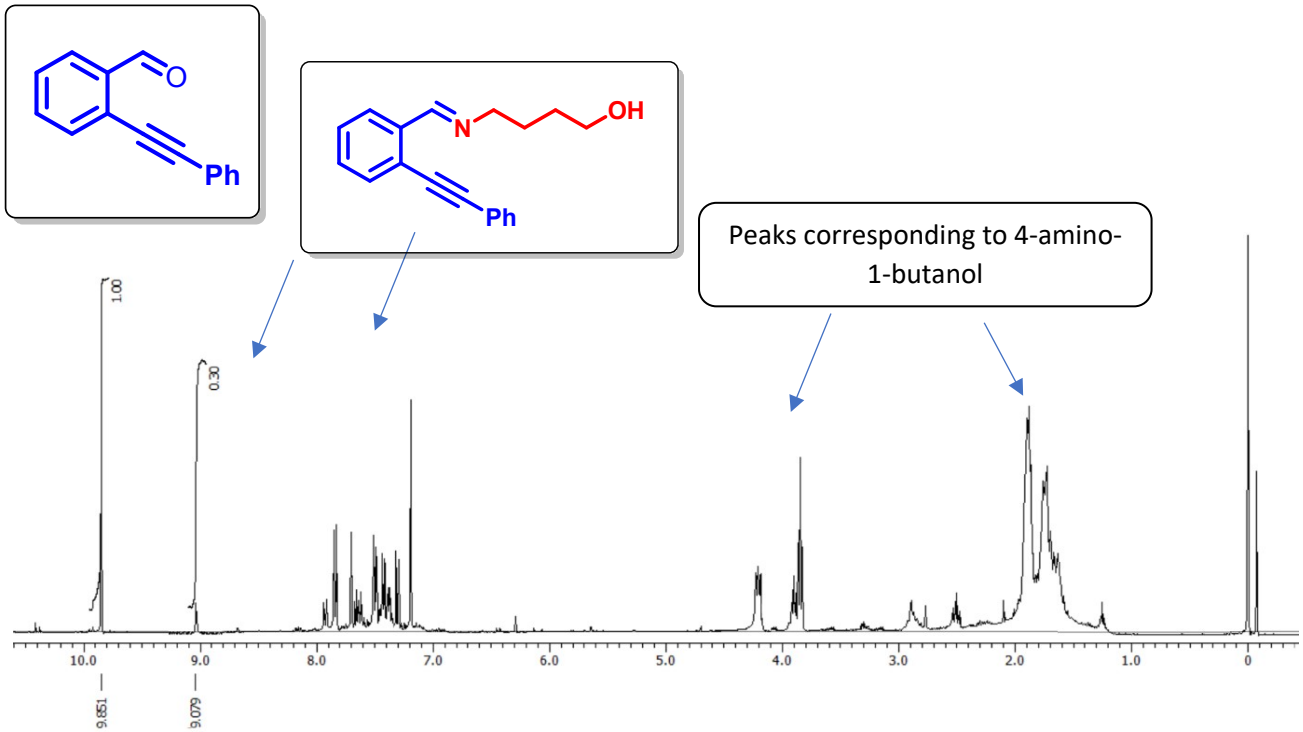
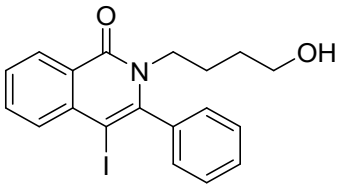
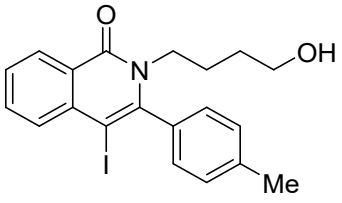


Figure S2: ^{1}H NMR of crude reaction mixture of (Scheme 6, i) showing relative ratio of **12** and **1a** in the reaction mixture.

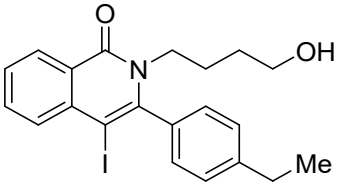
Characterization data for the products:



2-(4-hydroxybutyl)-4-iodo-3-phenylisoquinolin-1(2*H*)-one (3a).The product was obtained as a sticky semi-solid (86.1 mg, 82%): ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.40 (d, J = 8.0 Hz, 1H), 7.89 (d, J = 8.0 Hz, 1H), 7.71-7.67 (m, 1H), 7.52-7.48 (m, 1H), 7.36-7.31 (m, 2H), 7.28-7.23 (m, 3H), 3.86 (t, J = 7.6 Hz, 2H), 3.50 (t, J = 6.4 Hz, 2H), 2.68 (s, 1H), 1.66-1.59 (m, 4H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.7, 142.1, 137.8, 137.2, 134.0, 132.1, 129.4, 128.6, 128.1, 126.1, 80.5, 62.2, 48.1, 29.7, 25.6; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{19}H_{19}INO_2]$ 420.0460, found 420.0451.

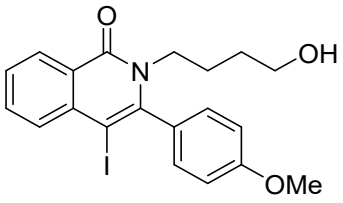


2-(4-hydroxybutyl)-4-iodo-3-(*p*-tolyl)isoquinolin-1(2*H*)-one (3b).The product was obtained as a brown solid (91.5 mg, 85%): mp 136–138 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.36 (dd, J = 8.0, 1.0 Hz, 1H), 7.85 (d, J = 8.0 Hz, 1H), 7.67-7.63 (m, 1H), 7.48-7.44 (m, 1H), 7.28 (d, J = 7.8 Hz, 2H), 7.12 (d, J = 8.1 Hz, 2H), 3.81 (t, J = 7.1 Hz, 2H), 3.45 (t, J = 6.5 Hz, 2H), 2.41 (s, 3H), 1.62-1.56 (m, 4H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.1, 145.5, 139.5, 137.3, 136.7, 133.4, 131.6, 129.6, 129.1, 128.1, 127.6, 125.2, 79.8, 61.5, 46.9, 30.7, 29.8, 21.5; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{20}H_{21}INO_2]$ 434.0617, found 434.0624.

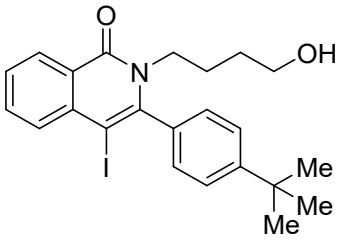


3-(4-ethylphenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2*H*)-one (3c). The product was obtained as a dark brown solid (89.4 mg, 80%): mp 140–142 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.36 (d, J = 9.1 Hz, 1H), 7.88 (d, J = 8.1 Hz, 1H), 7.70-7.69 (m, 1H), 7.49 (t, J = 8.1 Hz, 1H), 7.29 (d, J = 8.0 Hz, 2H), 7.12 (d, J = 8.1 Hz, 2H), 4.07 (t, J = 5.2 Hz, 2H), 3.64 (t, J = 5.3 Hz, 2H), 2.70 (q, J = 7.6 Hz, 2H), 1.62-1.56 (m, 4H), 1.25 (t, J = 7.6 Hz, 3H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.1, 145.7, 137.3, 136.9, 133.4, 131.6,

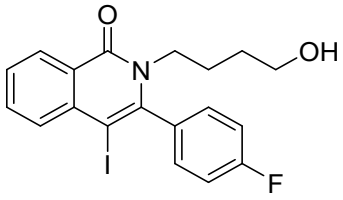
129.2, 128.4, 128.2, 127.6, 125.2, 79.8, 63.0, 46.9, 30.7, 29.7, 28.7, 15.4; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{21}H_{23}INO_2]$ 448.0773, found 448.0783.



2-(4-hydroxybutyl)-4-iodo-3-(4-methoxyphenyl)isoquinolin-1(2*H*)-one (3d). The product was obtained as a dark brown solid (87.5 mg, 78%): mp 152–156 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) 8.36 (d, J = 8.0 Hz, 1H), 7.86 (d, J = 8.1 Hz, 1H), 7.68-7.64 (m, 1H), 7.49-7.45 (m, 1H), 7.16 (d, J = 11.4 Hz, 2H), 6.99 (d, J = 8.8 Hz, 2H), 3.85-3.81 (m, 6H), 3.47 (t, J = 6.4 Hz, 2H), 1.63-1.55 (m, 4H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 160.1, 145.2, 137.3, 133.4, 132.0, 131.7, 130.6, 128.2, 127.6, 125.2, 114.2, 80.5, 61.7, 55.4, 46.9, 30.7, 29.7; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{20}H_{21}INO_3]$ 450.0566, found 450.0557.

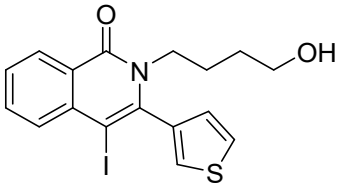


3-(4-(tert-butyl)phenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2*H*)-one (3e).The product was obtained as a brown solid (100.7 mg, 90%): mp 160–162 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.35 (d, J = 7.7 Hz, 1H), 7.85 (d, J = 8.2 Hz, 1H), 7.64 (t, J = 7.6 Hz, 1H), 7.48-7.44 (m, 3H), 7.15 (d, J = 8.2 Hz, 2H), 3.81 (t, J = 7.8 Hz, 2H), 3.45 (t, J = 6.3 Hz, 2H), 2.63 (s, 1H), 1.61-1.54 (m, 2H), 1.33 (s, 9H), 1.30-1.24 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.2, 152.7, 145.6, 137.3, 136.7, 133.4, 131.6, 128.8, 128.1, 127.6, 125.6, 80.0, 61.7, 47.6, 34.9, 31.3, 29.2, 25.0; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{23}H_{27}INO_2]$ 476.1086, found 476.1084.

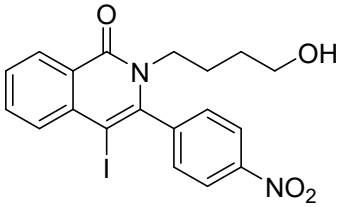


3-(4-fluorophenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2*H*)-one (3f). The product was obtained as a dark brown solid (95.0 mg, 87%): mp 142–144 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.36 (dd, J = 8.1, 1.0 Hz, 1H), 7.84 (d, J = 7.8 Hz, 1H), 7.68-7.64 (m, 1H), 7.50-7.46 (m, 1H), 7.26-7.22 (m, 2H), 7.21-7.16 (m, 2H + residual $CDCl_3$),

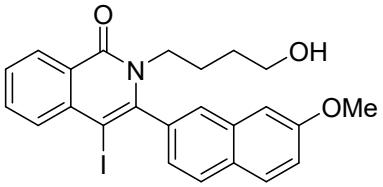
3.80 (t, J = 7.0 Hz, 2H), 3.49 (t, J = 6.3 Hz, 2H), 1.61-1.56 (m, 4H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 163.1 (d, J_{C-F} = 250.6 Hz), 162.0, 161.8, 144.3, 137.2, 135.6 (d, J_{C-F} = 3.63 Hz), 133.6, 131.7, 131.5, 131.4, 128.1 (d, J_{C-F} = 31.2 Hz), 125.3, 116.3 (d, J_{C-F} = 25.4 Hz), 80.2, 61.8, 47.0, 30.6, 29.8; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{19}H_{18}FINO_2]$ 438.0366, found 438.0382.



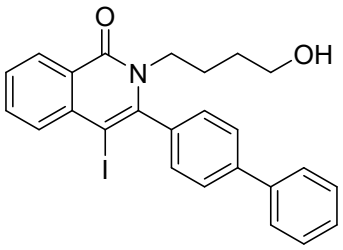
2-(4-hydroxybutyl)-4-iodo-3-(thiophen-3-yl)isoquinolin-1(2*H*)-one (3g). The product was obtained as a dark brown solid (87.1 mg, 82%): mp 120-122 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.35 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 8.1 Hz, 1H), 7.68-7.64 (m, 1H), 7.50-7.46 (m, 2H), 7.33-7.32 (m, 1H), 7.02 (dd, J = 4.9, 1.2 Hz, 1H), 3.99-3.91 (m, 1H), 3.77-3.70 (m, 1H), 3.17 (t, J = 6.5 Hz, 1H), 1.66-1.57 (m, 4H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.0, 140.9, 139.3, 133.5, 131.7, 128.2, 128.0, 127.9, 126.9, 126.5, 125.3, 80.6, 61.7, 46.9, 30.7, 29.7; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{17}H_{17}INO_2S]$ 426.0025, found 426.0013.



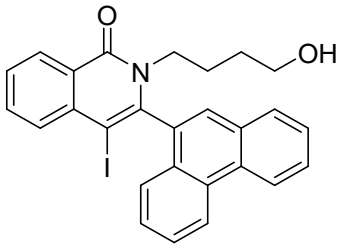
2-(4-hydroxybutyl)-4-iodo-3-(4-nitrophenyl)isoquinolin-1(2*H*)-one (3h). The product was obtained as a dark brown solid (102.0 mg, 88%): mp 160–164 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.21 (t, J = 8.5 Hz, 2H), 7.78 (dd, J = 7.4, 4.4 Hz, 1H), 7.58 (d, J = 8.3 Hz, 2H), 7.49-7.45 (m, 1H), 7.31-7.27 (m, 1H), 7.23 (d, J = 7.8 Hz, 1H), 3.88 (t, J = 7.3 Hz, 2H), 3.67 (t, J = 6.1 Hz, 2H), 1.84-1.76 (m, 2H), 1.65-1.58 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.2, 149.9, 146.3, 140.2, 136.2, 133.5, 129.8, 128.6, 127.1, 124.9, 124.3, 77.8, 61.8, 48.4, 29.4, 25.2; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{19}H_{18}IN_2O_4]$ 465.0311, found 465.0326.



2-(4-hydroxybutyl)-4-iodo-3-(7-methoxynaphthalen-2-yl)isoquinolin-1(2*H*)-one (3i). The product was obtained as a dark brown solid (118.5 mg, 95%): mp 200-202 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.12 (s, 1H), 7.75 (s, 2H), 7.68 (d, J = 8.8 Hz, 1H), 7.38 (d, J = 8.4 Hz, 1H), 7.17-7.11 (m, 2H), 7.02-6.99 (m, 1H), 6.88-6.84 (m, 1H), 6.72 (d, J = 8.7 Hz, 1H), 5.49 (s, 1H), 4.74 (t, J = 8.0 Hz, 2H), 3.89 (s, 3H), 3.71 (t, J = 6.2 Hz, 2H), 2.04-1.97 (m, 2H), 1.72-1.65 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 184.3, 159.3, 136.6, 135.7, 130.7, 130.6, 130.4, 129.6, 127.9, 127.0, 126.5, 126.0, 125.9, 123.0, 121.6, 120.7, 119.6, 105.7, 82.5, 61.6, 55.4, 51.2, 29.3, 27.4; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{24}H_{23}INO_3]$ 500.0723, found 500.0710.

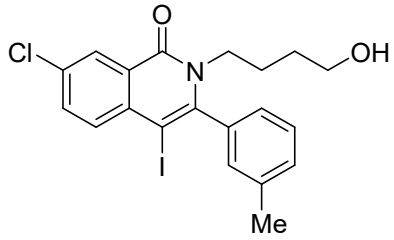


3-([1,1'-biphenyl]-4-yl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3j). The product was obtained as a dark brown solid (111.3 mg, 90%): mp 190-192 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.43 (dd, J = 8.0, 1.2 Hz, 1H), 7.92 (d, J = 8.1 Hz, 1H), 7.77 (d, J = 8.1 Hz, 2H), 7.74-7.69 (m, 3H), 7.55-7.53 (m, 1H), 7.51-7.47 (m, 2H), 7.41-7.35 (m, 3H), 3.92 (t, J = 7.8 Hz, 2H), 3.54 (t, J = 6.3 Hz, 2H), 2.97 (s, 1H), 1.71-1.64 (m, 2H), 1.43-1.36 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.1, 145.1, 142.0, 139.7, 138.4, 137.2, 133.5, 131.6, 129.7, 128.9, 128.1, 127.9, 127.7, 127.3, 127.1, 125.1, 79.8, 61.7, 47.7, 29.2, 25.2; HRMS (ESI) $[M+Na]^+$ Calcd for $[C_{25}H_{22}INaNO_2]$ 518.0593, found 518.0599.

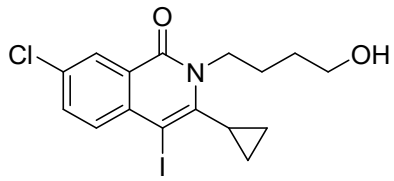


2-(4-hydroxybutyl)-4-iodo-3-(phenanthren-9-yl)isoquinolin-1(2H)-one (3k). The product was obtained as a dark brown solid (115.4 mg, 89%): mp 200-204°C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.75-8.69 (m, 2H), 8.45 (dt, J = 8.0, 1.6 Hz, 1H), 7.98-

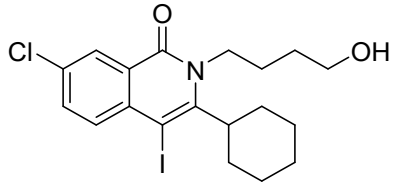
7.89 (m, 2H), 7.78-7.62 (m, 5H), 7.52-7.36 (m, 3H), 4.97 (s, 1H), 3.79 (t, J = 6.1 Hz, 2H), 3.38-3.25 (m, 2H), 2.15-2.07 (m, 2H), 1.81-1.74 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.3, 143.3, 142.2, 139.9, 138.6, 137.4, 133.6, 131.8, 130.6, 129.9, 129.1, 128.3, 128.1, 127.9, 127.5, 127.3, 125.3, 80.0, 61.9, 47.9, 29.4, 25.4; HRMS (ESI) $[M+Na]^+$ Calcd for $[C_{27}H_{23}INNaO_2]$ 542.0593, found 542.0587.



7-chloro-2-(4-hydroxybutyl)-4-iodo-3-(*m*-tolyl)isoquinolin-1(2*H*)-one (5a). The product was obtained as a dark brown solid (103.9 mg, 89%): mp 150–152 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.30 (d, J = 2.3 Hz, 1H), 7.77 (d, J = 8.8 Hz, 1H), 7.54 (dd, J = 8.8, 2.3 Hz, 1H), 7.35 (t, J = 7.6 Hz, 1H), 7.25 (d, J = 7.5 Hz, 1H), 7.01 (d, J = 7.8 Hz, 2H), 3.85-3.70 (m, 2H), 3.44 (t, J = 6.4 Hz, 2H), 2.63 (s, 1H), 2.36 (s, 3H), 1.60-1.52 (m, 2H), 1.33-1.26 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.1, 146.0, 139.3, 138.8, 135.9, 133.8, 133.7, 133.4, 130.3, 129.7, 128.8, 127.3, 126.2, 125.9, 78.3, 61.8, 48.0, 29.3, 25.2, 21.6; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{20}H_{20}ClINO_2]$ 468.0227, found 468.0252.

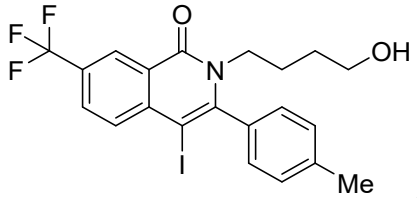


7-chloro-3-cyclopropyl-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2*H*)-one(5b). The product was obtained as a creamish white sticky solid (83.4 mg, 80%): ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.21 (d, J = 2.5 Hz, 1H), 7.78 (d, J = 8.8 Hz, 1H), 7.48 (dd, J = 8.8, 2.3 Hz, 1H), 4.46 (t, J = 7.6 Hz, 2H), 3.64 (t, J = 6.3 Hz, 2H), 2.64 (s, 1H), 1.86-1.79 (m, 1H), 1.78-1.70 (m, 2H), 1.60-1.53 (m, 2H), 1.37-1.32 (m, 2H), 0.89-0.85 (m, 2H) ; ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.7, 144.5, 136.1, 133.5, 133.4, 132.9, 127.1, 125.6, 81.4, 62.0, 45.1, 29.8, 25.5, 19.2, 14.4; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{16}H_{18}ClINO_2]$ 418.0071, found 418.0085.

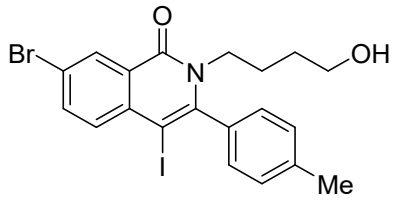


7-chloro-3-cyclohexyl-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2*H*)-one (5c). The product was obtained as a creamish white sticky solid

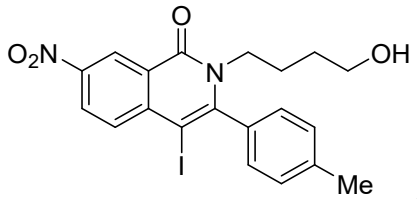
(86.0 mg, 75%): ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.23 (d, J = 2.5 Hz, 1H), 7.81 (d, J = 8.8 Hz, 1H), 7.50 (dd, J = 8.8, 2.3 Hz, 1H), 4.49 (t, J = 7.6 Hz, 2H), 3.67 (t, J = 6.3 Hz, 2H), 2.63 (s, 1H), 2.16-2.07 (m,1H), 1.89-1.18 (m, 14H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.8, 144.6, 136.3, 133.6, 133.5, 133.1, 127.2, 125.7, 81.5, 62.1, 51.5, 46.1, 32.8, 29.9, 26.1, 25.9.; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{19}H_{24}ClINO_2]$ 460.0540, found 460.0551.



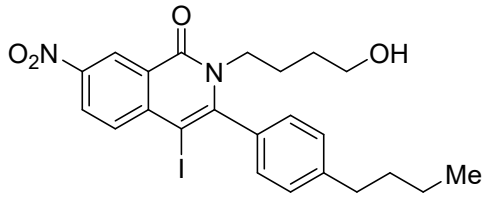
2-(4-hydroxybutyl)-4-iodo-3-(p-tolyl)-7-(trifluoromethyl)isoquinolin-1(2*H*)-one (5d). The product was obtained as a white solid (112.7 mg, 90%): mp 172–174 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.07 (d, J = 8.8 Hz, 1H), 7.91 (dd, J = 8.6, 1.9 Hz, 1H), 7.40 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 8.0 Hz, 2H), 3.95 (t, J = 5.6 Hz, 2H), 3.63 (t, J = 5.6 Hz, 2H), 2.51 (s, 3H), 2.37 (s, 1H), 1.73-1.62 (m, 4H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.5, 147.9, 139.9, 139.8, 136.3, 132.7, 129.5 (q, J_{C-F} = 13.1 Hz), 129.3, 128.3 (q, J_{C-F} = 260.1 Hz), 125.9 (q, J_{C-F} = 3.6 Hz), 125.0, 78.3, 61.6, 47.2, 30.6, 29.7, 21.6; HRMS (ESI) $[M+Na]^+$ Calcd for $[C_{21}H_{19}F_3INaNO_2]$ 524.0310, found 524.0316.



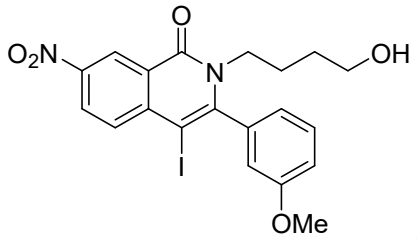
7-bromo-2-(4-hydroxybutyl)-4-iodo-3-(p-tolyl)isoquinolin-1(2*H*)-one (5e). The product was obtained as a dark brown solid (117.3 mg, 92%): mp 172–174 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.45 (d, J = 1.5 Hz, 1H), 7.68-7.67 (m, 2H), 7.26 (d, J = 7.8 Hz, 2H), 7.08 (d, J = 8.0 Hz, 2H), 3.78 (t, J = 7.9 Hz, 2H), 3.44 (t, J = 6.4 Hz, 2H), 2.62 (s, 1H), 2.38 (s, 3H), 1.58-1.50 (m, 2H), 1.32-1.25 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.0, 146.1, 139.7, 136.5, 136.4, 136.2, 133.5, 130.5, 129.6, 129.0, 126.2, 121.6, 78.8, 61.7, 48.0, 29.4, 25.2, 21.6; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{20}H_{20}BrINO_2]$ 511.9722, found 513.9721.



2-(4-hydroxybutyl)-4-iodo-7-nitro-3-(*p*-tolyl)isoquinolin-1(2*H*)-one (5f). The product was obtained as a dark brown solid (113.5 mg, 95%): mp 150–152 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.12 (d, J = 2.5 Hz, 1H), 8.33 (dd, J = 8.9, 2.5 Hz, 1H), 7.96 (d, J = 8.9 Hz, 1H), 7.31 (d, J = 7.8 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 3.82 (t, J = 8.0 Hz, 2H), 3.47 (t, J = 6.3 Hz, 2H), 2.67 (s, 1H), 2.41 (s, 3H), 1.62-1.54 (m, 2H), 1.35-1.28 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.1, 149.8, 146.2, 141.7, 140.0, 136.0, 133.3, 129.7, 128.4, 126.9, 124.8, 124.2, 77.6, 61.7, 48.2, 29.2, 25.0, 21.5; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{20}H_{20}IN_2O_4]$ 479.0468, found 479.0487.

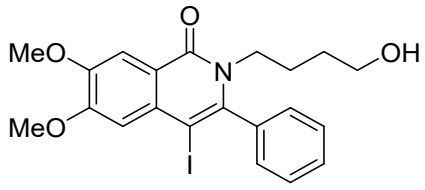


2-(4-hydroxybutyl)-4-iodo-7-nitro-3-(4-pentylphenyl)isoquinolin-1(2*H*)-one (5g). The product was obtained as a dark brown solid (120.1 mg, 90%): mp 130-132 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.19 (d, J = 2.5 Hz, 1H), 8.41 (dd, J = 9.0, 2.5 Hz, 1H), 8.05 (d, J = 9.0 Hz, 1H), 7.38 (d, J = 8.0 Hz, 2H), 7.21 (d, J = 8.3 Hz, 2H), 3.90 (t, J = 7.9 Hz, 2H), 3.51 (t, J = 6.4 Hz, 2H), 2.74 (t, J = 7.8 Hz, 2H), 1.72-1.62 (m, 6H), 1.45-1.36 (m, 4H), 0.97 (t, J = 7.4 Hz, 3H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.2, 150.0, 146.3, 145.1, 141.8, 136.3, 133.4, 129.0, 128.5, 127.0, 124.9, 124.3, 77.7, 61.7, 48.4, 35.5, 33.3, 29.4, 25.1, 22.4, 14.0; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{23}H_{25}IN_2O_4]$ 521.0937, found 521.0972.

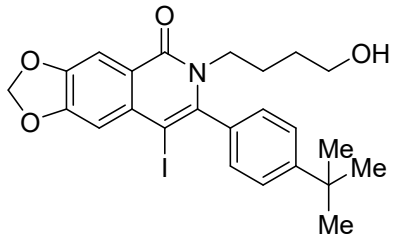


2-(4-hydroxybutyl)-4-iodo-3-(3-methoxyphenyl)-7-nitroisoquinolin-1(2*H*)-one (5h). The product was obtained as a dark brown solid (116.1 mg, 94%): mp 142–144 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.14 (d, J = 2.5 Hz, 1H), 8.35 (dd, J = 9.0, 2.5 Hz, 1H), 7.98 (d, J = 9.0 Hz, 1H), 7.42 (t, J = 7.9 Hz, 1H), 7.02-6.99 (m, 1H), 6.83-6.81 (m, 1H), 6.77 (t, J = 2.0 Hz, 1H), 3.86-3.82 (m, 5H), 3.45 (t, J = 6.4 Hz, 2H), 1.67-1.58 (m, 2H), 1.36-1.29 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.0, 159.8, 149.3, 146.2,

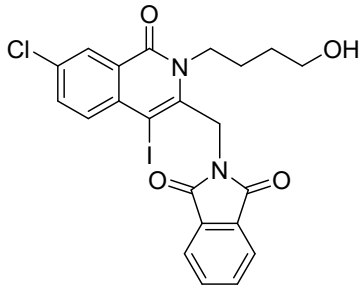
141.7, 139.9, 133.3, 130.3, 126.9, 124.8, 124.1, 120.8, 115.3, 114.3, 79.9, 61.5, 55.4, 48.3, 29.2, 25.2; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{20}H_{20}IN_2O_5]$ 495.0417, found 495.0425.



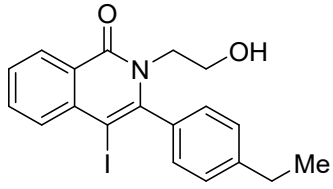
2-(4-hydroxybutyl)-4-iodo-6,7-dimethoxy-3-phenylisoquinolin-1(2*H*)-one (5i). The product was obtained as a dark brown solid (83.8 mg, 70%): mp 192–194 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.79 (s, 1H), 7.51-7.43 (m, 3H), 7.27-7.24 (m, 3H), 3.96 (s, 6H), 3.82-3.79 (m, 2H), 2.96-2.93 (m, 2H), 1.61-1.55 (m, 4H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.0, 159.8, 149.3, 141.7, 139.9, 133.3, 130.3, 126.9, 124.8, 124.1, 120.8, 115.3, 114.3, 107.2, 101.3, 80.5, 61.5, 55.4, 48.3, 29.2, 25.2.; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{21}H_{23}INO_4]$ 480.0672, found 480.0706.



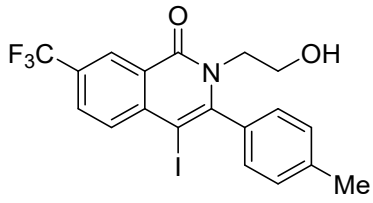
7-(4-(*tert*-butyl)phenyl)-6-(4-hydroxybutyl)-8-iodo-[1,3]dioxolo[4,5-*g*]isoquinolin-5(6*H*)-one (5j). The product was obtained as a dark brown solid (64.9 mg, 50%): mp 200–204 °C: ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.58 (d, J = 8.2 Hz, 2H), 7.45-7.39 (m, 2H), 7.31 (dd, J = 8.2, 2.7 Hz, 2H), 6.29 (s, 2H), 4.12 (t, J = 6.0 Hz, 2H), 3.63 (s, 1H), 2.94 (t, J = 6.2 Hz, 2H), 1.49 (t, J = 25.1 Hz, 4H), 1.35 (s, 9H); ^{13}C{1H} NMR (100 MHz, DMSO-d_6) δ 158.4, 151.6, 147.0, 143.6, 136.6, 131.5, 129.2, 125.4, 124.8, 114.0, 109.7, 102.9, 79.2, 61.3, 45.5, 31.1, 30.4, 29.0; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{24}H_{27}INO_4]$ 520.0985, found 520.0988.



2-((7-chloro-2-(4-hydroxybutyl)-4-iodo-1-oxo-1,2-dihydroisoquinolin-3-yl)methyl)isoindoline-1,3-dione (5k). The product was obtained as a white sticky solid (26.8 mg, 20%): ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 1.8 Hz, 1H), 7.90 (d, J = 8.5 Hz, 1H), 7.76-7.73 (m, 1H), 7.60-7.55 (m, 1H), 7.48 (dd, J = 8.4, 1.9 Hz, 2H), 7.40 (dt, J = 7.7, 1.4 Hz, 1H), 3.72 (t, J = 7.8 Hz, 2H), 3.31 (t, J = 7.0 Hz, 2H), 2.76 (s, 2H), 1.84 (t, J = 5.9 Hz, 2H), 1.72 (t, J = 5.3 Hz, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) : The amount of the product 3v was less, so ^{13}C NMR could not be recorded; HRMS (ESI) $[M+Na]^+$ Calcd for $[C_{22}H_{18}ClINaN_2O_4]$ 558.9897, found 558.9885.

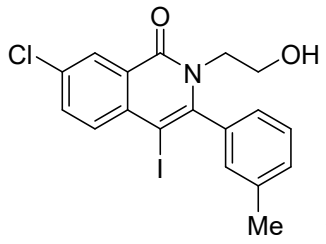


3-(4-ethylphenyl)-2-(2-hydroxyethyl)-4-iodoisoquinolin-1(2*H*)-one (6a). The product was obtained as a white sticky solid (83.8 mg, 80%): mp 152–156 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.36 (d, J = 9.1 Hz, 1H), 7.88 (d, J = 8.1 Hz, 1H), 7.70-7.69 (m, 1H), 7.49 (t, J = 8.1 Hz, 1H), 7.29 (d, J = 8.0 Hz, 2H), 7.12 (d, J = 8.1 Hz, 2H), 4.07 (t, J = 5.2 Hz, 2H), 3.64 (t, J = 5.3 Hz, 2H), 2.70 (q, J = 7.6 Hz, 2H), 1.25 (t, J = 7.6 Hz, 3H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 164.4, 145.9, 145.5, 137.6, 136.8, 133.9, 131.8, 129.2, 128.5, 128.2, 127.9, 124.9, 80.8, 63.0, 51.1, 28.7, 15.2; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{19}H_{19}INO_2]$ 420.0460, found 420.0461.

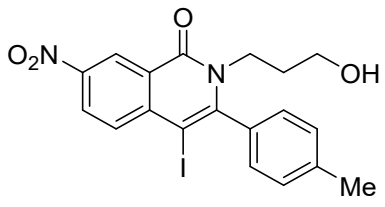


2-(2-hydroxyethyl)-4-iodo-3-(p-tolyl)-7-(trifluoromethyl)isoquinolin-1(2H)-one (6b). The product was obtained as a yellow solid (100.7 mg, 85%): mp 182–166 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 1H), 7.99 (d, J = 8.5 Hz, 1H), 7.83 (dd, J = 8.6, 1.9 Hz, 1H), 7.28 (d, J = 7.8 Hz, 2H), 7.10 (d, J = 8.1 Hz, 2H), 4.06

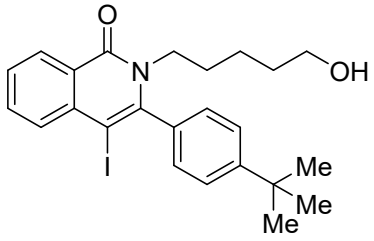
(t, J = 5.5 Hz, 2H), 3.63 (t, J = 5.5 Hz, 2H), 2.39 (s, 3H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.3, 146.9, 139.0, 135.2, 131.8, 131.3, 128.9, 128.7, 128.3 (q, J_{C-F} = 260.1 Hz), 127.9, 124.9, 123.7, 78.1, 61.0, 49.9, 20.5; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{19}H_{16}F_3INO_2]$ 474.0718, found 474.0725.



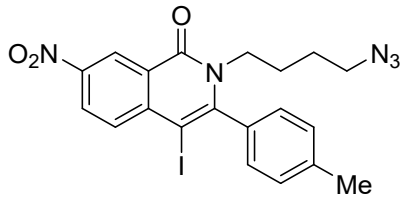
7-chloro-2-(2-hydroxyethyl)-4-iodo-3-(m-tolyl)isoquinolin-1(2*H*)-one (6c). The product was obtained as a dark brown solid (95.2 mg, 87%): mp 142–146 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.32 (d, J = 2.3 Hz, 1H), 7.79 (d, J = 8.8 Hz, 1H), 7.55 (dd, J = 8.8, 2.3 Hz, 1H), 7.36 (t, J = 7.6 Hz, 1H), 7.26 (d, J = 7.5 Hz, 1H), 7.02 (d, J = 7.8 Hz, 2H), 3.86-3.71 (m, 2H), 3.45 (t, J = 6.4 Hz, 2H), 2.65 (s, 1H), 2.37 (s, 3H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.1, 146.0, 139.3, 138.8, 135.9, 133.8, 133.7, 133.4, 130.3, 129.7, 128.8, 127.3, 126.2, 125.9, 78.3, 77.4, 77.1, 76.8, 61.8, 48.0, 21.6; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{18}H_{16}ClINO_2]$ 439.9914, found 439.9901.



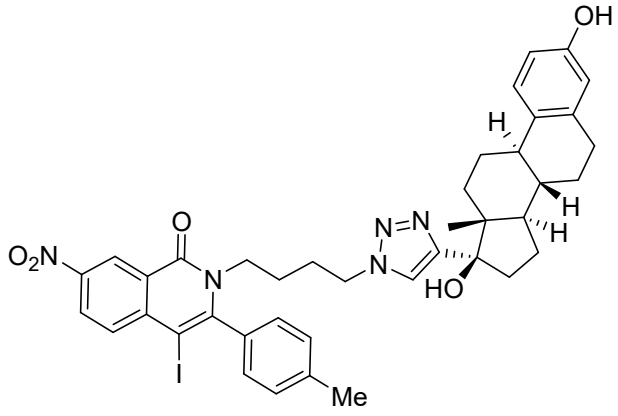
2-(3-hydroxypropyl)-4-iodo-7-nitro-3-(*p*-tolyl)isoquinolin-1(2*H*)-one (6d). The product was obtained as a dark brown solid (105.6 mg, 91%): mp 132–136 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.12 (d, J = 2.5 Hz, 1H), 8.33 (dd, J = 8.9, 2.5 Hz, 1H), 7.95 (d, J = 8.9 Hz, 1H), 7.30 (d, J = 7.8 Hz, 2H), 7.11 (d, J = 8.0 Hz, 2H), 4.06 (t, J = 6.3 Hz, 2H), 3.43 (t, J = 5.6 Hz, 2H), 2.53 (s, 3H), 1.62-1.56 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ162.0, 150.7, 147.1, 142.6, 141.0, 137.0, 134.3, 130.6, 129.3, 127.9, 125.7, 125.1, 77.6, 57.0, 43.6, 30.2, 21.4.; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{19}H_{17}IN_2O_4]$ 465.0311, found 465.0301.



