Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

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Contents

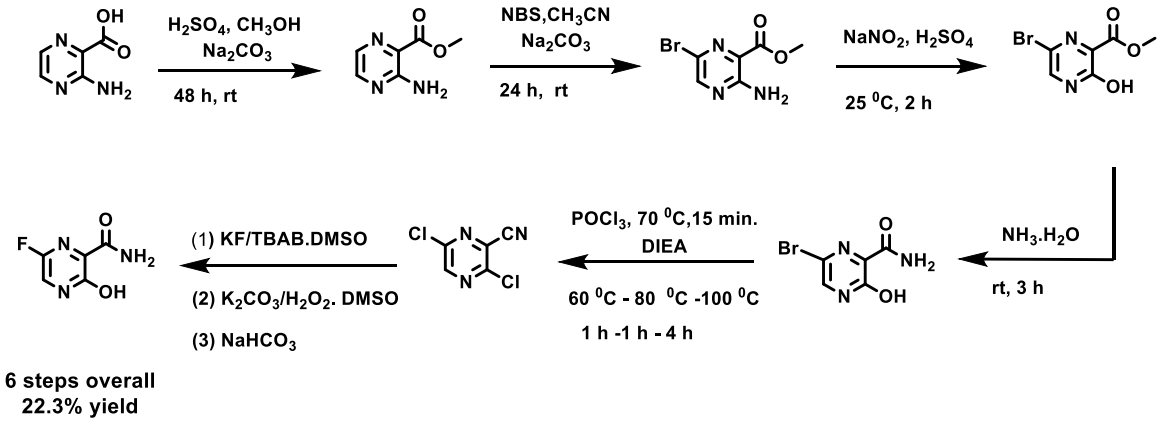
Section-1 General information:

Solvents are purchased from Merck Ltd. and used after distillation, and some of them were dried using literature procedures. Reagents were purchased from Sigma-Aldrich, Sisco Research Laboratories Pvt Ltd, and spectrochem Private Limited. Reactions were monitored by thin layer chromatography (TLC) and purified by column chromatography using 100-200 mesh size silica gel. A suitable mixture of (Ethyl acetate-hexane) or (Methanol-DCM) was used for elution. The ^{1}H, ^{13}C, and ^{19}F NMR spectra were recorded by Brucker ADVANCE III 400 Spectrometer by using $CDCl_3$ and DMSO-d_6 solvent. Chemical shift values were measured by ppm (parts per million) with reference to the solvent residual peak. Proton coupling patterns are described as singlet(s), doublet (d), triplet (t), quartet (q), and brs indicates a broad signal. Mass spectra were recorded by using a Bruker's micrOTOF-Q II system with electron spray ionization technique (ESI-MS). X-ray was recorded in a microfocus Rigaku Oxford XtalAB SuperNova Single X-ray diffraction system using a copper X-ray source at room temperature or 100K. The crystal data parameters are given in table 1 and 2.

Section-2:

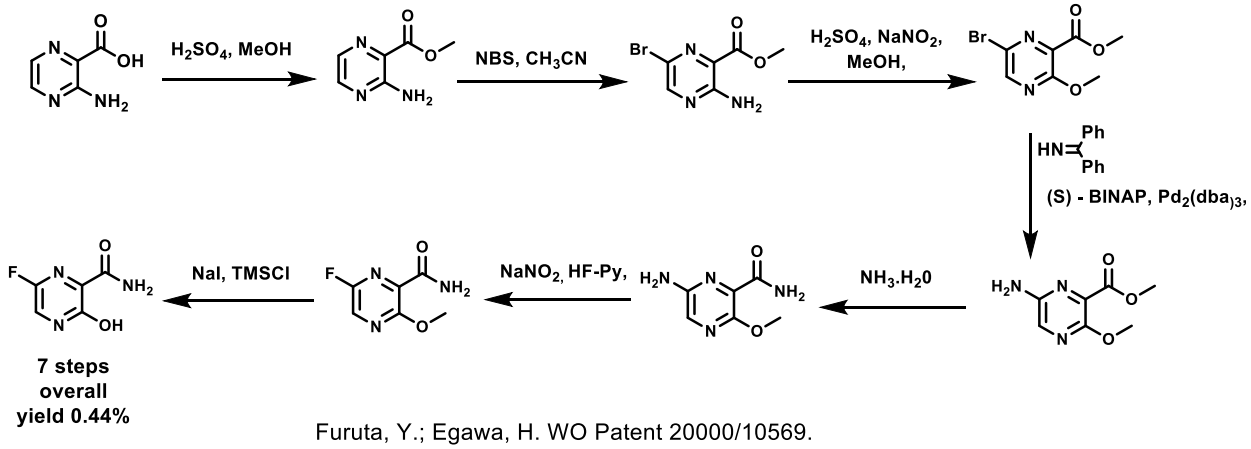
Previously reported Schemes:

Scheme-1



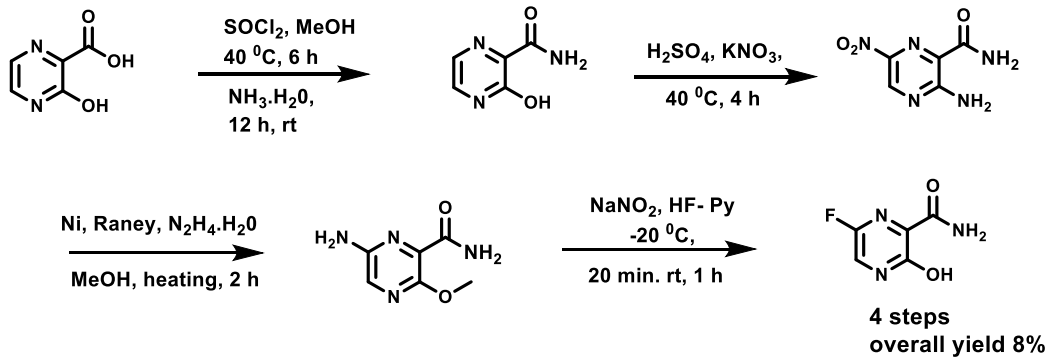
Liu, F.L.; Li, C. Q.; Xiang, H. Y.; Feng, S., Chem. Pap. **2017**, 71, 2153.

Scheme-2



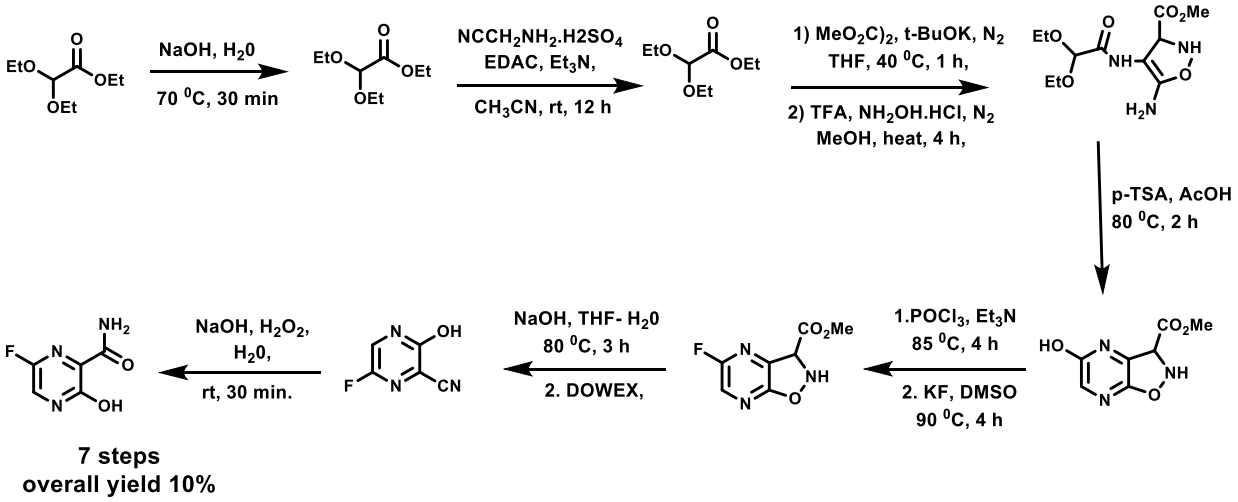
Furuta, Y.; Egawa, H. WO Patent 20000/10569.

Scheme-3



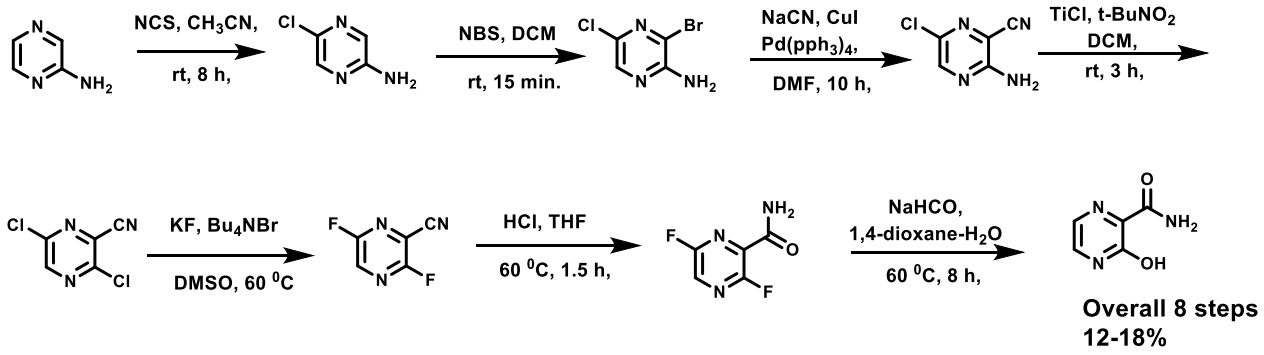
Shi, F.; Li, Z.; Kong, L.; Xie, Y.; Zhang, T.; Xu, W. Drug Discoveries Ther. 2014, 8, 117.

Scheme-4



Nakamura, K.; Murakami, T.; Naitou, H.; Hanaki, N.; Watanabe, K. US Patent 20150051396.

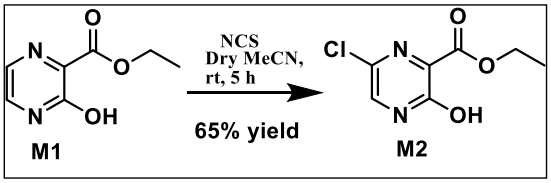
Scheme-5



Guo, Q.; Xu, M.; Guo, S.; Zhu, F.; Xie, Y.; Shen, J. *Chem. Pap*. **2019**, *73*,1043.

Section-3: Synthetic Procedure:

6-chloro-3-hydroxypyrazine-2-ethyl ester (M2):



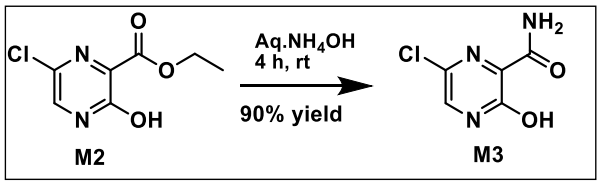
To a 100 mL RB flask, the solution of 3-hydroxypyrazine -2-ethyl ester, (**M1) (**500 mg, 1 equivalent., 2.975 mmol**)** in 20 mL dry acetonitrile, (595 mg, 1 equivalent, 4.462 mmol) of NCS was added. The mixture was stirred at rt for 5 h in dark conditions. The colour of the reaction mixture turned to reddish brown during this period. Thin-layer chromatography (TLC) indicates the complete consumption of the reactant. The reaction was stopped and solvent was evaporated using a rotary evaporator. The crude product was purified using column chromatography (EtOAc: Hexane 2:3) to get 6-chloro-3-hydroxypyrazine-2-ethyl ester **(M2)** as a white solid, 390 mg, Yield: 65%.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 13.35 (s, 1 H), 8.45 (s, 1 H), 4.57 (q, 2 H, *J* = 8 Hz and 4 Hz), 1.49 (t, 3 H, *J* = 8 Hz).

^{13}C NMR (400 MHz, $CDCl_3$, δ ppm): 167.74, 161.44, 148.41, 139.67, 124.87,63.82, 14.13.

HRMS (ESI-MS): calc. for $C_7H_7ClN_2O_3Na$, $[M+Na]^+$: 225.0037, found: 224.9907.

