SUPPLEMENTARY MATERIALS

Microwave-assisted synthesis of 1-(4-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one and its activities as an antioxidant, sunscreen, and antibacterial

Ihsan Ikhtiarudin[1*], Nesa Agistia[1], Neni Frimayanti[1], Tria Harlianti[1], Jasril[2]

[1] Department of Pharmacy, Sekolah Tinggi Ilmu Farmasi Riau, Jl. Kamboja, Kel. Simpang Baru, Kec. Tampan, Panam, Pekanbaru, 28293, Indonesia

[2] Department of Chemistry, FMIPA Universitas Riau, Jl. H.R. Subrantas Km. 12,5 Panam, Pekanbaru, 28293, Indonesia

***Corresponding author: ihsanikhtiarudin@stifar-riau.ac.id**

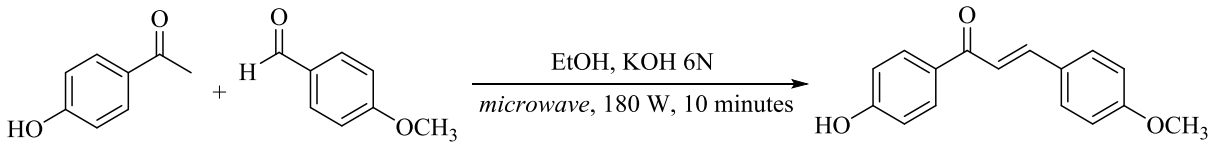


Figure 1. Synthesis route of chalcone analog

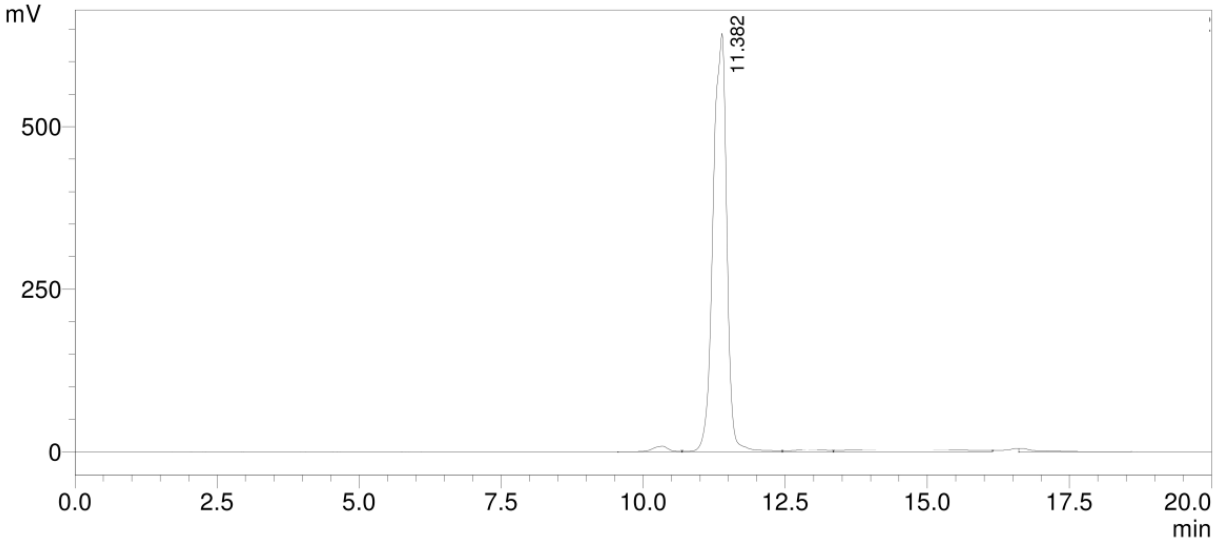


Figure 2. HPLC chromatogram of chalcone analog

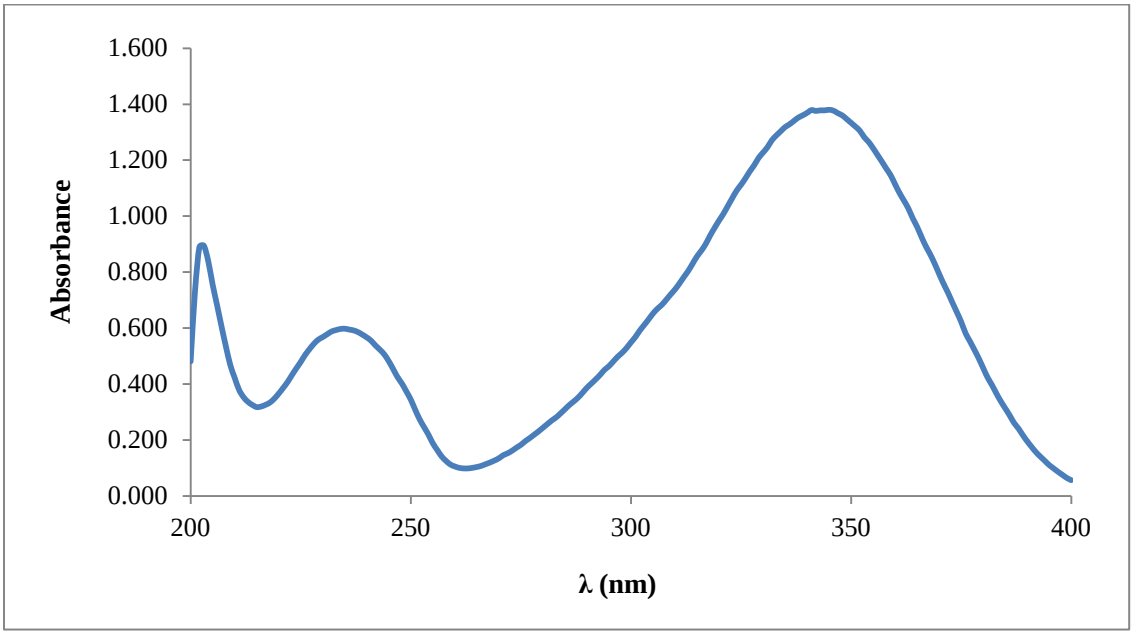