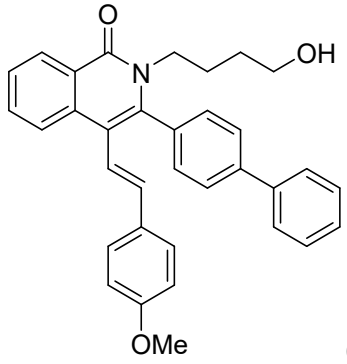
3-(4-(tert-butyl)phenyl)-2-(5-hydroxypentyl)-4-iodoisoquinolin-1(2*H*)-one (6e). The product was obtained as a dark brown solid (85.5 mg, 70%): mp 162–164 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.30 (d, J = 7.7 Hz, 1H), 7.80 (d, J = 8.2 Hz, 1H), 7.60 (t, J = 7.6 Hz, 1H), 7.43-7.39 (m, 3H), 7.10 (d, J = 8.2 Hz, 2H), 3.76 (t, J = 7.8 Hz, 2H), 3.40 (t, J = 6.3 Hz, 2H), 2.59 (s, 1H), 1.56-1.49 (m, 2H), 1.28-1.22 (m, 11H), 0.83-0.77 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 162.1, 152.6, 145.5, 137.2, 136.6, 133.3, 131.5, 128.7, 128.0, 127.5, 125.5, 79.9, 61.6, 47.5, 34.8, 31.2, 29.1, 25.0, 20.7; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{24}H_{29}INO_2]$ 490.1243, found 490.1224.



2-(4-azidobutyl)-4-iodo-7-nitro-3-(p-tolyl)isoquinolin-1(2*H*)-one (7f). The product was obtained as a dark brown solid (113.1 mg, 90%): mp 172–176 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.21 (d, J = 2.3 Hz, 1H), 8.41-8.37 (m, 1H), 8.01 (dd, J = 8.9, 3.7 Hz, 1H), 7.32 (d, J = 7.7 Hz, 2H), 7.12 (d, J = 7.8 Hz, 2H), 3.85-3.81 (m, 2H), 3.09-3.05 (m, 2H), 2.43 (s, 3H), 1.62-1.54 (m, 2H), 1.37-1.30 (m, 2H).; ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.1, 149.7, 146.4, 141.8, 140.2, 136.1, 133.4, 129.8, 128.5, 127.1, 125.0, 124.4, 77.6, 50.6, 48.0, 29.7, 26.0, 21.5; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{20}H_{19}IN_5O_3]$ 504.0533, found 504.0547.

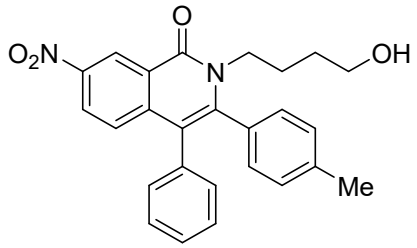


2-(4-(4-((8S,9R,13R,14R,17R)-3,17-dihydroxy-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[a]phenanthren-17-yl)-1H-1,2,3-triazol-1-yl)butyl)-4-iodo-7-nitro-3-(p-tolyl)isoquinolin-1(2*H*)-one (9). The product was obtained as a white solid (139.8 mg, 70%): mp 182-184 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.32 (d, J = 2.5 Hz, 1H), 8.49 (dd, J = 9.0, 2.5 Hz, 1H), 8.11 (d, J = 9.0 Hz, 1H), 7.42 (d, J = 7.8 Hz, 2H), 7.17 (d, J = 8.5 Hz, 3H), 6.68 (dd, J = 8.4, 2.6 Hz, 2H), 6.61 (d, J = 2.5 Hz, 1H), 3.93 (t, J = 7.9 Hz, 2H), 3.15 (t, J = 6.8 Hz, 2H), 2.84-2.82 (m, 2H), 2.63 (s, 3H), 2.46 (d, J = 8.0 Hz, 1H), 2.26-2.20 (m, 2H), 2.09-2.02 (m, 4H), 1.94-1.87 (m, 2H), 1.78-1.69 (m, 4H), 1.47-1.41 (m, 4H), 0.89 (s, 3H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.3, 153.7, 146.4, 140.2, 138.0, 135.9, 133.4, 132.0, 129.8, 128.4, 127.1, 126.4, 124.4, 115.3, 112.8, 87.4, 79.9, 74.0, 49.3, 47.1, 43.4, 39.3, 38.8, 32.7, 29.6, 29.6, 27.2, 26.3, 22.7, 12.7; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{40}H_{43}IN_5O_5]$ 822.2128, found 822.2194.



(*E*)-3-([1,1'-biphenyl]-4-yl)-2-(4-hydroxybutyl)-4-(4-methoxystyryl)isoquinolin-1(2*H*)-one (11j). The product was obtained as a dark brown solid (87.7 mg, 70%): mp 192–196 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.64-7.55 (m, 7H), 7.43-7.28 (m, 8H), 7.07 (d, J = 8.7 Hz, 1H), 6.78 (d, J = 8.8 Hz, 2H), 6.71 (d, J = 8.7 Hz, 1H), 6.40 (t, J = 4.7 Hz, 1H), 5.47-5.57 (1H), 3.95 (t, J = 7.6 Hz, 2H), 3.73 (s, 3H), 3.51 (t, J = 6.3 Hz, 2H), 1.65 (q, J = 7.5 Hz, 2H), 1.37 (dd, J = 20.0, 6.7 Hz, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$)

δ162.0, 150.7, 147.1, 142.6, 141.0, 137.0, 134.3, 130.6, 129.3, 127.9, 125.7, 125.1, 77.6, 57.0, 43.6, 30.2, 21.4.; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{34}H_{32}NO_3]$ 502.2382, found 502.2397.

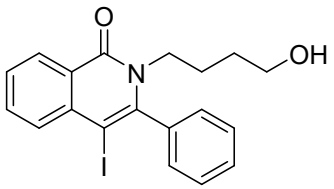


2-(4-hydroxybutyl)-7-nitro-4-phenyl-3-(*p*-tolyl)isoquinolin-1(2*H*)-one (13f). The product was obtained as a dark brown solid (90.7 mg, 85%): mp 210–212 °C: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.28 (d, J = 2.5 Hz, 1H), 8.19 (dd, J = 9.0, 2.5 Hz, 1H), 7.20-7.13 (m, 4H), 7.02-6.97 (m, 6H), 3.84 (t, J = 7.9 Hz, 2H), 3.50 (t, J = 6.3 Hz, 2H), 2.23 (s, 3H), 1.68-1.61 (m, 2H), 1.40-1.33 (m, 2H); ^{13}C{1H} NMR (100 MHz, $CDCl_3$) δ 161.5, 145.7, 145.5, 141.5, 138.7, 135.4, 131.2, 130.8, 129.4, 128.8, 128.5, 128.3, 127.3, 126.9, 125.8, 124.3, 118.8, 61.8, 46.1, 29.3, 25.0, 21.3; HRMS (ESI) $[M+H]^+$ Calcd for $[C_{26}H_{24}N_2O_4]$ 428.1736, found 428.1742.

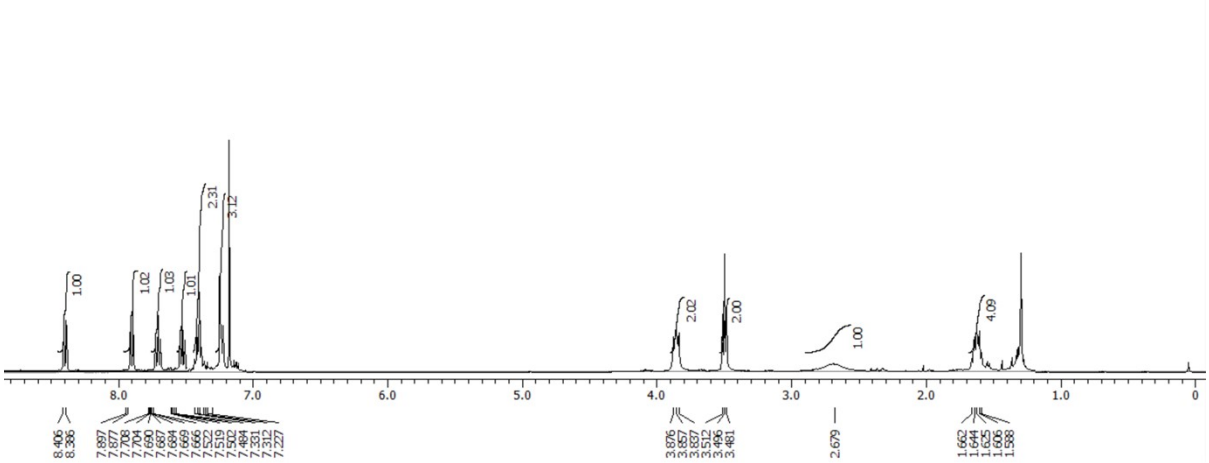
Copies of ^{1}H, ^{13}C and HRMS

Copies of ^{1}H and ^{13}C NMR

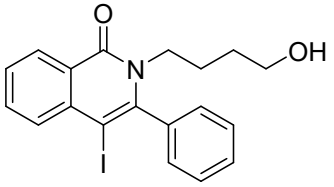
^{1}H NMR (400 MHz, $CDCl_3$)



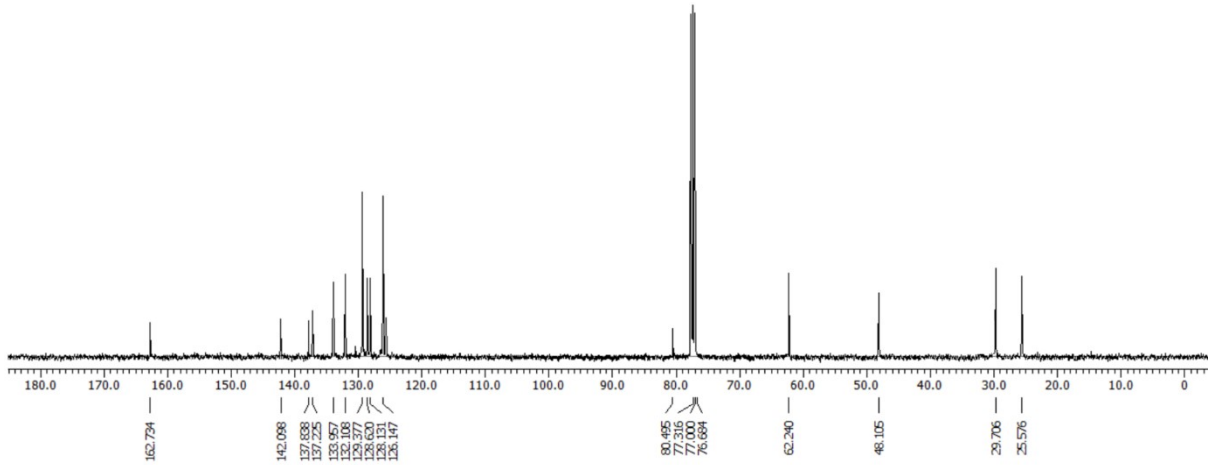
2-(4-hydroxybutyl)-4-iodo-3-phenylisoquinolin-1(2H)-one (3a)



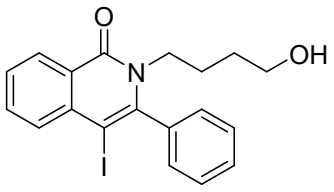
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



2-(4-hydroxybutyl)-4-iodo-3-phenylisoquinolin-1(2H)-one (3a)



HRMS



2-(4-hydroxybutyl)-4-iodo-3-phenylisoquinolin-1(2H)-one (3a)

Qualitative Compound Report

Data File	MSH-411.d	Sample Name	MSH-411
Sample Type	Sample	Position	P1-C9
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	18-01-2023 14:30:20
IRM Calibration Status	[redacted]	DA Method	Default.m
Comment			

Sample Group — Info. 3
Acquisition SW — 6200 series TOF/6500 series
Version — Q-TOF B.05.01 (B5125)

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
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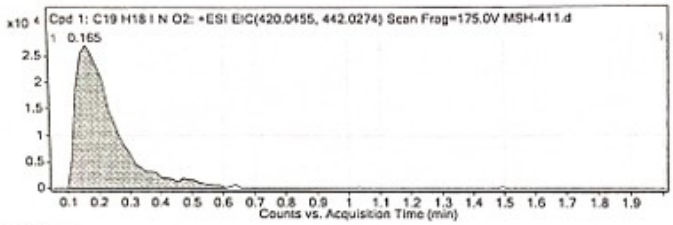
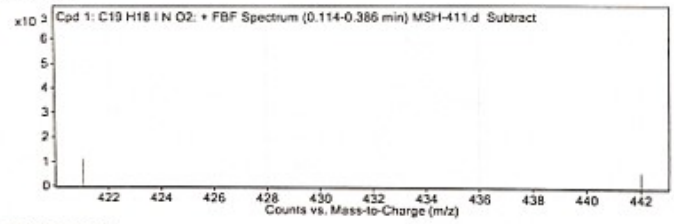
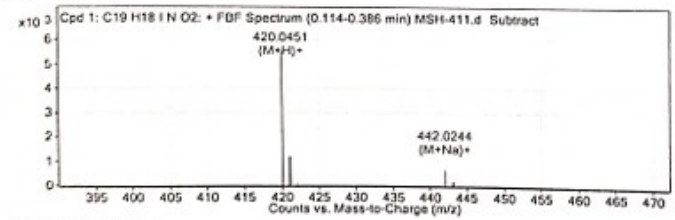
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C19 H18 I N O2	420.0451	0.165	Find By Formula	419.0378



MS Spectrum



MS Zoomed Spectrum

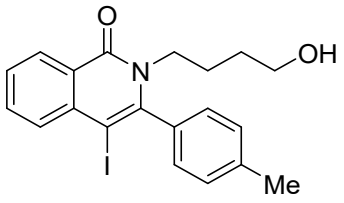


MS Spectrum Peak List

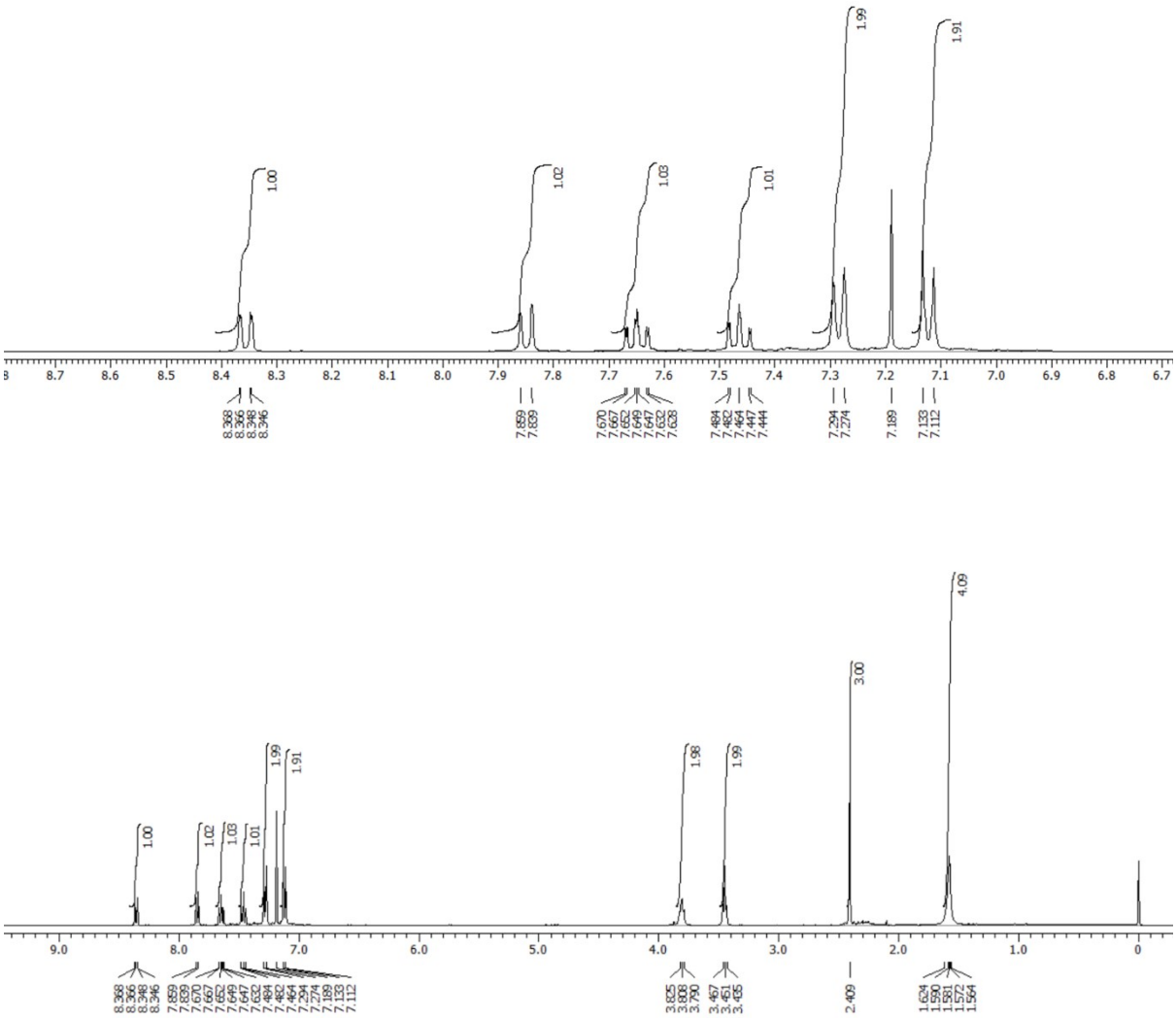
m/z	z	Abund	Formula	Ion
420.0451	1	5535.12	C19H19INO2	(M+H)+
421.0492	1	1135.04	C19H19INO2	(M+H)+
442.0244	1	622.2	C19H18INNaO2	(M+Na)+
443.0339	1	190.1	C19H18INNaO2	(M+Na)+

--- End Of Report ---

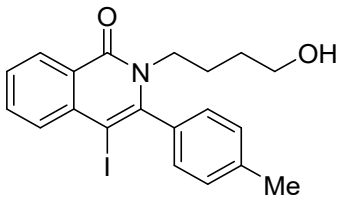
^{1}H NMR (400 MHz, $CDCl_3$)



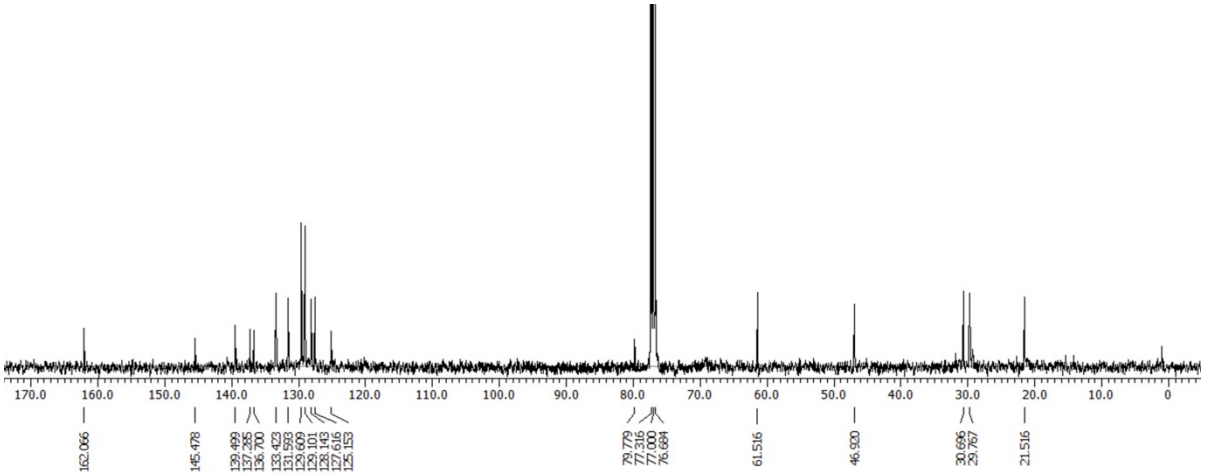
2-(4-hydroxybutyl)-4-iodo-3-(p-tolyl)isoquinolin-1(2H)-one (3b)



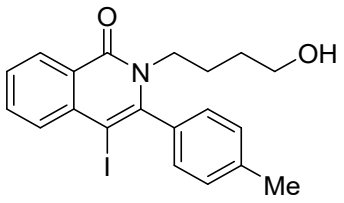
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



2-(4-hydroxybutyl)-4-iodo-3-(p-tolyl)isoquinolin-1(2H)-one (3b)



HRMS



2-(4-hydroxybutyl)-4-iodo-3-(p-tolyl)isoquinolin-1(2H)-one (3b)

Qualitative Compound Report

Data File	MSH-408.d	Sample Name	MSH-408
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IRM Calibration Status	[redacted]	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
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Compound Label	m/z	RT	Algorithm	Mass
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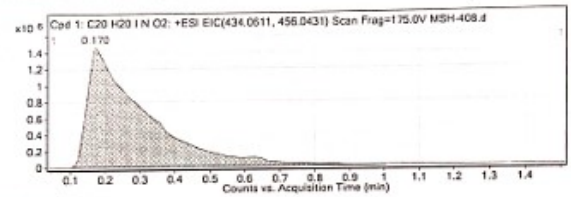
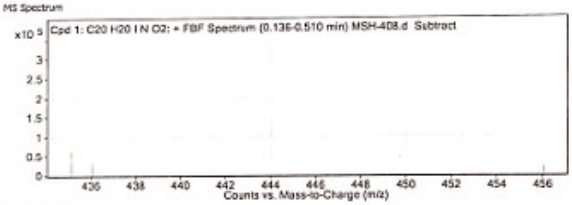
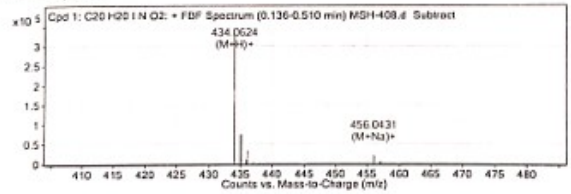


MS Spectrum



MS Zoomed Spectrum



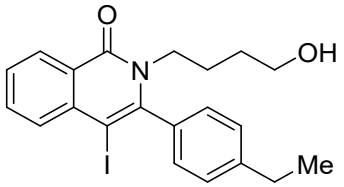
MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
434.0624	1	302859.38	C20H21INO2	(M+H)+
435.065	1	65551.63	C20H21INO2	(M+H)+
436.0585	1	34441.77	C20H21INO2	(M+H)+
437.0598	1	7383.41	C20H21INO2	(M+H)+
438.0633	1	958.57	C20H21INO2	(M+H)+
456.0431	1	19898.95	C20H20INNaO2	(M+Na)+
457.0463	1	4270	C20H20INNaO2	(M+Na)+

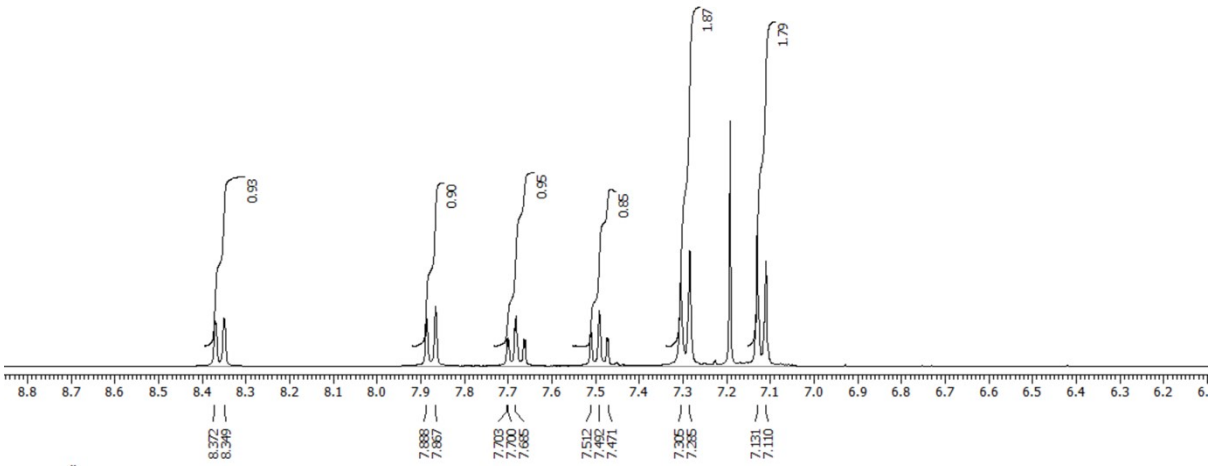
-- End Of Report --

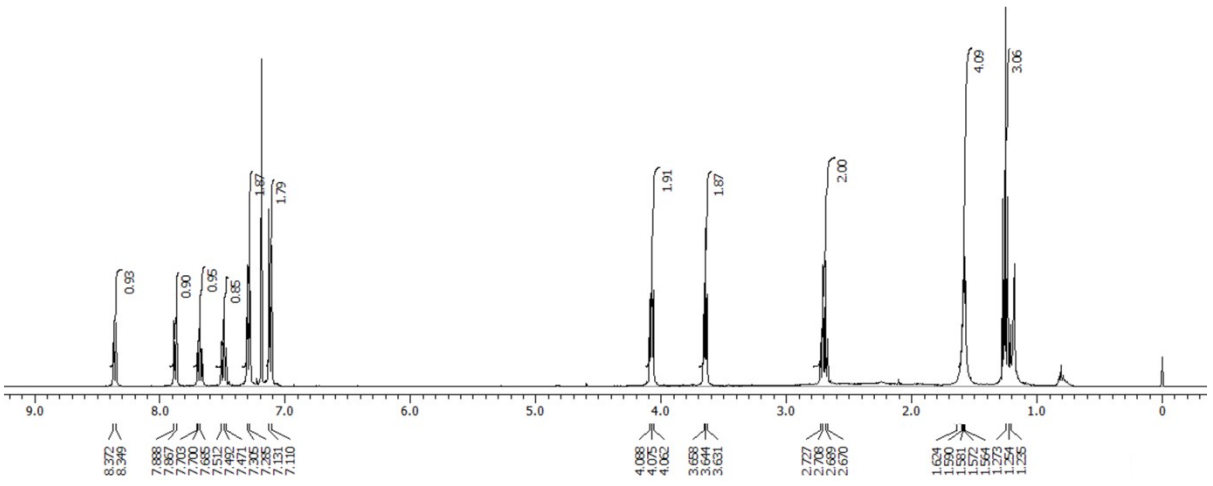
Agilent Technologies Page 1 of 1 Printed at: 16:56 on:19-01-2023

^{1}H NMR

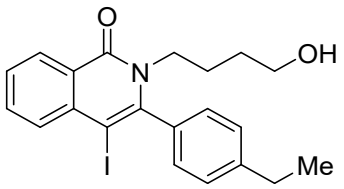


3-(4-ethylphenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3c)

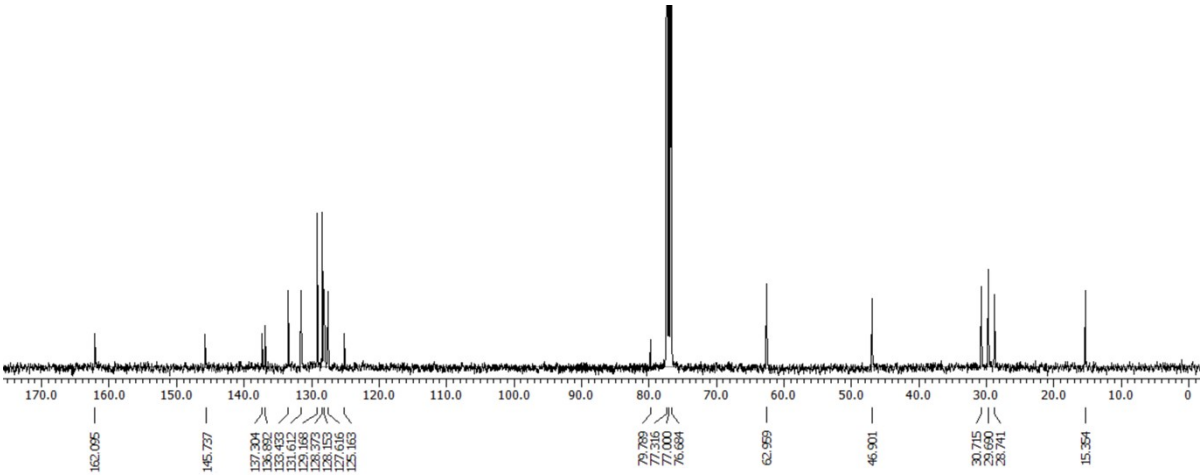




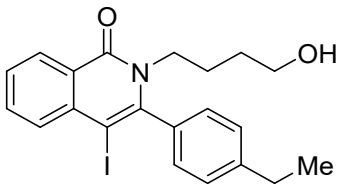
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



3-(4-ethylphenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3c)



HRMS



3-(4-ethylphenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3c)

Qualitative Compound Report

Data File	MSH-387B.d	Sample Name	MSH-387B
Sample Type	Sample	Position	P1-B5
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	17-05-2022 14:55:21
IRM Calibration Status	Success	DA Method	Default.m
Comment			

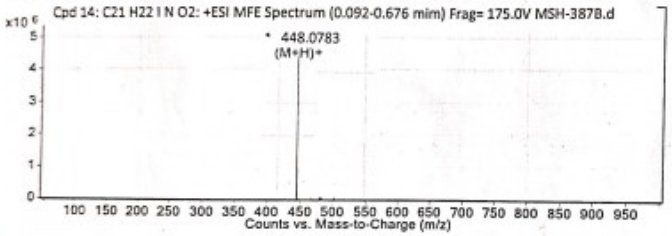
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

Compound Table

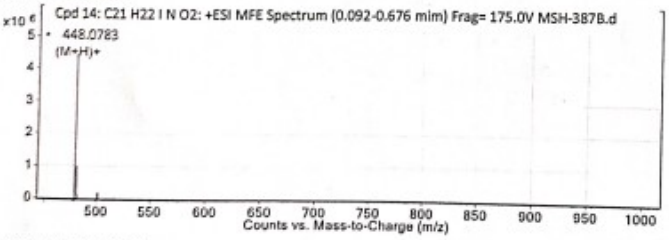
Compound Label	RT	Mass	Formula	MFG Formula	MFG Diff (ppm)	DB Formula
Cpd 14: C21 H22 I N O2	0.166	447.0695	C21 H22 I N O2	C21 H22 I N O2	-9.31	C21 H22 I N O2

Compound Label	m/z	RT	Algorithm	Mass
Cpd 14: C21 H22 I N O2	448.0783	0.166	Find by Molecular Feature	447.0695

MFE MS Spectrum



MFE MS Zoomed Spectrum



MS Spectrum Peak List

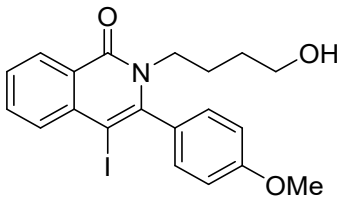
m/z	z	Abund	Formula	Ion
448.0783	1	4463637.5	C21 H23 I N O2	(M+H)+
449.0346	1	1017697.78	C21 H23 I N O2	(M+H)+
450.0367	1	130964.93	C21 H23 I N O2	(M+H)+
451.0392	1	11722.32	C21 H23 I N O2	(M+H)+
470.0593	1	171836.52	C21 H22 I N Na O2	(M+Na)+
502.0157	1	37555.65	C21 H22 I N Na O2	(M+Na)+

Agilent Technologies

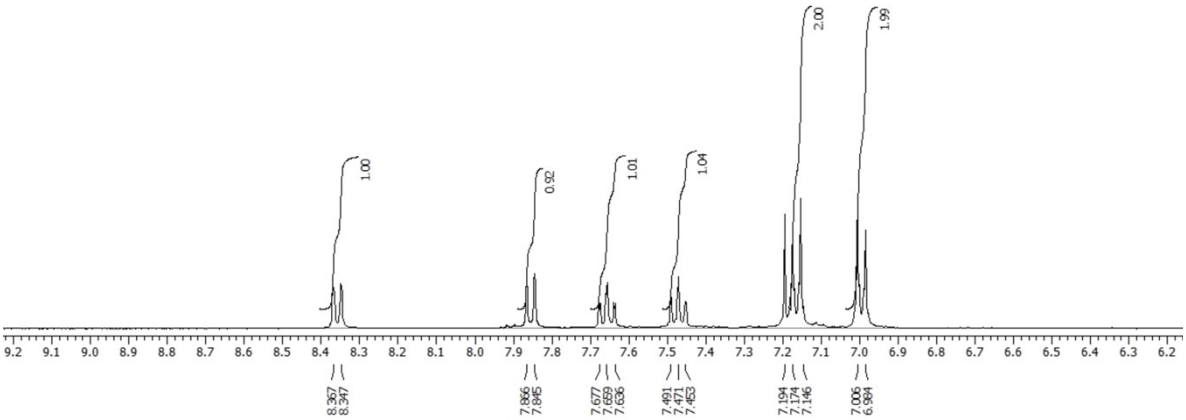
Page 1 of 2

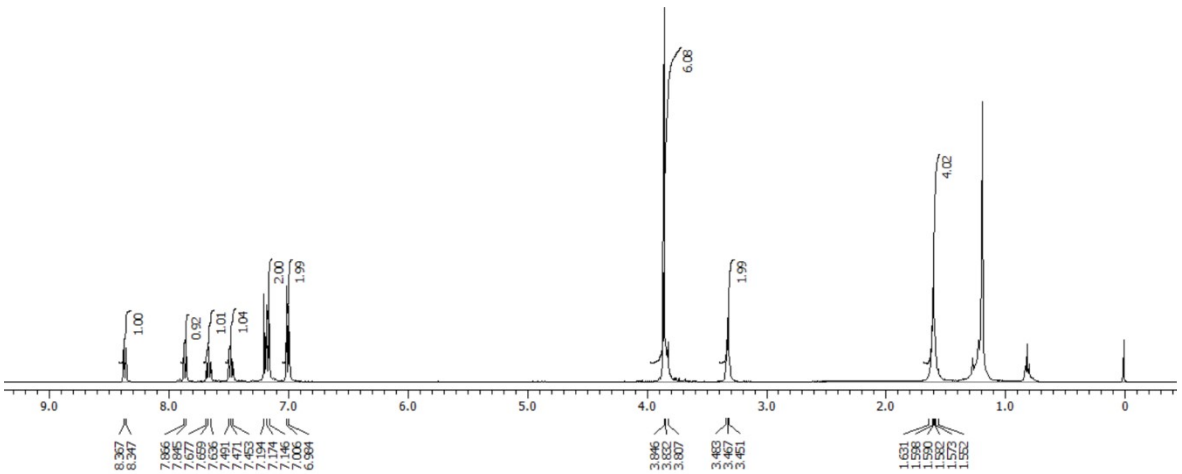
Printed at: 23:16 on:24-10-2023

^{1}H NMR (400 MHz, $CDCl_3$)

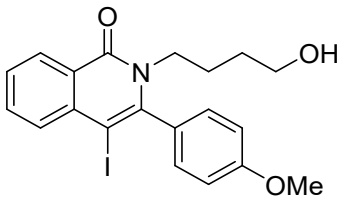


2-(4-hydroxybutyl)-4-iodo-3-(4-methoxyphenyl)isoquinolin-1(2H)-one (3d)

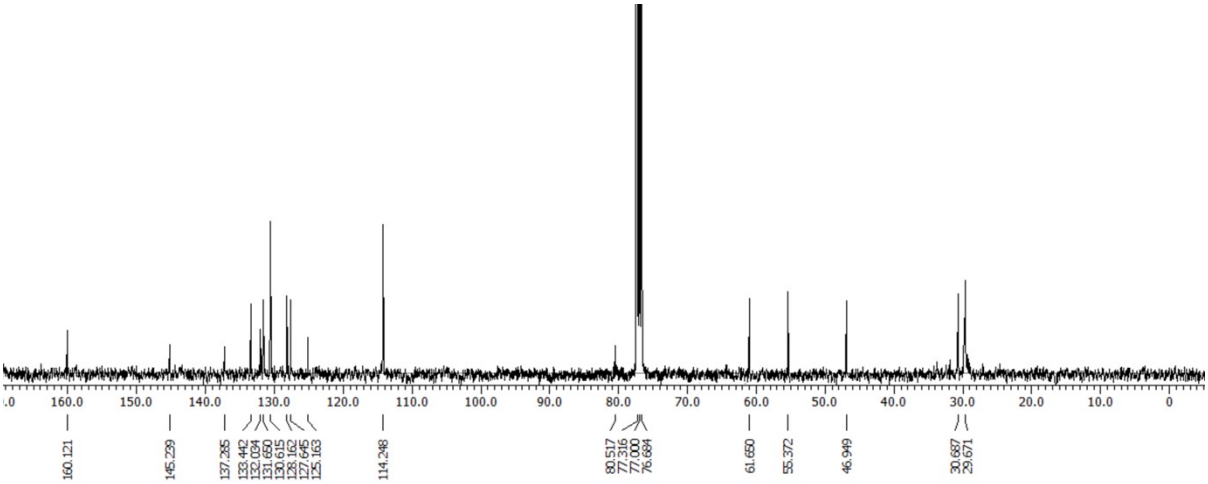




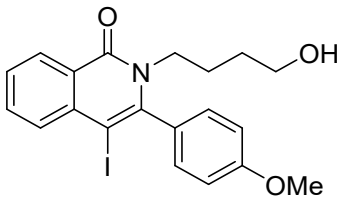
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