6-Chloro-3-hydroxypyrazinamide (M3):



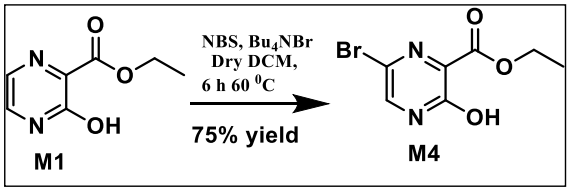
In a 100 mL RB flask, (500 mg, 1 equivalent, 2.687 mmol) of 6-Chloro-3-hydroxypyrazine-2-ethyl ester **(M2)** was added to 25 mL of 50% Aq. NH_4OH solution. The mixture was stirred at rt for 4 h. Thin-layer chromatography (TLC) indicates the complete consumption of the reactant. The colour of the reaction mixture turned to faint yellow during this period. The reaction was stopped and solvent was evaporated using a rotary evaporator. The crude product was purified using column chromatography (MeOH: DCM 3:97) to get 6-chloro-3-hydroxypyrazinamide **(M3)** as a white solid 385 mg, Yield: 90%.

^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.10 (s, 1 H), 8.18 (s, 2 H), 7.81 (s, 1 H).

^{13}C NMR (700 MHz, DMSO-d_6, δ ppm): 168.36, 164.11, 163.50, 146.14, 129.78.

HRMS (ESI-MS): calc. for $C_5H_4ClN_3O_2$ Na, $[M+Na]^+$: 172.9987, found: 173.1035.

6-bromo-3-hydroxypyrazine-2-ethyl ester (M4):



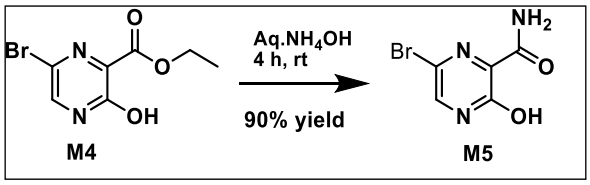
To a 100 mL sealed tube, the solution of 3-hydroxy-pyrazine 2-ethyl ester, (**M1) (**500 mg, 1 equivalent, 2.975 mmol**)**, in 25 mL dry DCM, (794 mg, 1equivalent, 4.462 mmol) of NBS and (95 mg, 0.1 equivalent. 0.297mmol) of TBAB as a catalytic amount was added and stirred for 5 min. The mixture was stirred at 65 ^{o}C for 6 h under dark conditions. The colour of the reaction mixture turned to brownish red during this period. Thin-layer chromatography (TLC) indicates the complete consumption of the reactant. The reaction was stopped and the solvent was evaporated using a rotary evaporator. The crude product was purified using column chromatography (EtOAc: Hexane 35:65) to get 6-bromo-3-hydroxypyrazine-2-ethyl ester **(M4)** as a white solid, 551 mg Yield: 75%.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 11.35 (s, 1 H), 8.52 (s, 1 H), 4.57 (q, 2 H, *J* = 8 Hz), 1.48 (t, 3 H, *J* = 8 Hz and 4 Hz).

^{13}C NMR (400 MHz, $CDCl_3$, δ ppm): 167.87, 162.07, 151.50, 129.70, 126.06, 63.94, 14.12.

HRMS (ESI-MS): calc. for $C_7H_7BrN_2O_3Na$, $[M+Na]^+$: 268.9536, and found: 268.9532.

6-Bromo-3-hydroxy-pyrazinamide (M5):



In a 100 mL RB flask, (500 mg, 1 equivalent, 2.687 mmol) of 6-Bromo-3-hydroxypyrazine-2-ethyl ester **(M4)** was added to 25 mL of 50% Aq. NH_4OH solution. The mixture was stirred at rt for 4 h. The colour of the reaction mixture turned to yellowish milky during this period. Thin-layer chromatography (TLC) indicates the complete consumption of the reactant. The reaction was stopped and the solvent was evaporated using a rotary evaporator. The crude product was purified using column chromatography (MeOH: DCM 3:93) to get, 6-Bromo-3-hydroxy-pyrazinamide (**M5)** as a white solid with 397 mg, Yield: 90%.

^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.16 (s, 1 H,), 8.23 (s, 2 H,), 7.85 (s, 1 H).

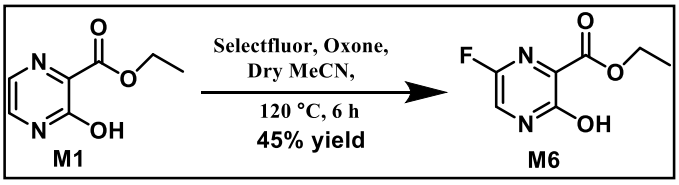
^{13}C NMR (700 MHz, $CDCl_3$, δ ppm): 168.63, 161.55, 150.89, 128.14, 126.94.

HRMS (ESI-MS): calc. for $C_5H_4BrN_3O_2$+Na^+, $[M+Na]^+$: 239.9379, found: 239.9371.

This reaction was performed in a gram scale as follows.

To a 100 mL sealed tube, the solution of 3-hydroxy-pyrazine 2-ethyl ester, (**M1) (**6 g, 1 equivalent, 35.68 mmol**)**, in 250 mL dry DCM, (6.35 g, 1 equivalent) of NBS and (1.15 g, 0.1 equivalent) of TBAB as a catalytic amount was added and stirred for 10 min. The mixture was stirred further at 65 ^{o}C for 6 h under dark conditions. The reaction was stopped after 6 hours and solvent was evaporated. The product was then separated by column chromatography. This product thus obtained (6.7 g) was stirred in 25 % ammonium hydroxide (100 mL) for 4 h. After this time the solution saturated with NaCl and extracted with chloroform and subjected to chromatographic separation to yield 5.32 g of the product (90 % yield).

6-fluoro-3-hydroxypyrazine-2-ethyl ester (M6):



To a 100 mL sealed tube, the solution of 3-hydroxypyrazine 2-ethyl ester, (**M1)** (500 mg, 1 equivalent, 2.975 mmol), in 20 mL dry acetonitrile, 518 mg (1 equivalent, 1.462 mmol) of Selectflour was added. The mixture was heated at 120 °C for 6 h. The colour of the reaction mixture turned to yellowish red during this period. Thin- layer chromatography (TLC) indicates the complete consumption of the reactant. The reaction was stopped and solvent was evaporated using a rotary evaporator. The crude product was purified using column chromatography (EtOAc: Hexane 1:4) to get 6-fluoro-3-hydroxypyrazine-2-ethyl ester **(M6**) as white solid 249 mg, Yield: 45%.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 11. 23 (s, 1H), 8.34 (d, 1 H, *J*=8 Hz), 4.57 (q, 2 H, *J* = 8 Hz and 4 Hz), 1.50 (t, 3 H, *J* = 8 Hz).

^{19}F NMR (400 MHz, $CDCl_3$, δ ppm): -88.64, (s, 1 F).

^{13}C NMR (400 MHz, $CDCl_3$, δ ppm): 167.58, 161.13, 152.07, 138.06, 137.37, 63.85, 14.23.

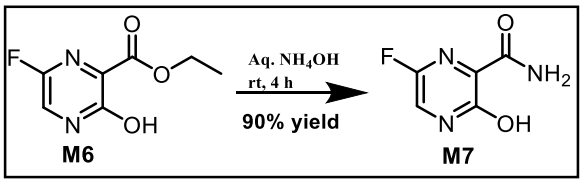
HRMS (ESI-MS): calc. for $C_7H_7FN_2O_3Na$, $[M+Na]^+$: 209.0333; Found: 209.0325.

Reaction optimization and controlled experiment (M6):

Table S1:

SL No.	Fluorinating agent	Additive	Temperature (°C)	Yield (%)
1	Selectflour	…….	RT	ND
2	Selectflour	Oxone	60	21
3	Selectflour	Oxone	80	29
4	Selectflour	Oxone	100	38
5	Selectflour	…….	120	33
6	**Selectflour**	**Oxone**	**120**	**45**
7	NFSI	…….	RT	ND
8	NFSI	Oxone	RT	ND

6-fluoro-3-hydroxypyrazinamide (M7):



In a 100 mL RB flask, (500 mg, 1 equivalent., 2.687 mmol) of 6-fluoro-3-hydroxypyrazine-2-ethyl ester **(M6)** was added to 25 mL of 50% Aq. NH_4OH solution. The mixture was stirred at rt for 4 h. The colour of the reaction mixture turned to yellowish milky during this period. Thin-layer chromatography (TLC) indicates the complete consumption of the reactant. The reaction was stopped and solvent was evaporated using a rotary evaporator. The crude product was purified using column chromatography (EtOAc: Hexane 3:2) to get 6-fluoro-3-hydroxypyrazinamide **(M7)** as a white solid, 380 mg, with Yield 90%.

^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 13.41 (s, 1 H), 8.77 (s, 1 H), 8.50 (s, 2 H).

^{13}C NMR (400 MHz, $CDCl_3$, δ ppm): 173.68, 164.97, 150.95, 140.84, 126.86.

HRMS (ESI-MS): calc. for $C_5H_4FN_3O_2Na$, $[M+Na]^+$: 180.0180, found: 180.0201.

Spectra analysis

Section-4

Spectral data(^{1}H,^{13}C) of starting material (Commercially available).

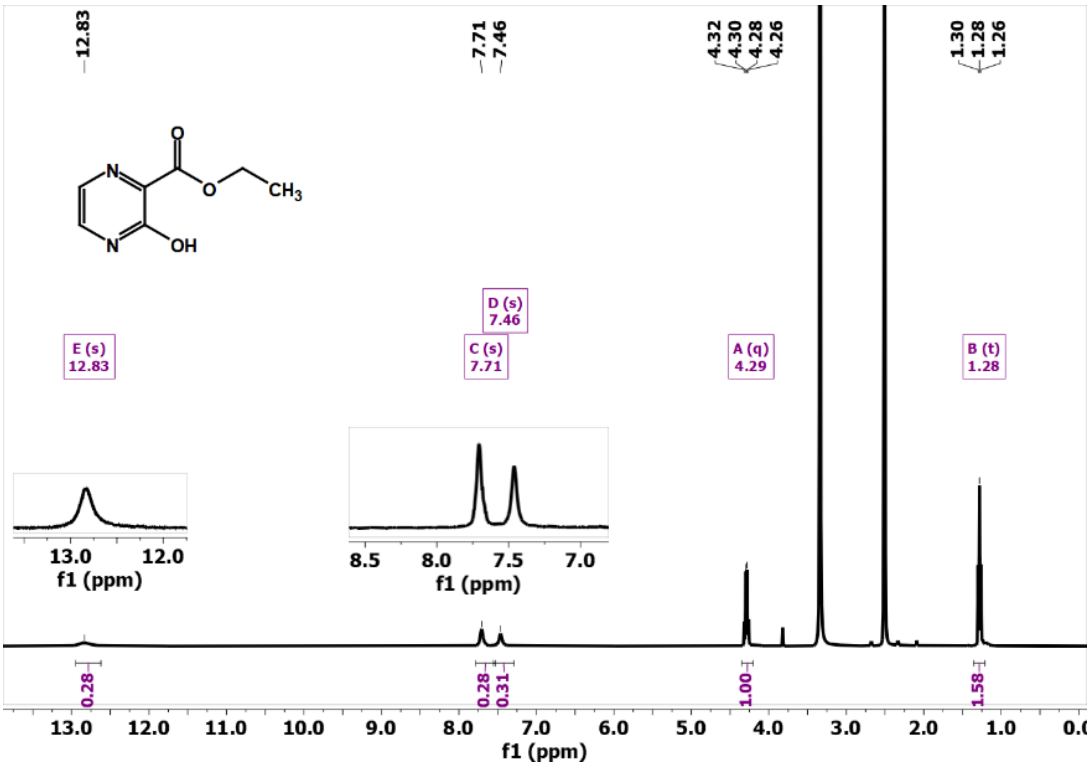


Fig. S1 ^{1}H NMR Spectra of **M1** in DMSO-d_6 (400 MHz).