Figure 3. UV spectra of chalcone analog

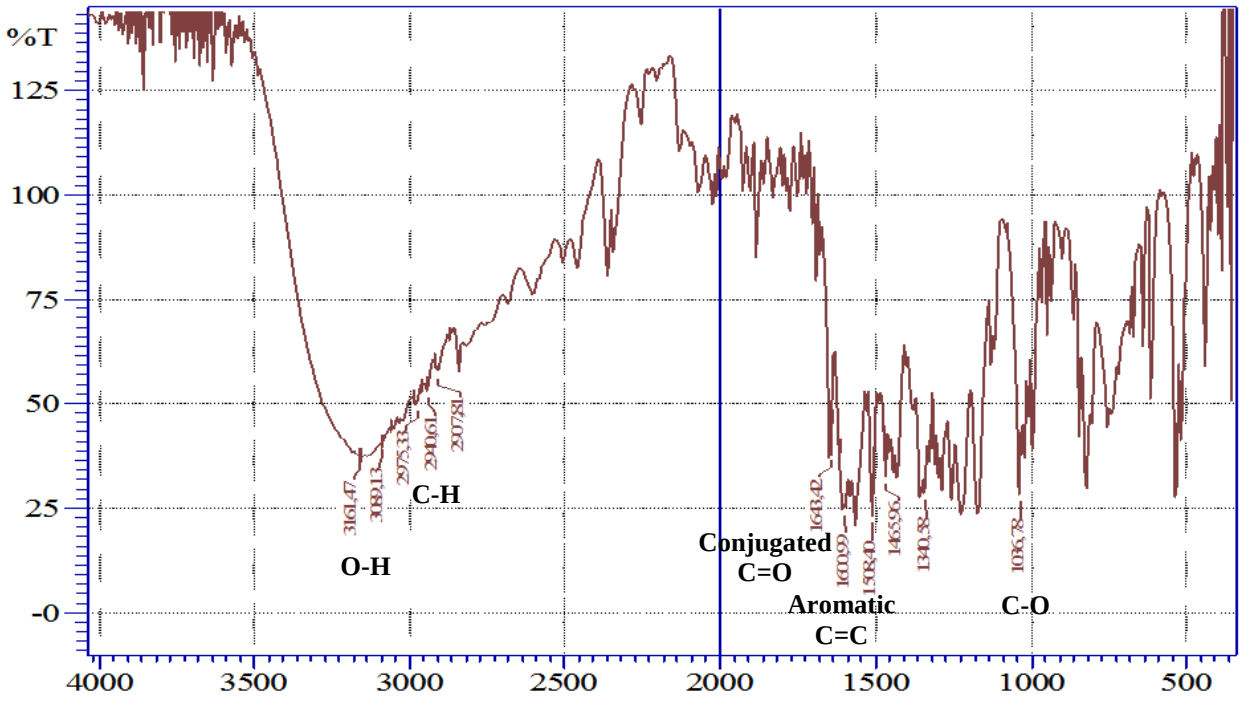


Figure 4. FT-IR spectra of chalcone analog

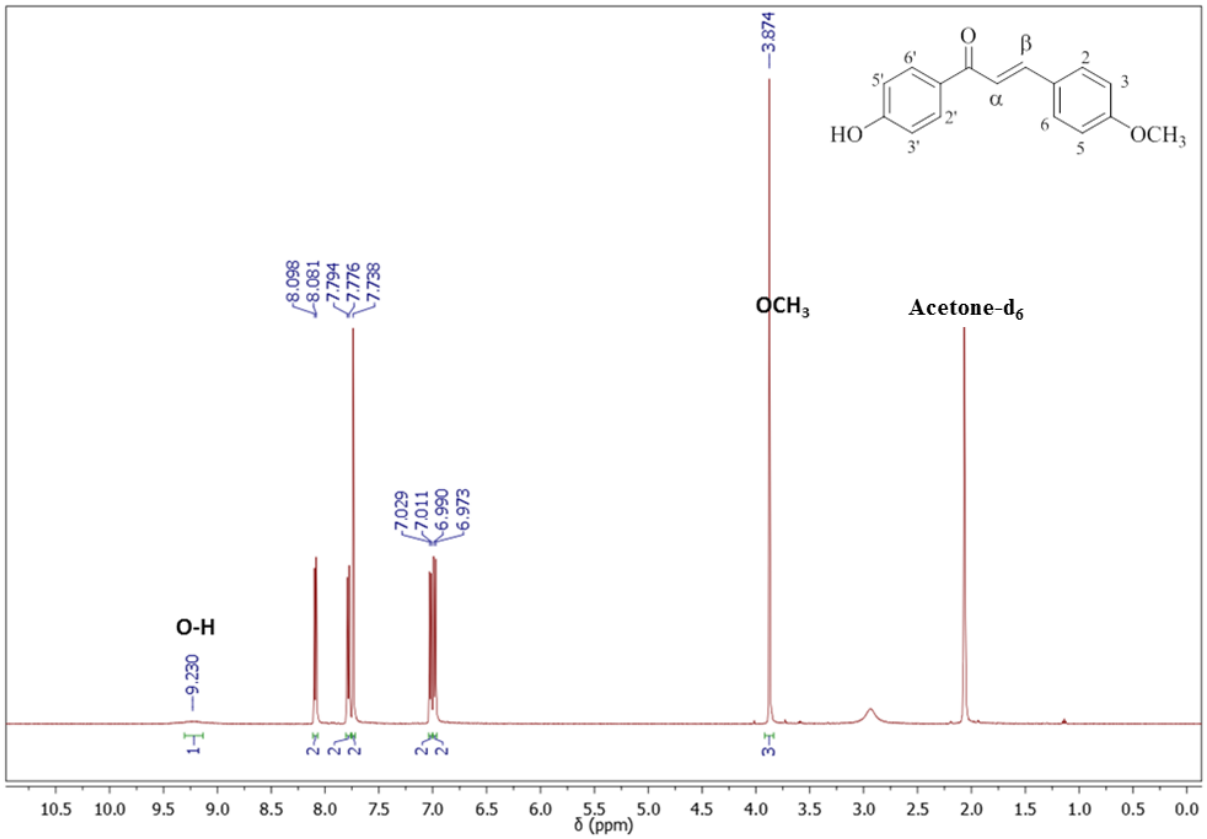
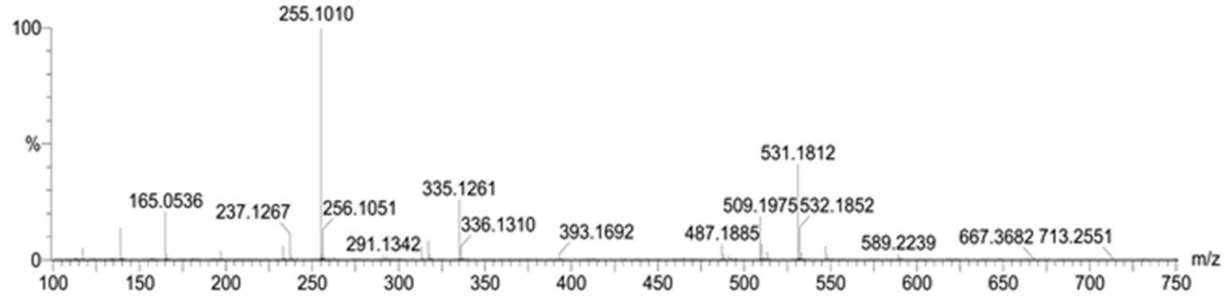
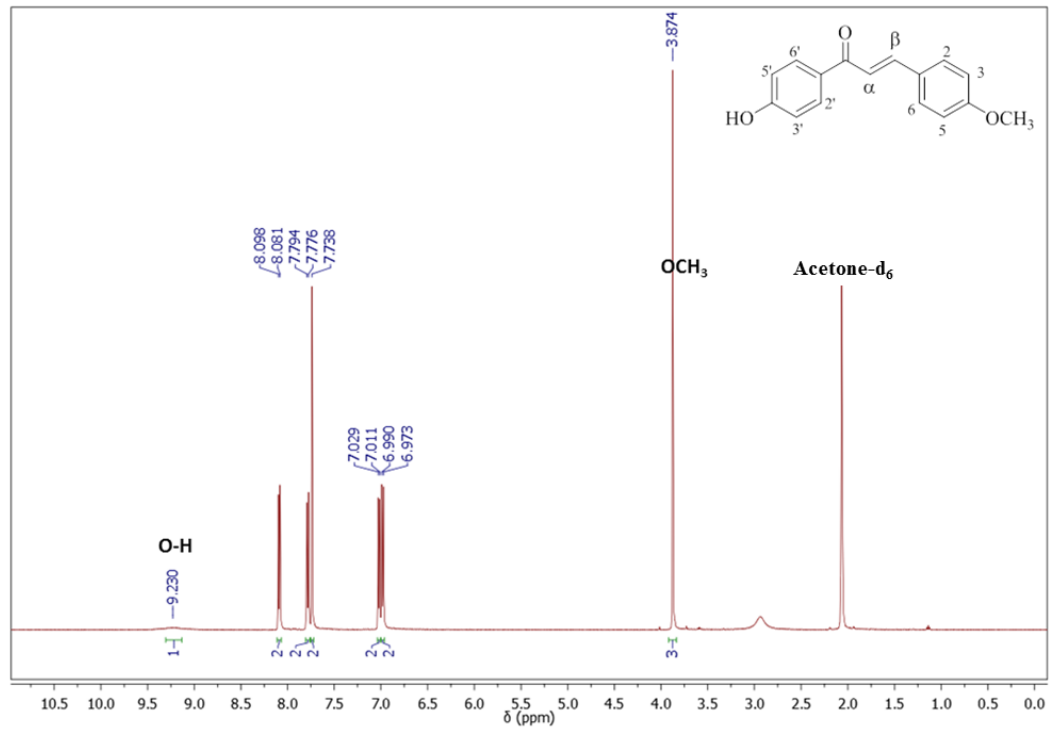


