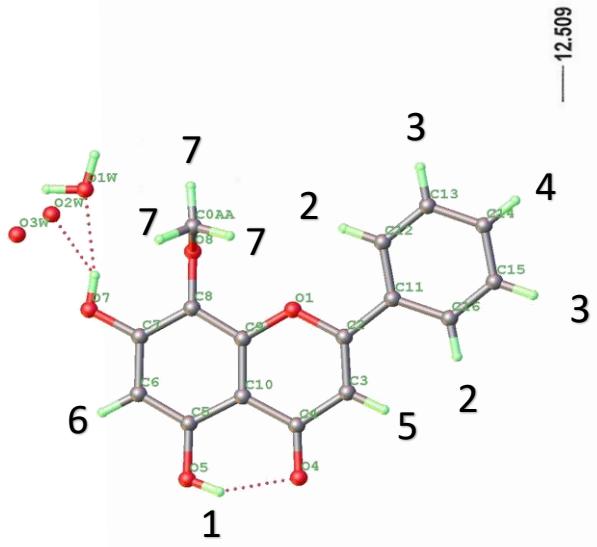
^{1}H NMR of Wogonin Crystallized from Methanol: Sample 3b

(Wog 1.5H_2O)

1H NMR Sample 3b in d6-DMSO



BRUKER

NAME	Wog-MeOH
EXPNO	1
PROCNO	1
Date_	20200415
Time	14.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	1290
DW	60.800 usec
DE	6.00 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	15.80 usec
PL1	-1.00 dB
PL1W	12.17476940 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300037 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

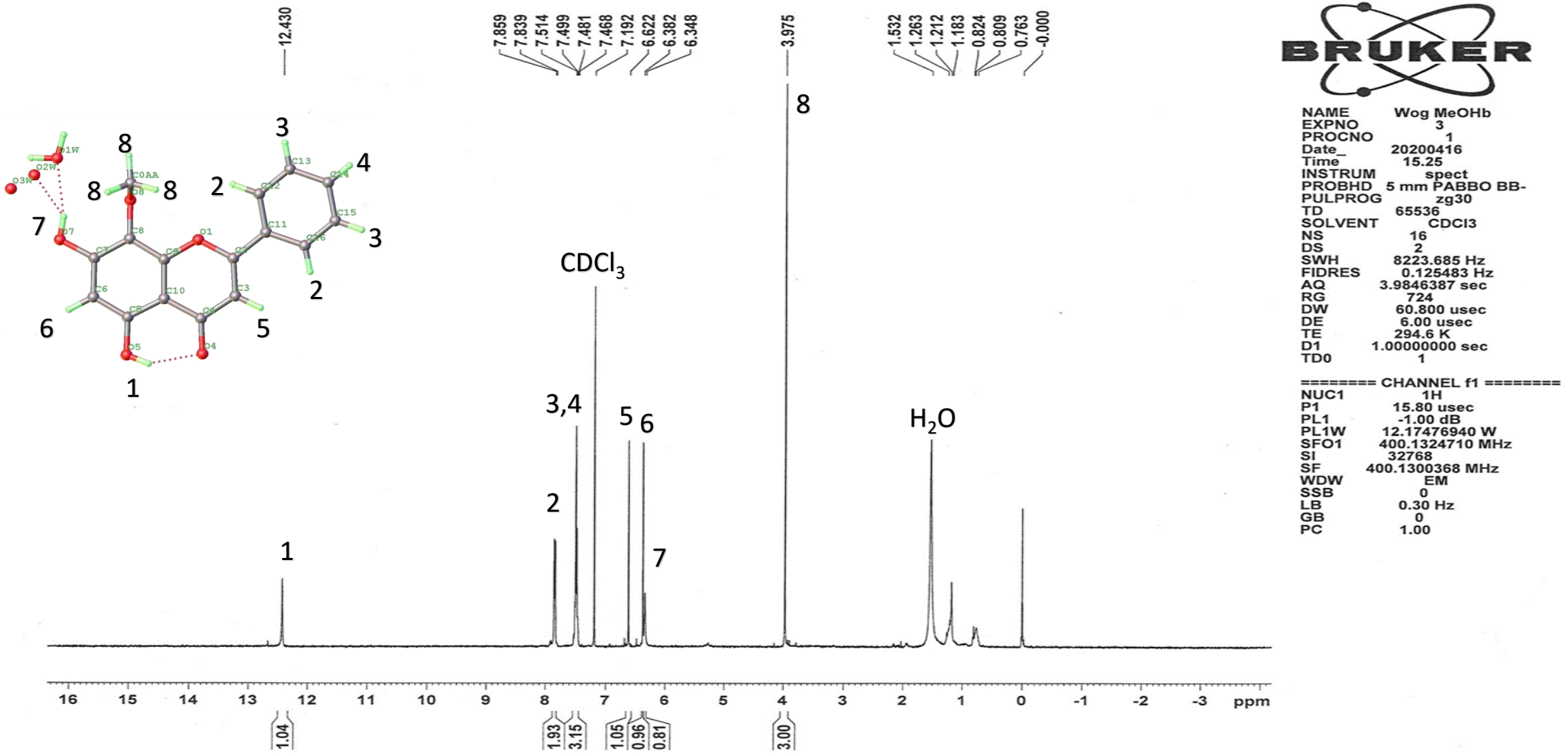
1H NMR (400 MHz, DMSO): δ = 12.4509 (s, 2H, 5−OH), 8.087~8.0866(d, 2H, Ar−H) , 7.628~7.610 (d 2H+1H, Ar−H), 7.003 (s, 1H, CHCO), 6.304 (s, 1H, Ar−H), 3.853 (s, 3H, $ArOCH_3$) ppm.