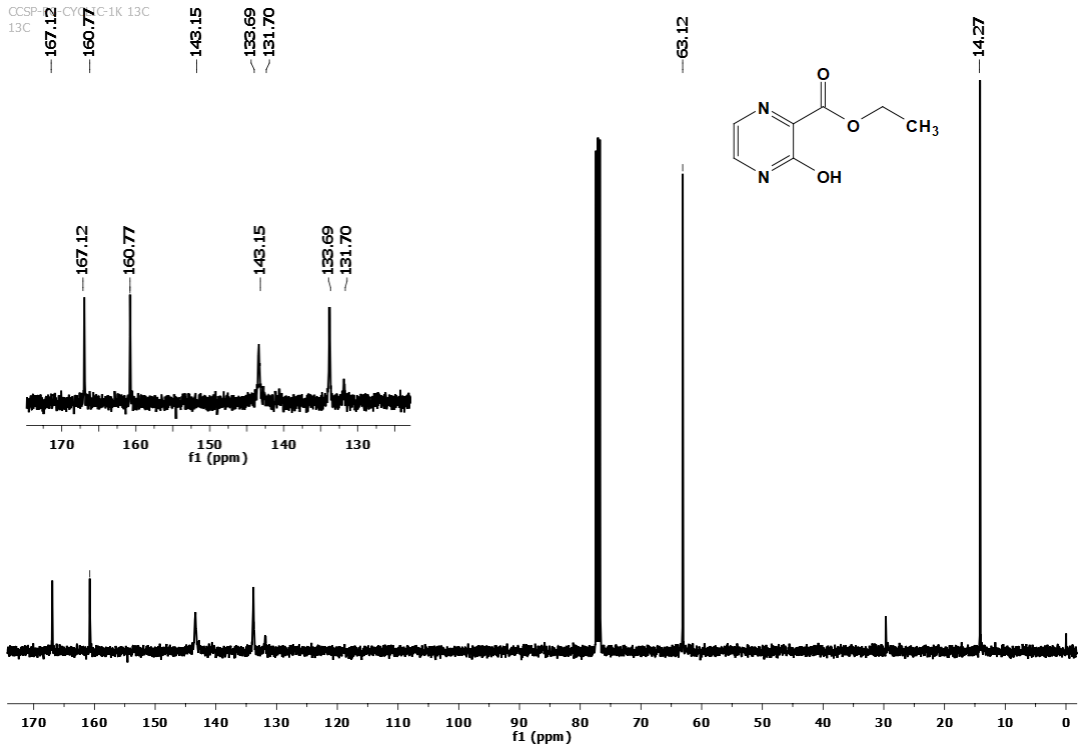


Fig. S2 ^{13}C NMR Spectra of **M1** in Chloroform-d (400 MHz).

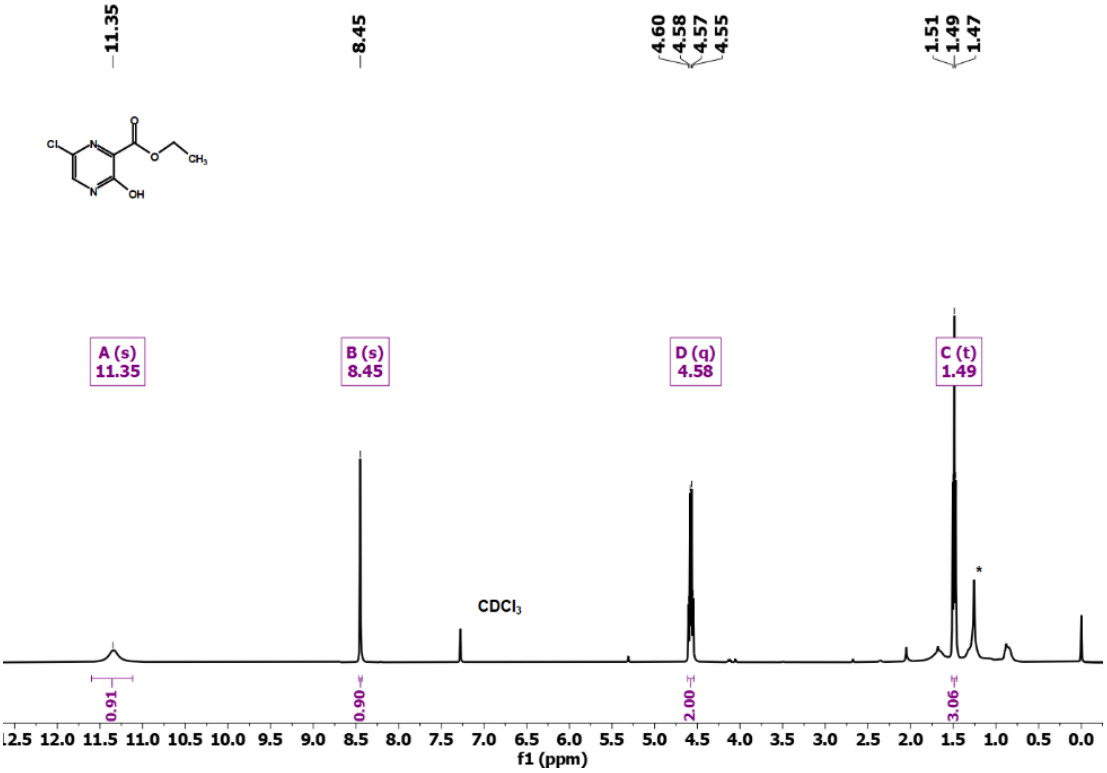


Fig. S3 ^{1}H NMR Spectra of **M2** in Chloroform-d (400 MHz). **(*)** indicates the grease peak.

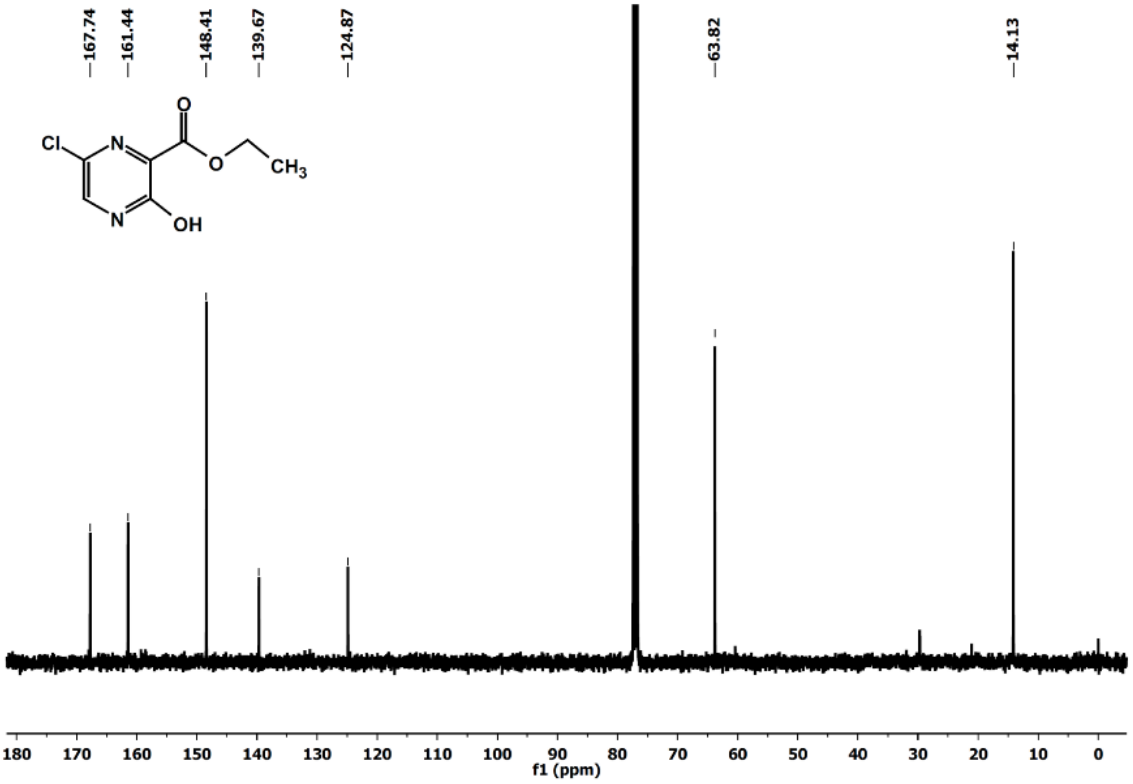


Fig. S4 ^{13}C NMR Spectra of **M2** in Chloroform-d (400 MHz).

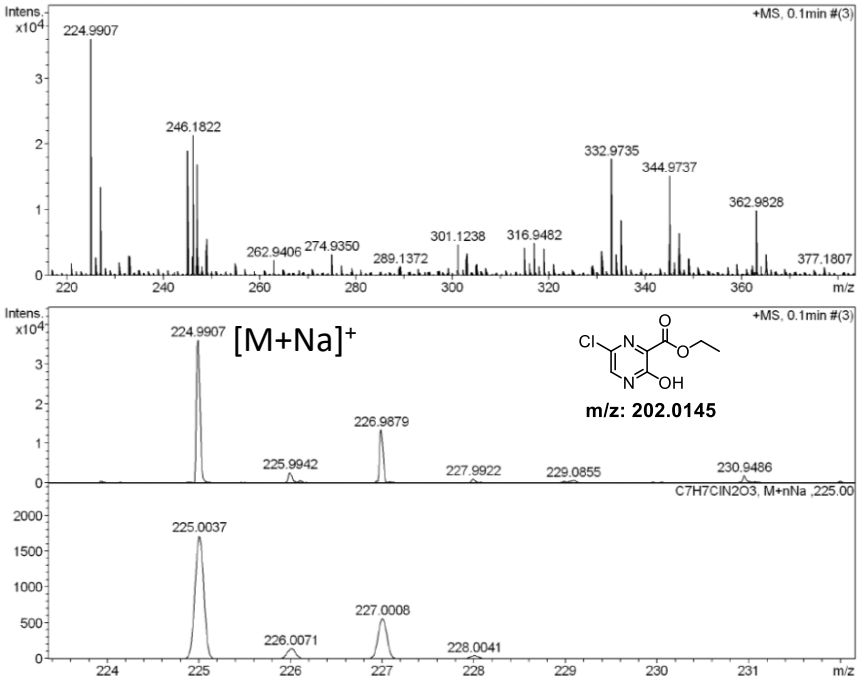


Fig. S5: ESI-MS (HRMS) Mass Spectra of **M2.**

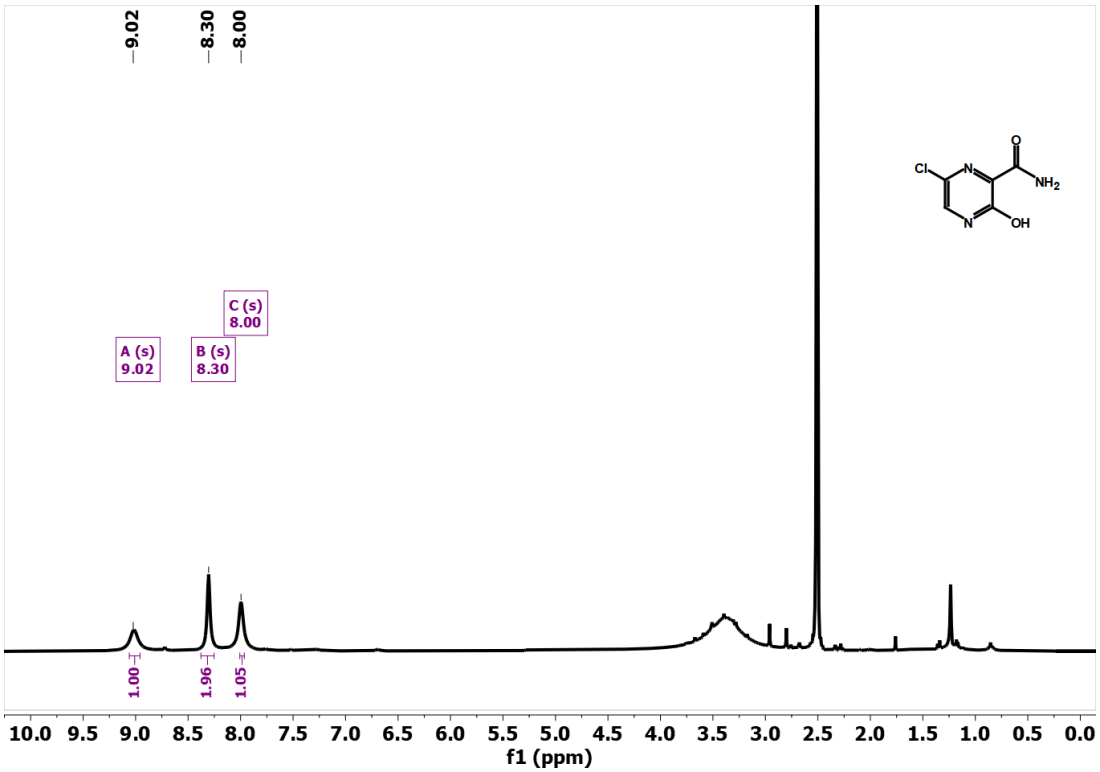


Fig. S6: ^{1}H NMR Spectra of **M3** in DMSO- d_6 (400 MHz).