Figure 5. ^{1}H NMR spectra of chalcone analog

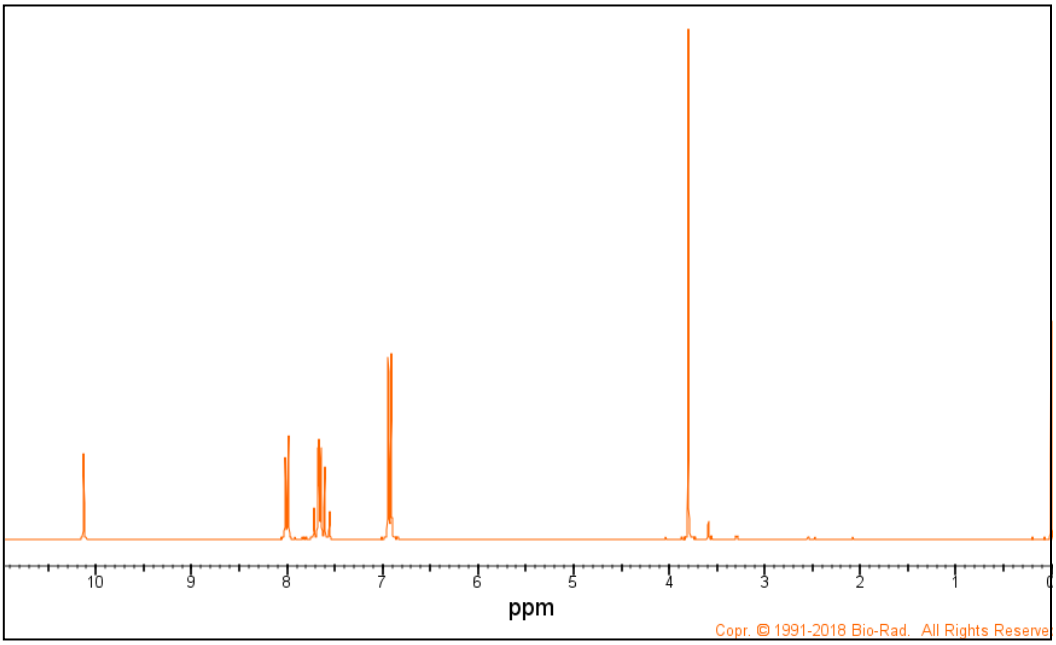


Minimum:				-1.5			
Maximum:		10.0	15.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
255.1010	255.1021	-1.1	-4.3	9.5	439.1	0.0	C16 H15 O3

Figure 6. HRMS spectra of chalcone analog



(a)



(b)

Figure 7. Comparison of ^{1}H NMR spectra of synthesized chalcone analog and literatur (a) The spectra was measured in acetone-d_6 using Agilent 500 (b) The spectra is measured in polysol using Bruker 300 (downloaded from Spectralbase.com) (http://spectrabase.com/spectrum/8HbcjN7xboX?a=SPECTRUM_8HbcjN7xboX)