2-(4-hydroxybutyl)-4-iodo-3-(4-methoxyphenyl)isoquinolin-1(2H)-one (3d)



HRMS



2-(4-hydroxybutyl)-4-iodo-3-(4-methoxyphenyl)isoquinolin-1(2H)-one (3d)

Qualitative Compound Report

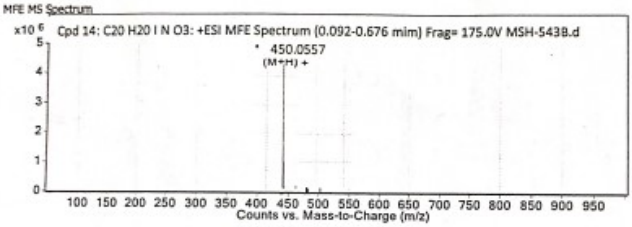
Data File	MSH-543B.d	Sample Name	MSH-543B
Sample Type	Sample	Position	P1-B5
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	18-05-2022 15:55:21
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group	Info.	3	
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

Compound Table

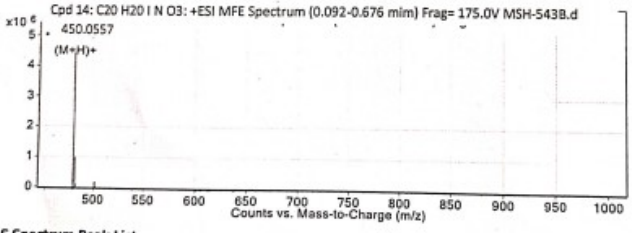
Compound Label	RT	Mass	Formula	MFG Formula	MFG Diff (ppm)	DB Formula
Cpd 14: C20 H20 I N O3	0.166	449.0488	C20 H20 I N O3	C20 H20 I N O3	-9.31	C20 H20 I N O3

Compound Label	m/z	RT	Algorithm	Mass
Cpd 14: C20 H20 I N O3	450.0557	0.166	Find by Molecular Feature	449.0488

MFE MS Spectrum



MFE MS Zoomed Spectrum

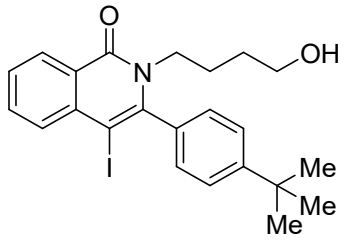


MS Spectrum Peak List

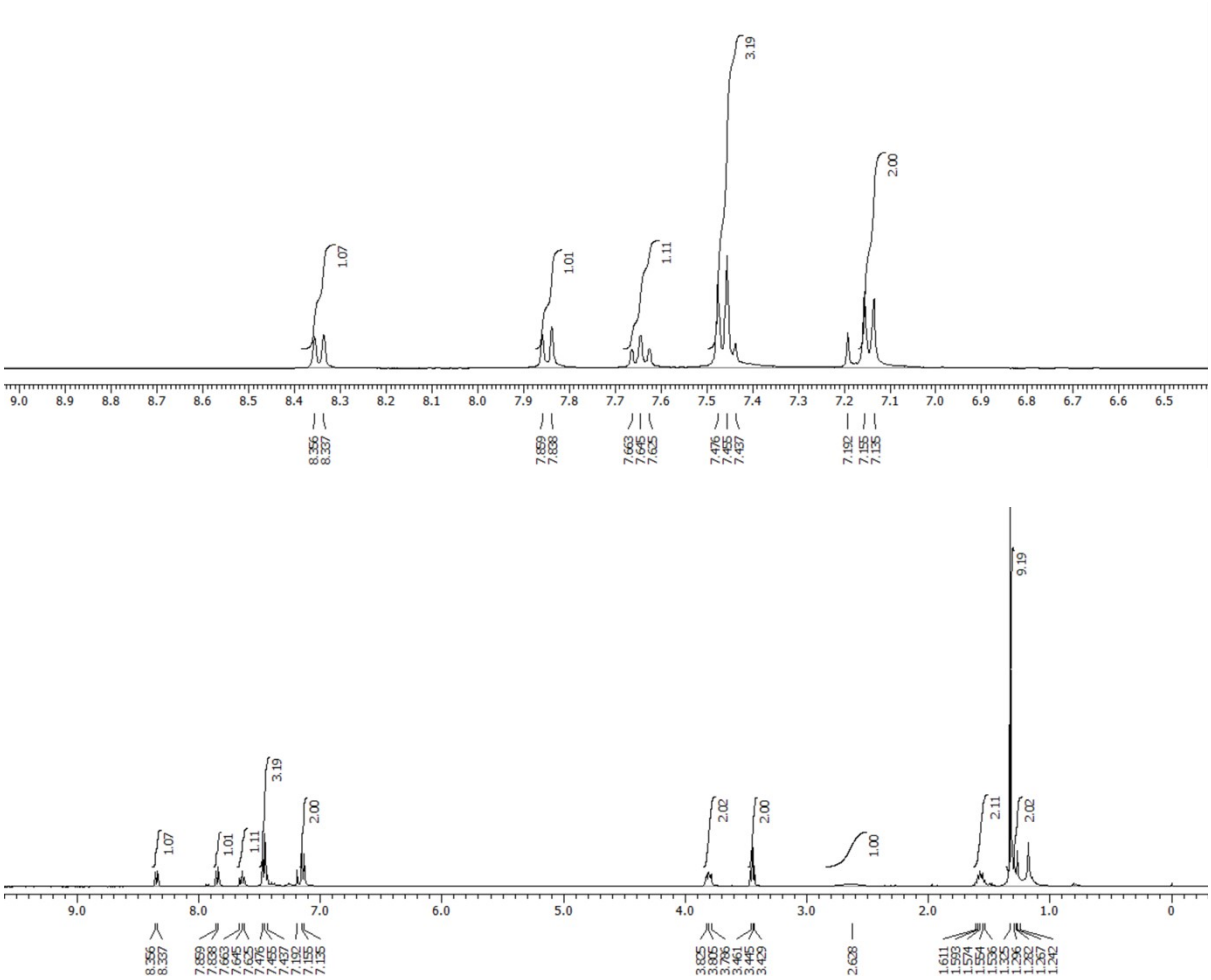
m/z	z	Abund	Formula	Ion
450.0557	1	5463637.5	C20 H20 I N O3	(M+H)+
451.0346	1	1017697.78	C20 H20 I N O3	(M+H)+
452.0367	1	130904.93	C20 H20 I N O3	(M+H)+
453.0392	1	11722.32	C20 H20 I N O3	(M+H)+
472.0386	1	171836.52	C20 H19 I N Na O2	(M+Na)+
473.0157	1	37555.65	C20 H19 I N Na O2	(M+Na)+

Page 1 of 2 Printed at: 23:16 on:24-10-2023

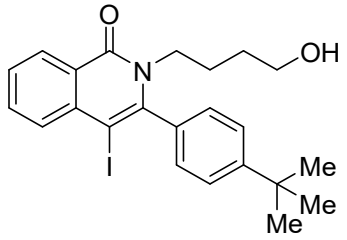
^{1}H NMR (400 MHz, $CDCl_3$)



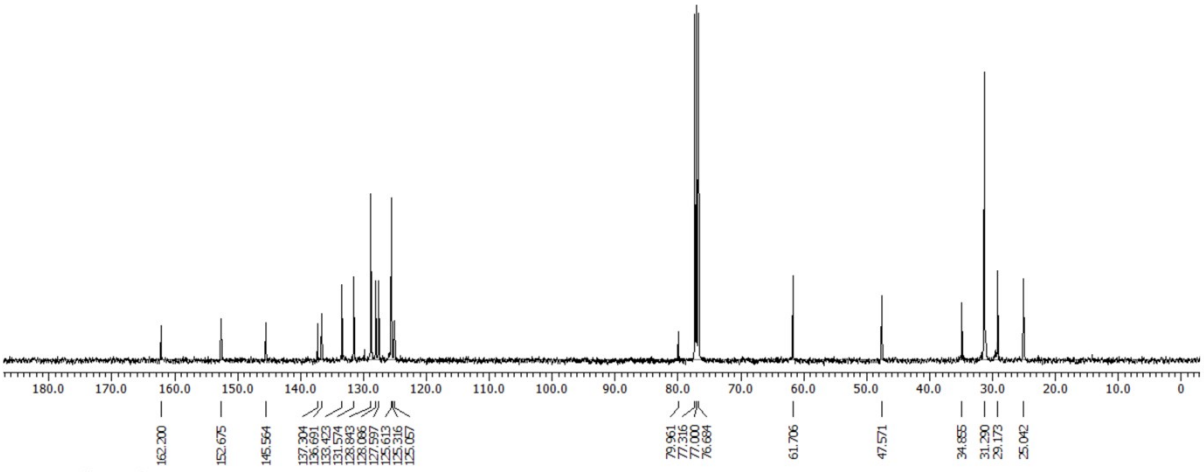
3-(4-(tert-butyl)phenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3e)



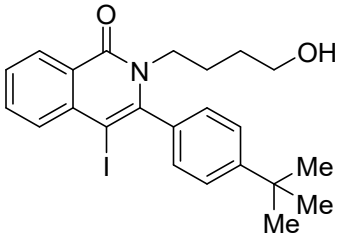
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



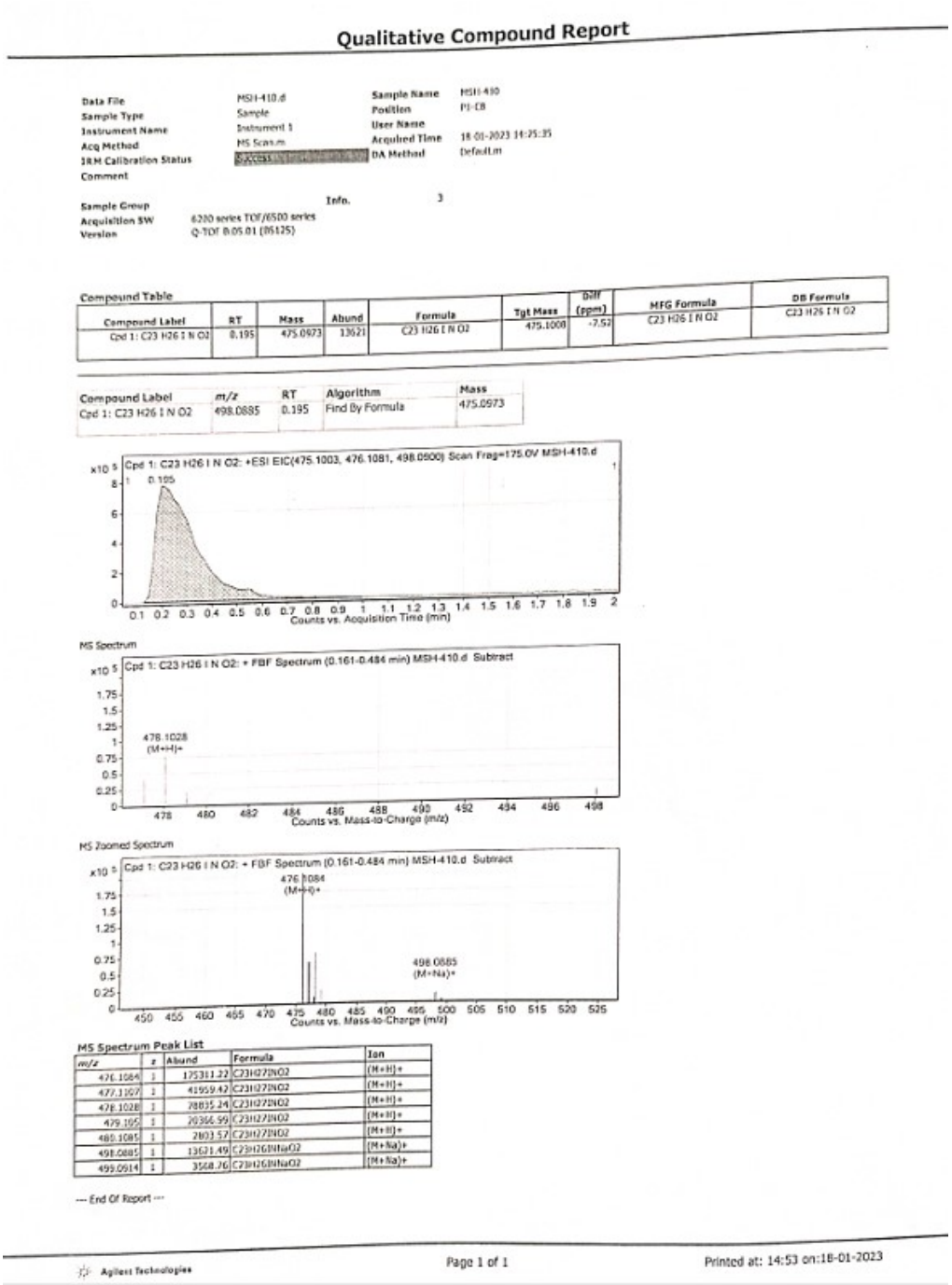
3-(4-(tert-butyl)phenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3e)



HRMS



3-(4-(tert-butyl)phenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3e)



Qualitative Compound Report

Data File MSH-410.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method MS Scan.m
IRM Calibration Status
Comment
Sample Name MSH-410
Position P1-C8
User Name
Acquired Time 18-01-2023 14:25:35
DA Method Default.m

Sample Group Info. 3
Acquisition SW Version 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C23 H26 I N O2	0.195	475.0973	13621	C23 H26 I N O2	475.1008	-7.52	C23 H26 I N O2	C23 H26 I N O2

Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C23 H26 I N O2	498.0885	0.195	Find By Formula	475.0973

MS Spectrum

MS Zoomed Spectrum

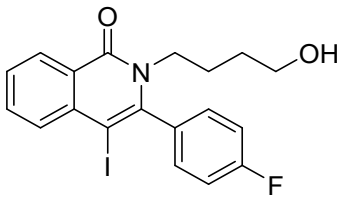
MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
476.1084	1	175381.22	C23H27INO2	(M+H)+
477.1107	1	41959.42	C23H27INO2	(M+H)+
478.1028	1	78835.24	C23H27INO2	(M+H)+
479.105	1	20366.59	C23H27INO2	(M+H)+
480.1085	1	2803.57	C23H27INO2	(M+H)+
498.0885	1	13621.49	C23H26INNaO2	(M+Na)+
499.0914	1	3568.76	C23H26INNaO2	(M+Na)+

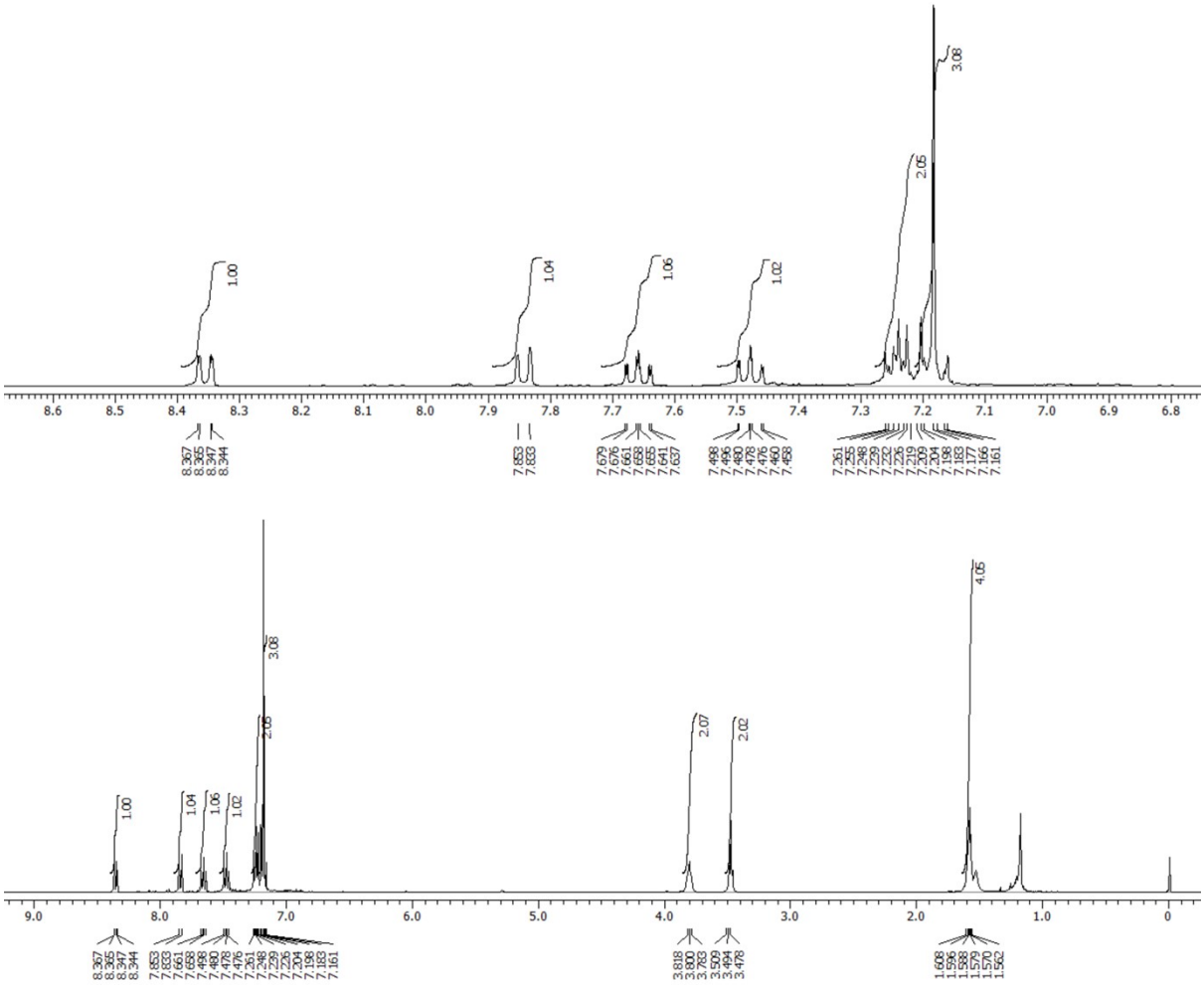
--- End Of Report ---

Agilent Technologies Page 1 of 1 Printed at: 14:53 on:18-01-2023

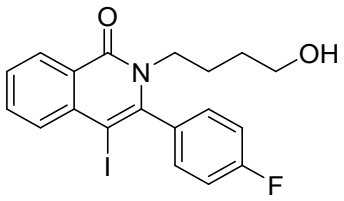
^{1}H NMR (400 MHz, $CDCl_3$)



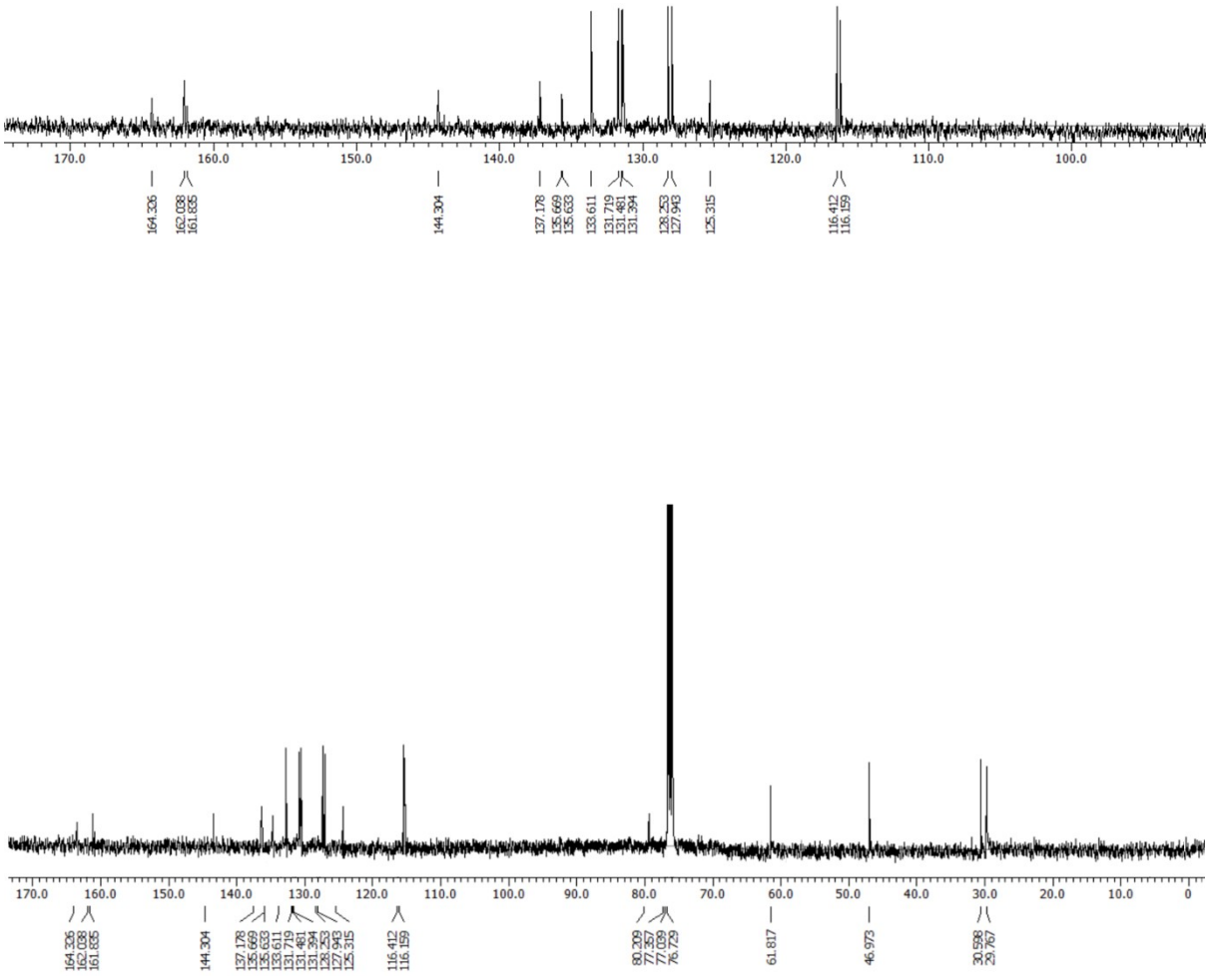
3-(4-fluorophenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3f)



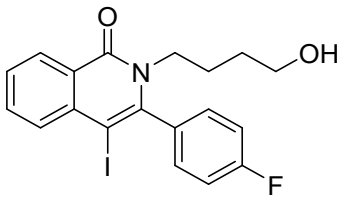
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



3-(4-fluorophenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3f)



HRMS



3-(4-fluorophenyl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3f)

Qualitative Compound Report

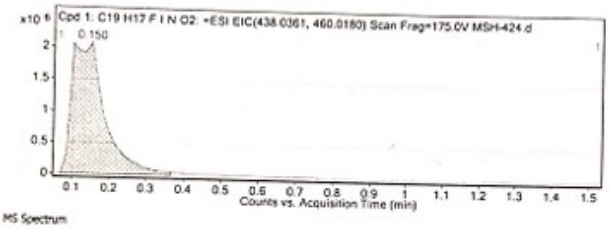
Data File MSH-424.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method MS Scan.m
IRM Calibration Status Success
Comment
Sample Name MSH-424
Position P1-B5
User Name
Acquired Time 19.10.2023 13:07:43
DA Method Default.m

Sample Group
Acquisition SW Version 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)
Info. 3

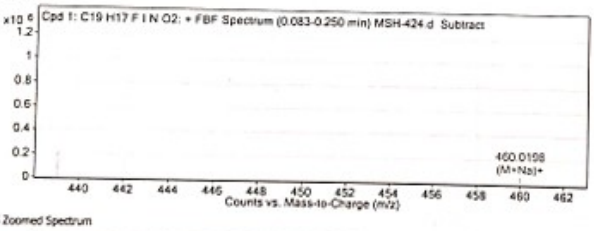
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C19 H17 F I N O2	0.15	437.0309	64699	C19 H17 F I N O2	437.0288	4.78	C19 H17 F I N O2	C19 H17 F I N O2

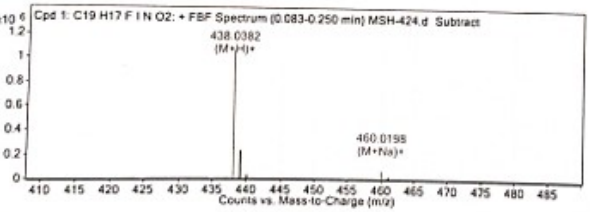
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C19 H17 F I N O2	460.0198	0.15	Find By Formula	437.0309



MS Spectrum



MS Zoomed Spectrum

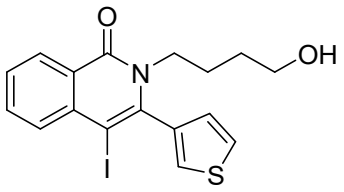


MS Spectrum Peak List

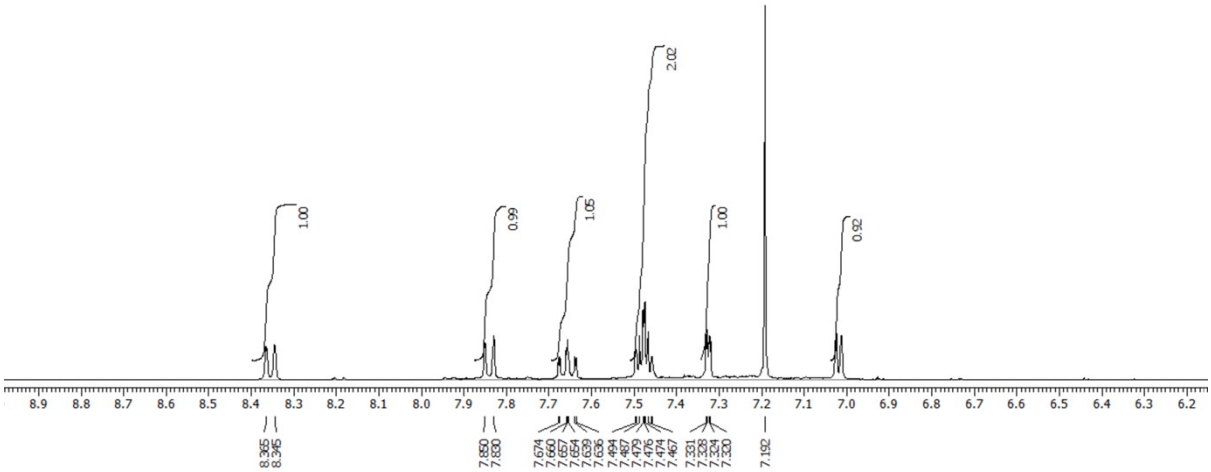
m/z	z	Abund	Formula	Ion
438.0382	1	1065868.88	C19H18F2NO2	(M+H)+
439.0414	1	222754.39	C19H18F2NO2	(M+H)+
440.0433	1	26897.15	C19H18F2NO2	(M+H)+
441.0456	1	2476	C19H18F2NO2	(M+H)+
460.0198	1	64699.44	C19H17F2NNaO2	(M+Na)+
461.0233	1	13489.45	C19H17F2NNaO2	(M+Na)+
462.0255	1	1717.83	C19H17F2NNaO2	(M+Na)+
463.0286	1	153.38	C19H17F2NNaO2	(M+Na)+

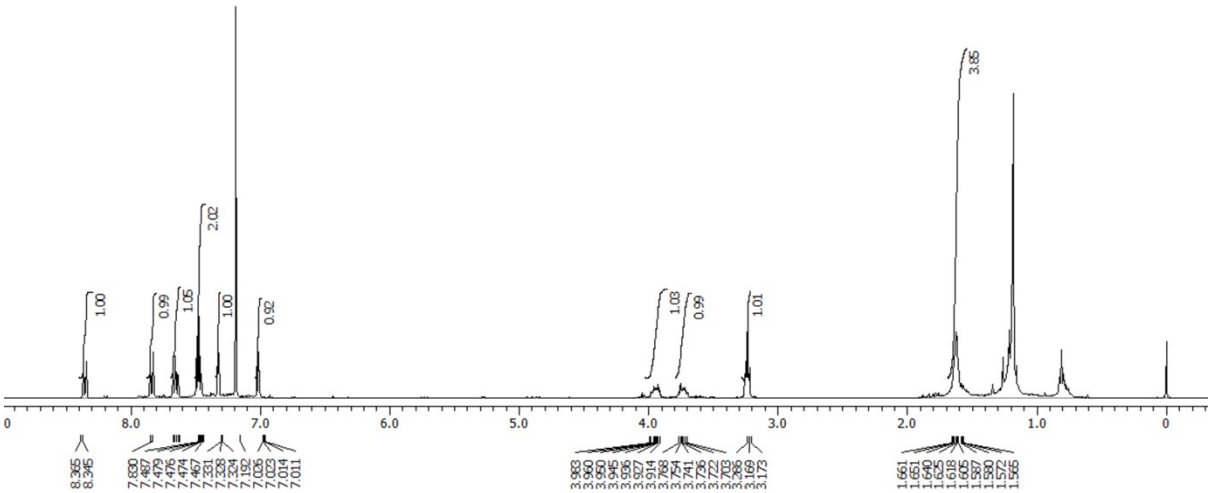
--- End Of Report ---

^{1}H NMR (400 MHz, $CDCl_3$)

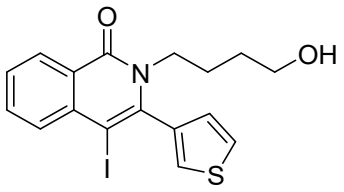


2-(4-hydroxybutyl)-4-iodo-3-(thiophen-3-yl)isoquinolin-1(2H)-one (3g)

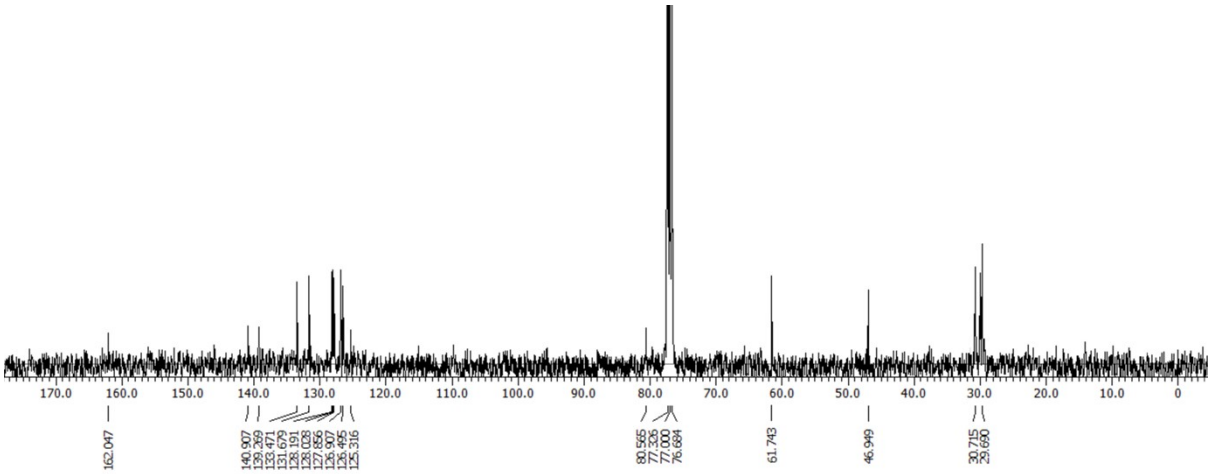




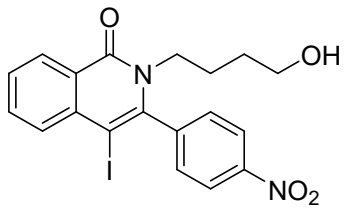
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



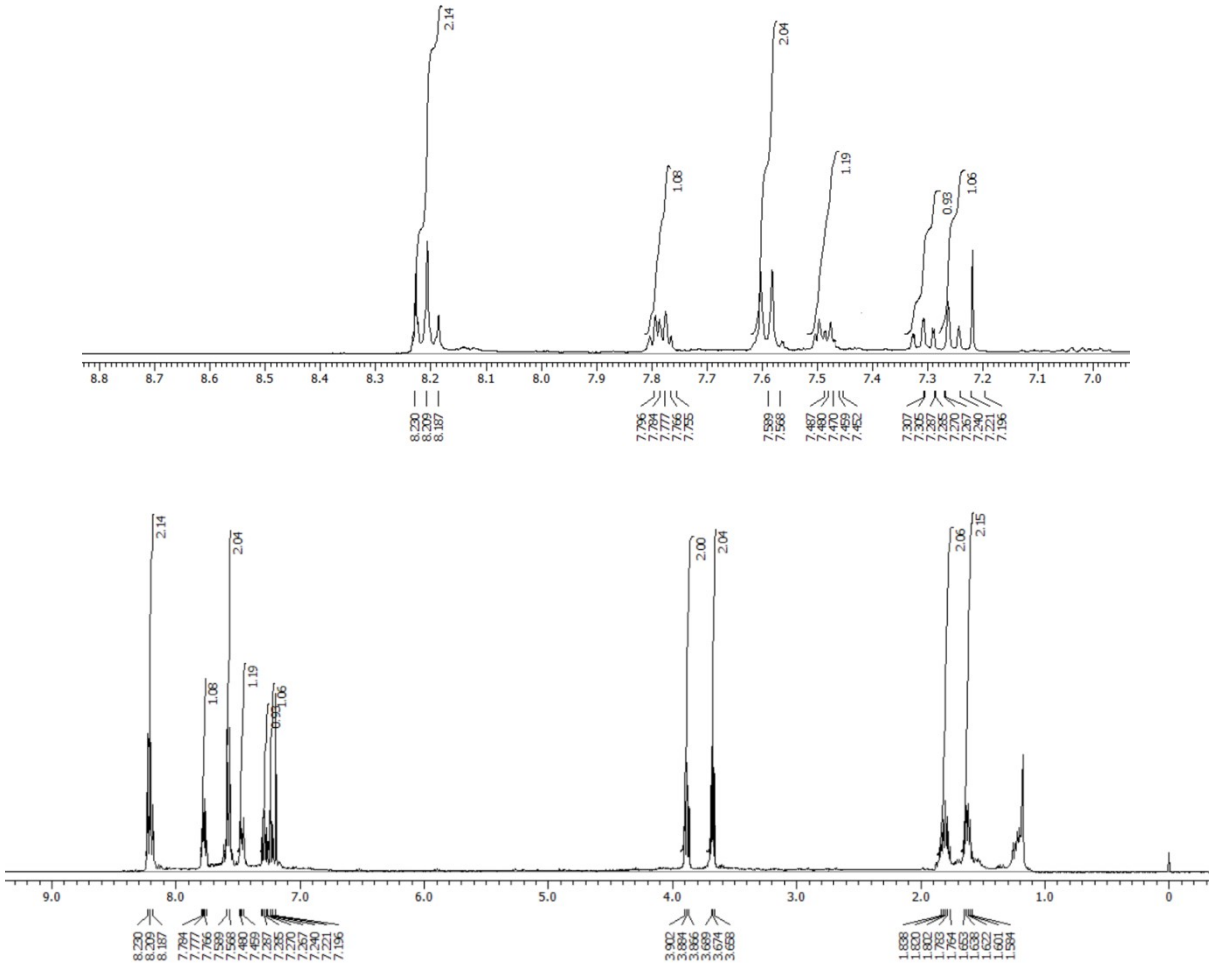
2-(4-hydroxybutyl)-4-iodo-3-(thiophen-3-yl)isoquinolin-1(2H)-one (3g)



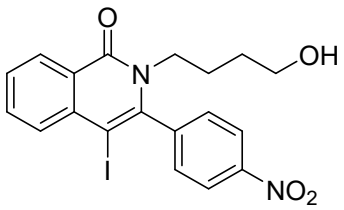
^{1}H NMR (400 MHz, $CDCl_3$)



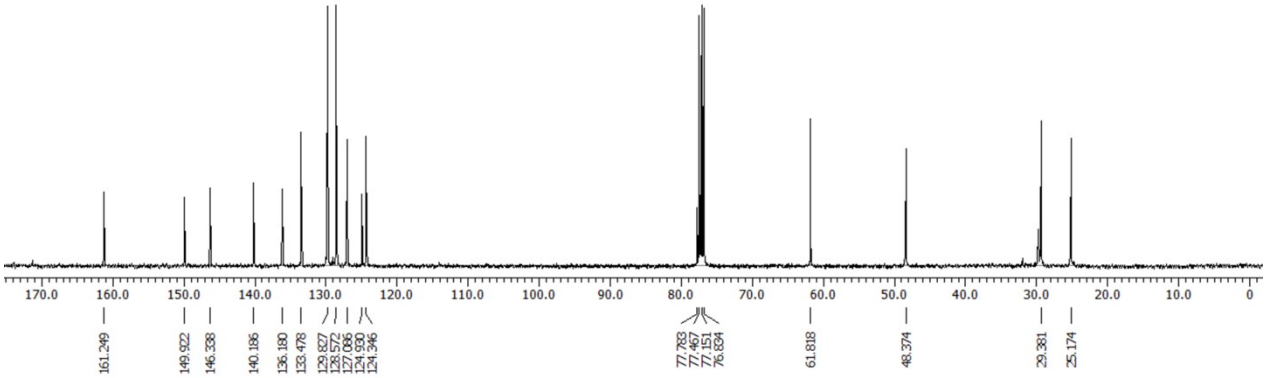
2-(4-hydroxybutyl)-4-iodo-3-(4-nitrophenyl)isoquinolin-1(2H)-one (3h)