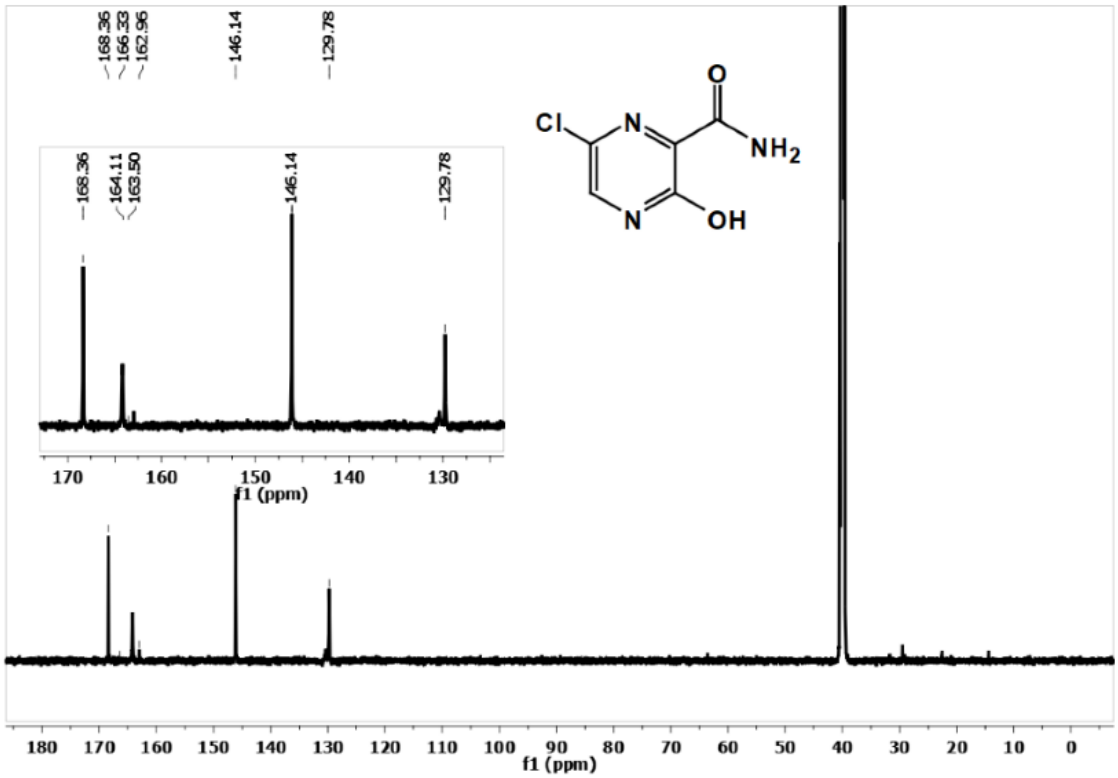


Fig. S7: ^{13}C NMR Spectra of **M3** in DMSO- d_6 (700 MHz).

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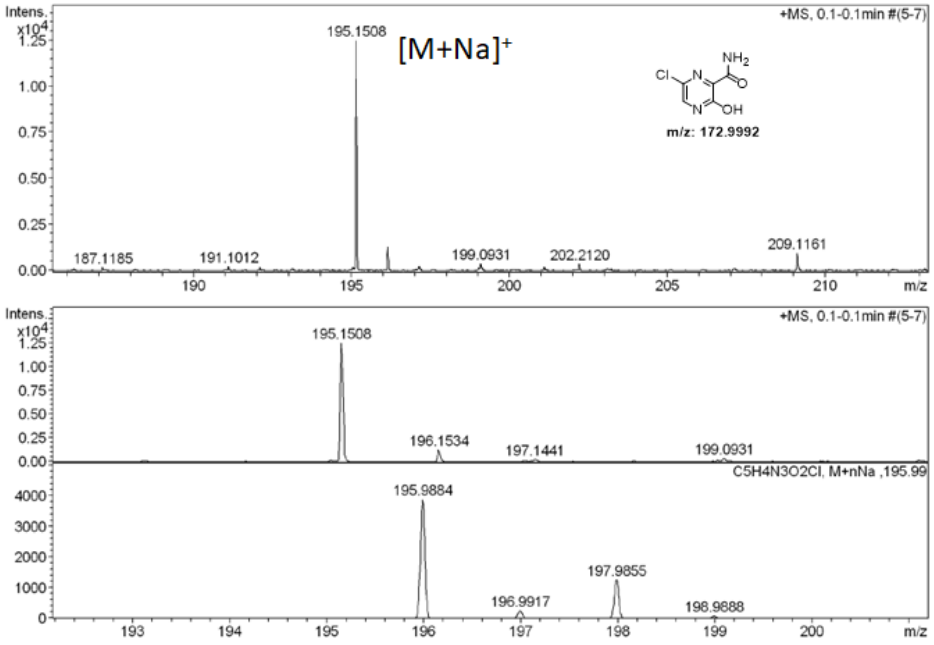


Fig. S8: ESI-MS (HRMS) Mass Spectra of **M3**.

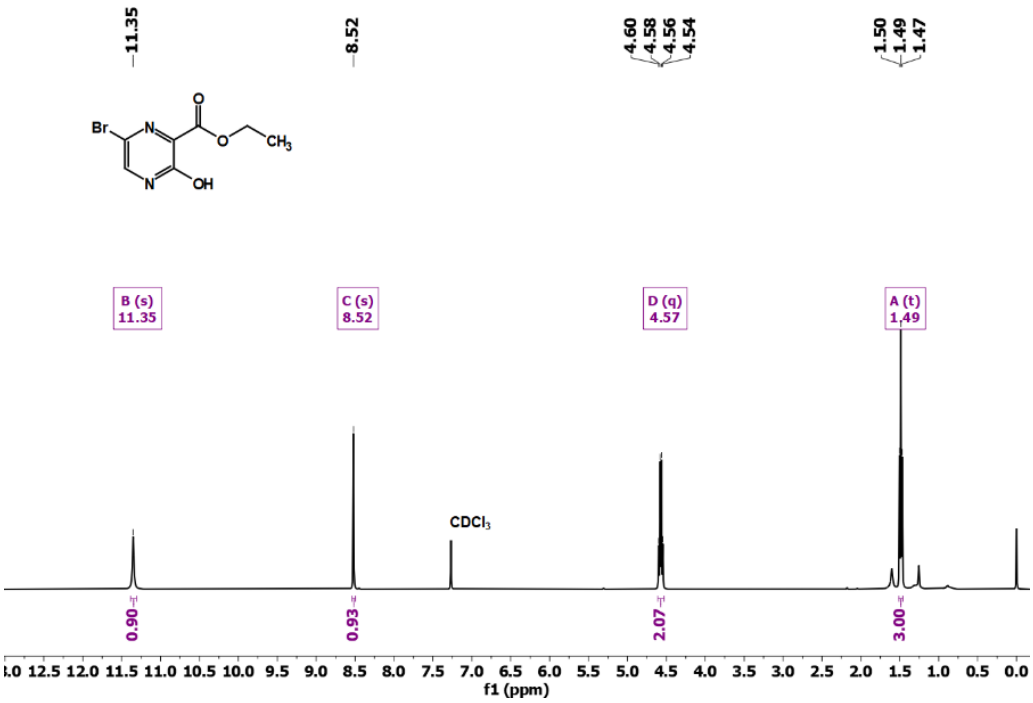


Fig. S9: ^{1}H NMR Spectra of **M4** in chloroform-d (400 MHz).

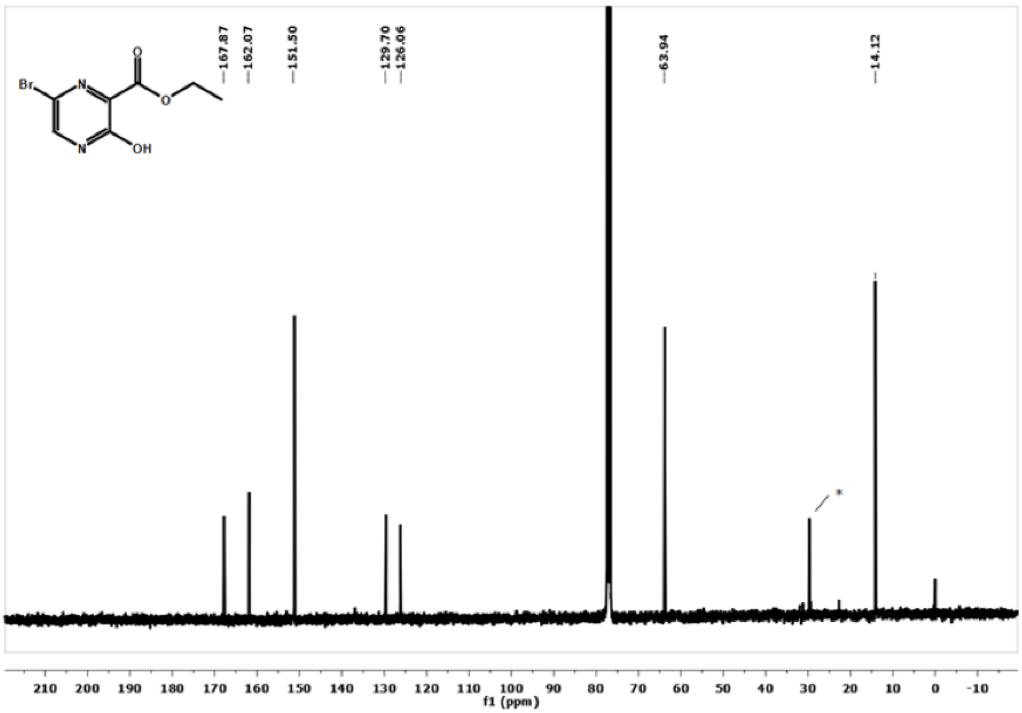


Fig. S10: ^{13}C NMR Spectra of **M4** in chloroform-d (400 MHz). **(*)** indicates the grease peak.