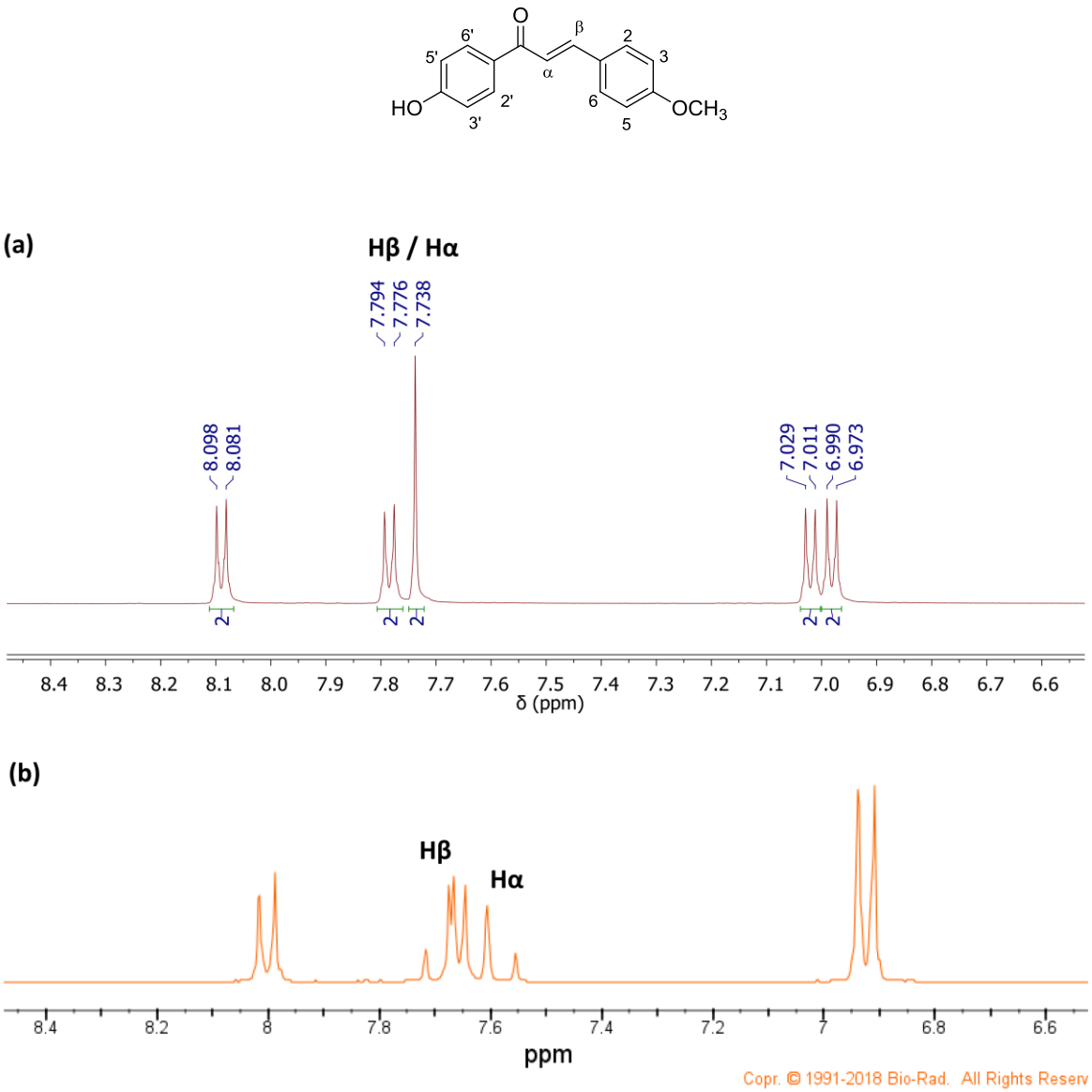
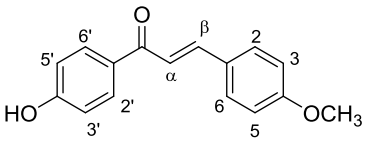


Figure 8. Comparison of proton signals in aromatic region, 6.5 – 8.5 ppm (a) in acetone-d_6 (b) in polysol

Table 1. Comparison of proton chemical shift of chalcone analog when measured in various solvents