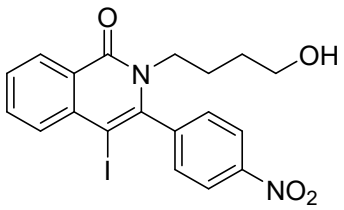
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



2-(4-hydroxybutyl)-4-iodo-3-(4-nitrophenyl)isoquinolin-1(2H)-one (3h)



HRMS



2-(4-hydroxybutyl)-4-iodo-3-(4-nitrophenyl)isoquinolin-1(2H)-one (3h)

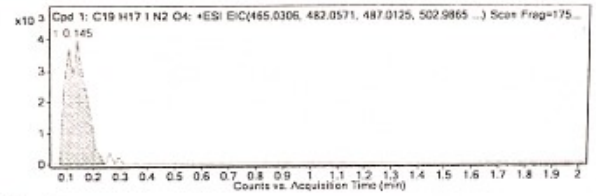
Qualitative Compound Report

Data File	MSH-578.d	Sample Name	MSH-578
Sample Type	Sample	Position	P1-B4
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	24.08.2023 13:55:38
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

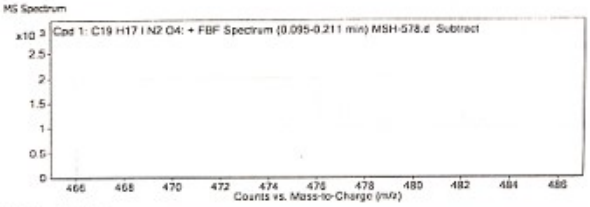
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C19 H17 I N2 O4	0.145	464.0252	2381	C19 H17 I N2 O4	464.0233	4	C19 H17 I N2 O4	C19 H17 I N2 O4

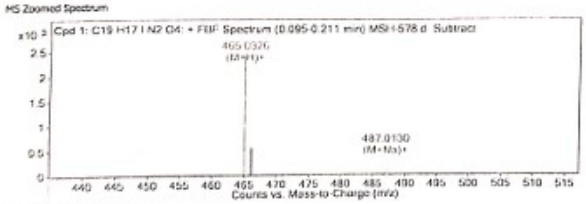
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C19 H17 I N2 O4	465.0326	0.145	Find By Formula	464.0252



MS Spectrum



MS Zoomed Spectrum

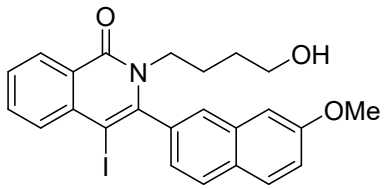


MS Spectrum Peak List

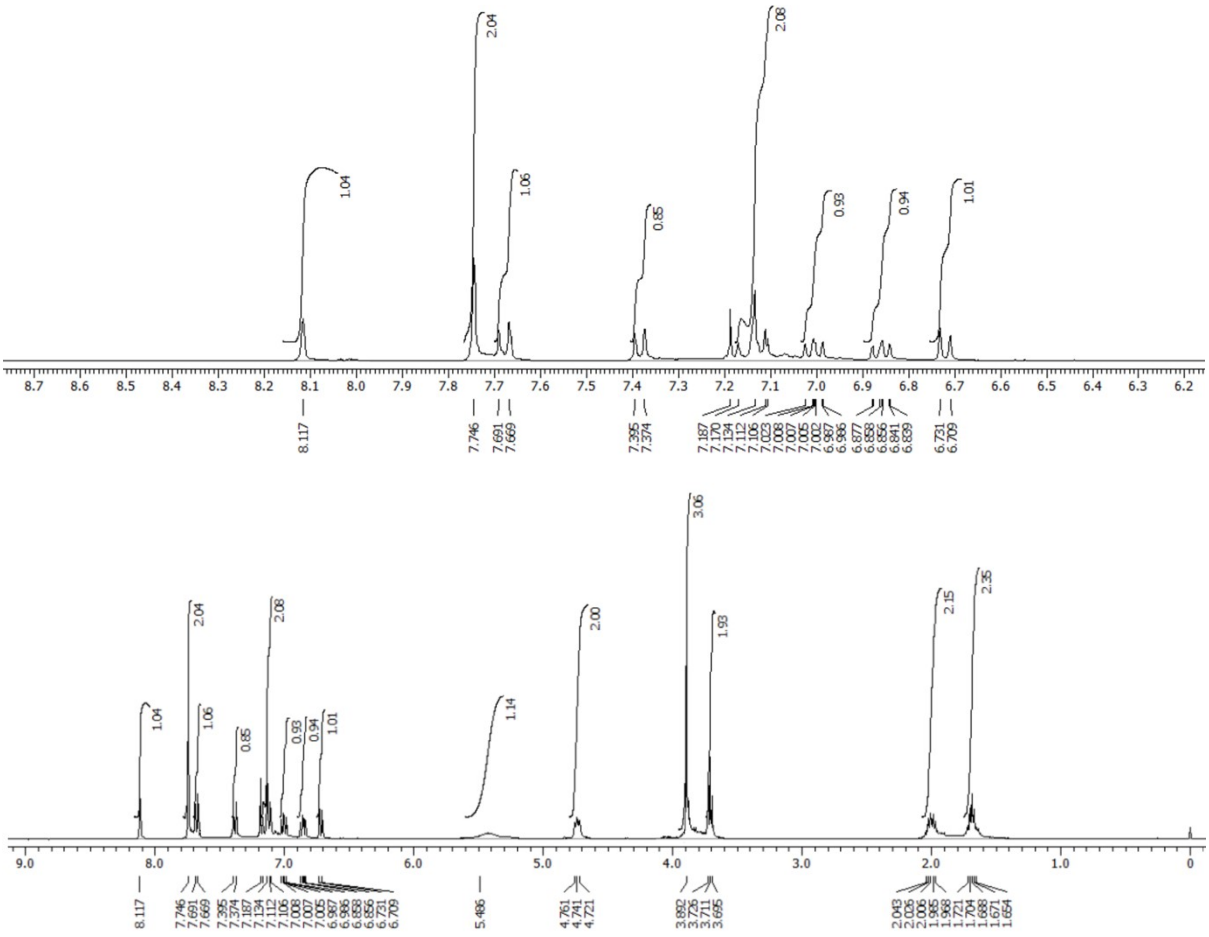
m/z	z	Abund	Formula	Ion
465.0326	1	2380.85	C19H18IN2O4	(M+H)+
466.0351	1	575.07	C19H18IN2O4	(M+H)+
487.013	1	97.83	C19H17IN2NaO4	(M+Na)+

--- End Of Report ---

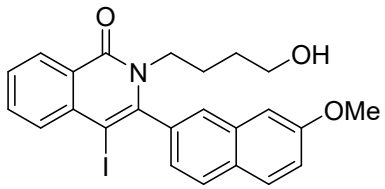
^{1}H NMR (400 MHz, $CDCl_3$)



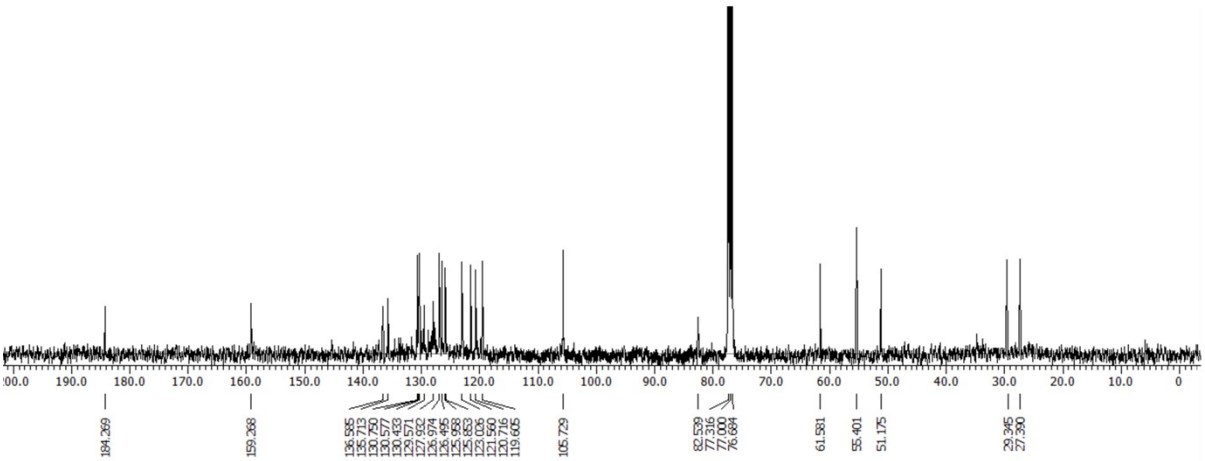
2-(4-hydroxybutyl)-4-iodo-3-(7-methoxynaphthalen-2-yl)isoquinolin-1(2H)-one (3i)



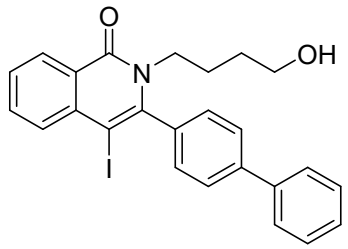
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



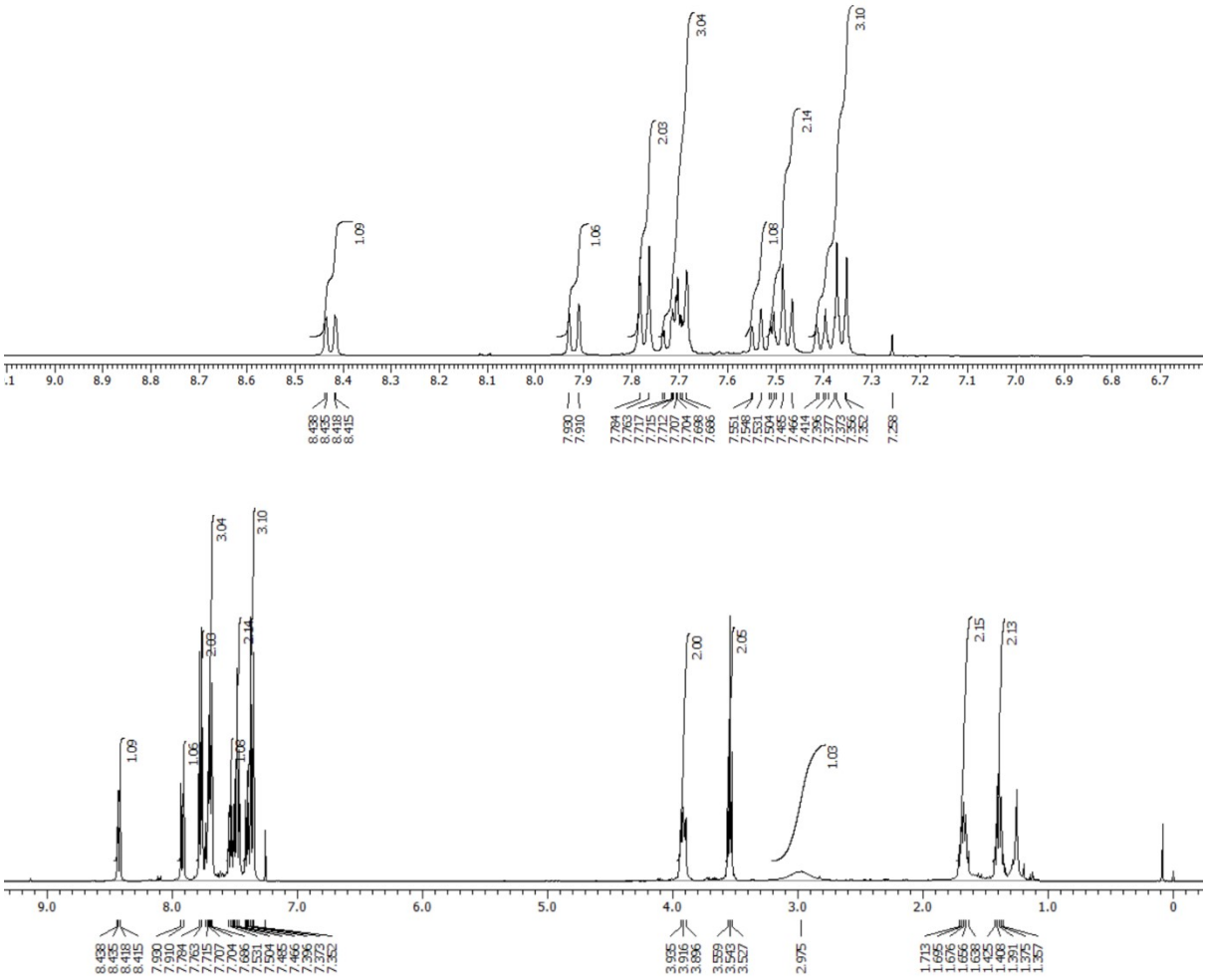
2-(4-hydroxybutyl)-4-iodo-3-(7-methoxynaphthalen-2-yl)isoquinolin-1(2H)-one (3i)



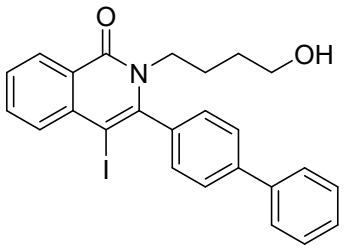
^{1}H NMR (400 MHz, $CDCl_3$)



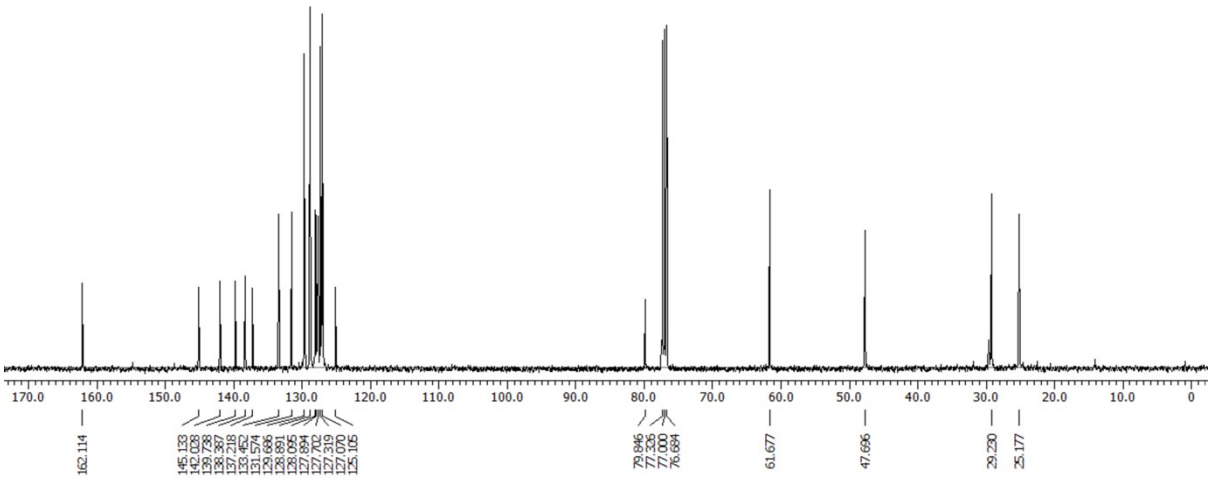
3-([1,1'-biphenyl]-4-yl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3j)



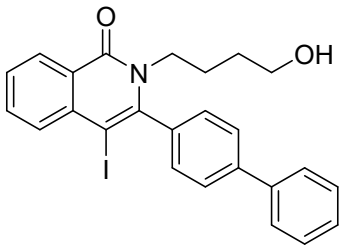
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



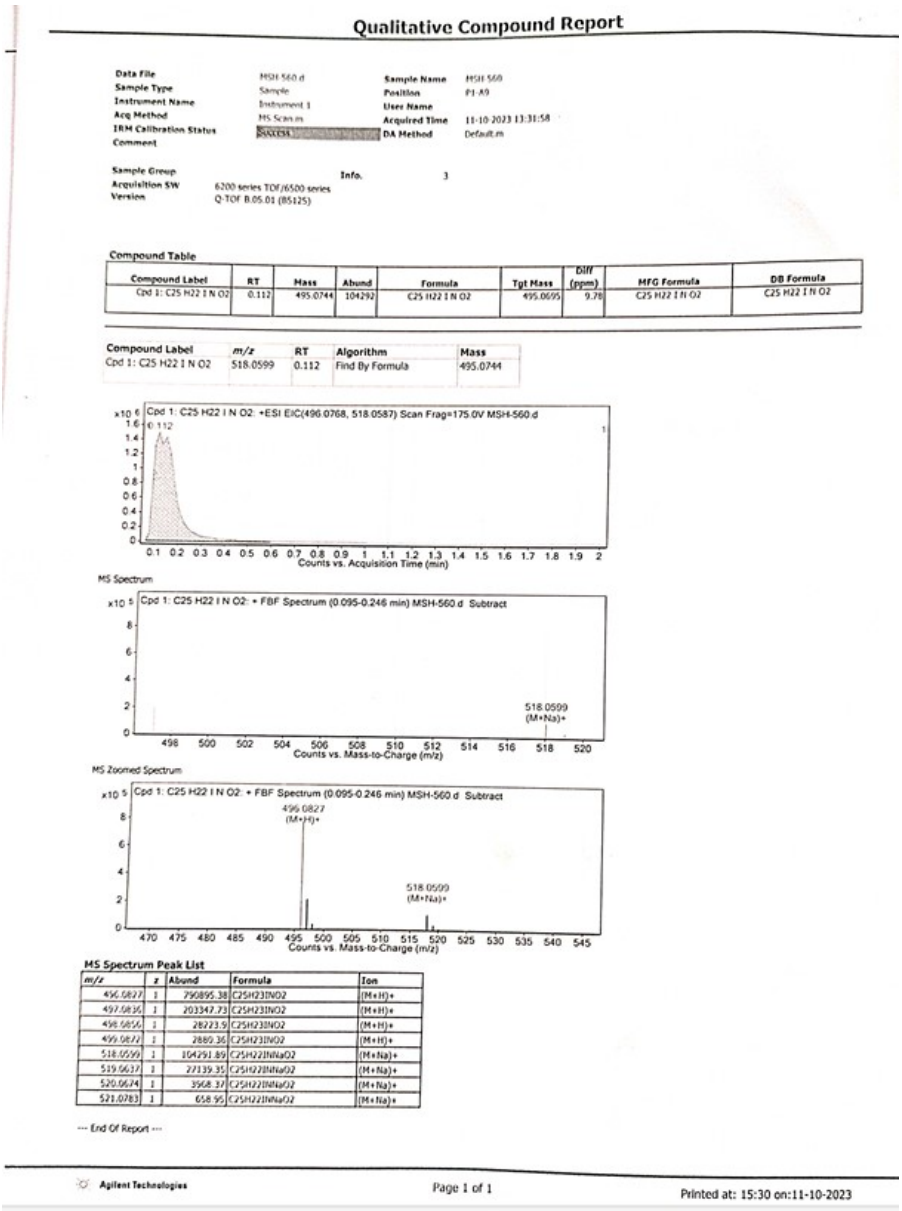
3-([1,1'-biphenyl]-4-yl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3*j*)



HRMS



3-([1,1'-biphenyl]-4-yl)-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (3j)



Qualitative Compound Report

Data File	MSH-560.d	Sample Name	MSH-560
Sample Type	Sample	Position	P1-A9
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	11-10-2023 13:31:58
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group	Info.	3	
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (85125)		

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C25 H22 I N O2	0.112	495.0744	104292	C25 H22 I N O2	495.0695	9.78	C25 H22 I N O2	C25 H22 I N O2

Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C25 H22 I N O2	518.0599	0.112	Find By Formula	495.0744

MS Spectrum

MS Zoomed Spectrum

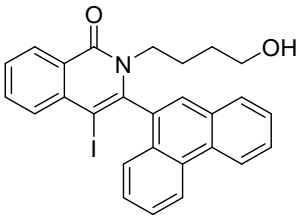
MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
496.0827	1	790895.38	C25H23INO2	(M+H)+
497.0836	1	203347.73	C25H23INO2	(M+H)+
498.0856	1	28223.9	C25H23INO2	(M+H)+
499.0872	1	2889.36	C25H23INO2	(M+H)+
518.0599	1	104291.89	C25H22INNaO2	(M+Na)+
519.0637	1	27139.35	C25H22INNaO2	(M+Na)+
520.0674	1	3568.37	C25H22INNaO2	(M+Na)+
521.0783	1	658.95	C25H22INNaO2	(M+Na)+

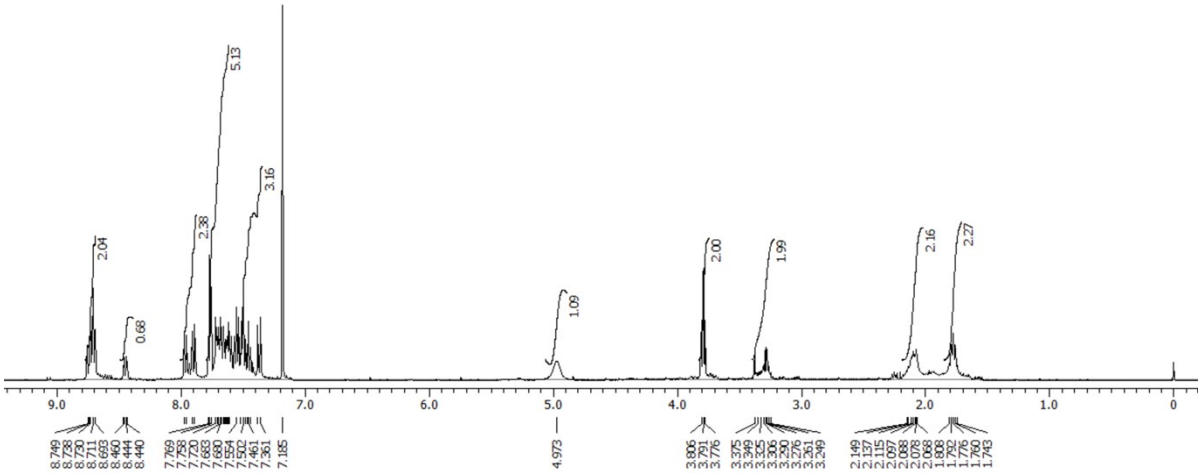
--- End Of Report ---

Agilent Technologies Page 1 of 1 Printed at: 15:30 on:11-10-2023

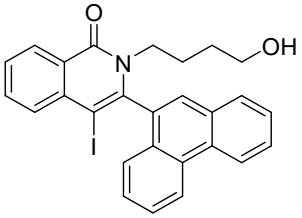
^{1}H NMR (400 MHz, $CDCl_3$)



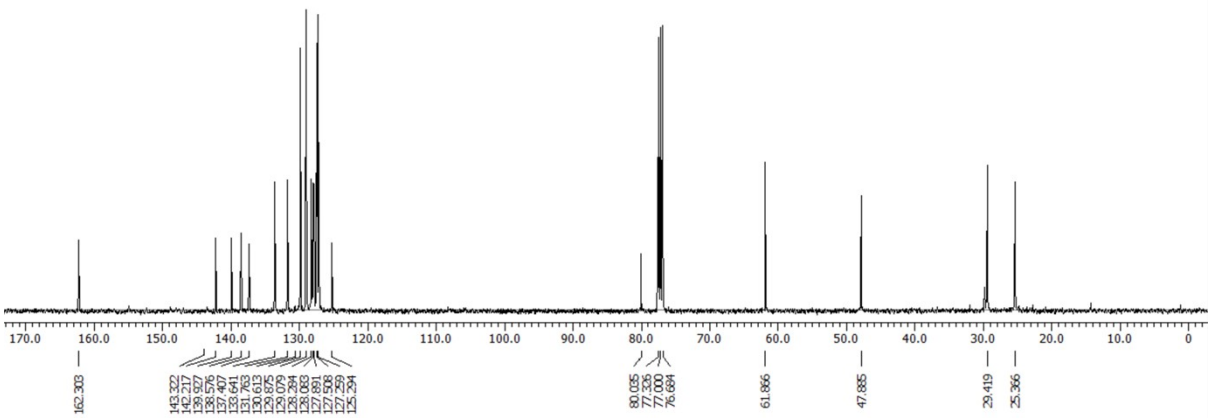
2-(4-hydroxybutyl)-4-iodo-3-(phenanthren-9-yl)isoquinolin-1(2H)-one (3k)



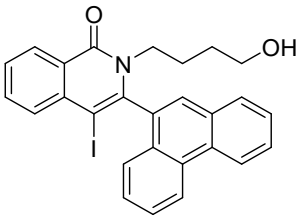
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



2-(4-hydroxybutyl)-4-iodo-3-(phenanthren-9-yl)isoquinolin-1(2H)-one (3k)



HRMS



2-(4-hydroxybutyl)-4-iodo-3-(phenanthren-9-yl)isoquinolin-1(2H)-one (3k)

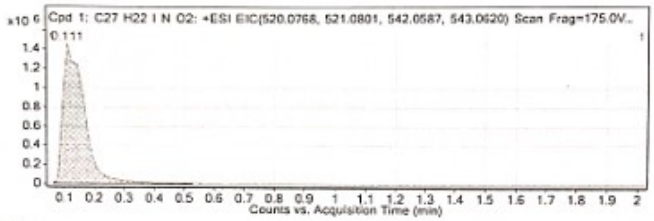
Qualitative Compound Report

Data File	MSH-562.d	Sample Name	MSH-562
Sample Type	Sample	Position	P1-D9
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	13-09-2023 15:30:46
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

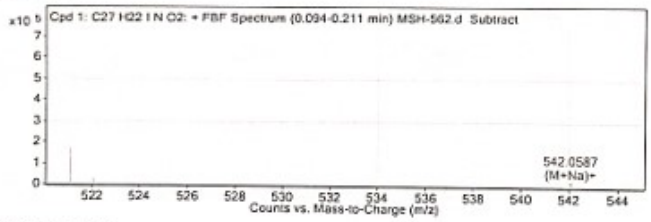
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C27 H22 I N O2	0.111	519.0732	631592	C27 H22 I N O2	519.0695	7.17	C27 H22 I N O2	C27 H22 I N O2

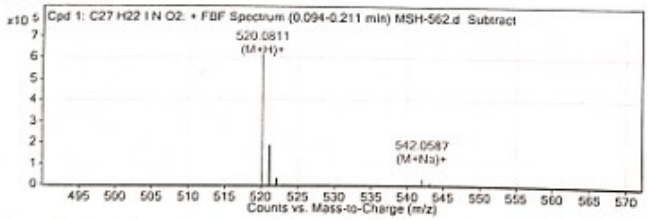
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C27 H22 I N O2	520.0811	0.111	Find By Formula	519.0732



MS Spectrum



MS Zoomed Spectrum

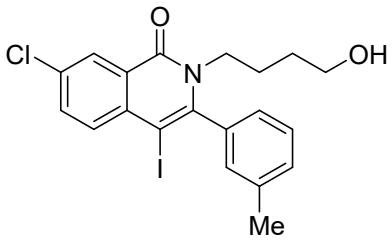


MS Spectrum Peak List

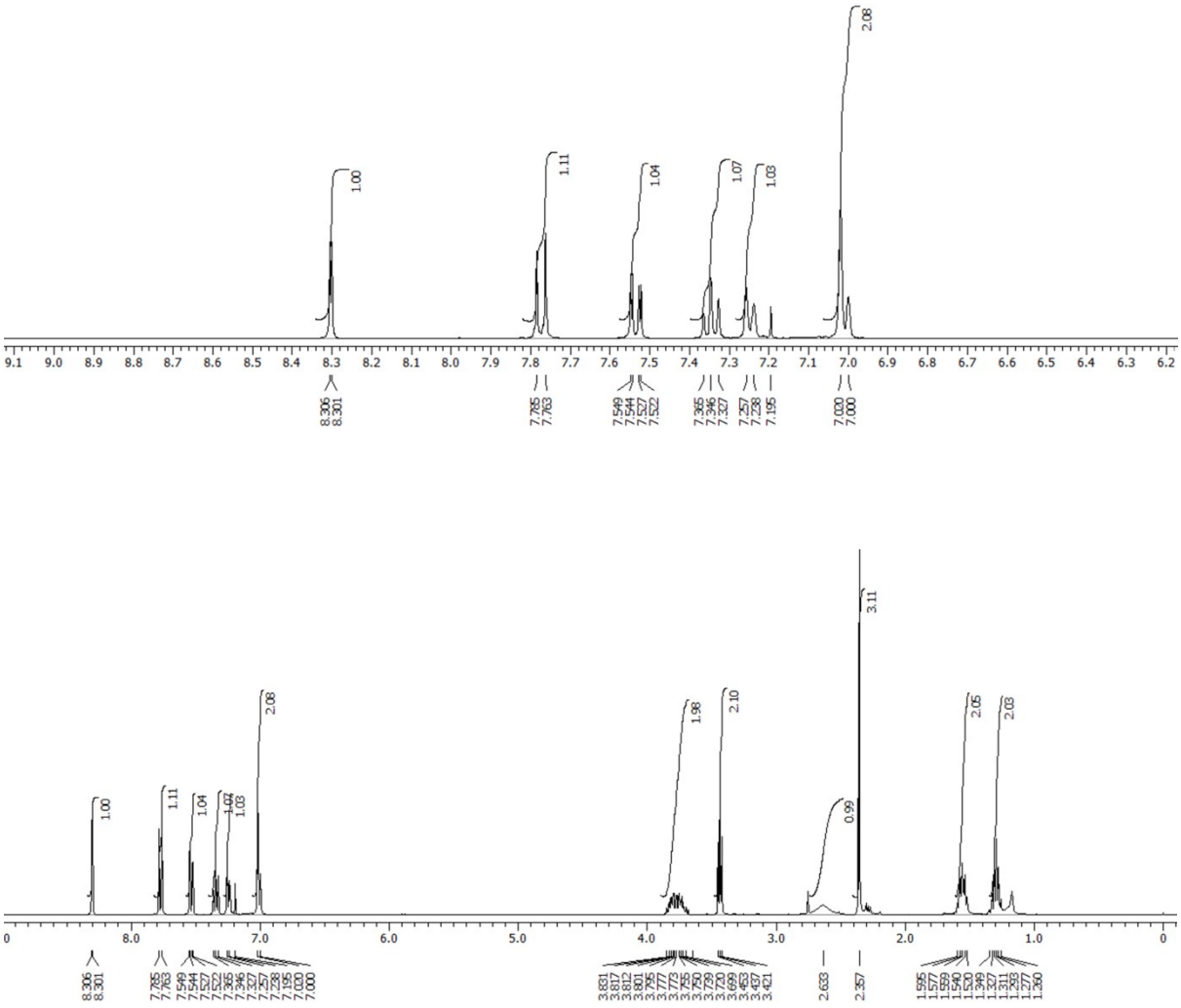
m/z	z	Abund	Formula	Ion
520.0811	1	631592.13	C27H23INO2	(M+H)+
521.0823	1	171683.02	C27H23INO2	(M+H)+
522.0846	1	25380.69	C27H23INO2	(M+H)+
523.0875	1	2644.58	C27H23INO2	(M+H)+
542.0587	1	25672.63	C27H22INNaO2	(M+Na)+
543.07	1	8617.74	C27H22INNaO2	(M+Na)+
544.0823	1	1793.04	C27H22INNaO2	(M+Na)+
545.0834	1	441.22	C27H22INNaO2	(M+Na)+

--- End Of Report ---

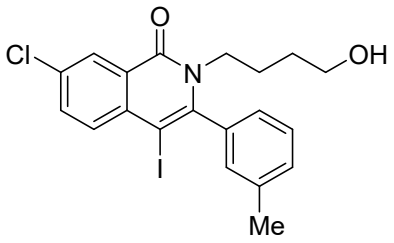
^{1}H NMR (400 MHz, $CDCl_3$)



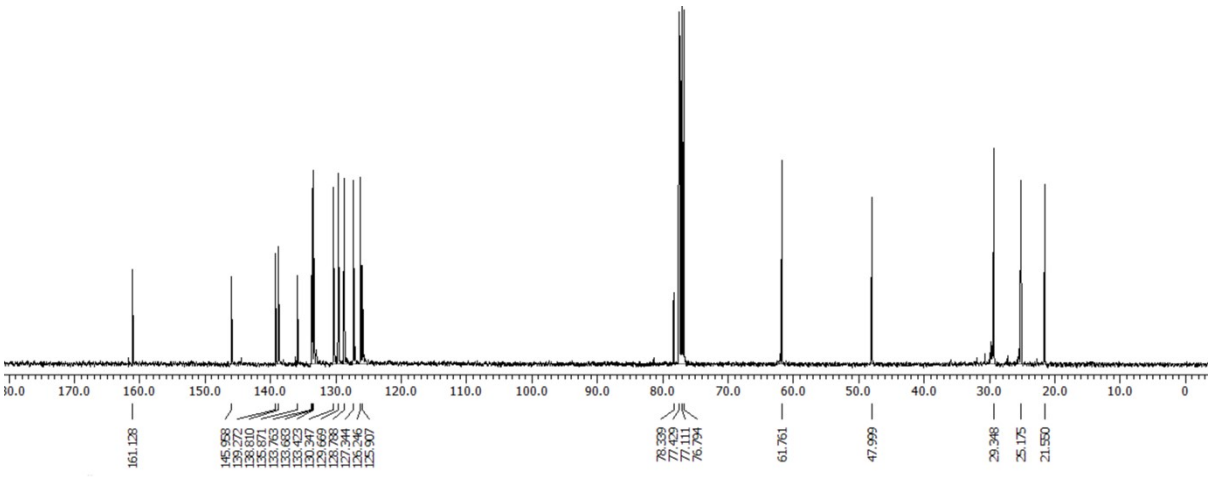
7-chloro-2-(4-hydroxybutyl)-4-iodo-3-(*m*-tolyl)isoquinolin-1(2H)-one (5a)



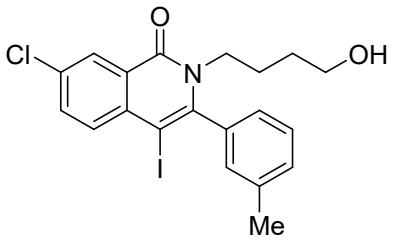
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



7-chloro-2-(4-hydroxybutyl)-4-iodo-3-(*m*-tolyl)isoquinolin-1(2*H*)-one (5a)



HRMS



7-chloro-2-(4-hydroxybutyl)-4-iodo-3-(*m*-tolyl)isoquinolin-1(2*H*)-one (5a)

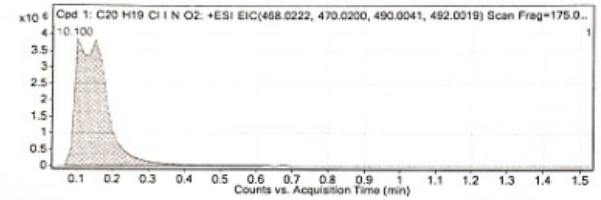
Qualitative Compound Report

Data File	MSH-596.d	Sample Name	MSH-596
Sample Type	Sample	Position	P1-B6
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	19-10-2023 13:11:28
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

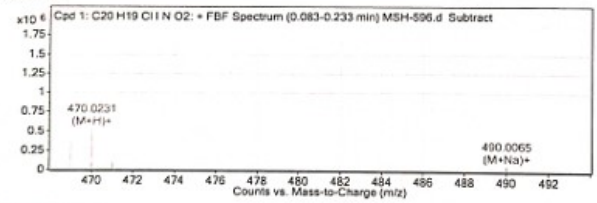
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C20 H19 Cl I N O2	0.1	467.018	69435	C20 H19 Cl I N O2	467.0149	6.64	C20 H19 Cl I N O2	C20 H19 Cl I N O2

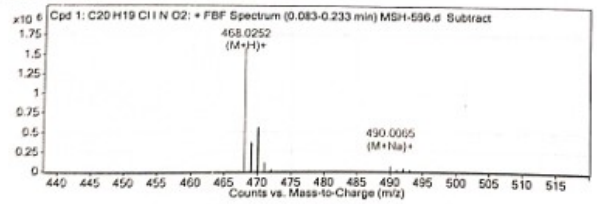
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C20 H19 Cl I N O2	490.0065	0.1	Find By Formula	467.018