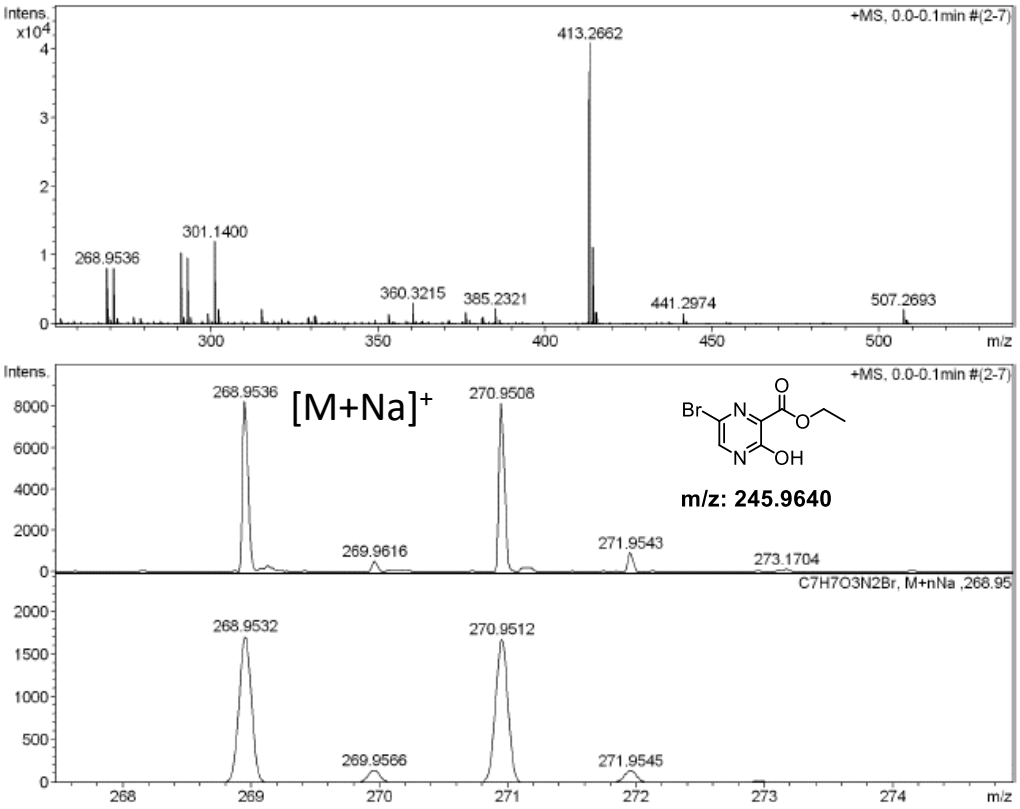


Fig. S11: ESI-MS (HRMS) Mass Spectra of **M4.**

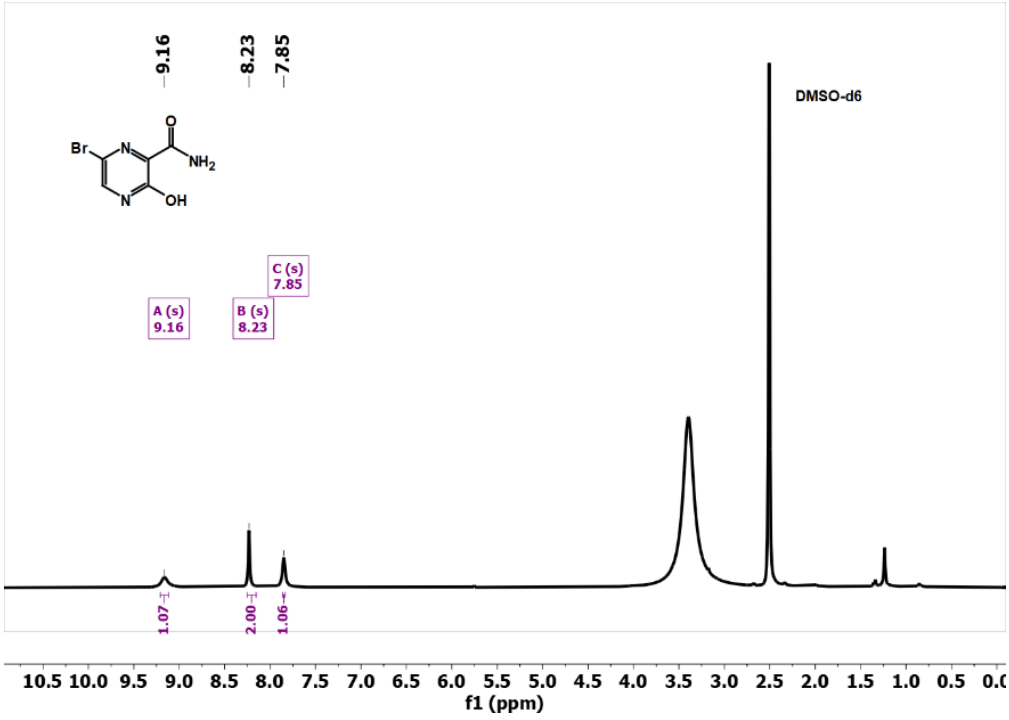


Fig. S12: ^{1}H NMR Spectra of **M5** in DMSO- d_6 (400 MHz).

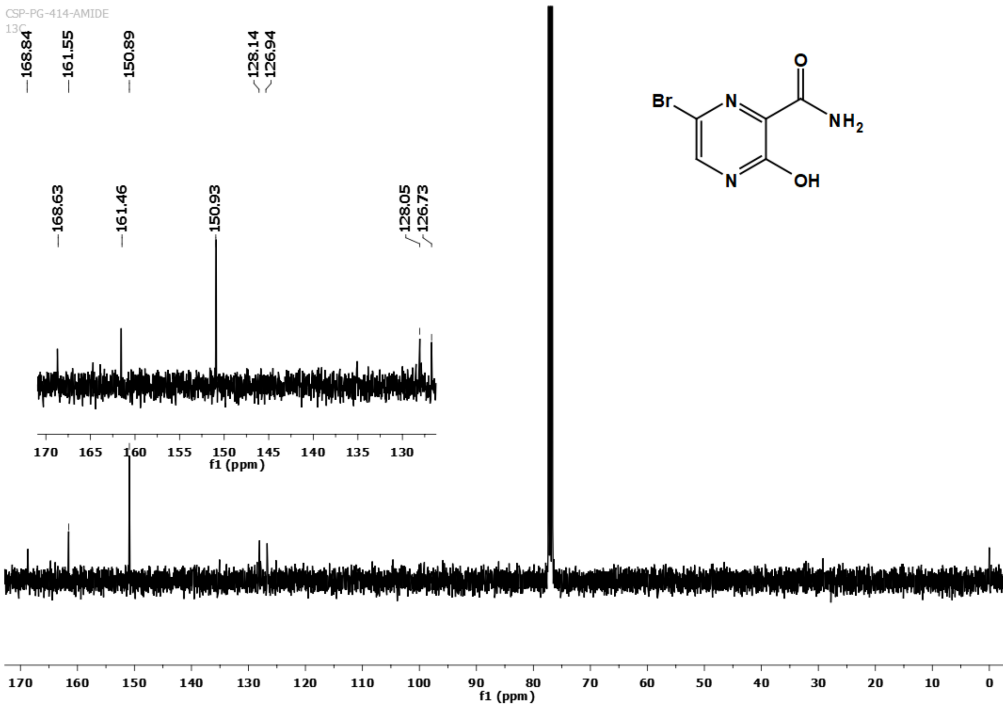


Fig. S13: ^{13}C NMR Spectra of **M5** in chloroform-d (700 MHz).

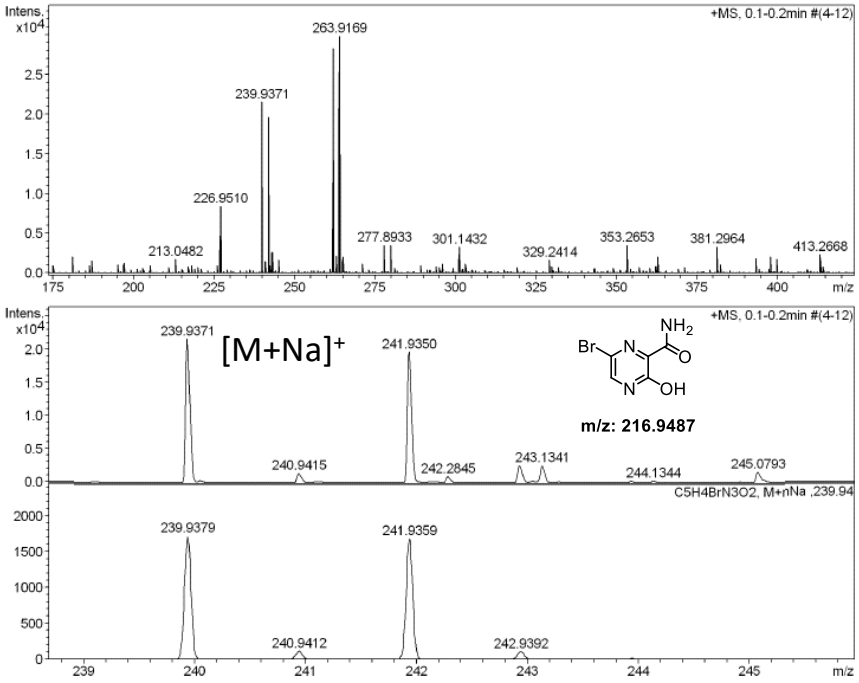


Fig. S14: ESI-MS (HRMS) Mass Spectra of **M5.**

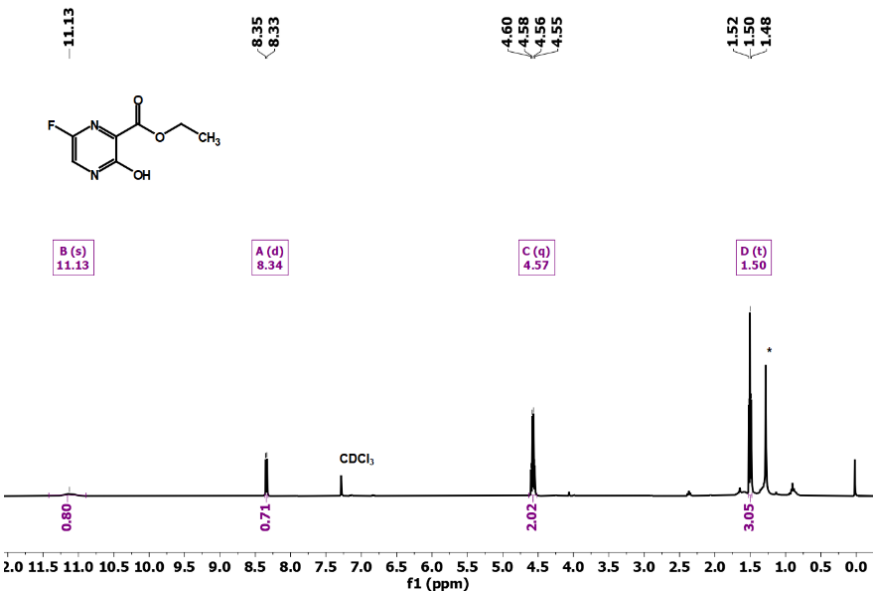


Fig. S15: ^{1}H NMR Spectra of **M6** in Chloroform-d (400 MHz), **(*)** indicates the grease peak.

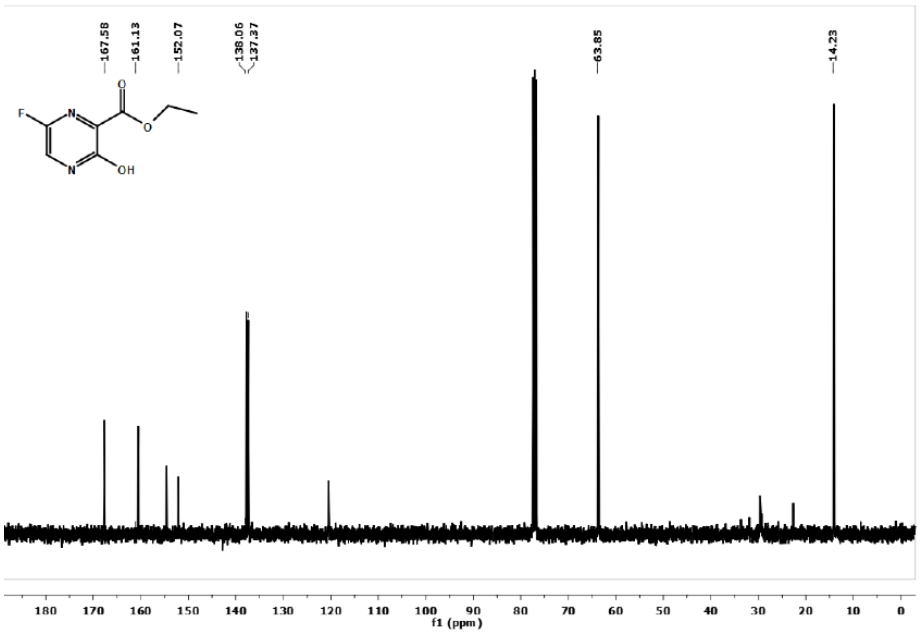


Fig. S16: ^{13}C NMR Spectra of **M6** in Chloroform-d (400 MHz).