Comment on 1H NMR of Wog 1.5(H2O) 3b in d6-DMSO

- To double check the result we run the sample using DMSO as what most of the previous method reported used in their analysis.
- If DMSO (δ = 2.50 ppm) is used as NMR solvent the chemical shift for the water is typically found at δ = 3.33 ppm.[1] NMR result showed a chemical shift at δ = 3.328 indicating the presence of water.
- MeOH may be seen in the following chemical shifts , δ CH_3 = 3.16 ppm and δ OH = 4.01ppm.[1] No peak appears in the MeOH region thus there is no MeOH in the sample.
- To confirm the peak at 3.33 is not due to MeOH we also ran the spectrum in d6-dmso, which will shift the water peak but not the CH_3 of MeOH.

1. Fulmer G. R. et al., Organometallics 2010, 29, 2176–2179

1H NMR Sample 3b in $CDCl_3$



BRUKER

NAME	Wog MeOHb
EXPNO	3
PROCNO	1
Date_	20200416
Time	15.25
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	724
DW	60.800 usec
DE	6.00 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	15.80 usec
PL1	-1.00 dB
PL1W	12.17476940 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300368 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1H NMR (400 MHz, $CDCl_3$): δ = 12.430 (s, 1H, 5-OH),, 7.859~7.839 (d, 2H, Ar−H), 7.514~7.468 (d, 2H+1H, Ar−H), 6.622 (s, 1H, CHCO), 6.382 (s, 1H, Ar−H), 6.348 (s,1H, 7-OH), 3.975 (s, 3H, $ArOCH_3$) ppm.

Comment on 1H NMR of Wog 1.5(H_2O) 3b in $CDCl_3$

- When $CDCl_3$ (δ = 7.192ppm) is used as NMR solvent the chemical shift for the water is typically found at δ = 1.56ppm. NMR result showed a chemical shift at δ = 1.532 indicating the presence of water.
- MeOH may be seen in the following chemical shifts , δ CH_3 = 3.49ppm and δ OH = 1.09ppm. No peak appeared in the CH_3 region thus there is no MeOH in the sample.
- The peaks in the 1.263 ~1.183 and 0.824 ~0.763, integrate to less than 1H and may be due to an impurity such as grease.

- *1 Fulmer G. R. et al., Organometallics 2010, 29, 2176–2179*