MS Spectrum



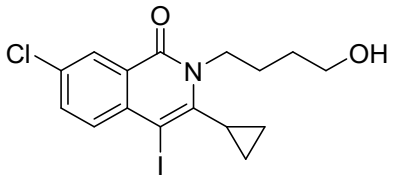
MS Zoomed Spectrum



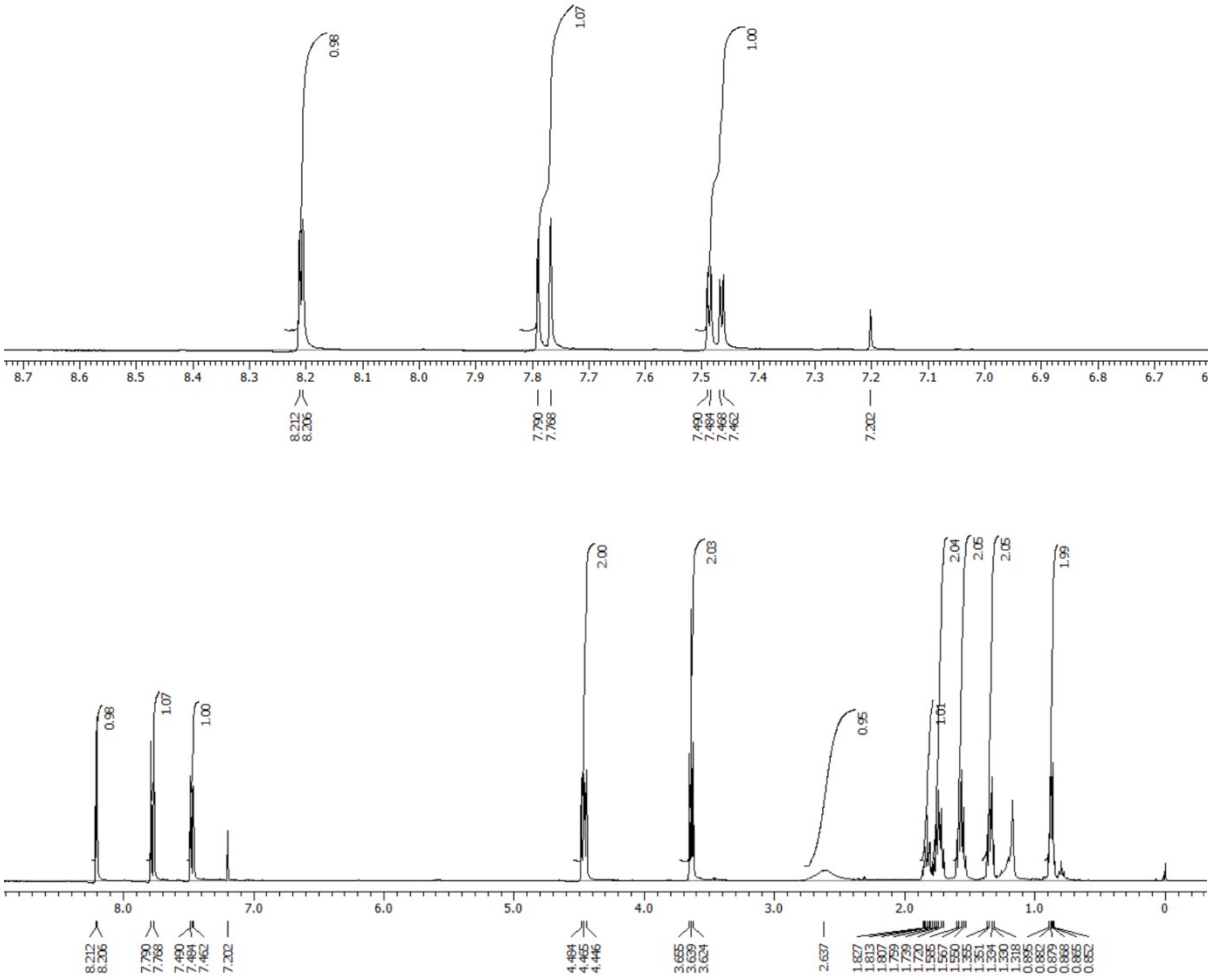
MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
468.0252	1	1592326.25	C20H20ClINO2	(M+H)+
469.0292	1	376870.94	C20H20ClINO2	(M+H)+
470.0231	1	532184.88	C20H20ClINO2	(M+H)+
471.0255	1	109512.77	C20H20ClINO2	(M+H)+
472.0278	1	12016.04	C20H20ClINO2	(M+H)+
473.0314	1	1146.1	C20H20ClINO2	(M+H)+
490.0065	1	69434.58	C20H19ClINNaO2	(M+Na)+

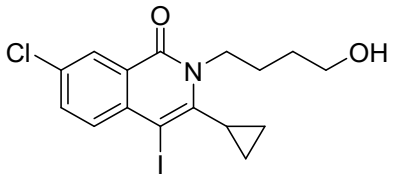
^{1}H NMR (400 MHz, $CDCl_3$)



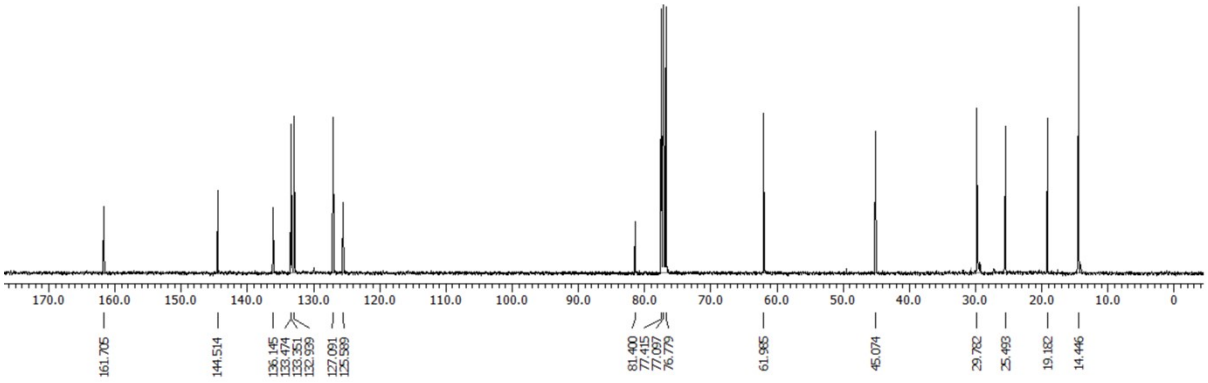
7-chloro-3-cyclopropyl-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2*H*)-one (5b)



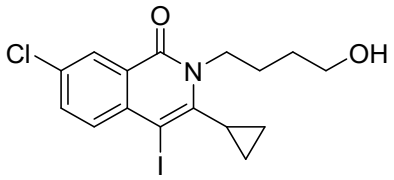
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



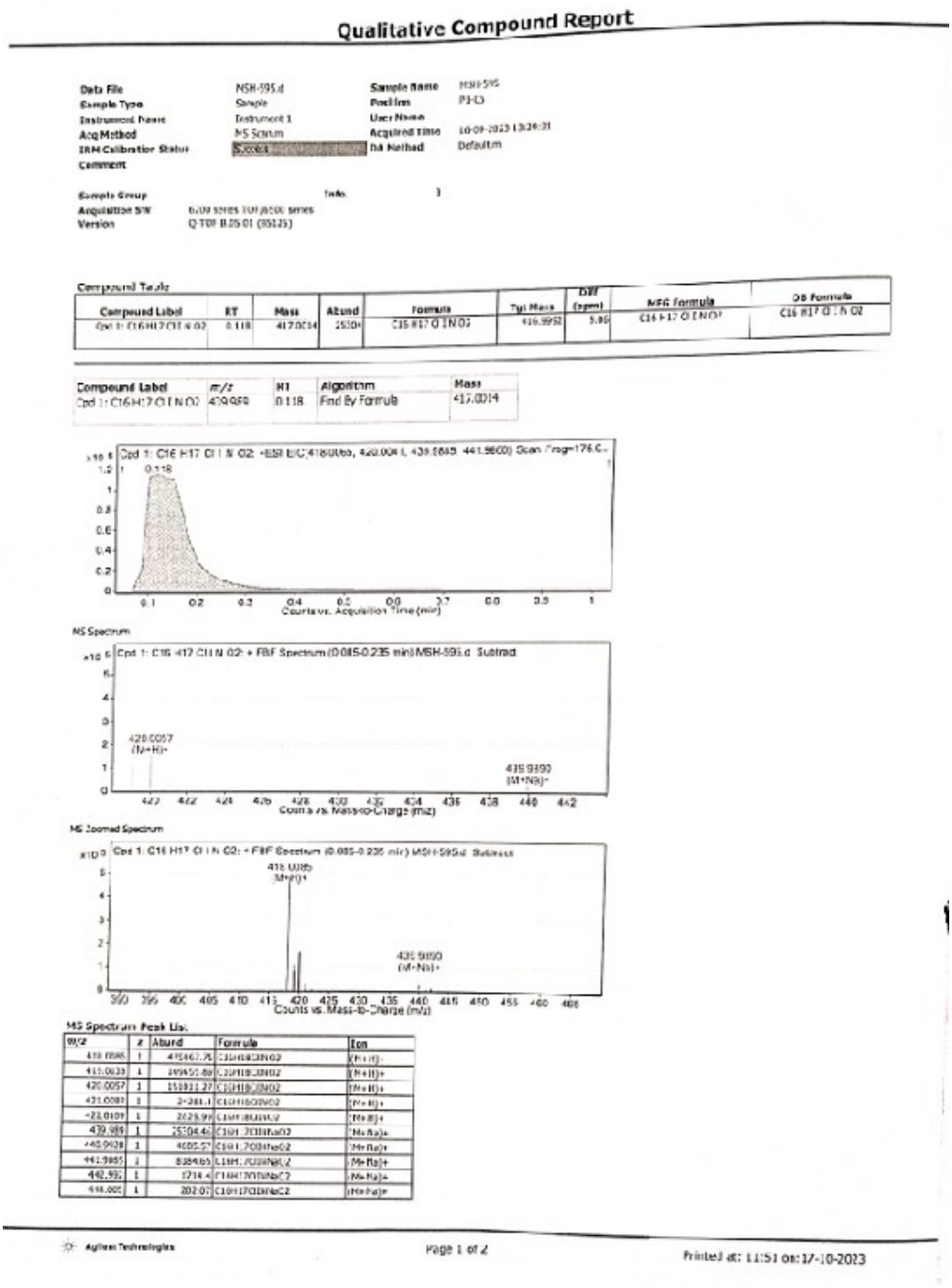
7-chloro-3-cyclopropyl-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (5b)



HRMS



7-chloro-3-cyclopropyl-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (5b)



Qualitative Compound Report

Data File MSH-595.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method MS Scrum
IRM Calibration Status Success
Comment
Sample Name MSH-595
Position P1-D
User Name
Acquired Time 10-09-2023 13:24:31
DA Method Default.m
Sample Group Info.
Acquisition S/W 6200 series TOF/6500 series
Version Q-TOF B.05.01 (05125)

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C16H17Cl I N O2	0.118	417.0014	2630	C16 H17 Cl I N O2	416.9952	5.86	C16 H17 Cl I N O2	C16 H17 Cl I N O2

Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C16H17Cl I N O2	439.9869	0.118	Find By Formula	417.0014

MS Spectrum

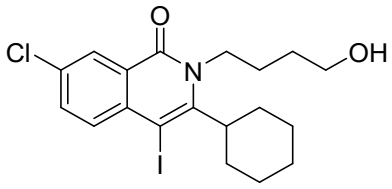
MS Zoomed Spectrum

MS Spectrum Peak List

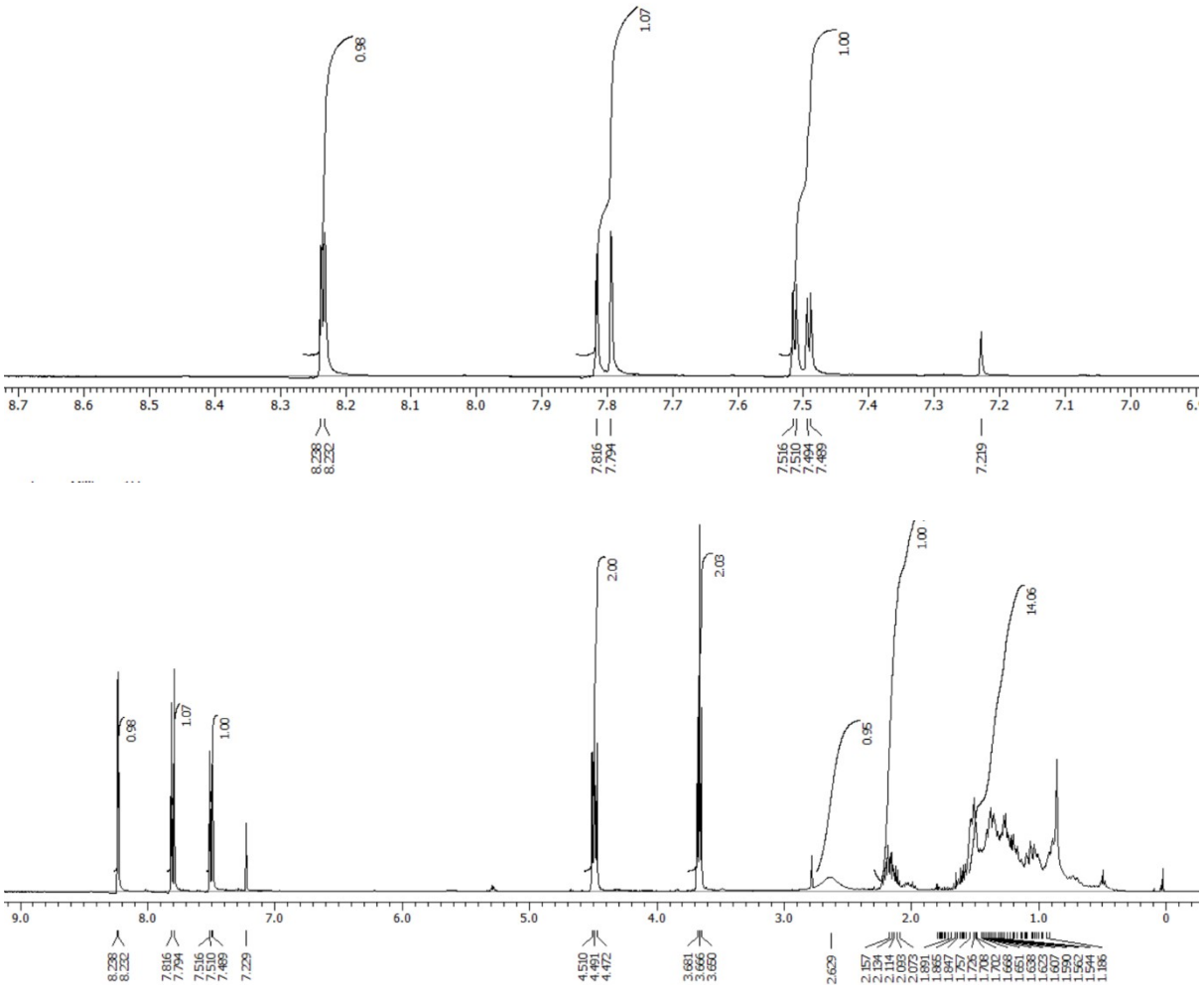
m/z	z	Abund	Formula	Ion
417.0085	1	479467.75	C16H18ClINO2	(M+H)+
419.0033	1	399551.69	C16H18ClINO2	(M+H)+
420.0057	1	151931.27	C16H18ClINO2	(M+H)+
421.0089	1	21281.1	C16H18ClINO2	(M+H)+
423.0101	1	2023.99	C16H18ClINO2	(M+H)+
439.9869	1	25704.46	C16H17ClINaO2	(M+Na)+
441.9839	1	4085.57	C16H17ClINaO2	(M+Na)+
443.9865	1	838.65	C16H17ClINaO2	(M+Na)+
445.985	1	1216.4	C16H17ClINaO2	(M+Na)+
446.005	1	202.07	C16H17ClINaO2	(M+Na)+

Agilent Technologies Page 1 of 2 Printed at: 11:51 on: 17-10-2023

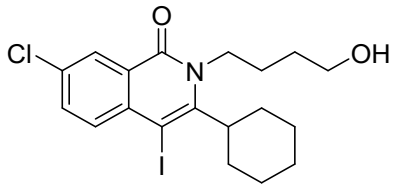
^{1}H NMR (400 MHz, $CDCl_3$)



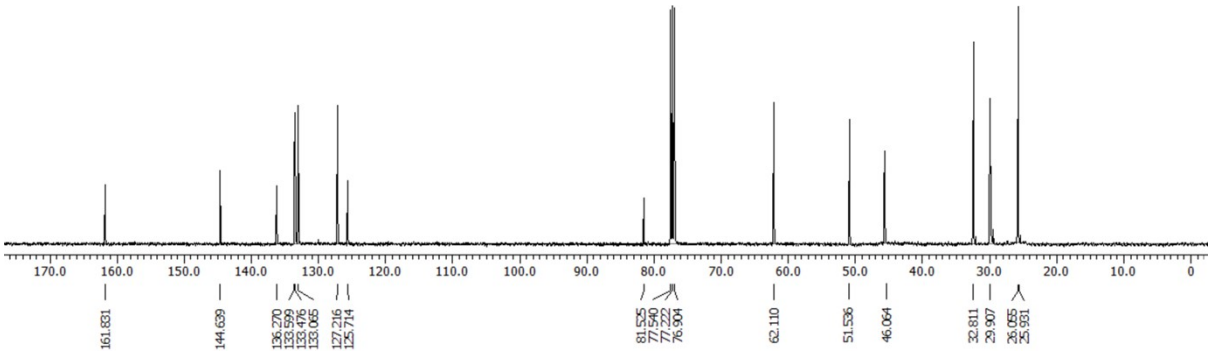
7-chloro-3-cyclohexyl-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (5c)



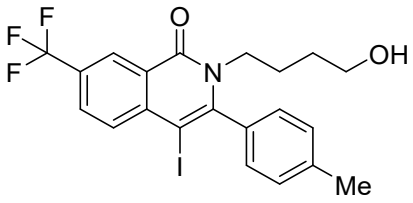
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



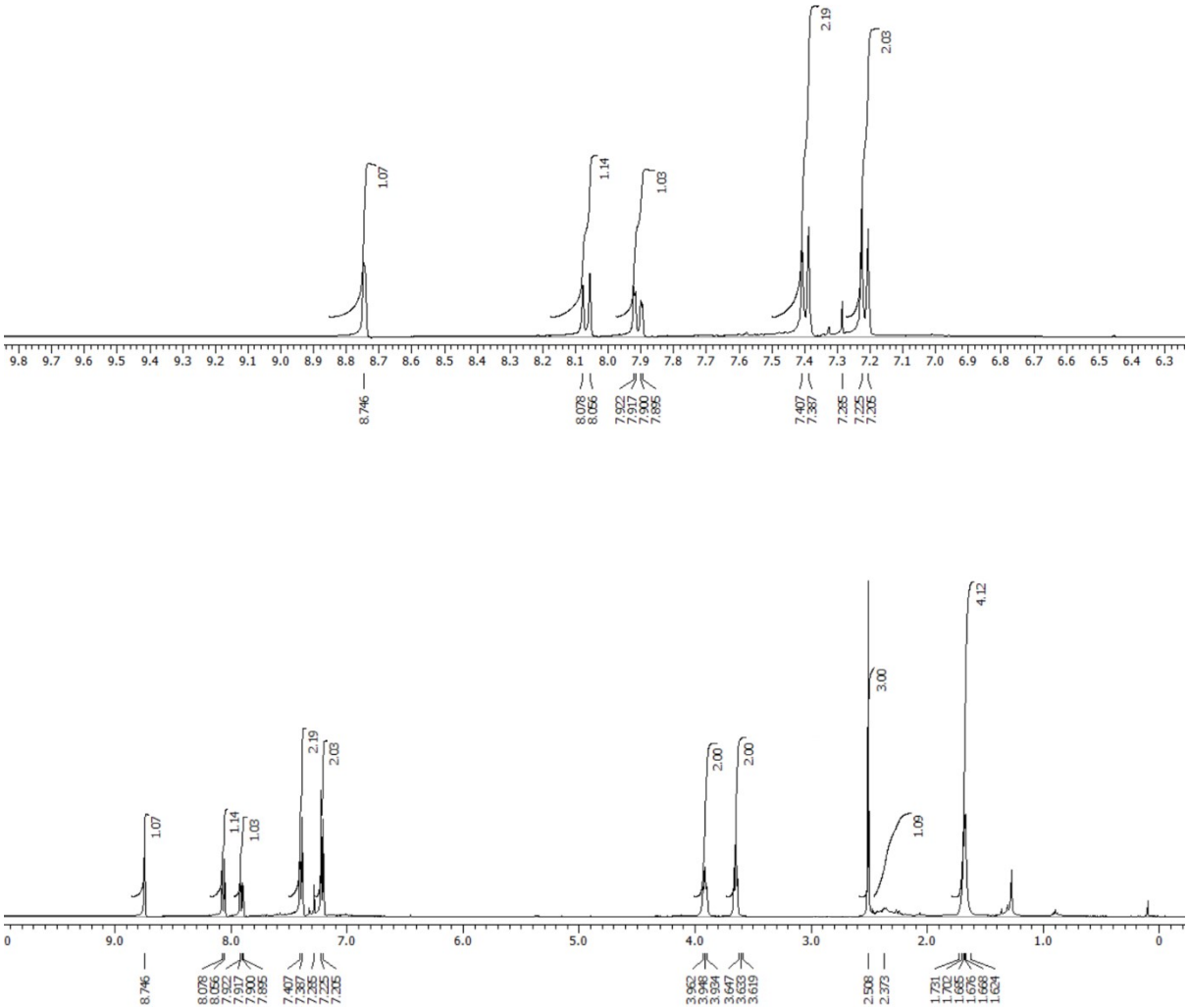
7-chloro-3-cyclohexyl-2-(4-hydroxybutyl)-4-iodoisoquinolin-1(2H)-one (5c)



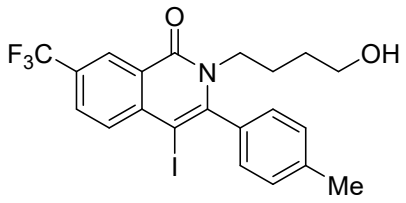
^{1}H NMR (400 MHz, $CDCl_3$)



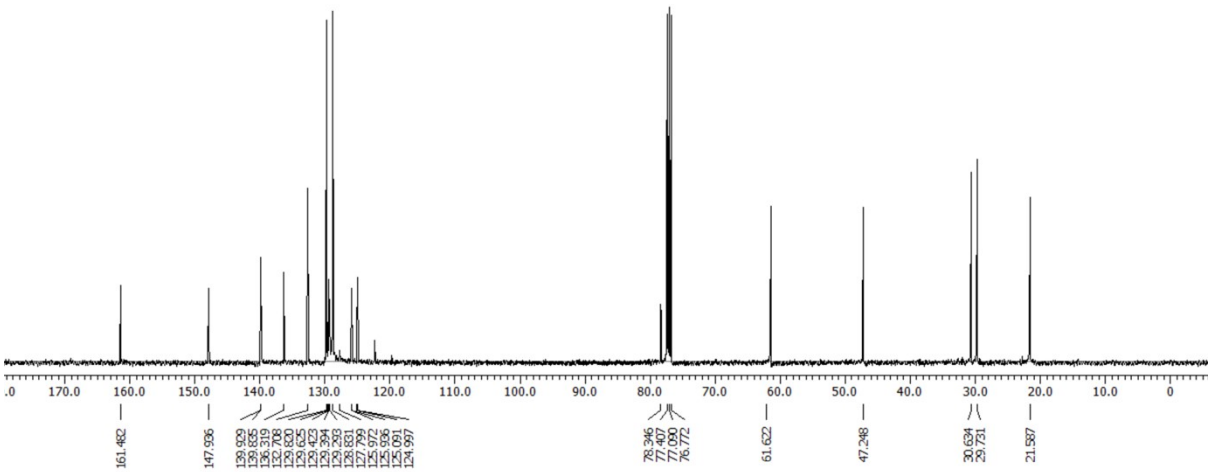
2-(4-hydroxybutyl)-4-iodo-3-(*p*-tolyl)-7-(trifluoromethyl)isoquinolin-1(2H)-one (5d)



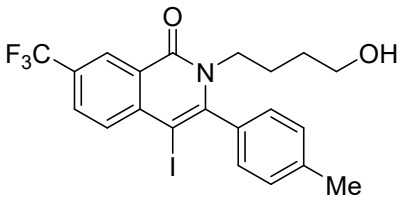
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



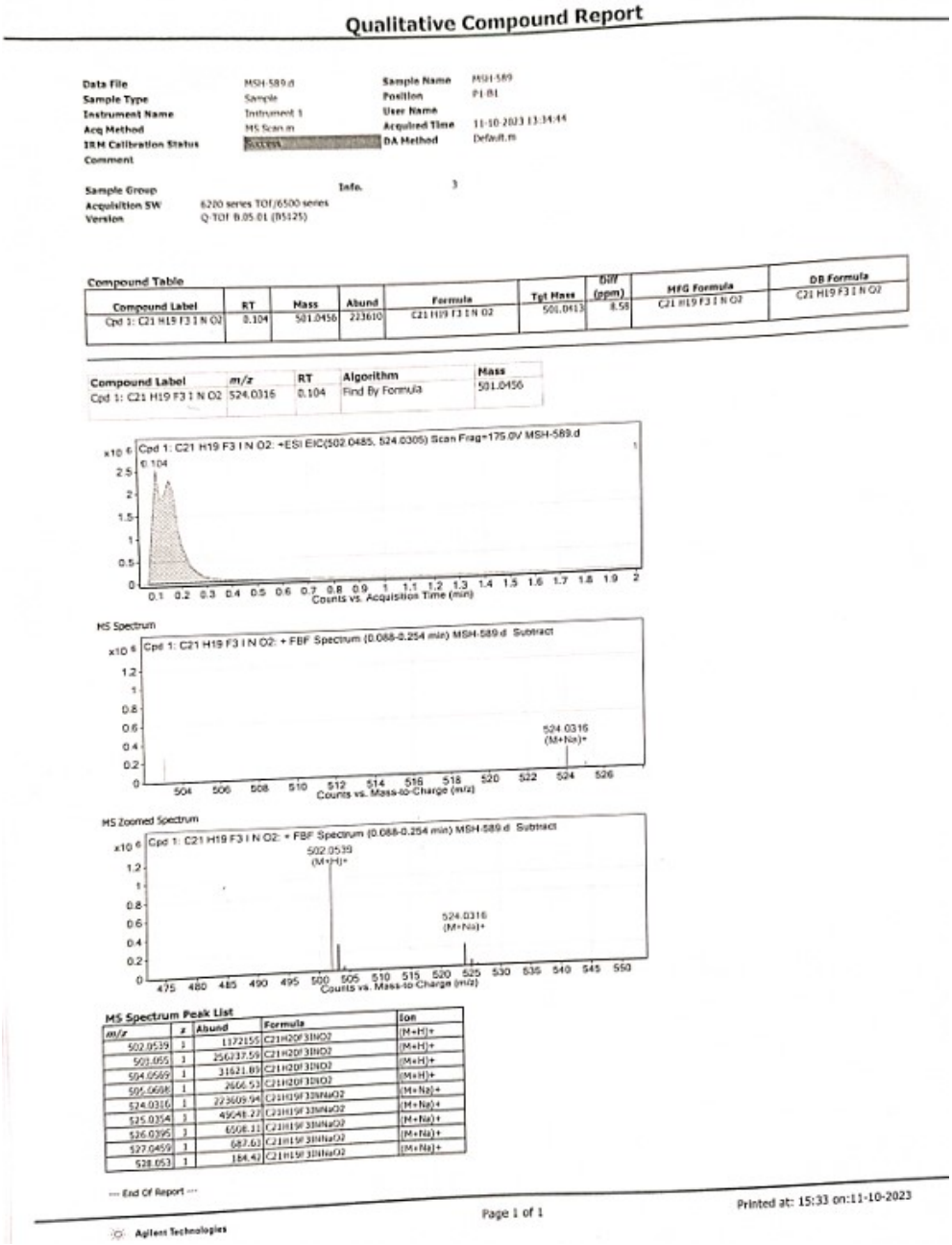
2-(4-hydroxybutyl)-4-iodo-3-(*p*-tolyl)-7-(trifluoromethyl)isoquinolin-1(2H)-one (5d)



HRMS



2-(4-hydroxybutyl)-4-iodo-3-(p-tolyl)-7-(trifluoromethyl)isoquinolin-1(2H)-one (5d)



Qualitative Compound Report

Data File MSH-589.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method MS Scan.m
IRM Calibration Status Success
Comment

Sample Name MSH-589
Position P1-B1
User Name
Acquired Time 11-10-2023 13:34:44
DA Method Default.m

Sample Group Info. 3
Acquisition SW Version 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C21 H19 F3 I N O2	0.104	501.0456	223610	C21 H19 F3 I N O2	501.0413	8.58	C21 H19 F3 I N O2	C21 H19 F3 I N O2

Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C21 H19 F3 I N O2	524.0316	0.104	Find By Formula	501.0456

MS Spectrum

MS Zoomed Spectrum

MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
502.0539	1	1372155	C21H20F3INO2	(M+H)+
503.055	1	256737.59	C21H20F3INO2	(M+H)+
504.0569	1	31621.89	C21H20F3INO2	(M+H)+
505.0608	1	2666.53	C21H20F3INO2	(M+H)+
524.0316	1	223609.94	C21H19F3INNaO2	(M+Na)+
525.0354	1	49048.27	C21H19F3INNaO2	(M+Na)+
526.0395	1	6508.31	C21H19F3INNaO2	(M+Na)+
527.0459	1	687.63	C21H19F3INNaO2	(M+Na)+
528.053	1	184.42	C21H19F3INNaO2	(M+Na)+

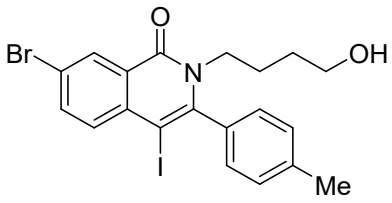
--- End Of Report ---

Agilent Technologies

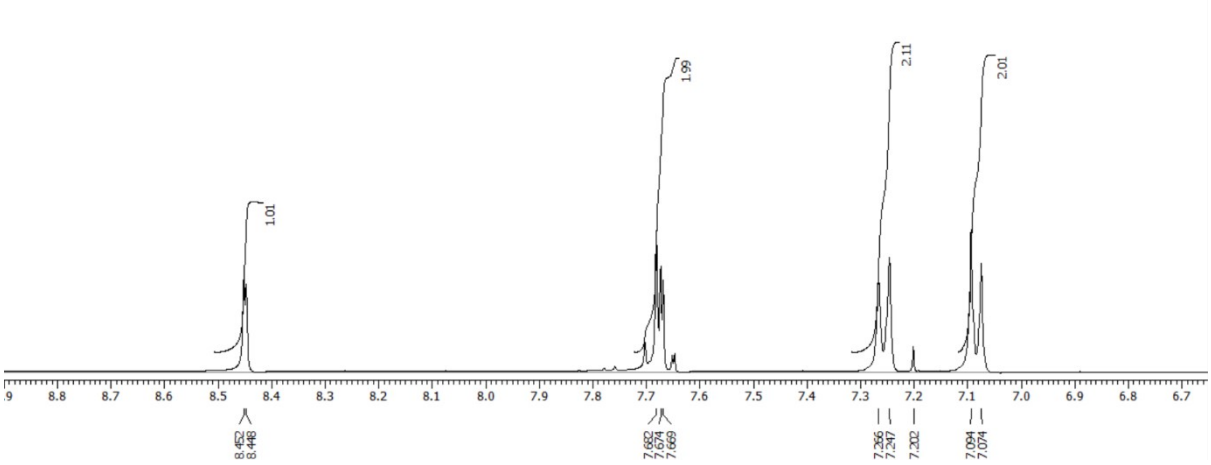
Page 1 of 1

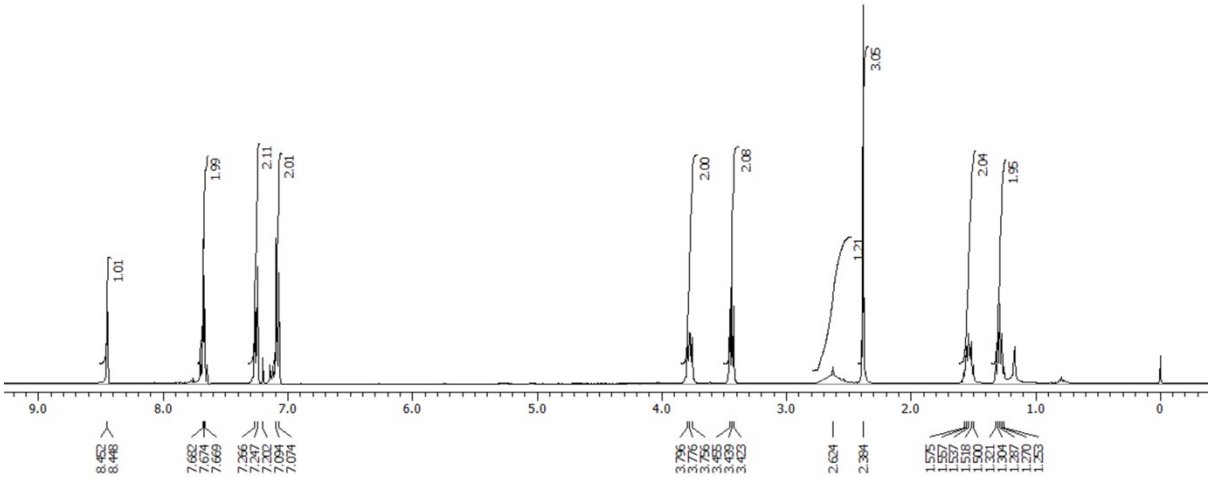
Printed at: 15:33 on:11-10-2023

^{1}H NMR (400 MHz, $CDCl_3$)

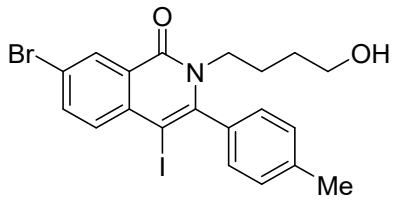


7-bromo-2-(4-hydroxybutyl)-4-iodo-3-(*p*-tolyl)isoquinolin-1(2H)-one (5e)

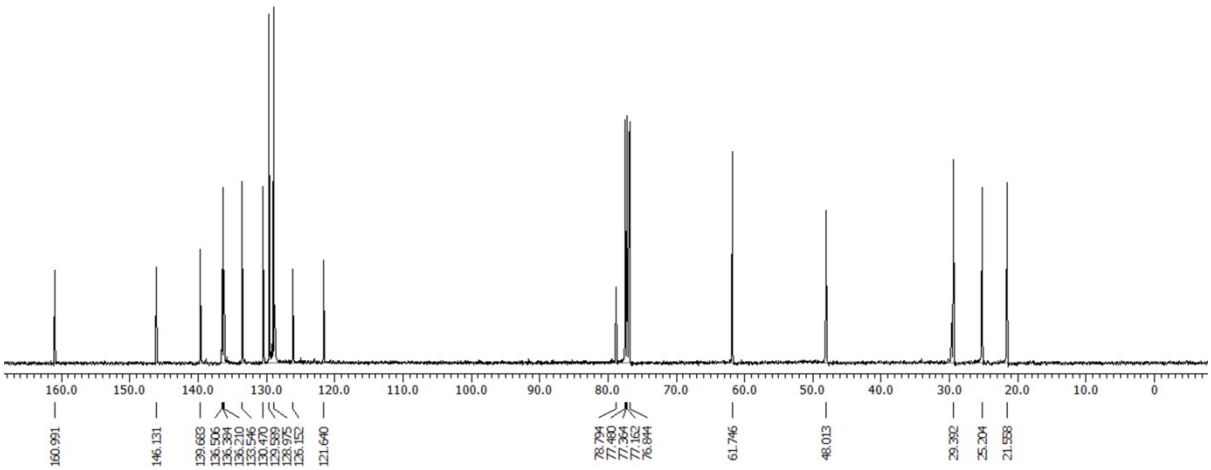




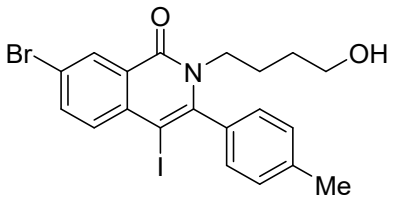
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