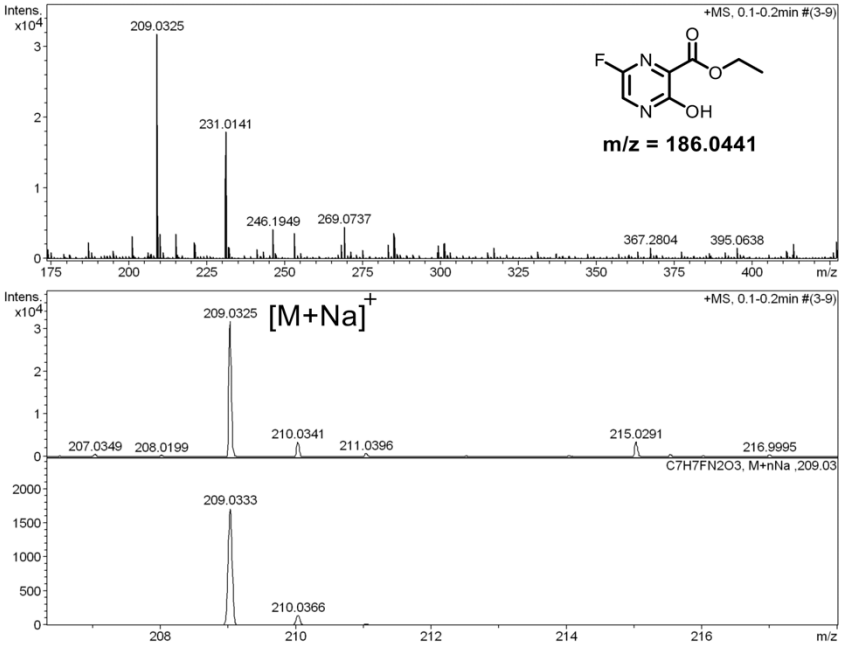


Fig. S17: ESI-MS (HRMS) Mass Spectra of **M6.**

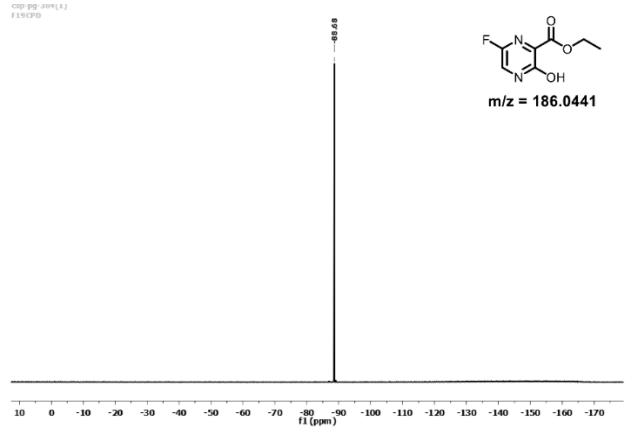


Fig. S18: ^{19}F NMR Spectra of **M6** in chloroform-d (400 MHz).

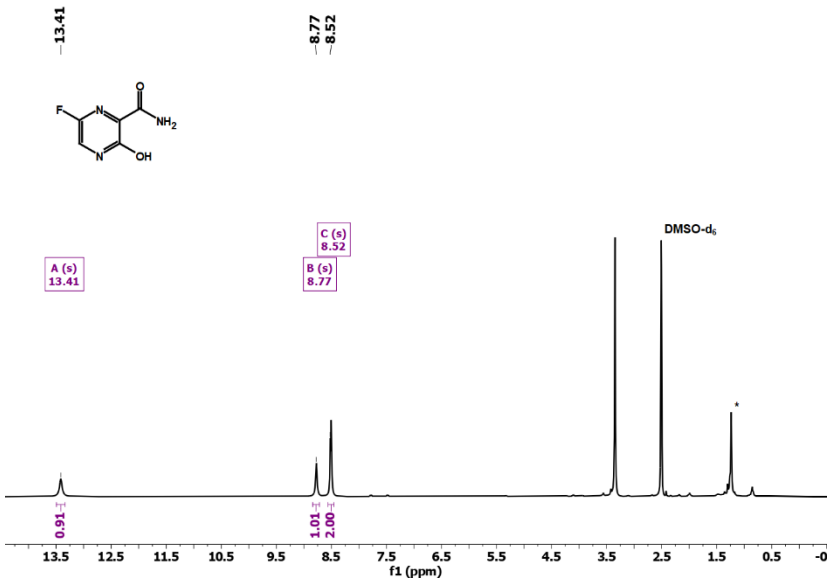


Fig. S19: ^{1}H NMR Spectra of **M7** in DMSO-d_6 (400 MHz).

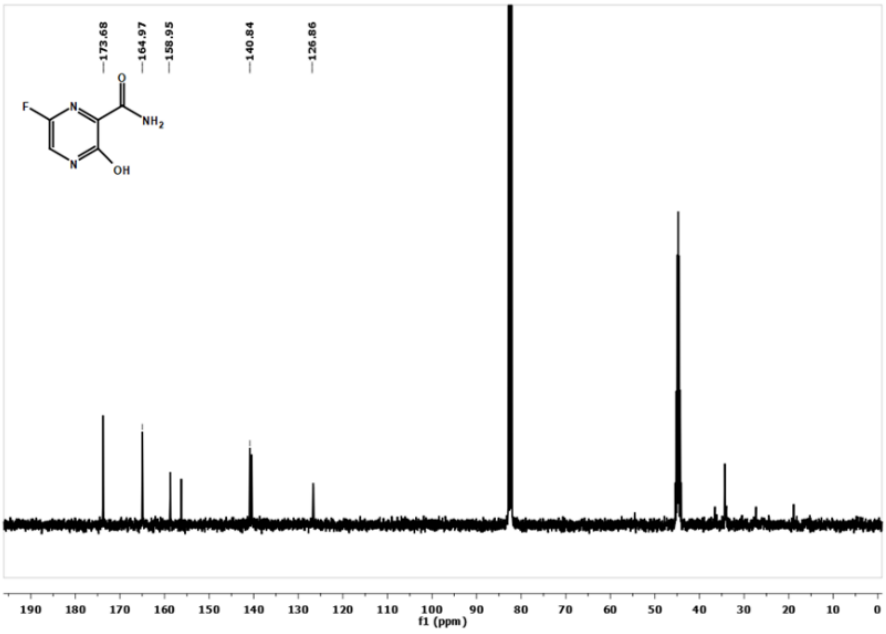


Fig. S20: ^{13}C NMR Spectra of **M7** in DMSO- d_6 and chloroform-d (400 MHz).

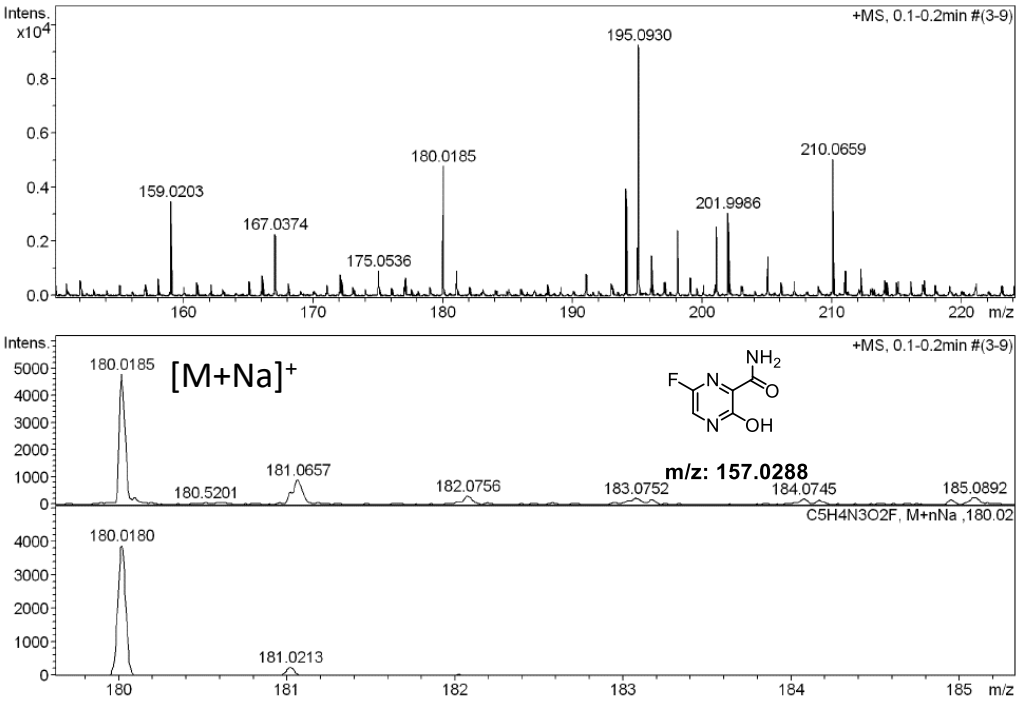


Fig. S21: ESI-MS (HRMS) Mass Spectra of **M7.**

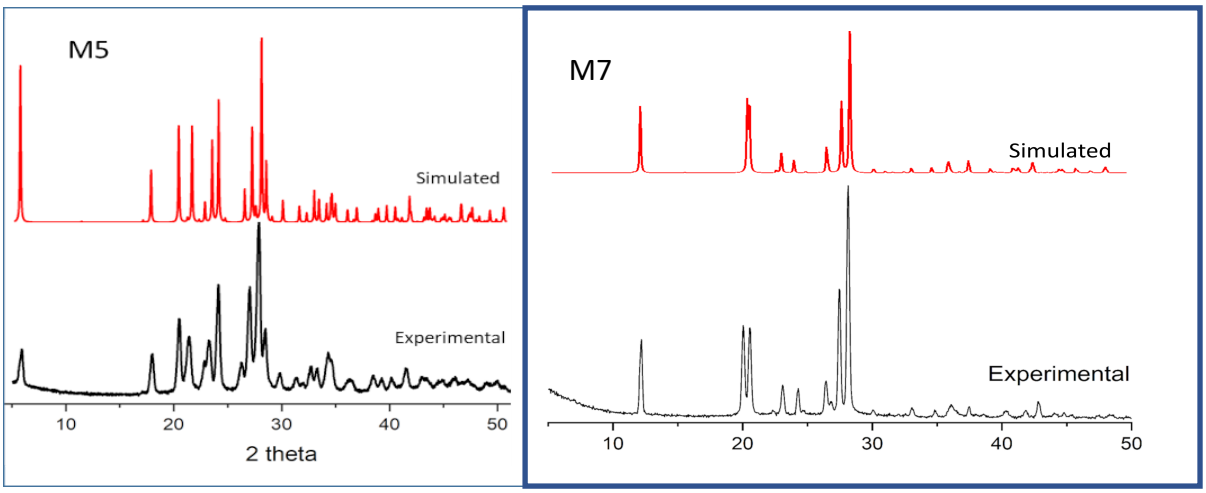


Fig. S22: Powder X-ray diffraction of M5 and M7. Experimental data indicate the simulated pattern form single crystal data.

Section-5

Crystallographic Analysis

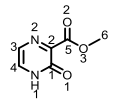
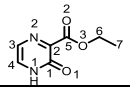
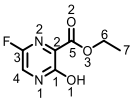
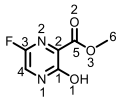
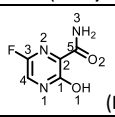
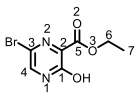
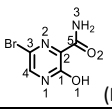
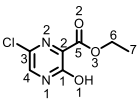
1. Solvent System and method:

The crystal structure of favipiravir and all its all-halide analogous molecules **(M1, M1a, M2, M4, M5, M6, M6a, M7)** were grown by slow evaporation method using chloroform and methanol solvent system in 5-7 days time period.

2. Structure Determination:

The crystal data of favipiravir and its halide analogous molecules **(M1, M1a, M2, M4, M5, M6, M6a, M7)** was collected on **Rigaku Oxford XtaLAB SuperNova diffractometer at 293 K** respectively.
The crystal parameters and other crystallographic results are given below.
The program package diagram **SHELXT1 and Olex2** was used for solve the crystal structure and **DIAMOND 4.6** was used for packing diagram**. ORTEP3** also used for single ortep diagram with atom numbering.