Proton Signals	δ (ppm) in acetone-d_6, 500 MHz	δ (ppm) in polysol, 300 MHz	δ (ppm) in $CDCl_3$, 200 MHz
2'/6'	8,09 (*d*, 2H, Ar-2',6'-H, *J* = 8,5 Hz)	8,00 (*d*, 2H)	7,99 (*d*, 2H, *J* = 8,6 Hz)
3'/5'	6,98 (*d*, 2H, Ar-3',5'-H, *J* = 8,5 Hz)	6,93 (*d*, 4H)*	6,93 (*d*, 4H, *J* = 7,2 Hz)*
2/6	7,79 (*d*, 2H, Ar-2,6-H, *J* = 9,0 Hz)	7,66 (*d*, 2H),	7,60 (*d*, 2H, *J* = 8,6 Hz)
3/5	7,02 (*d*, 2H, Ar-3,5-H, *J* = 9,0 Hz)	6,93 (*d*, 4H)*	6,93 (*d*, 4H, *J* = 7,2 Hz)*
α	7,74 (*s*, 2H)*	7,58 (*d*, 1H, H_α)	7,41 (*d*, 1H, H_α, *J* = 15,6 Hz)
β	7,74 (*s*, 2H)*	7,69 (*d*, 1H, H_β)	7,78 (*d*, 1H, H_β, *J* = 15,6 Hz)
-OH	9,23 (*br-s*, 1H)**	10,10 (*s*, 1H, Ar-4-OH)**	5,85 (*s*, 1H, Ar-4-OH)**
-OCH_3	3,87 (*s*, 3H, Ar-4-OCH_3)	3,80 (*s*, 3H)	3,86 (*s*, 3H, Ar-4-OCH_3)

* The signals highlighted in yellow are the overlapped signals. Generaly, the signals of α and β protons appear as two doublet signals (1H), with a coupling constant of 15-16 Hz. Nevertheless, in this case, the signals of protons α and β appear as a singlet (2H) signal. This can be caused by the effect of the solvent used in the measurement of the spectrum, so that the chemical environment of α and β protons in chalcone analog becomes equivalent. As a result, the spectrum does not show a spin splitting, even though the protons are not equivalent, structurally. When measured with polysol solvent [20], there is a slight spin splitting between α and β protons because the chemical environment becomes slightly different, but the 3'/5' and 3/5 proton signals were overlaped and appear as a doublet (4H) signal. When measured with the $CDCl_3$ solvent [22], the difference in the chemical environment of α and β protons becomes greater, as a result, the two doublet signals become further apart

** The signals highlighted in yellow are the signal of hydroxy proton. In the acetone-d_6 solvent, the hydroxy proton signal appears at a chemical shift of 9.23 ppm as a broad singlet. However, when measured in polysol and $CDCl_3$ solvents, the hydroxy proton signal appears as a singlet at a chemical shift of 10.10 ppm and 5.85 ppm, respectively.

^{1}H NMR spectrum (acetone-d_6, 500 MHz) (δ, ppm): 9.23 (*br-s*, ^{1}H, Ar-4-OH); 8.09 (*d*, 2H, Ar-2', 6'-H, *J* = 8.5 Hz); 7.79 (*d*, 2H, Ar-2,6-H, *J* = 9.0 Hz); 7,74 (*s*, 2H, H_α, H_β), 7.02 (*d*, 2H, Ar-3,5-H, *J* = 9.0 Hz); 6.98 (*d*, 2H, Ar-3', 5'-H, *J* = 8.5 Hz); 3.87 (*s*, 3H, Ar-4-OCH_3).

^{1}H NMR spectrum based on literature [20] (polysol, 300 MHz) (δ, ppm): 10.10 (*s*, ^{1}H, Ar-4-OH), 8.00 (*d*, 2H), 7.69 (*d*, ^{1}H, H_β), 7.66 (*d*, 2H), 7.58 (*d*, ^{1}H, H_α), 6.93 (*d*, 4H), 3.80 (*s*, 3H).

^{1}H NMR spectrum based on literature [21] ($CDCl_3$, 200 MHz) (δ, ppm): 7.99 (*d*, 2H, *J* = 8.6 Hz), 7.78 (*d*, ^{1}H, H_β, *J* = 15.6 Hz), 7.60 (*d*, 2H, J = 8.6 Hz), 7.41 (*d*, ^{1}H, H_α, J = 15.6 Hz), 6.93 (*d*, 4H, *J* = 7.2 Hz), 5.85 (*s*, ^{1}H, Ar-4-OH), 3.86 (*s*, 3H, Ar-4-OCH_3).