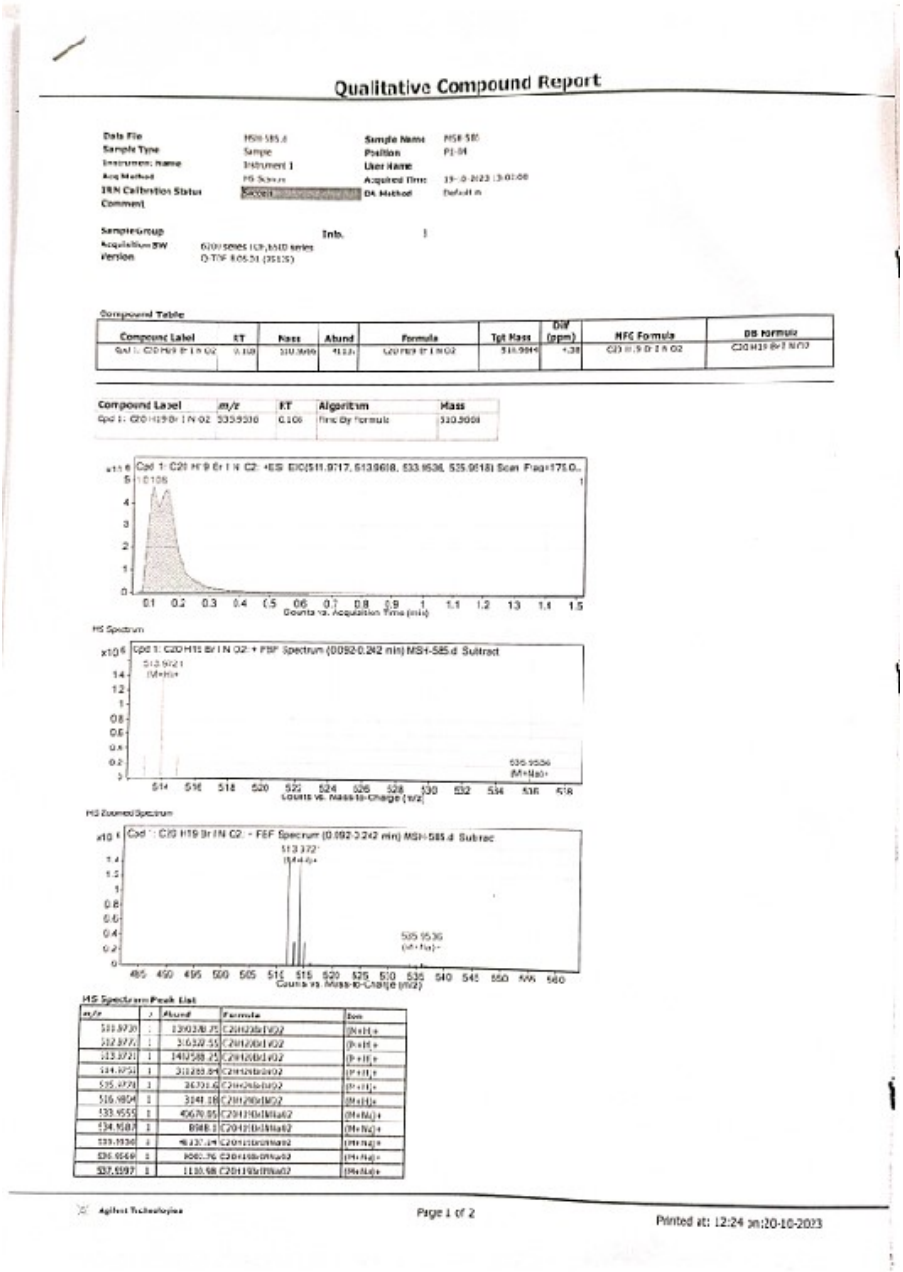
7-bromo-2-(4-hydroxybutyl)-4-iodo-3-(*p*-tolyl)isoquinolin-1(2H)-one (5e)



HRMS



7-bromo-2-(4-hydroxybutyl)-4-iodo-3-(*p*-tolyl)isoquinolin-1(2H)-one (5e)


Qualitative Compound Report

Data File MSH-585.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method MS Scan
IRM Calibration Status Success
Comment

Sample Name MSH-585
Position P1-D4
User Name
Acquired Time 19-10-2023 13:01:00
DA Method Default.m

SampleGroup
Acquisition SW Version 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)
Info. 1

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFC Formula	DB Formula
Cpd 1: C20 H19 Br I N O2	0.106	510.9959	4133	C20 H19 Br I N O2	510.9964	-1.28	C20 H19 Br I N O2	C20 H19 Br I N O2

Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C20 H19 Br I N O2	533.9930	0.106	Find By Formula	510.9959

MS Spectrum

MS Zoomed Spectrum

MS Spectrum Peak List

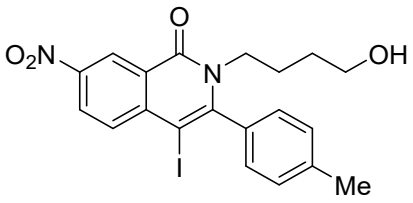
m/z	z	Abund	Formula	Ion
513.9720	1	139038.75	C20H20BrINO2	(M+H)+
512.9771	1	31632.55	C20H19BrINO2	(M+H)+
513.9721	1	140588.25	C20H19BrINO2	(M+H)+
514.9753	1	31289.84	C20H19BrINO2	(M+H)+
535.9570	1	24731.62	C20H18BrINNaO2	(M+Na)+
516.9804	1	3241.18	C20H19BrINO2	(M+H)+
533.9550	1	40670.95	C20H18BrINNaO2	(M+Na)+
534.9582	1	8988.1	C20H18BrINNaO2	(M+Na)+
535.9536	1	40237.09	C20H18BrINNaO2	(M+Na)+
536.9569	1	8981.76	C20H18BrINNaO2	(M+Na)+
537.9597	1	1110.98	C20H18BrINNaO2	(M+Na)+

Agilent Technologies

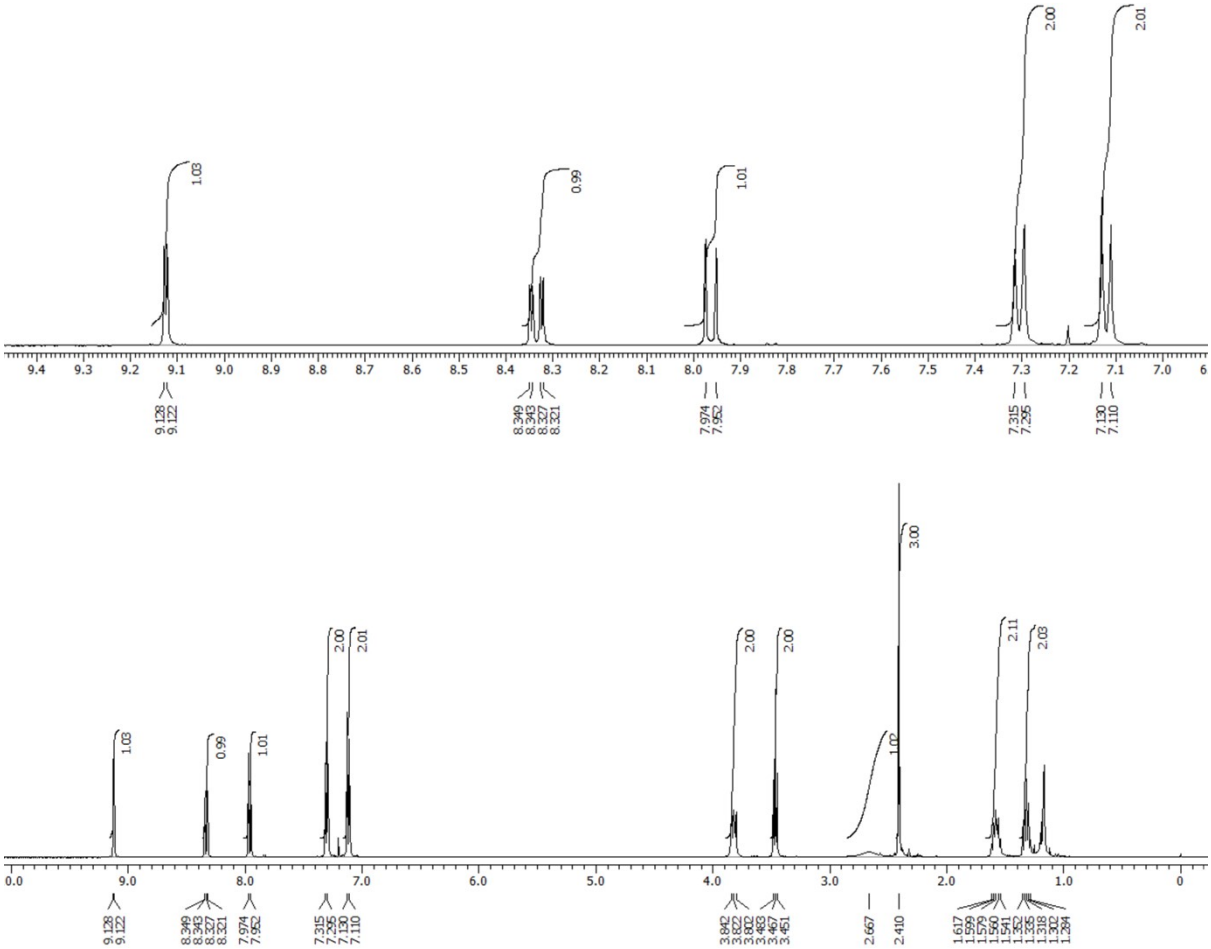
Page 1 of 2

Printed at: 12:24 on:20-10-2023

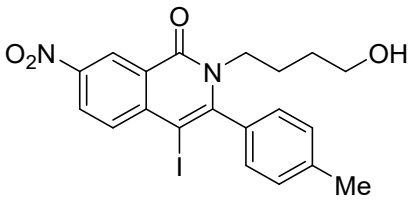
^{1}H NMR (400 MHz, $CDCl_3$)



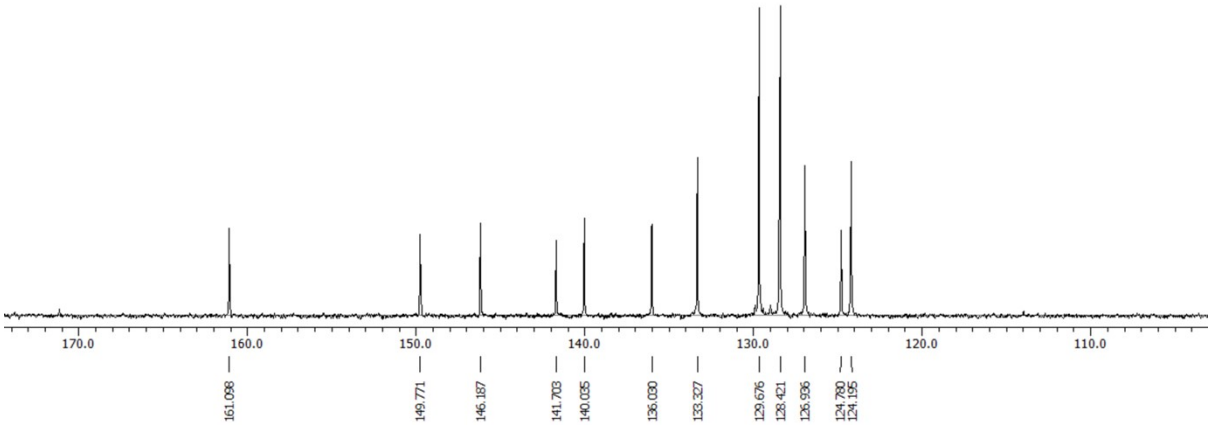
2-(4-hydroxybutyl)-4-iodo-7-nitro-3-(*p*-tolyl)isoquinolin-1(2H)-one (5f)

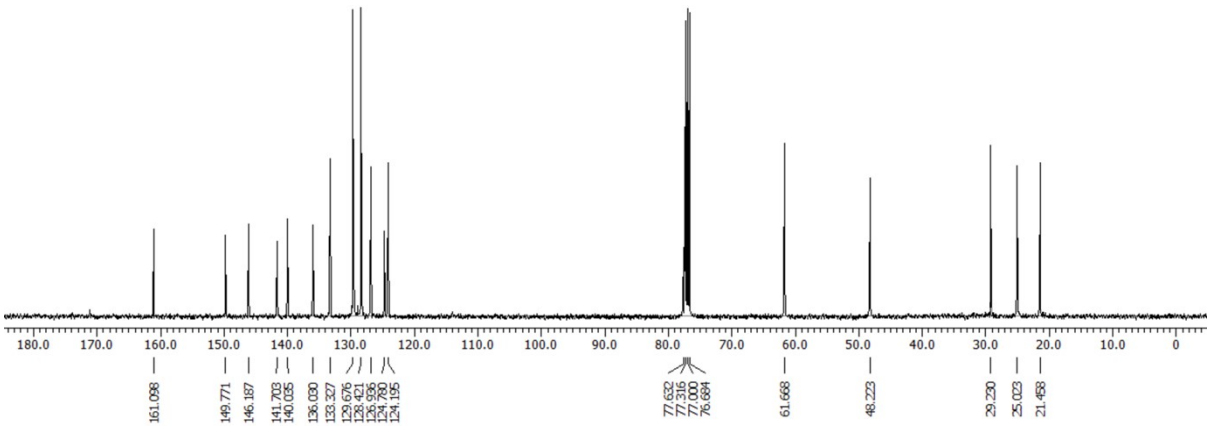


^{13}C {1H} NMR (100 MHz, $CDCl_3$)

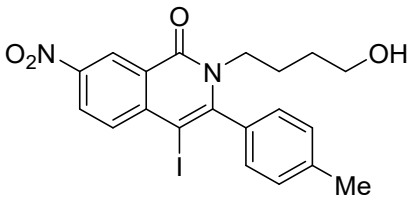


2-(4-hydroxybutyl)-4-iodo-7-nitro-3-(*p*-tolyl)isoquinolin-1(2H)-one (5f)





HRMS



2-(4-hydroxybutyl)-4-iodo-7-nitro-3-(*p*-tolyl)isoquinolin-1(2H)-one (5f)

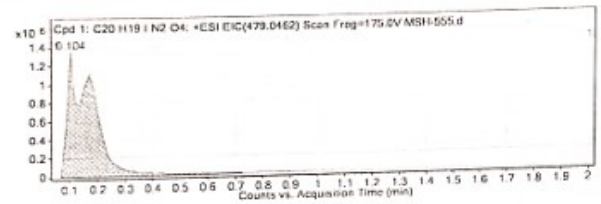
Qualitative Compound Report

Data File	MSH-555.d	Sample Name	MSH-555
Sample Type	Sample	Position	P1-B1
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	21-08-2023 13:38:21
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (85125)		

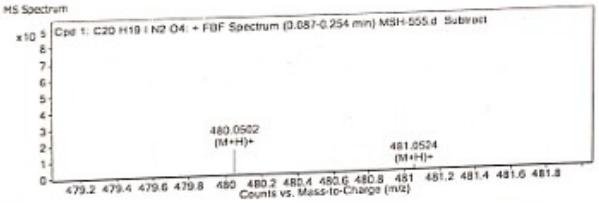
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C20 H19 I N2 O4	0.104	478.0411	725136	C20 H19 I N2 O4	478.039	4.41	C20 H19 I N2 O4	C20 H19 I N2 O4

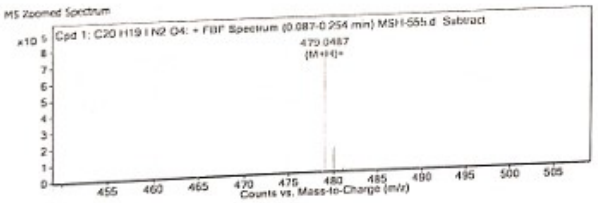
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C20 H19 I N2 O4	479.0487	0.104	Find By Formula	478.0411



MS Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
479.0487	1	725136.19	C20H20IN2O4	(M+H)+
480.0502	1	158009.52	C20H20IN2O4	(M+H)+
481.0524	1	21650.11	C20H20IN2O4	(M+H)+
482.0543	1	2280.99	C20H20IN2O4	(M+H)+

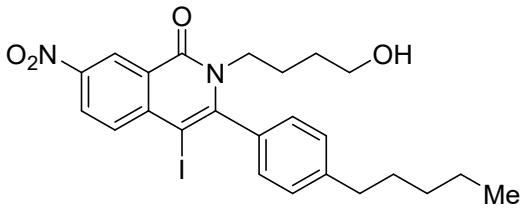
--- End Of Report ---

Agilent Technologies

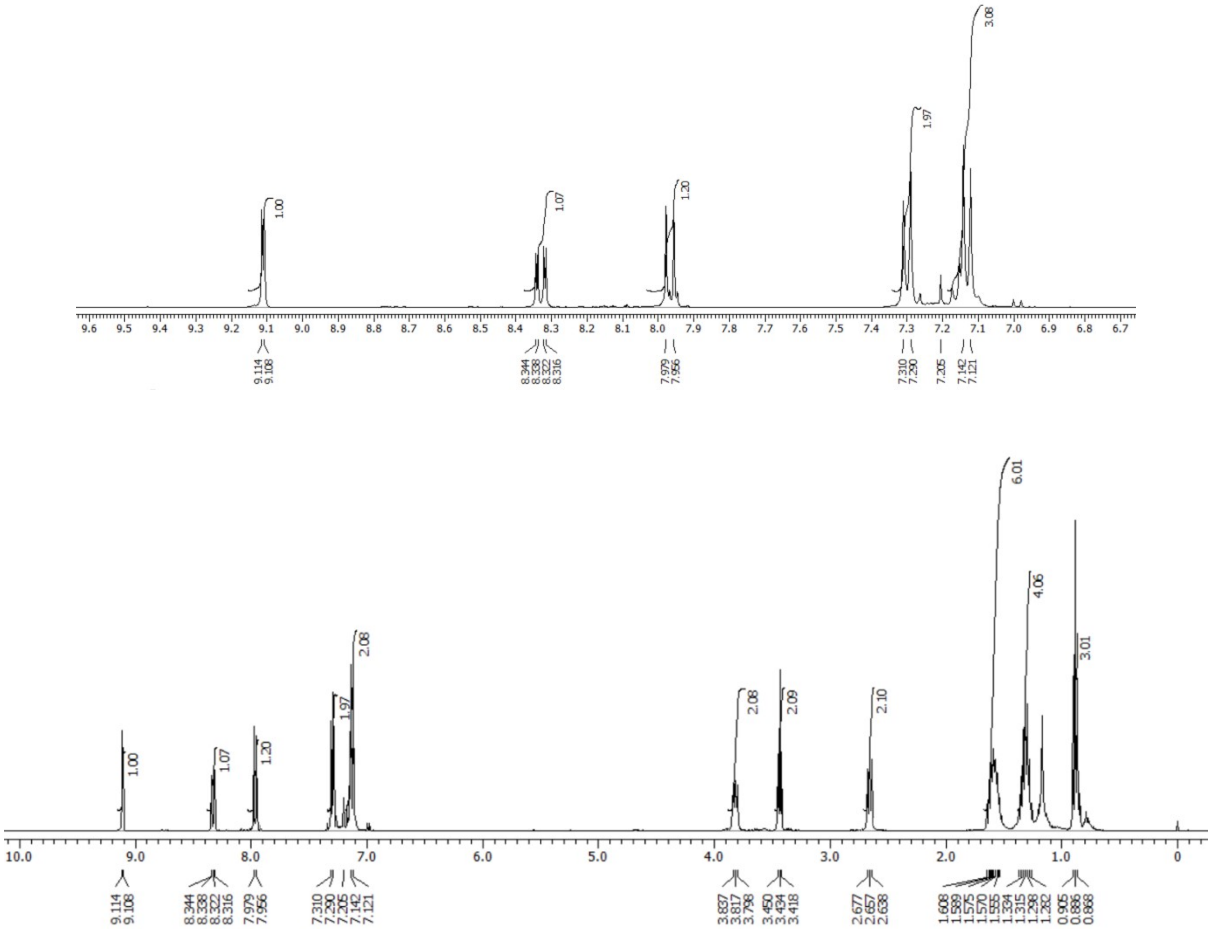
Page 1 of 1

Printed at: 16:30 on:21-08-2023

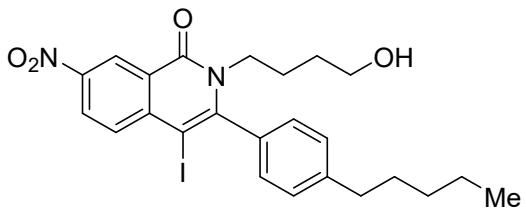
H NMR (400 MHz, $CDCl_3$)



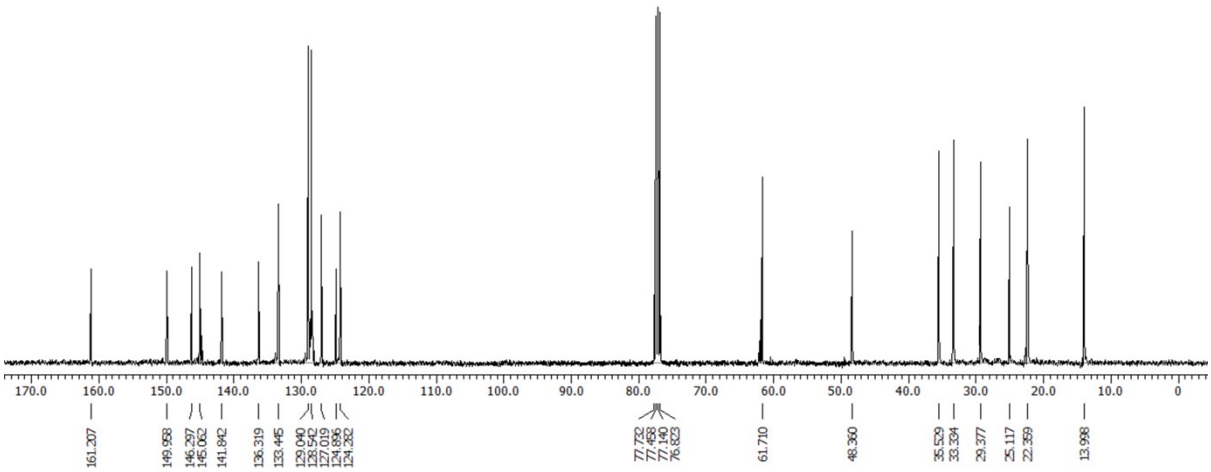
2-(4-hydroxybutyl)-4-iodo-7-nitro-3-(4-pentylphenyl)isoquinolin-1(2H)-one (5g)



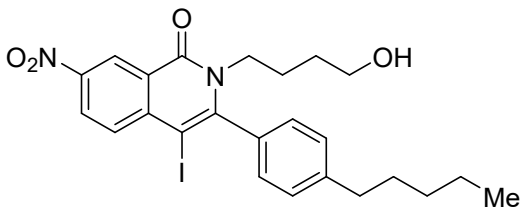
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



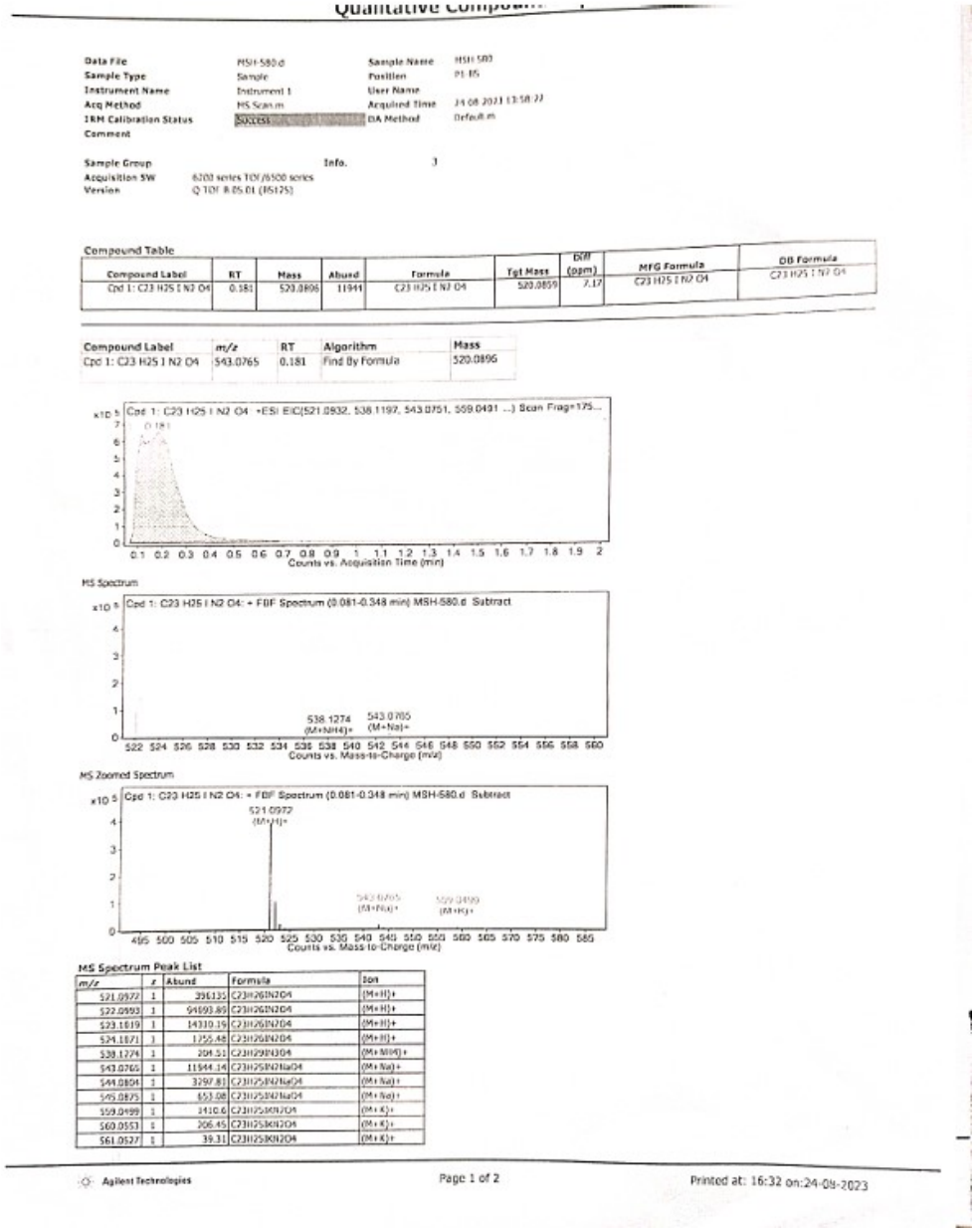
2-(4-hydroxybutyl)-4-iodo-7-nitro-3-(4-pentylphenyl)isoquinolin-1(2H)-one (5g)



HRMS



2-(4-hydroxybutyl)-4-iodo-7-nitro-3-(4-pentylphenyl)isoquinolin-1(2H)-one (5g)



Qualitative Compound...

Data File	MSH-580.d	Sample Name	MSH 580
Sample Type	Sample	Position	P1-B5
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	24.08.2023 13:58:22
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C23 H25 I N2 O4	0.181	520.0896	11944	C23 H25 I N2 O4	520.0859	7.17	C23 H25 I N2 O4	C23 H25 I N2 O4

Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C23 H25 I N2 O4	543.0765	0.181	Find By Formula	520.0896

MS Spectrum

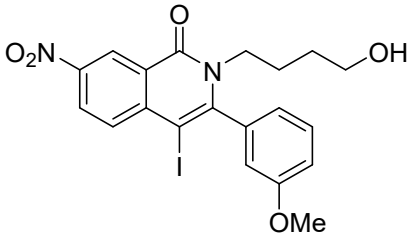
MS Zoomed Spectrum

MS Spectrum Peak List

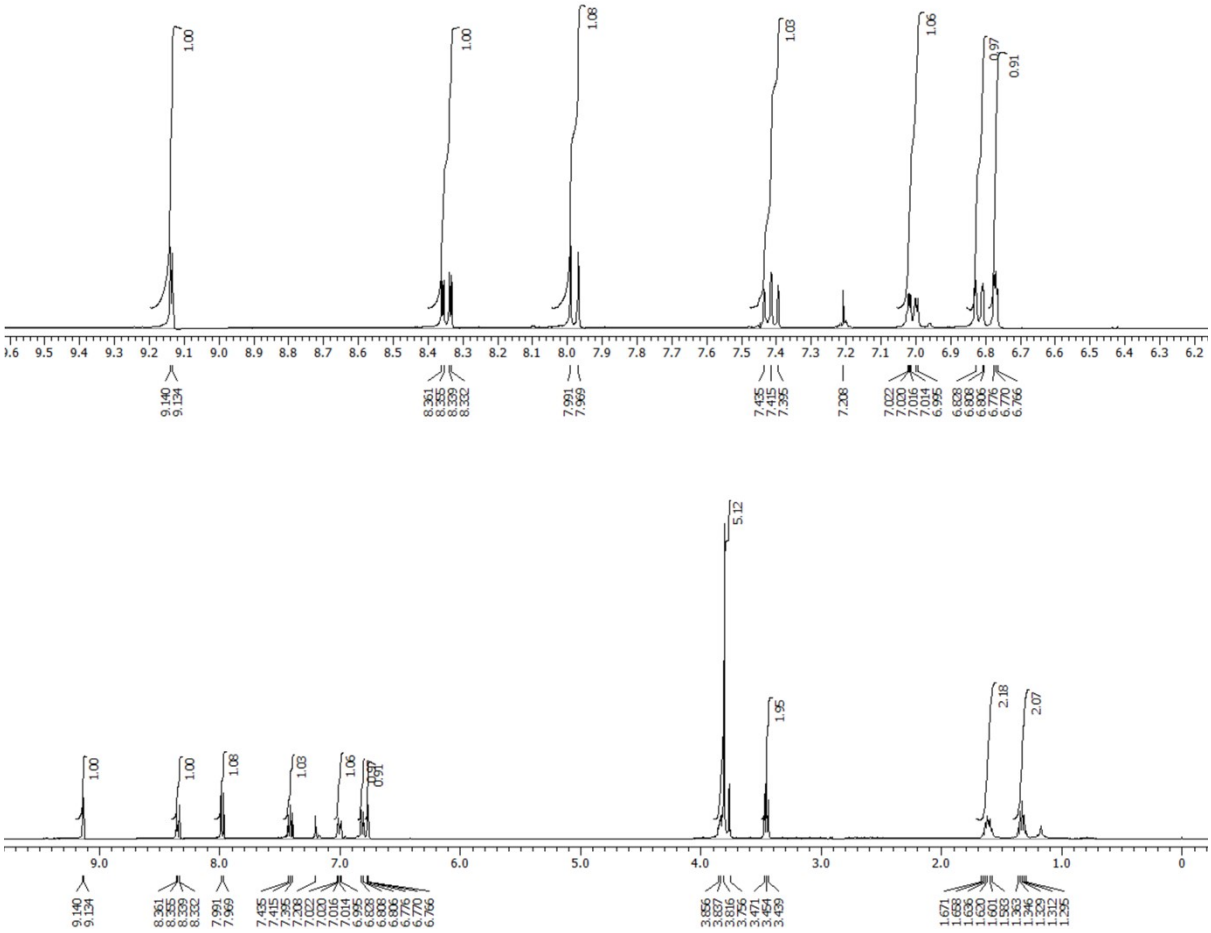
m/z	z	Abund	Formula	Ion
521.0972	1	396135	C23H26IN2O4	(M+H)+
522.0993	1	94693.89	C23H26IN2O4	(M+H)+
523.1019	1	14310.19	C23H26IN2O4	(M+H)+
524.1071	1	1755.48	C23H26IN2O4	(M+H)+
538.1274	1	204.51	C23H29IN3O4	(M+NH4)+
543.0765	1	11944.14	C23H25IN2NaO4	(M+Na)+
544.0804	1	3297.81	C23H25IN2NaO4	(M+Na)+
545.0875	1	653.08	C23H25IN2NaO4	(M+Na)+
559.0499	1	3410.6	C23H25IKN2O4	(M+K)+
560.0553	1	706.45	C23H25IKN2O4	(M+K)+
561.0527	1	39.31	C23H25IKN2O4	(M+K)+

Agilent Technologies Page 1 of 2 Printed at: 16:32 on:24-08-2023

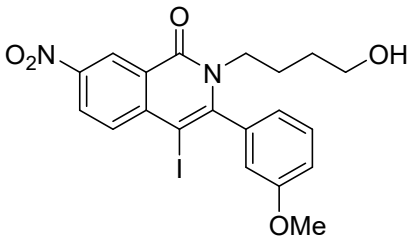
^{1}H NMR (400 MHz, $CDCl_3$)



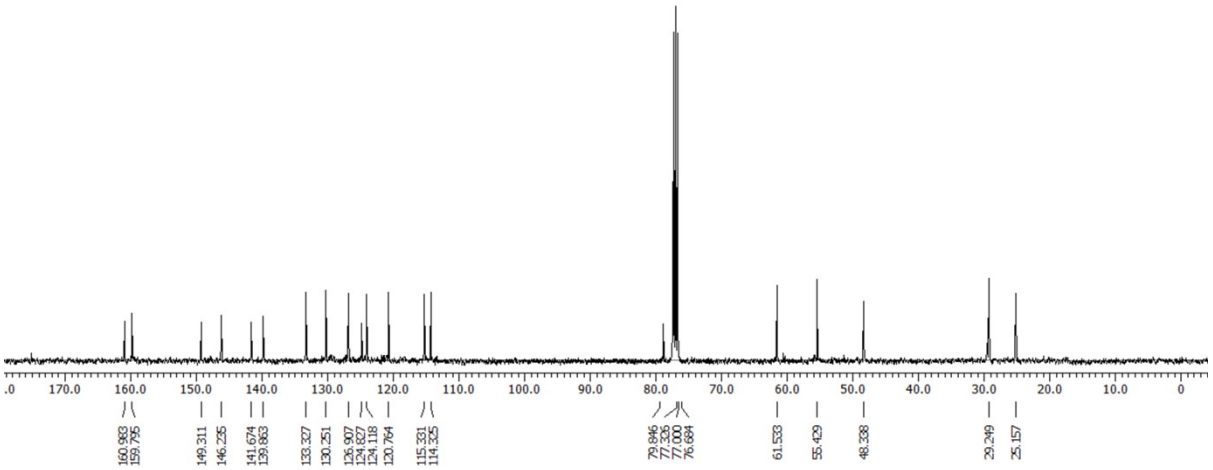
2-(4-hydroxybutyl)-4-iodo-3-(3-methoxyphenyl)-7-nitroisoquinolin-1(2H)-one (5h)



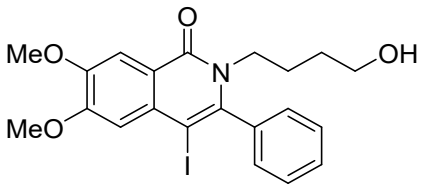
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



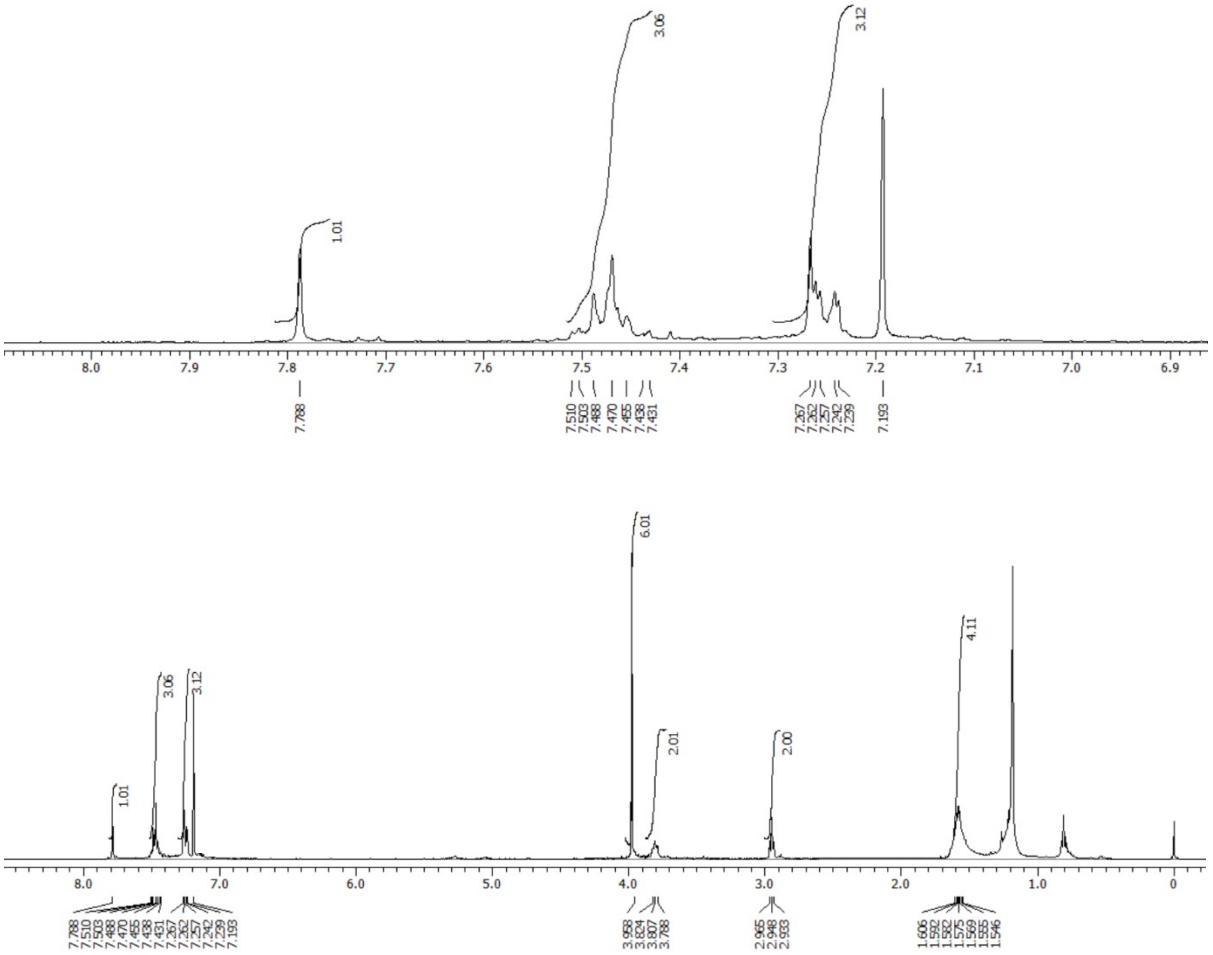
2-(4-hydroxybutyl)-4-iodo-3-(3-methoxyphenyl)-7-nitroisoquinolin-1(2H)-one (5h)