Table S2: Bond parameters

Bond length (In Å)	(N_1-C_1)	(C_1-O_1)	(C_5-O_2)	(C_5-N_3)	(C_2-C_5)	(C_3-F)	(C_3-Cl)	(C_3-Br)
 (M1a)	1.382 (3)	1.234 (2)	1.211 (2)	…….	1.496 (3)	…….	…….	…….
(M1) 	1.372 (2)	1.241 (2)	1.201 (2)	……..	1.504 (2)	…….	…….	…….
 (M6)	1.3422 (17)	1.3325 (16)	1.2157 (17)	……..	1.4868 (18)	1.3555 (15)	…….	…….
 (M6a)	1.338 (2)	1.3350 (19)	1.2169 (19)	……..	1.487 (2)	1.3467 (18)	…….	…….
 (M7)	1.334 (6)	1.338 (5)	1.250 (6)	1.312 (7)	……..	1.346 (6)	…….	…….
 (M4)	1.323 (5)	1.339 (4)	1.478 (5)	……..	1.212 (4)	…….	…….	1.896 (4)
 (M5)	1.330 (12)	1.341 (13)	1.245 (11)	1.318 (11)	1.484 (12)	…….	…….	1.904 (10)
 (M2)	1.327 (3)	1.337 (2)	1.211 (2)	……..	1.483 (3)	…….	1.496 (3)	…….

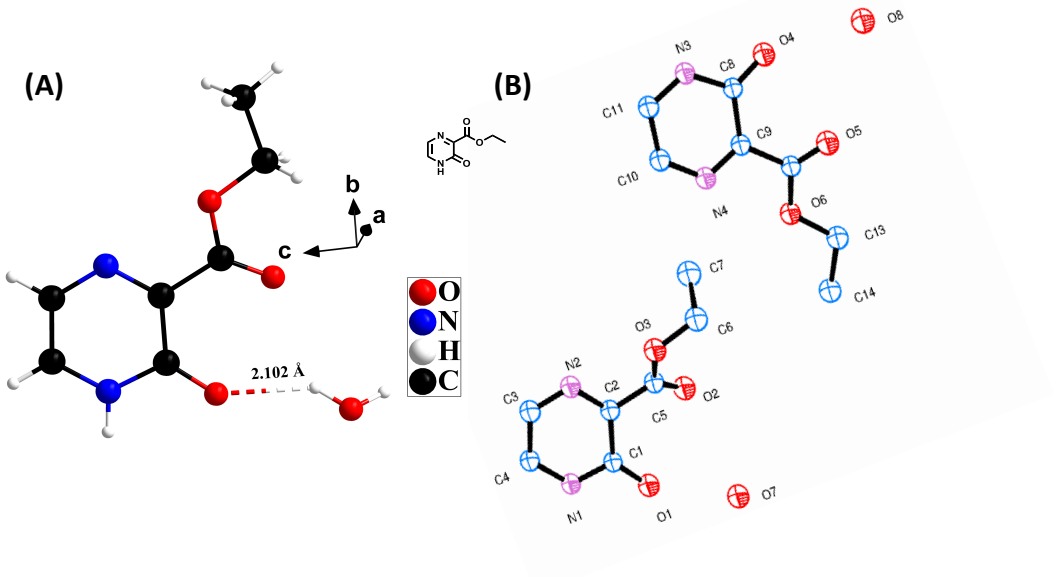


Fig. S23: (A) Crystal structure of **M1** having intermolecular H-bonding interaction with bond distance 2.102 Å (dotted white-red lines). **(B)** Perspective view of **M1** with atom numbering**,** where blue colour indicates **C** atom, pink colour indicates **N** atom, red colour indicates **O** atom. Thermal ellipsoids are drawn at the 35% probability level.

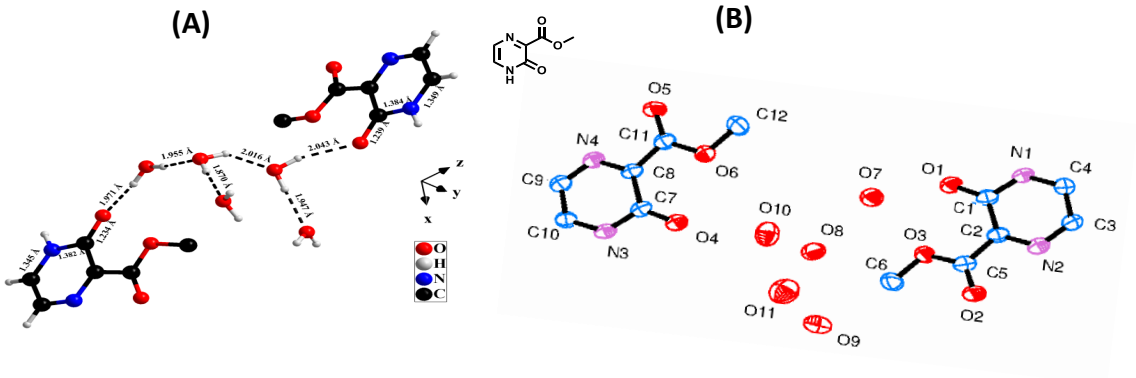


Fig. S24: (A) Crystal structure of **M1a** having intermolecular H-bonding interaction with bond distance range 1.971 Å to 2.043 Å (black dotted lines). **(B)** Perspective view of **M1a** with atom numbering where blue colour indicates **C** atom, pink colour indicates **N** atom, red colour indicates **O** atom. Thermal ellipsoids are drawn at the 35% probability level.

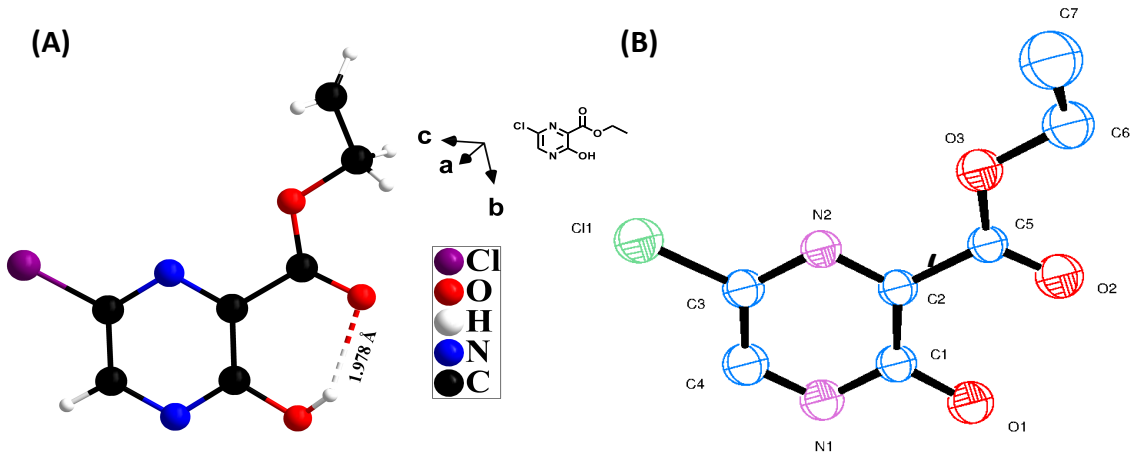


Fig. S25: (A) Crystal structure of **M2** having intramolecular H-bonding interaction with bond distance 1.978 Å (white-red dotted line). **(B)** Perspective view of **M2** with atom numbering**,** where blue colour indicates **C** atom, pink colour indicates **N** atom, red colour indicates **O** atom, and green colour indicate **Cl** atom. Thermal ellipsoids are drawn at the 50% probability level.

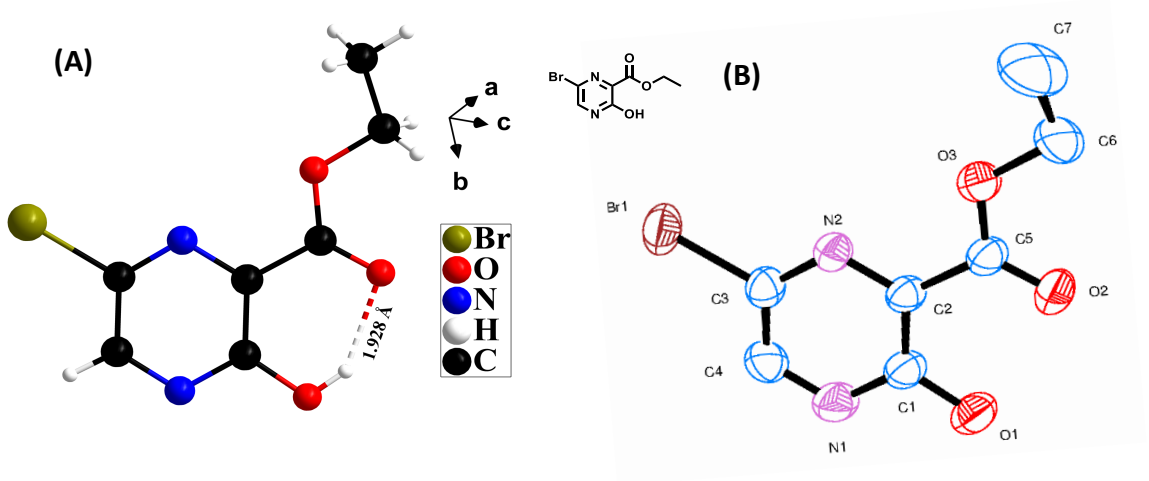


Fig. S26: (A) Crystal structure of **M4** having intramolecular H-bonding interaction with bond distance 1.928 Å (white-red dotted line). **(B)** Perspective view of **M4** with atom numbering**,** where blue colour indicates **C** atom, pink colour indicates **N** atom, red colour indicates **O** atom, and green colour indicate **F** atom. Thermal ellipsoids are drawn at the 50%. probability level.

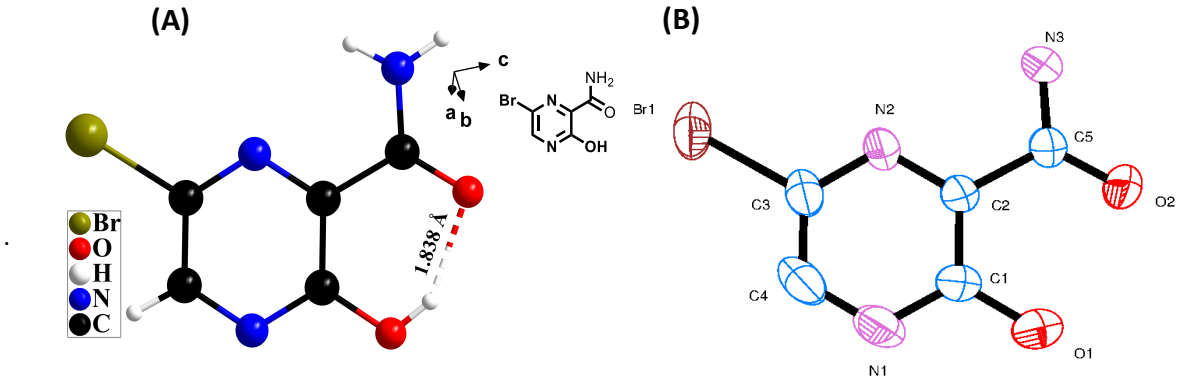


Fig. S27: (A) Crystal structure of **M5** having intramolecular H-bonding interaction with bond distance 1.8383 Å (white-red dotted line). **(B)** Perspective view of **M5** with atom numbering**,** where blue colour indicates **C** atom, pink colour indicates **N** atom, red colour indicates **O** atom, and brown colour indicate **Br** atom. Thermal ellipsoids are drawn at the 55% probability level.