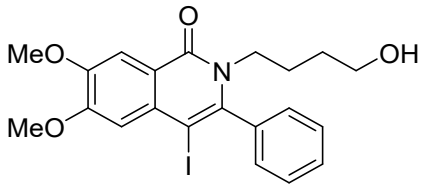
^{1}H NMR (400 MHz, $CDCl_3$)



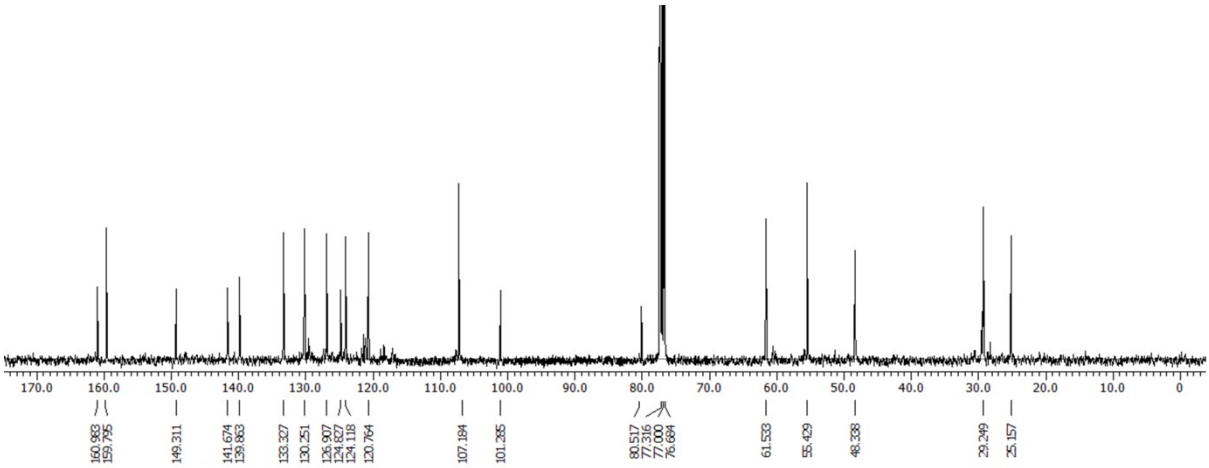
2-(4-hydroxybutyl)-4-iodo-6,7-dimethoxy-3-phenylisoquinolin-1(2H)-one (5i)



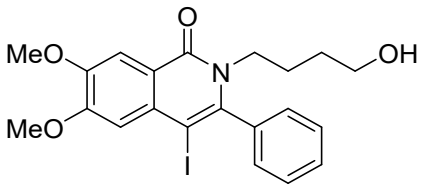
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



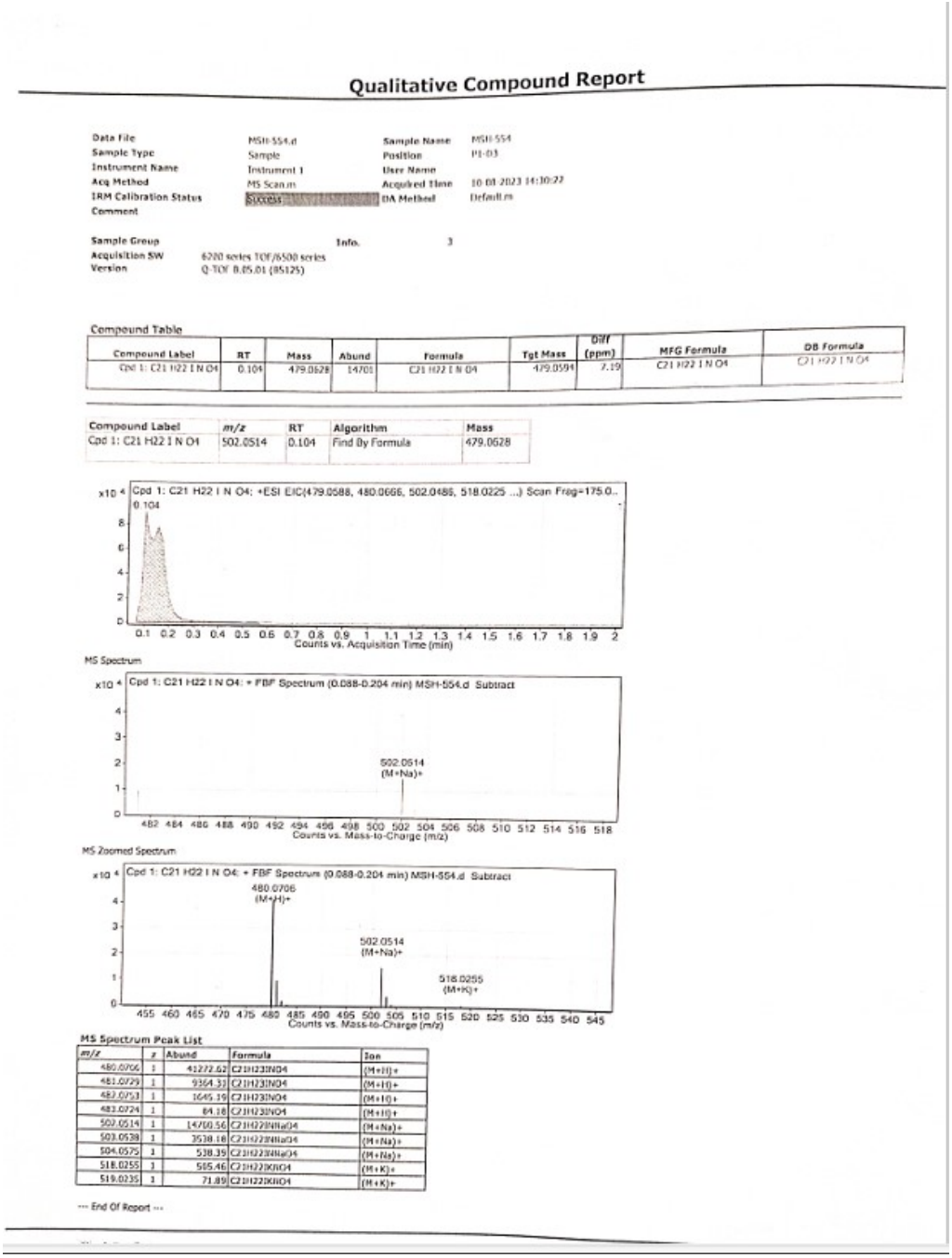
2-(4-hydroxybutyl)-4-iodo-6,7-dimethoxy-3-phenylisoquinolin-1(2H)-one (5i)



HRMS



2-(4-hydroxybutyl)-4-iodo-6,7-dimethoxy-3-phenylisoquinolin-1(2H)-one (5i)



Qualitative Compound Report

Data File	MSH-554.d	Sample Name	MSH-554
Sample Type	Sample	Position	P1-D3
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	10.03.2023 14:30:22
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C21 H22 I N O4	0.104	479.0628	14701	C21 H22 I N O4	479.0594	7.19	C21 H22 I N O4	C21 H22 I N O4

Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C21 H22 I N O4	502.0514	0.104	Find By Formula	479.0628

MS Spectrum

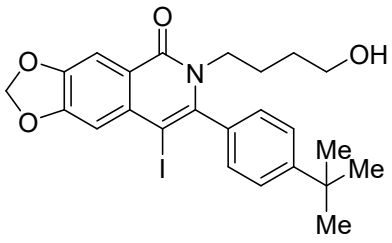
MS Zoomed Spectrum

MS Spectrum Peak List

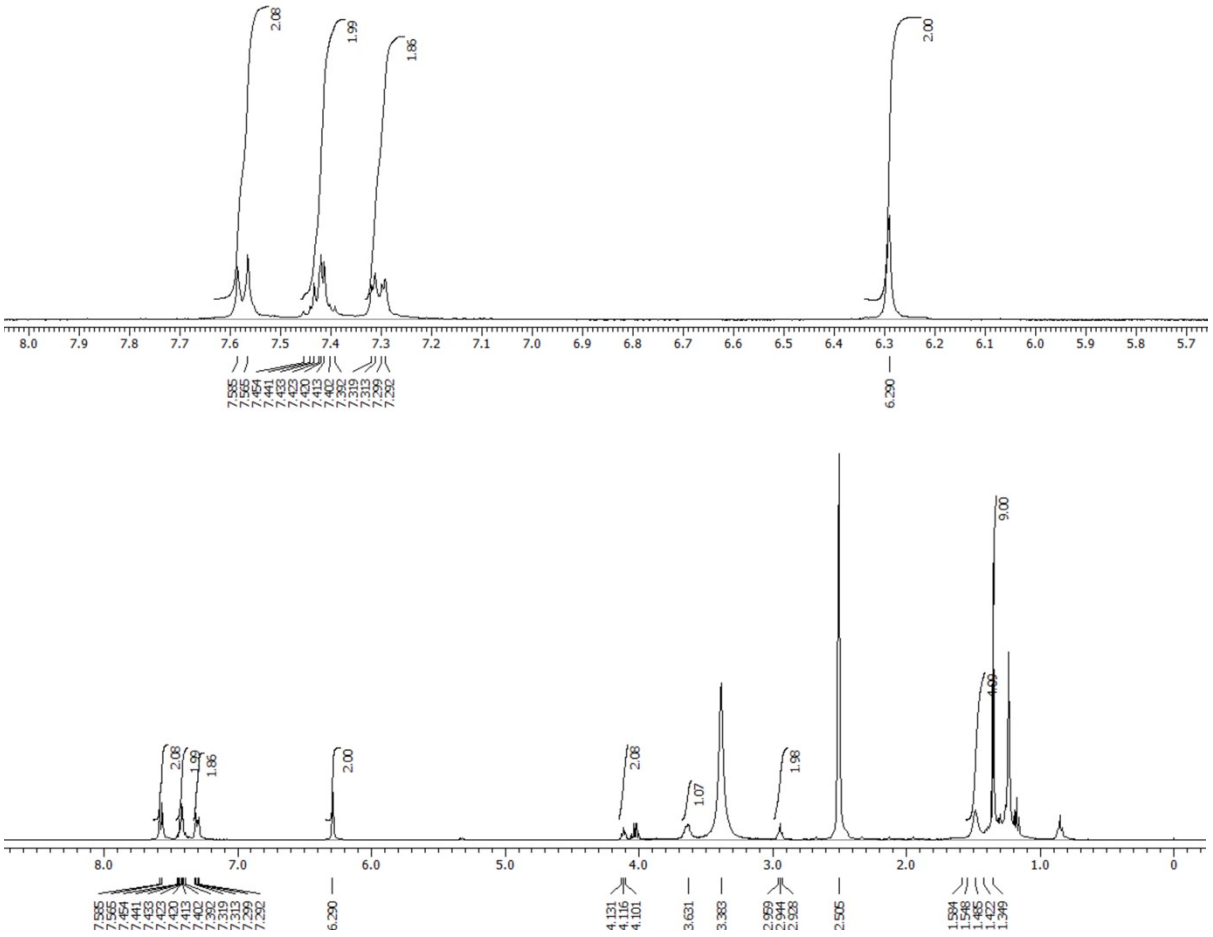
m/z	z	Abund	Formula	Ion
480.0706	1	41272.62	C21H23INO4	(M+H)+
481.0729	1	9364.33	C21H23INO4	(M+H)+
482.0753	1	1645.19	C21H23INO4	(M+H)+
483.0724	1	84.18	C21H23INO4	(M+H)+
502.0514	1	14700.56	C21H22INNaO4	(M+Na)+
503.0538	1	3538.18	C21H22INNaO4	(M+Na)+
504.0575	1	538.39	C21H22INNaO4	(M+Na)+
518.0255	1	585.46	C21H22IKNO4	(M+K)+
519.0235	1	71.89	C21H22IKNO4	(M+K)+

--- End Of Report ---

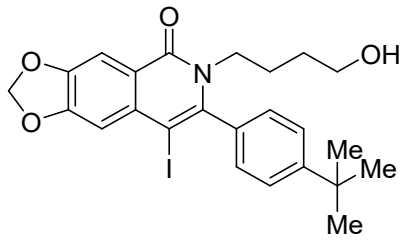
^{1}H NMR (400 MHz, $CDCl_3$)



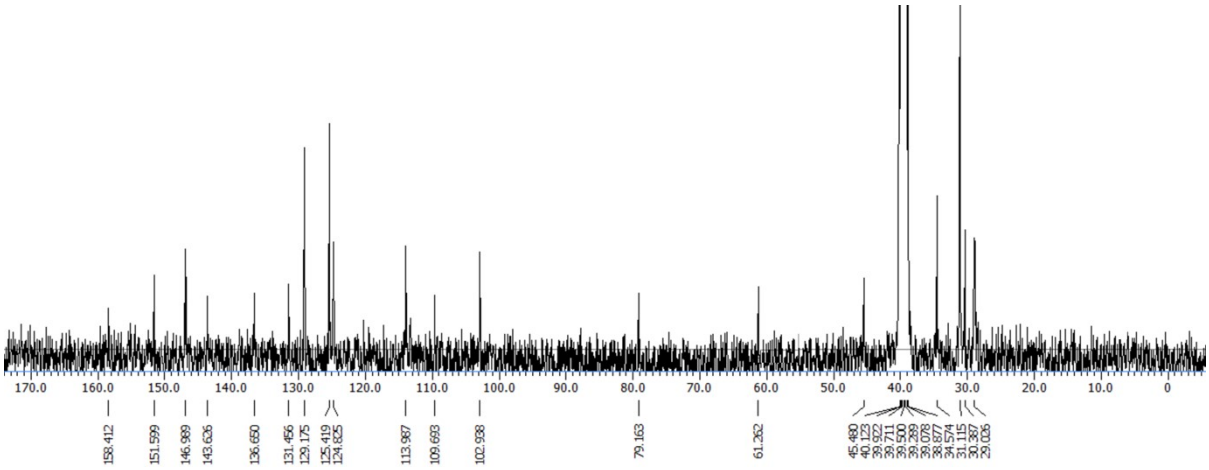
7-(4-(tert-butyl)phenyl)-6-(4-hydroxybutyl)-8-iodo-[1,3]dioxolo[4,5-g]isoquinolin-5(6H)-one (5j)



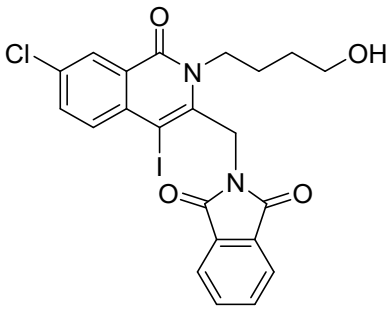
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



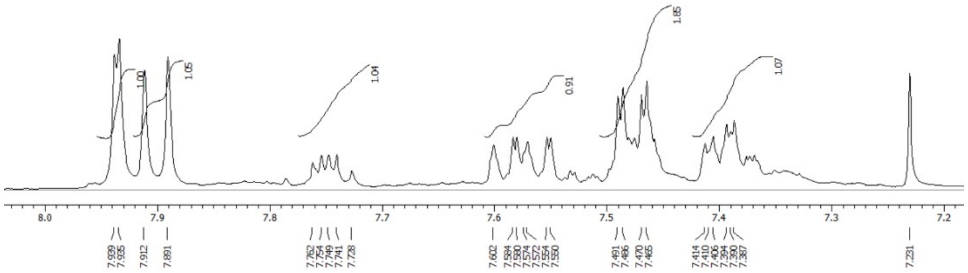
7-(4-(tert-butyl)phenyl)-6-(4-hydroxybutyl)-8-iodo-[1,3]dioxolo[4,5-g]isoquinolin-5(6H)-one (5j)

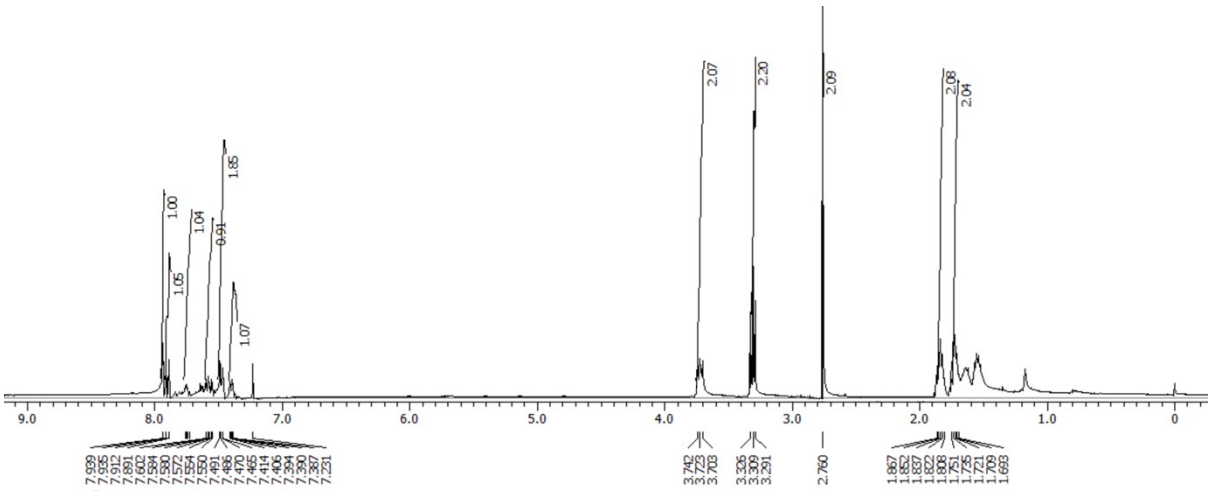


^{1}H NMR (400 MHz, $CDCl_3$)

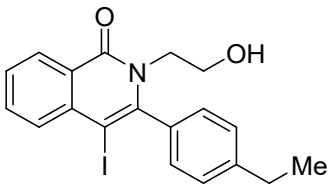


2-((7-chloro-2-(4-hydroxybutyl)-4-iodo-1-oxo-1,2-dihydroisoquinolin-3-yl)methyl)isoindoline-1,3-dione (5k)

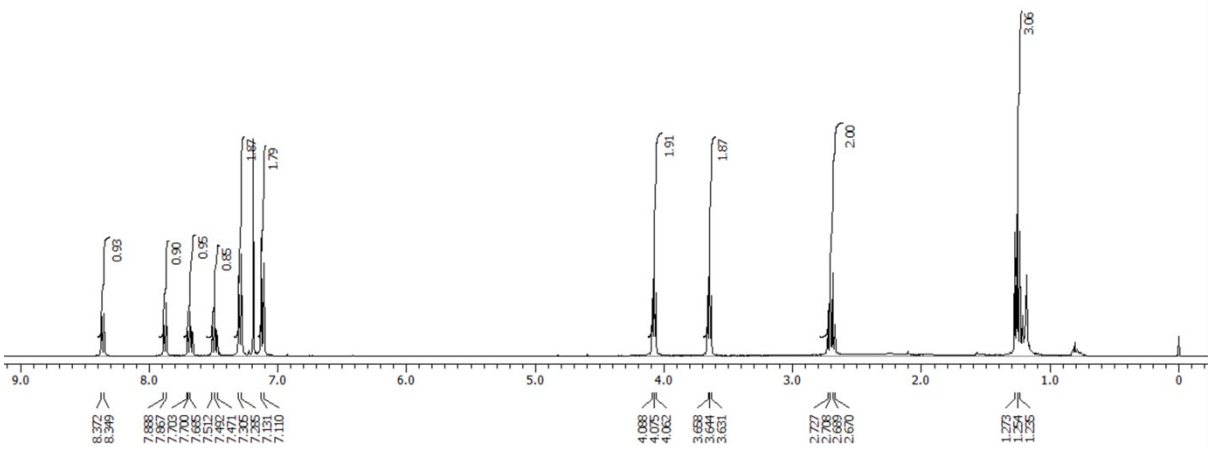




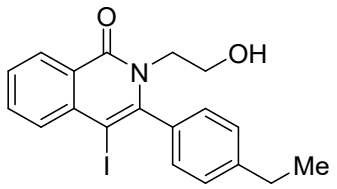
^{1}H NMR (400 MHz, $CDCl_3$)



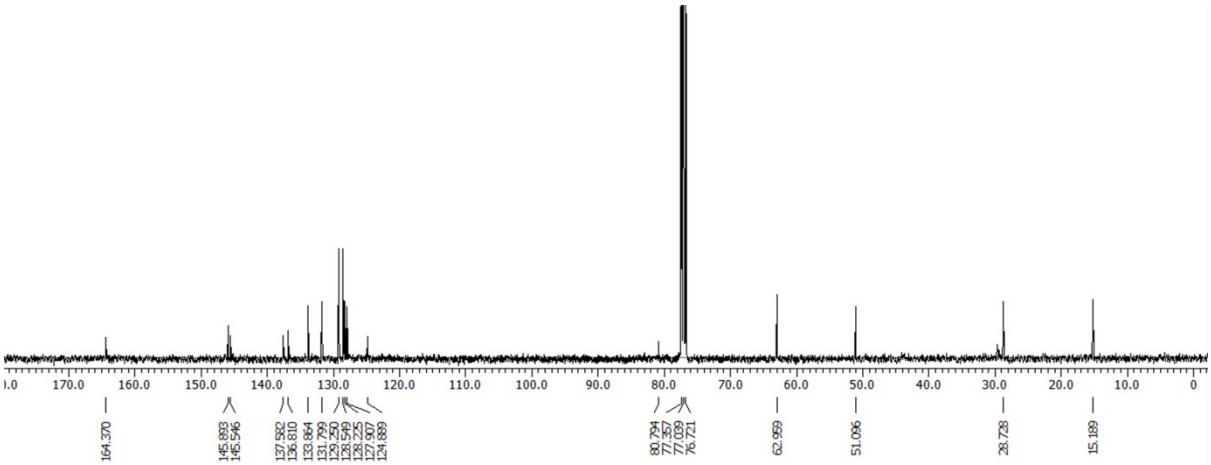
3-(4-ethylphenyl)-2-(2-hydroxyethyl)-4-iodoisoquinolin-1(2H)-one (6a)



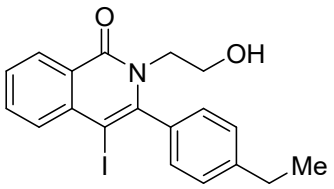
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



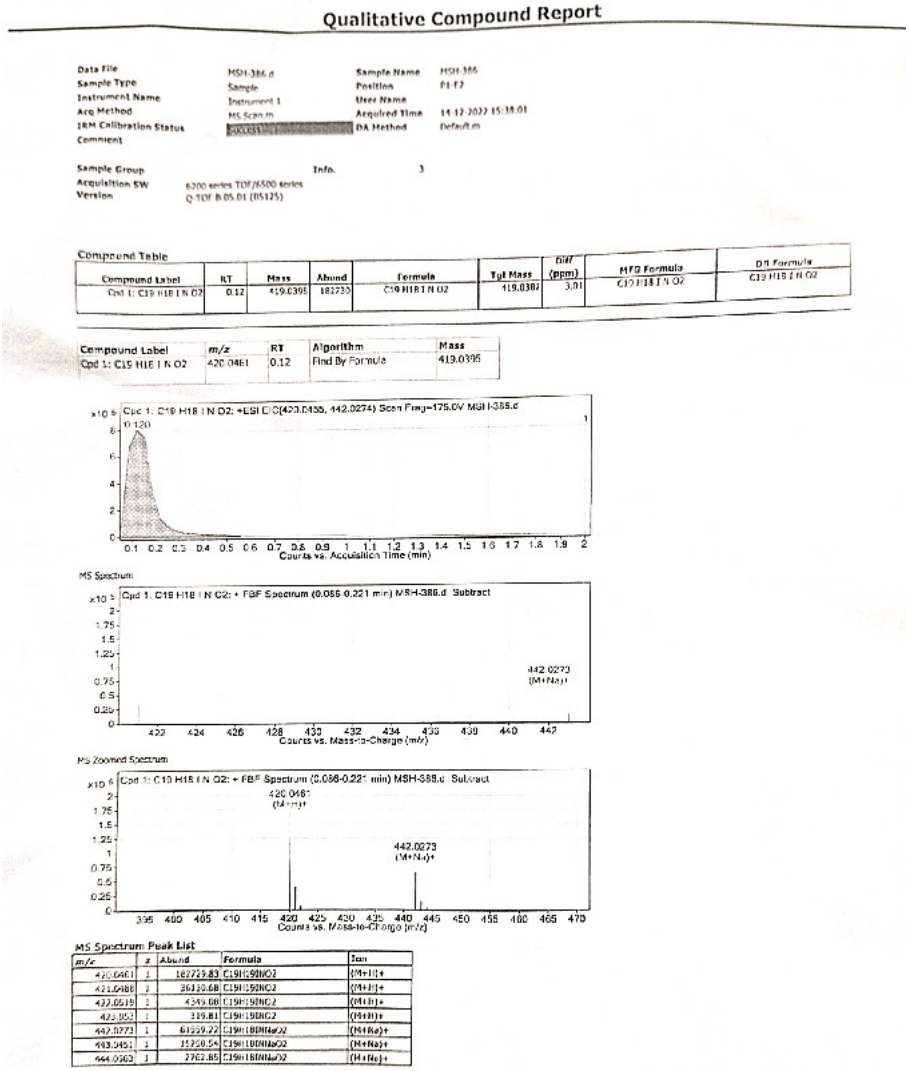
3-(4-ethylphenyl)-2-(2-hydroxyethyl)-4-iodoisoquinolin-1(2H)-one (6a)



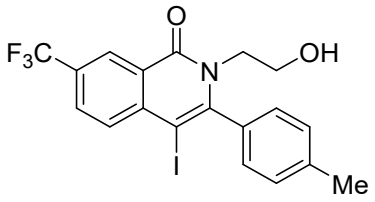
HRMS



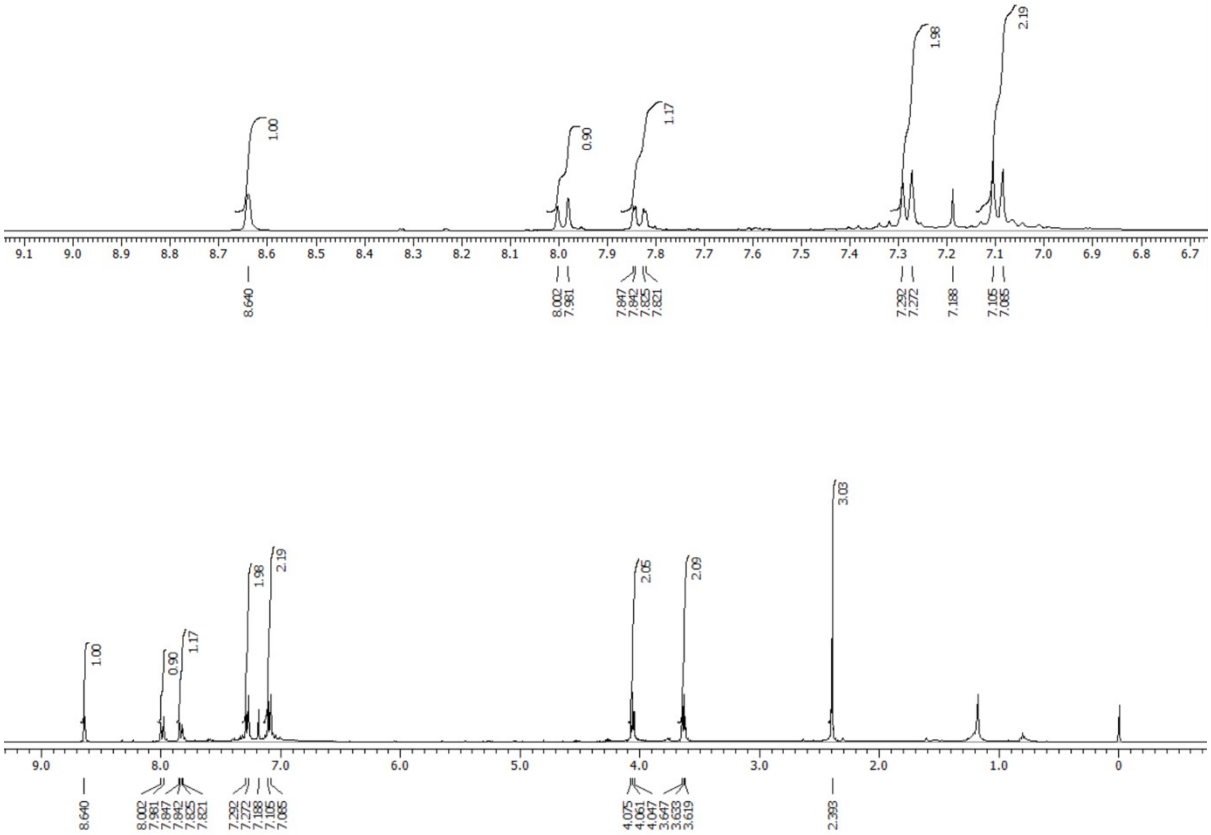
3-(4-ethylphenyl)-2-(2-hydroxyethyl)-4-iodoisoquinolin-1(2H)-one (6a)



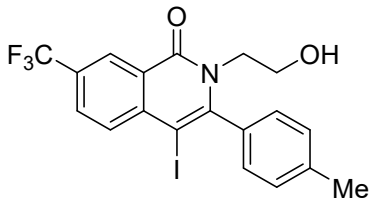
^{1}H NMR



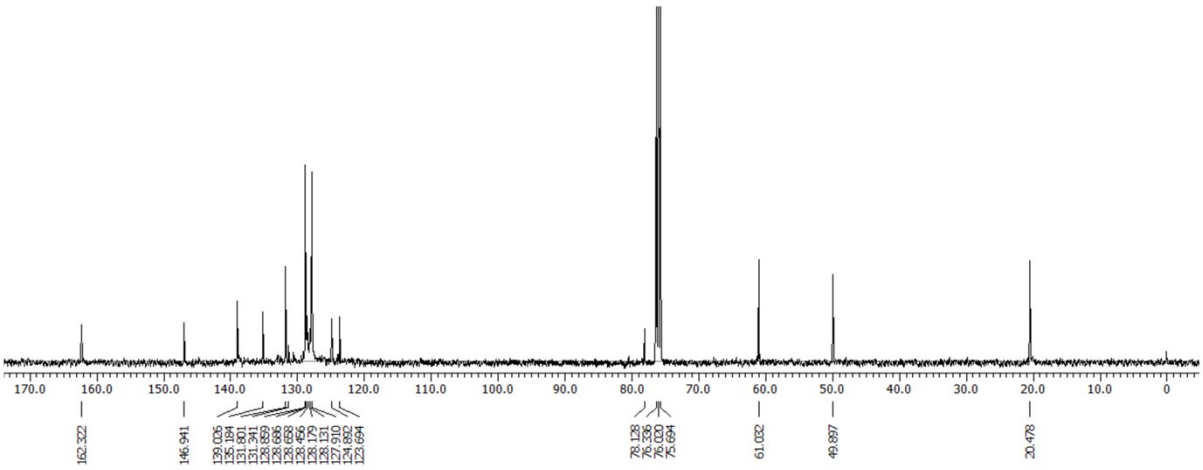
2-(2-hydroxyethyl)-4-iodo-3-(p-tolyl)-7-(trifluoromethyl)isoquinolin-1(2H)-one (6b)



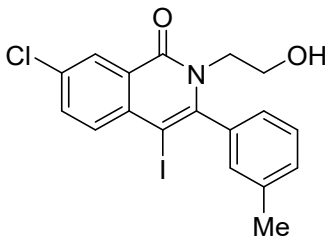
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



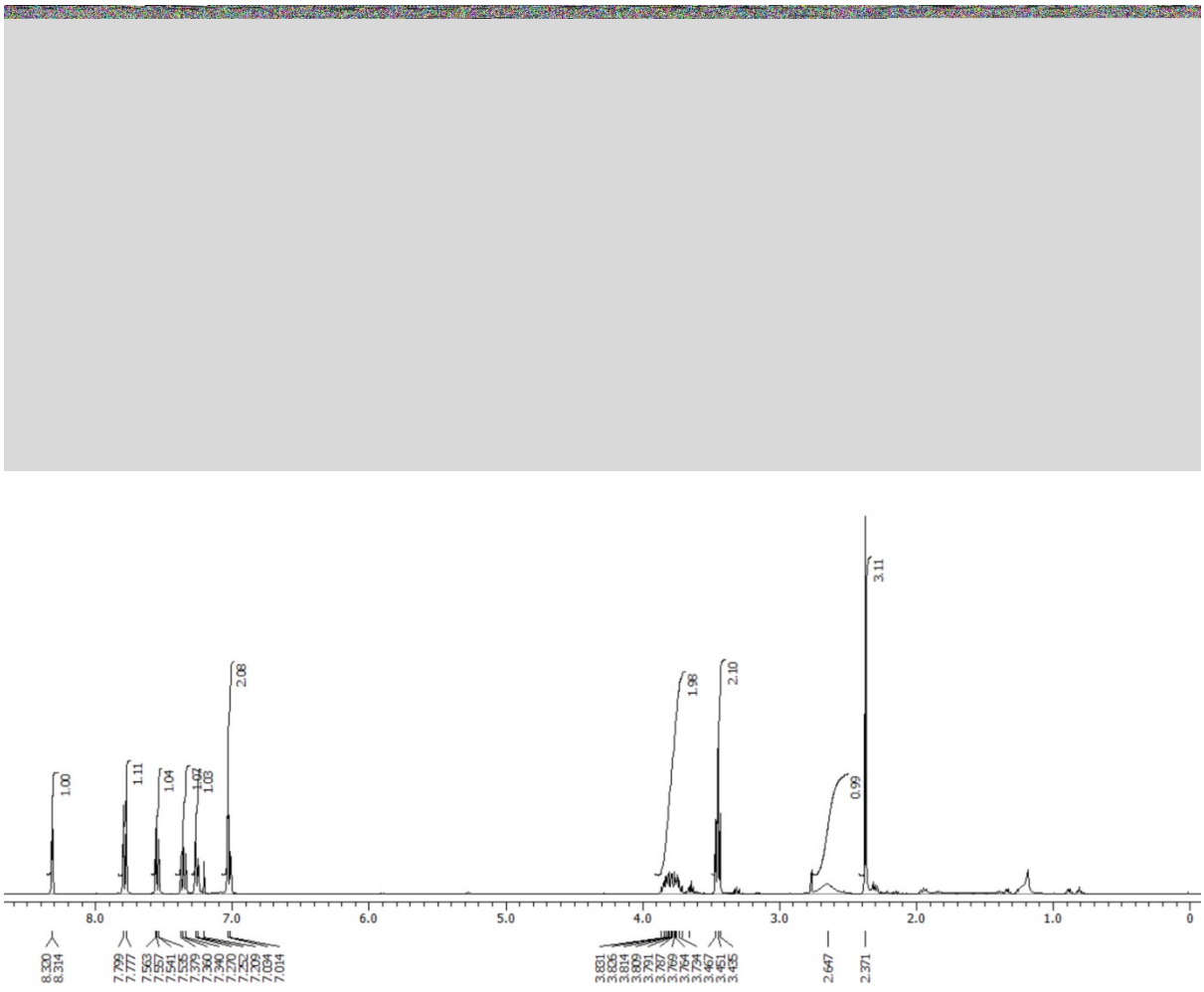
2-(2-hydroxyethyl)-4-iodo-3-(p-tolyl)-7-(trifluoromethyl)isoquinolin-1(2H)-one (6b)



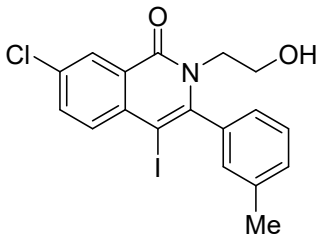
^{1}H NMR



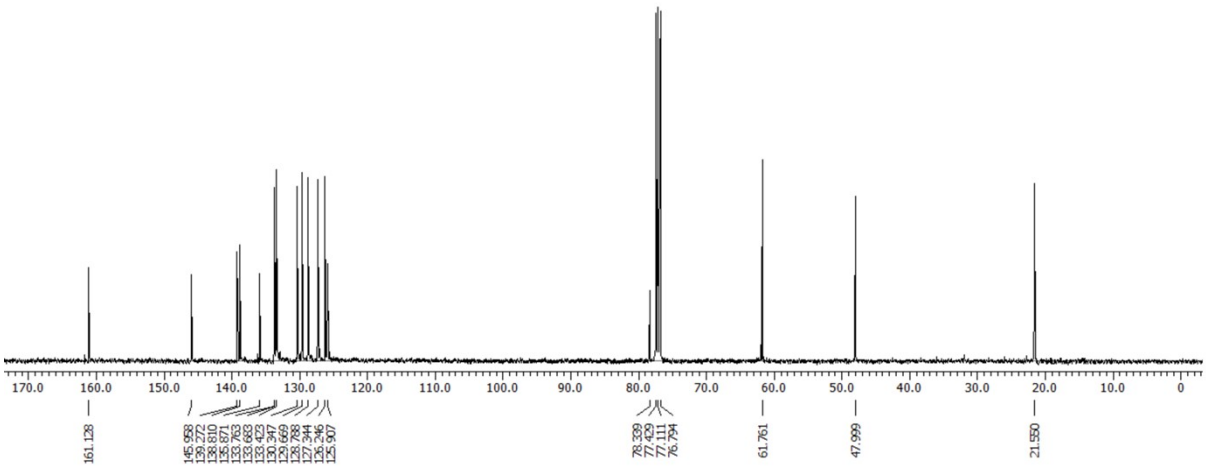
7-chloro-2-(2-hydroxyethyl)-4-iodo-3-(m-tolyl)isoquinolin-1(2H)-one (6c)



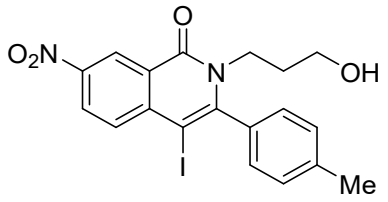
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



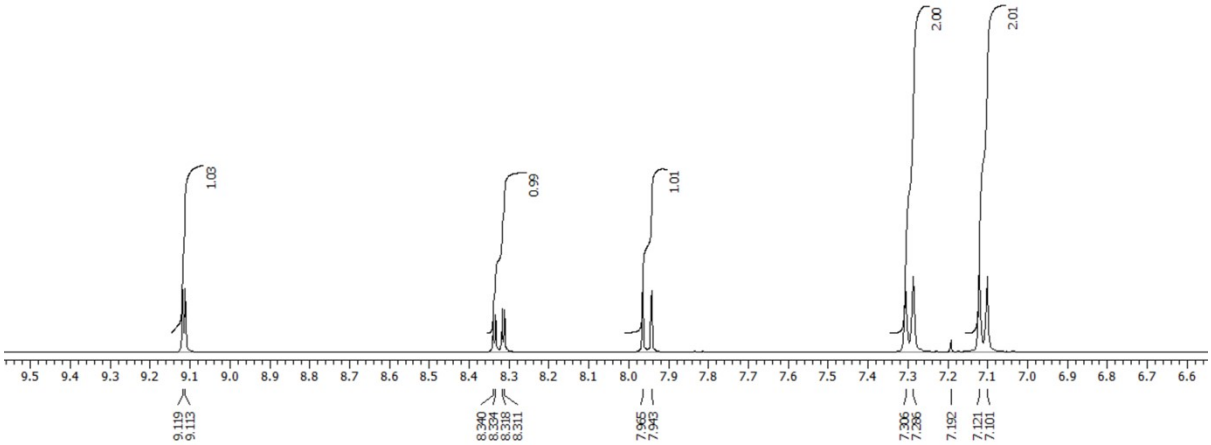
7-chloro-2-(2-hydroxyethyl)-4-iodo-3-(m-tolyl)isoquinolin-1(2H)-one (6c)

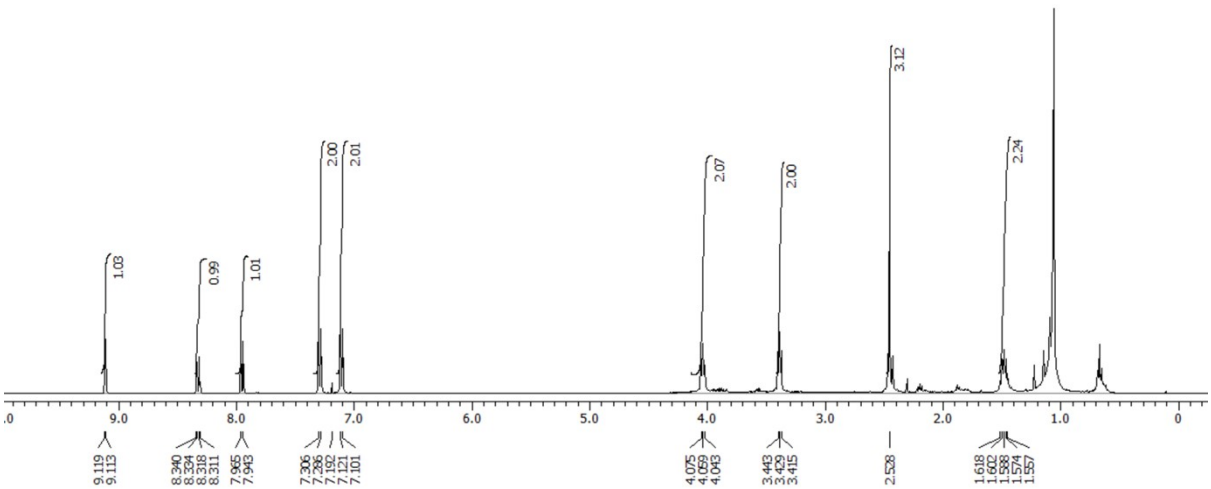


^{1}H NMR (400 MHz, $CDCl_3$)

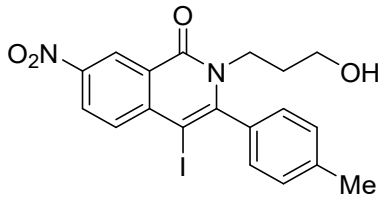


2-(3-hydroxypropyl)-4-iodo-7-nitro-3-(p-tolyl)isoquinolin-1(2H)-one (6d)

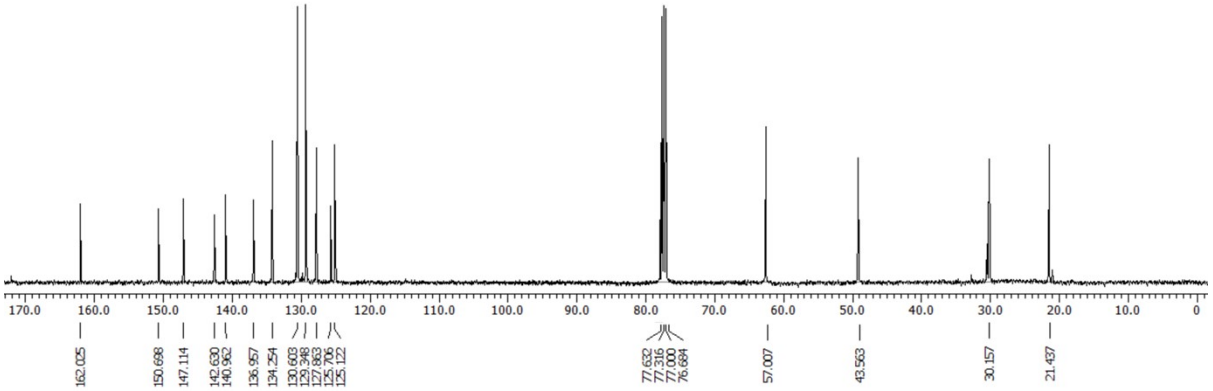




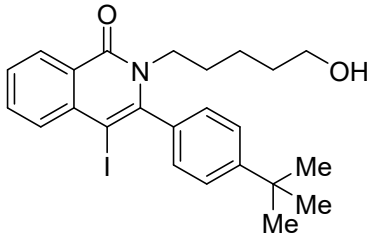
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



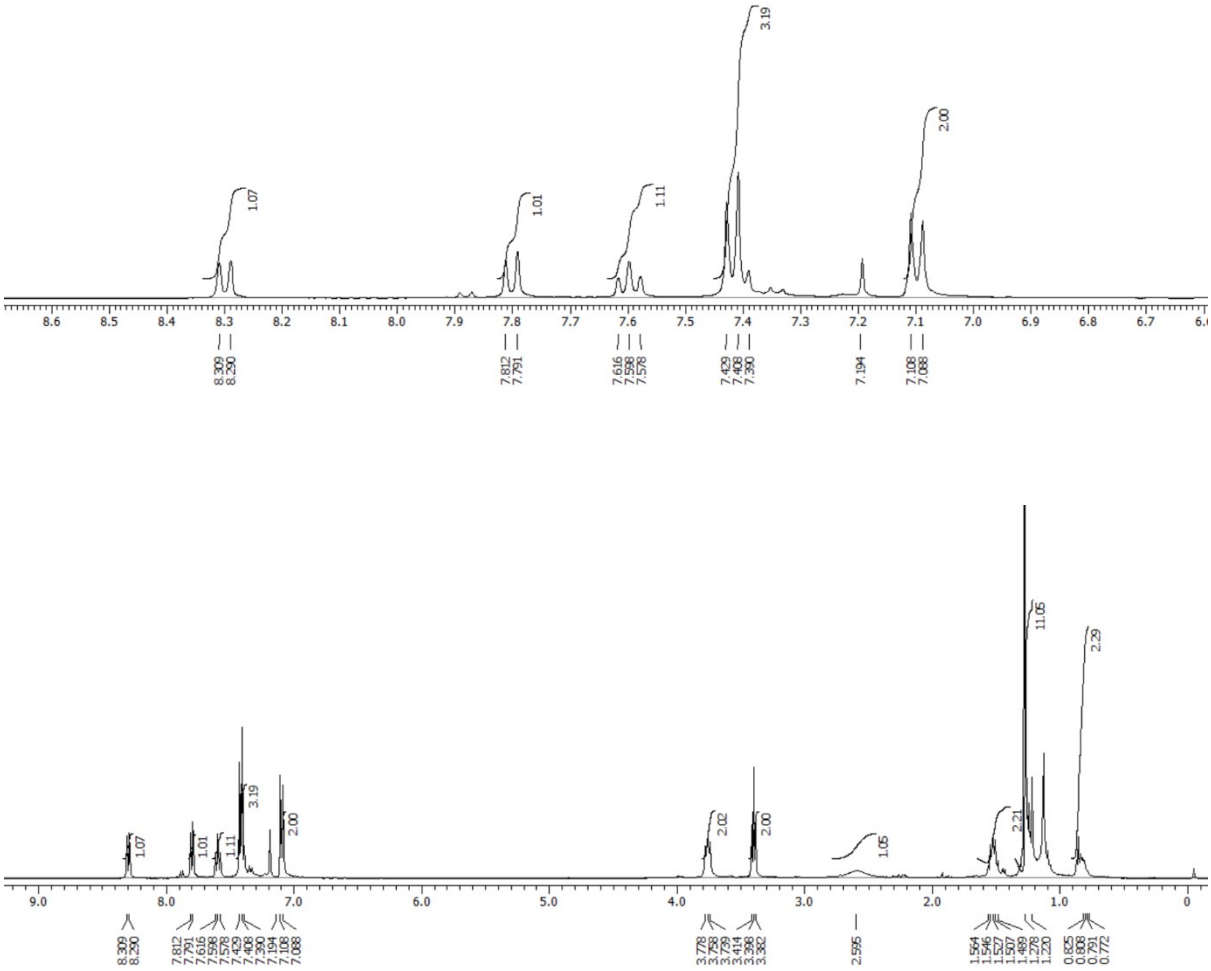
2-(3-hydroxypropyl)-4-iodo-7-nitro-3-(p-tolyl)isoquinolin-1(2H)-one (6d)



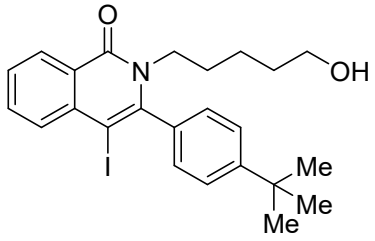
^{1}H NMR (400 MHz, $CDCl_3$)



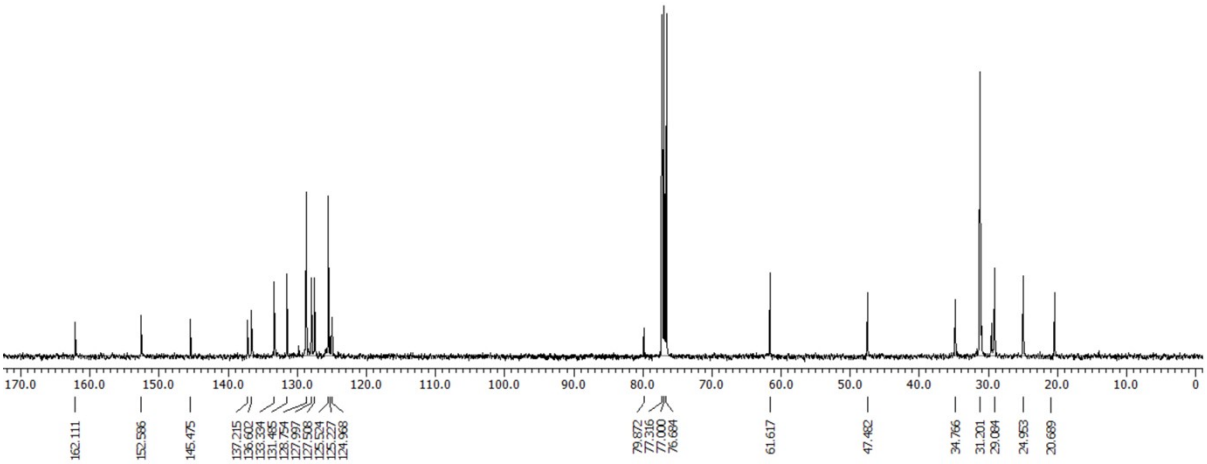
3-(4-(tert-butyl)phenyl)-2-(5-hydroxypentyl)-4-iodoisoquinolin-1(2H)-one (6e)



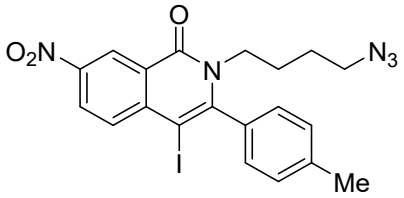
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



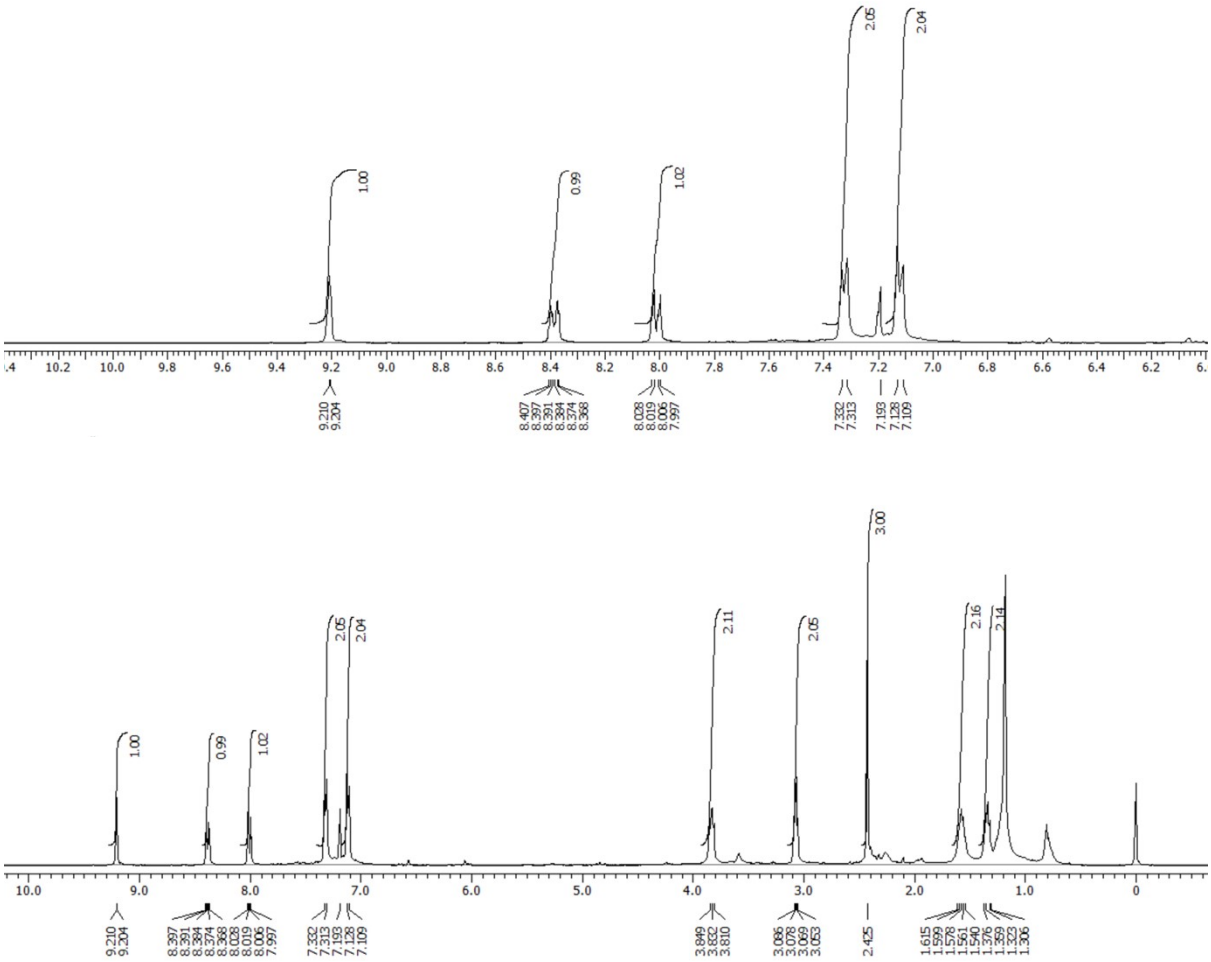
3-(4-(tert-butyl)phenyl)-2-(5-hydroxypentyl)-4-iodoisoquinolin-1(2H)-one (6e)



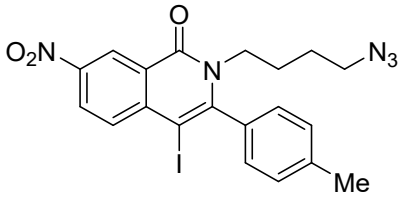
^{1}H NMR (400 MHz, $CDCl_3$)



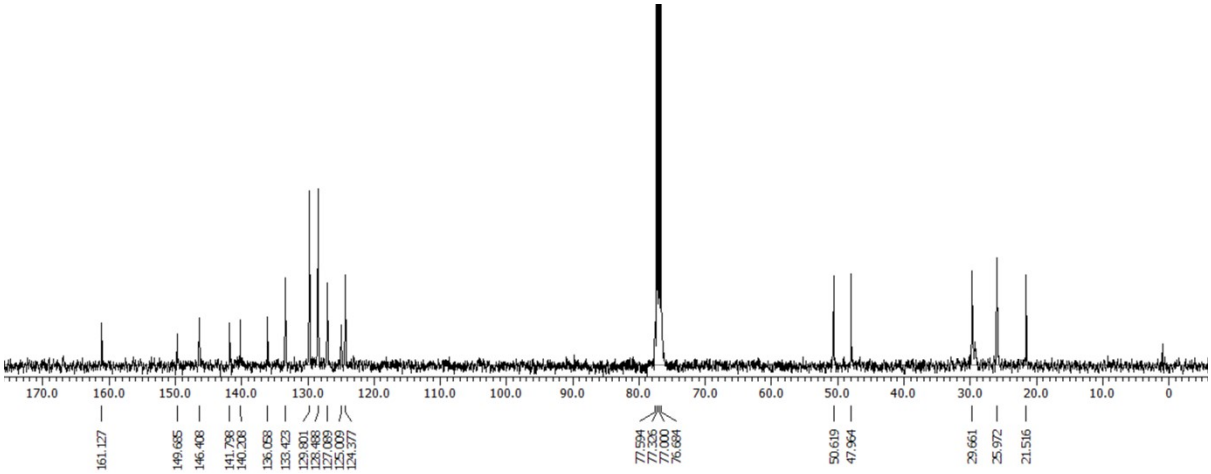
2-(4-azidobutyl)-4-iodo-7-nitro-3-(p-tolyl)isoquinolin-1(2H)-one (7f)



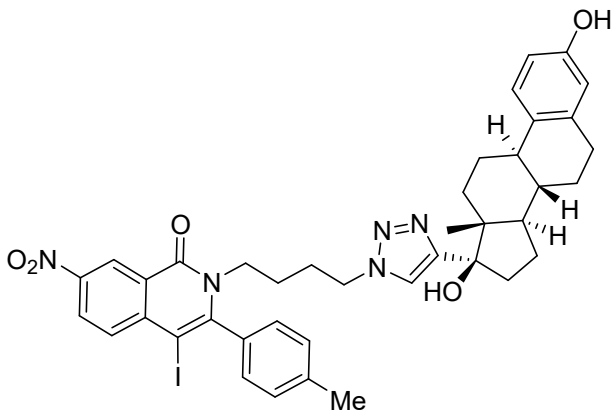
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



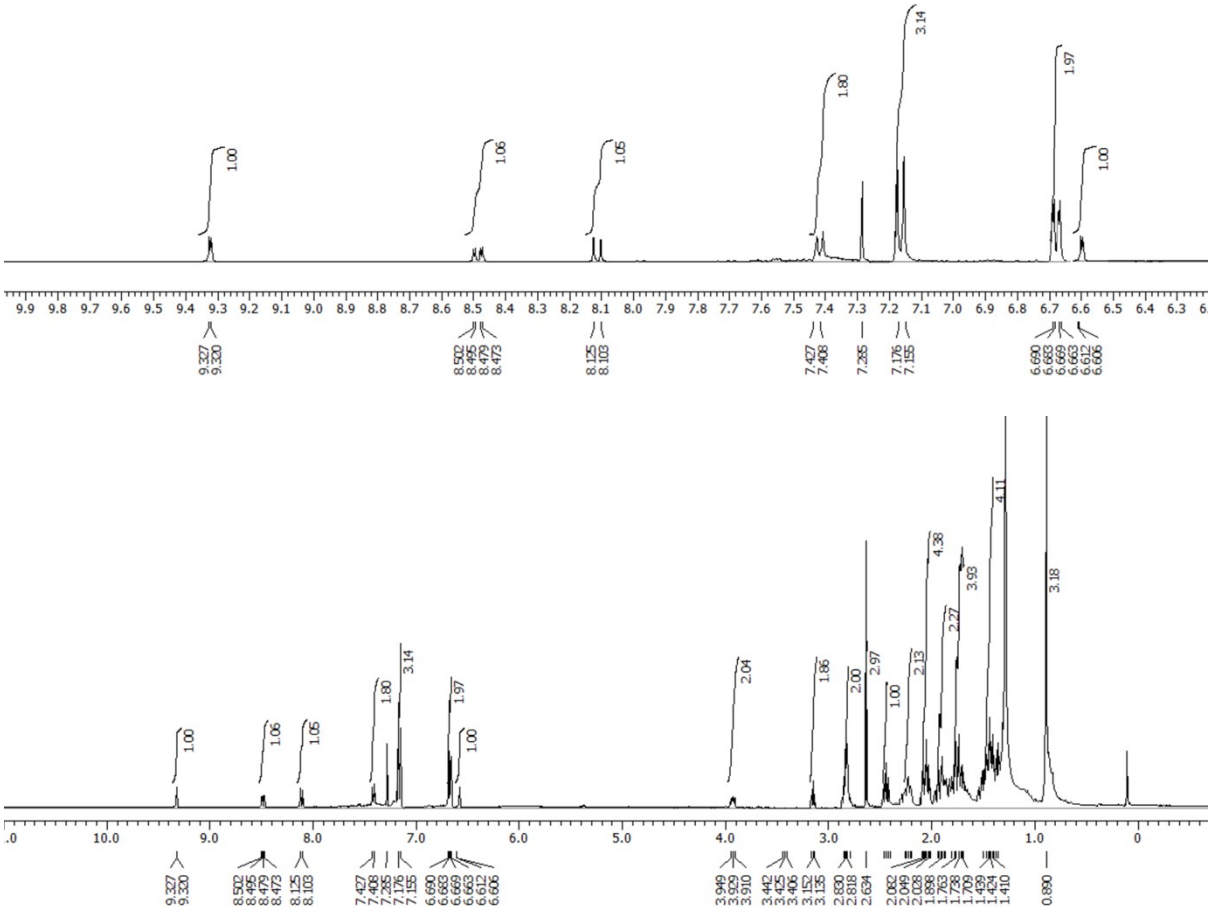
2-(4-azidobutyl)-4-iodo-7-nitro-3-(p-tolyl)isoquinolin-1(2H)-one (7f)



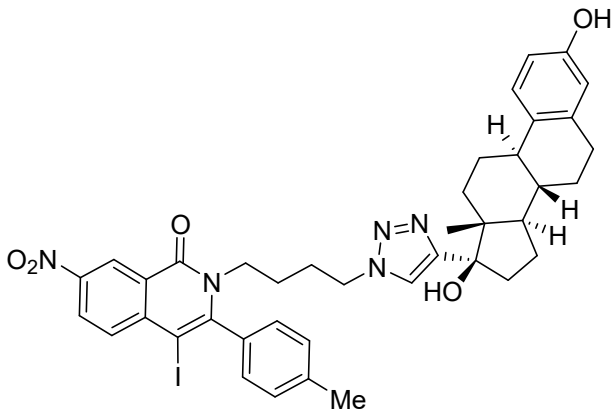
^{1}H NMR (400 MHz, $CDCl_3$)



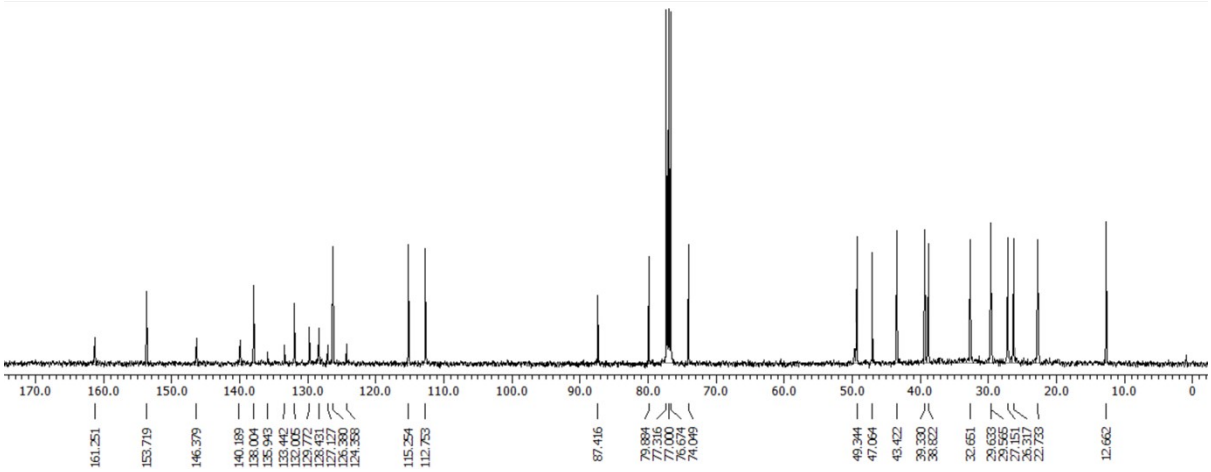
2-(4-(4-((8S,9R,13R,14R,17R)-3,17-dihydroxy-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-17-yl)-1H-1,2,3-triazol-1-yl)butyl)-4-iodo-7-nitro-3-(p-tolyl)isoquinolin-1(2H)-one (9)



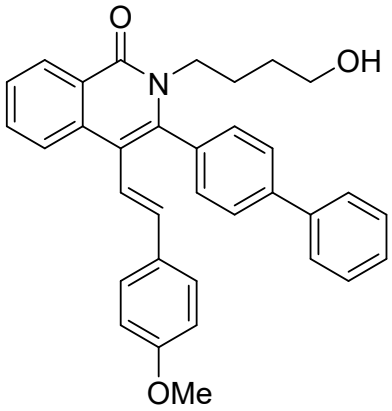
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



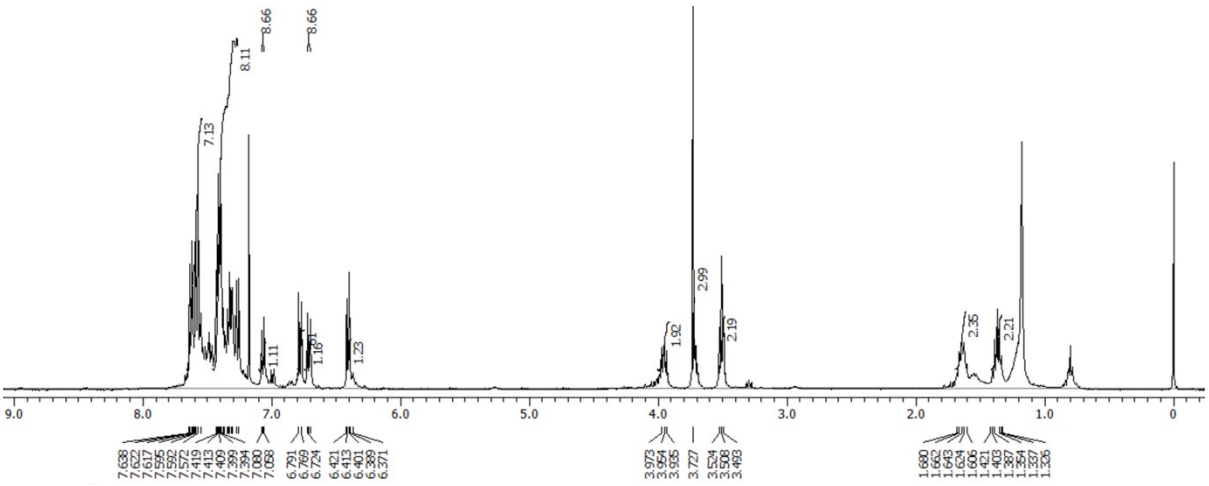
2-(4-(4-((8S,9R,13R,14R,17R)-3,17-dihydroxy-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-17-yl)-1H-1,2,3-triazol-1-yl)butyl)-4-iodo-7-nitro-3-(p-tolyl)isoquinolin-1(2H)-one (9)



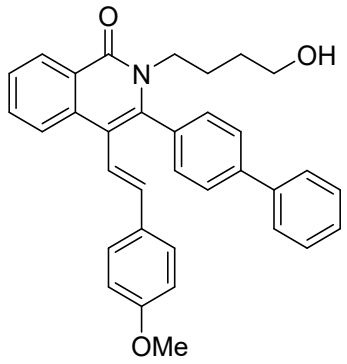
^{1}H NMR (400 MHz, $CDCl_3$)



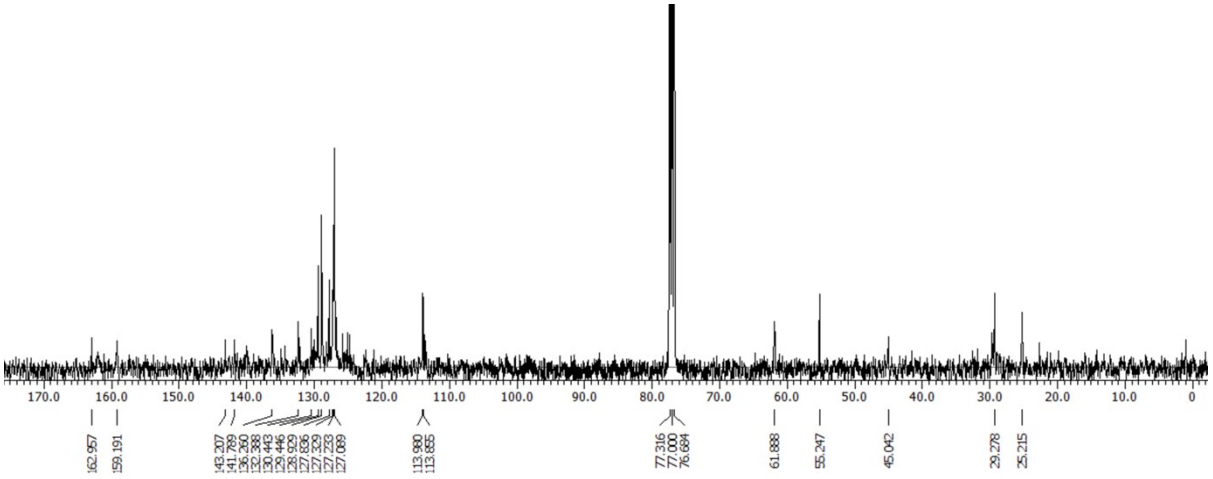
(E)-3-([1,1'-biphenyl]-4-yl)-2-(4-hydroxybutyl)-4-(4-methoxystyryl)isoquinolin-1(2H)-one (11j)



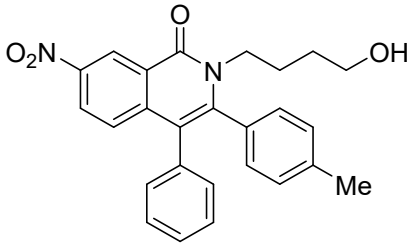
^{13}C {1H} NMR (100 MHz, $CDCl_3$)



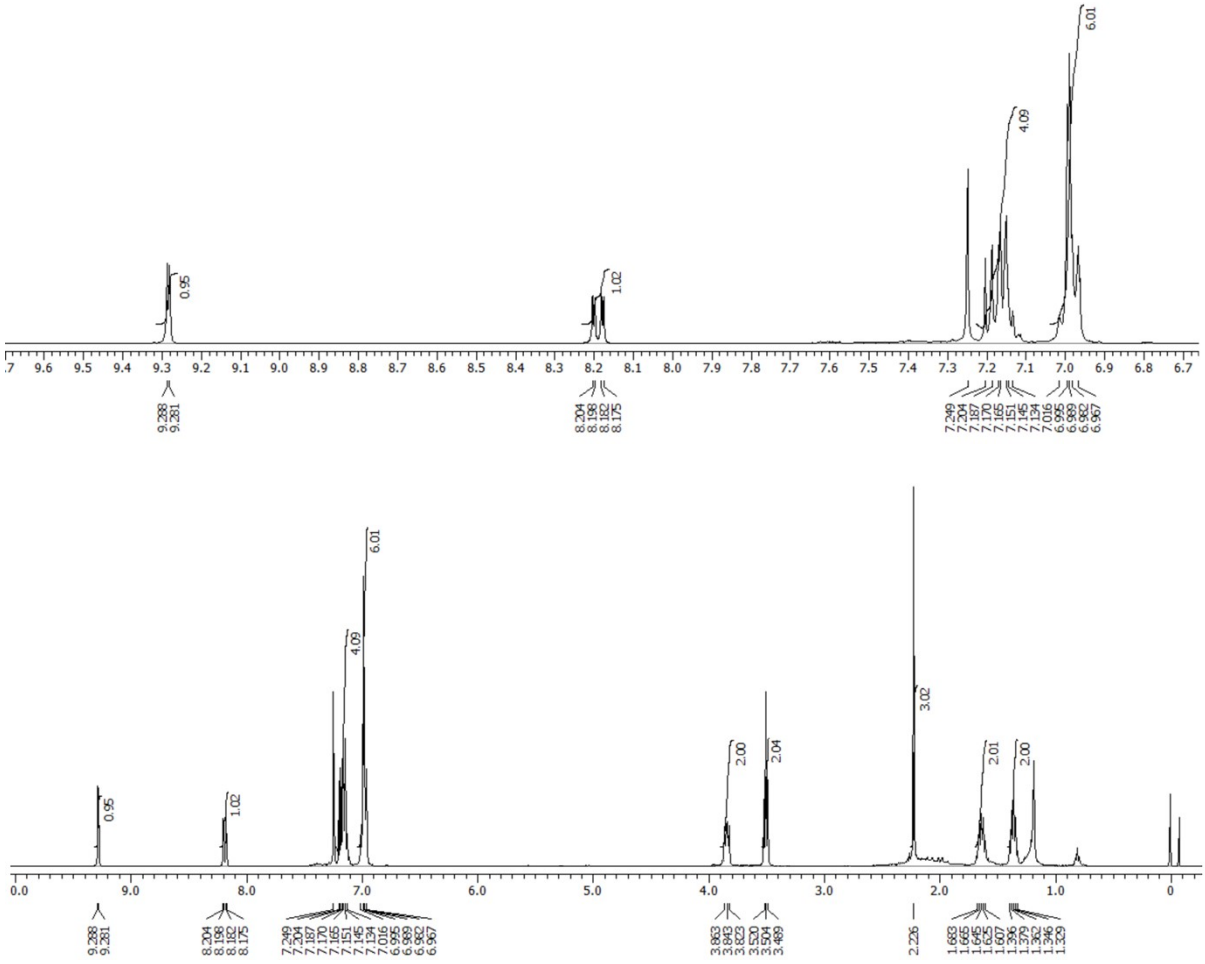
(E)-3-([1,1'-biphenyl]-4-yl)-2-(4-hydroxybutyl)-4-(4-methoxystyryl)isoquinolin-1(2H)-one (11j)



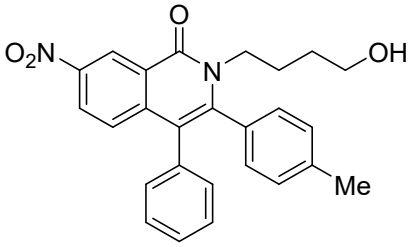
^{1}H NMR (400 MHz, $CDCl_3$)



2-(4-hydroxybutyl)-7-nitro-4-phenyl-3-(p-tolyl)isoquinolin-1(2H)-one (13f)



^{13}C {1H} NMR (100 MHz, $CDCl_3$)



2-(4-hydroxybutyl)-7-nitro-4-phenyl-3-(p-tolyl)isoquinolin-1(2H)-one (13f)