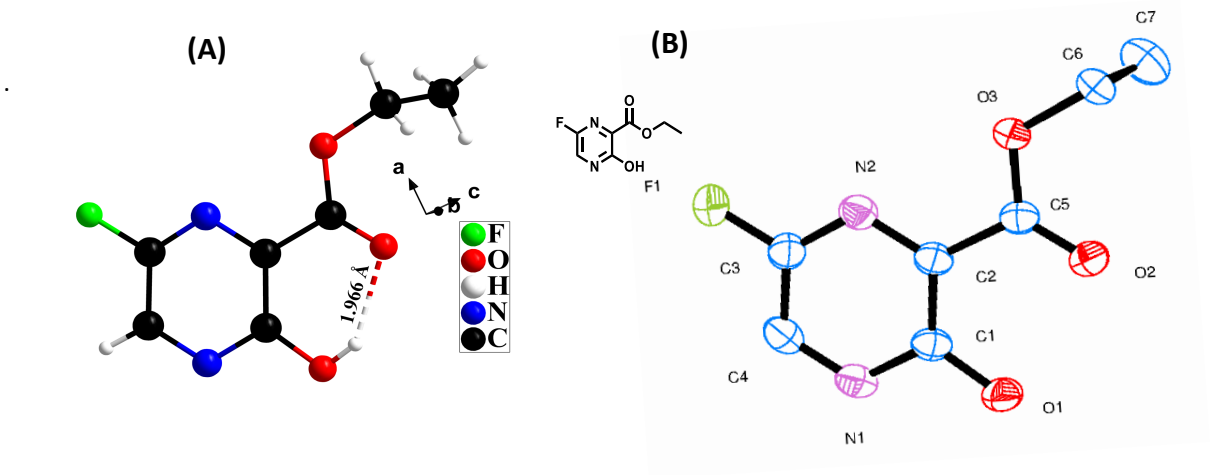


Fig. S28: (A) Crystal structure of **M6** having intramolecular H-bonding interaction with bond distance 1.966 Å (white-red dotted line). **(B)** Perspective view of **M6** with atom numbering**,** where blue colour indicates **C** atom, pink colour indicates **N** atom, red colour indicates **O** atom, and green colour indicate **F** atom. Thermal ellipsoids are drawn at the 45% probability level.

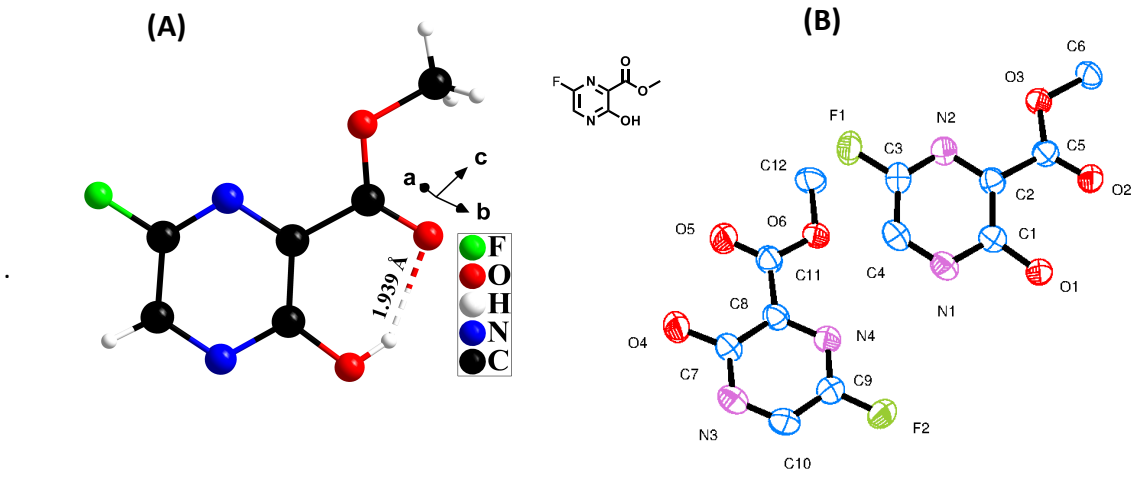


Fig. S29: (A) Crystal structure of **M6a** having intramolecular H-bonding interaction with bond distance 1.939 Å (white-red dotted line)**. (B)** Perspective view of **M6a** with atom numbering**,** where blue colour indicates **C** atom, pink colour indicates **N** atom, red colour indicates **O -** atom, and green colour indicate **F - atom.** Thermal ellipsoids are drawn at the 40% probability level.

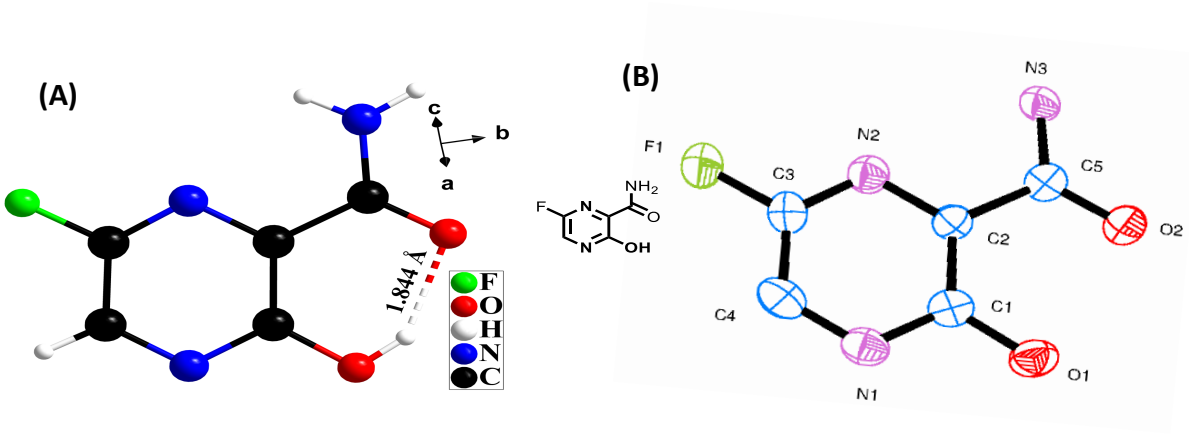


Fig. S30: (A) Crystal structure of **favipiravir (M7)** having intramolecular H-bonding interaction with bond distance 1.844 Å (white-red dotted line). **(B)** Perspective view of **M7** where blue colour indicates **C** atom, pink colour indicates **N** atom, red colour indicates **O** atom, and green colour indicate **F** atom. Thermal ellipsoids are drawn at the 50% probability level.

Table S1. Crystallographic parameters of **M2**, **M4**, **M5 and M6**.

Identification code	**M2**	**M4**	**M5**	**M6**
Empirical formula	$C_{14}H_{12}Cl_2N_4O_6$	$C_7H_7BrN_2O_3$	$C_5H_4BrN_3O_2$	$C_7H_7FN_2O_3$
Formula weight	403.18	247.06	218.02	186.15
Temperature/K	300(1)	298.8(5)	293(2)	100.00(10)
Crystal system	tetragonal	tetragonal	monoclinic	monoclinic
Space group	$P4_2/n$	$P4_2/n$	$P2_1$	C2/c
a/Å	18.7318(5)	18.4161(7)	4.26356(16)	20.2620(5)
b/Å	18.7318(5)	18.4161(7)	5.3334(2)	4.72480(10)
c/Å	5.1839(2)	5.5450(2)	15.8471(6)	16.6253(4)
α/°	90	90	90	90
β/°	90	90	93.394(4)	90.480(2)
γ/°	90	90	90	90
Volume/$Å^3$	1818.93(12)	1880.60(16)	359.72(2)	1591.55(6)
Z	4	8	2	8
$\rho_{calc}g/cm^3$	1.472	1.745	2.013	1.554
μ/mm^{-1}	3.576	5.824	7.439	1.201
F(000)	824.0	976.0	212.0	768.0
Crystal size/mm^3	0.27× 0.16 × 0.14	0.27 × 0.17 × 0.15	0.3× 0.16 × 0.14	0.5 × 0.3 × 0.1
Radiation	CuKα (λ = 1.54184)	CuKα (λ = 1.54184)	CuKα (λ = 1.54184)	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.442 to 150.62	9.606 to 150.686	11.186 to 150.212	8.728 to 154.964
Index ranges	$-23 \le h \le 23$, $-23 \le k \le 23$, $-4 \le l \le 6$	$-22 \le h \le 23$, $-22 \le k \le 23$, $-6 \le l \le 6$	$-5 \le h \le 4$, $-6 \le k \le 6$, $-19 \le l \le 19$	$-25 \le h \le 25$, $-5 \le k \le 5$, $-20 \le l \le 20$
Reflections collected	7563	7769	5212	3864
Independent reflections	1850 [R_{int} = 0.0384, R_{sigma} = 0.0300]	1909 [R_{int} = 0.0408, R_{sigma} = 0.0329]	1447 [R_{int} = 0.0391, R_{sigma} = 0.0269]	1618 [R_{int} = 0.0227, R_{sigma} = 0.0298]
Data/restraints/parameters	1850/0/125	1909/0/121	1447/1/102	1618/0/120
Goodness-of-fit on F^2	1.050	1.052	1.171	1.098
Final R indexes [I>=2σ (I)]	R_1 = 0.0411, wR_2 = 0.1148	R_1 = 0.0422, wR_2 = 0.0993	R_1 = 0.0576, wR_2 = 0.1669	R_1 = 0.0387, wR_2 = 0.1064
Final R indexes [all data]	R_1 = 0.0505, wR_2 = 0.1223	R_1 = 0.0576, wR_2 = 0.1074	R_1 = 0.0589, wR_2 = 0.1679	R_1 = 0.0414, wR_2 = 0.1090
Largest diff. peak/hole / e $Å^{-3}$	0.26/-0.23	0.47/-0.44	0.51/-0.63	0.20/-0.28
Flack parameter	NA	NA	0.05(8)	NA
CCDC Number	2155451	2155452	2155453	2048891

Table S1. Crystallographic parameters of **M1**, **M1a**, **M6a** and **M7**

Identification code	**M1**	**M1a**	**M6a**	**M7**
Empirical formula	$C_{14}H_{18}N_4O_7$	$C_{12}H_{22}N_4O_{11}$	$C_{12}H_{10}F_2N_4O_6$	$C_5H_4FN_3O_2$
Formula weight	354.32	398.33	344.24	157.10
Temperature/K	293(2)	99.99(10)	100.00(10)	100.01(10)
Crystal system	monoclinic	orthorhombic	triclinic	orthorhombic
Space group	P2/c	Pbca	P-1	$Pna2_1$
a/Å	20.5834(5)	11.35633(17)	4.1240(2)	9.0668(5)
b/Å	5.22440(10)	12.7400(3)	12.7162(6)	14.8508(10)
c/Å	15.7133(4)	24.1766(5)	14.3351(9)	4.5755(3)
α/°	90	90	67.238(5)	90
β/°	111.662(3)	90	82.602(5)	90
γ/°	90	90	81.791(4)	90
Volume/$Å^3$	1570.41(7)	3497.87(12)	683.88(7)	616.09(7)
Z	4	8	2	4
$\rho_{calc}g/cm^3$	1.499	1.513	1.672	1.6936
μ/mm^{-1}	1.042	1.173	1.344	1.348
F(000)	744.0	1680.0	352.0	321.4
Crystal size/mm^3	0.200 × 0.270 × 0.300	0.5 × 0.3 × 0.1	0.5 × 0.3 × 0.1	0.310 × 0.250 × 0.200
Radiation	CuKα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.246 to 150.354	7.312 to 155.554	7.578 to 154.824	11.44 to 151.18
Index ranges	$-25 \le h \le 25$, $-6 \le k \le 6$, $-19 \le l \le 17$	$-12 \le h \le 14$, $-15 \le k \le 10$, $-30 \le l \le 27$	$-5 \le h \le 4$, $-15 \le k \le 16$, $-18 \le l \le 16$	$-9 \le h \le 10$, $-18 \le k \le 18$, $-5 \le l \le 5$
Reflections collected	12472	13459	6256	4293
Independent reflections	3121 [R_{int} = 0.0414, R_{sigma} = 0.0275]	3611 [R_{int} = 0.0520, R_{sigma} = 0.0502]	2748 [R_{int} = 0.0324, R_{sigma} = 0.0364]	1116 [R_{int} = 0.0823, R_{sigma} = 0.0548]
Data/restraints/parameters	3121/0/237	3611/15/277	2748/0/221	1116/1/101
Goodness-of-fit on F^2	1.029	1.036	1.050	1.011
Final R indexes [I>=2σ (I)]	R_1 = 0.0493, wR_2 = 0.1358	R_1 = 0.0511, wR_2 = 0.1240	R_1 = 0.0526, wR_2 = 0.1473	R_1 = 0.0657, wR_2 = 0.1933
Final R indexes [all data]	R_1 = 0.0521, wR_2 = 0.1387	R_1 = 0.0638, wR_2 = 0.1316	R_1 = 0.0572, wR_2 = 0.1518	R_1 = 0.0783, wR_2 = 0.2010
Largest diff. peak/hole / e $Å^{-3}$	0.37/-0.28	0.38/-0.31	0.46/-0.25	0.42/-0.40
Flack parameter	NA	NA	NA	0.2(7)
CCDC Number	2048893	2048892	2048893	2048895